AN EXPERIMENTAL STUDY OF CEMENTING PASTE BACKFILL

By
Megan Louise Walske
B.E.(hons)

This thesis is submitted for the
Degree of Doctor of Philosophy

THE UNIVERSITY OF WESTERN AUSTRALIA

School of Civil, Environmental and Mining Engineering

2014
To my Dad,

Grant Alfred Walske.

Another beginning and another end…
This thesis focuses on experimental element testing of cementing paste backfill (CPB) to examine and improve on existing laboratory testing techniques. Specifically, the work focuses on developing a framework to account for differences in curing conditions between in situ and laboratory environments, given the recognised improvement in mechanical properties of in situ cured CPB. This has been explored within the effective stress framework by making use of a hydration cell testing apparatus.

The existing hydration cell set-up was modified to allow control over the rate of temperature increase and final temperature of specimens during curing, to replicate in situ curing conditions. The combination of elevated curing temperature and effective stress generation was found to significantly increase the mechanical properties of CPB compared with curing at elevated effective stress and ambient temperatures.

The current procedure for curing under effective stress can be expensive and time consuming. As such, the standard test method for chemical shrinkage of hydraulic cement paste (ASTM C1608-07) was investigated as a potential index/screening test for use in the design of CPB mixes through comparison of the chemical shrinkage-induced strain generated in both the ASTM and hydration cell tests. The appropriate use of this ASTM standard with CPB was validated despite the high w/c ratio of typical CPB mixes. However, a reliable connection between the shrinkage in the ASTM and pore pressure reduction measured in the hydration cell was not established.

An existing self-weight consolidation solution, which shows the pore pressure distribution within a consolidating soil specimen, was modified to include the effects of self-desiccation. A series of design charts were developed to determine the chemical shrinkage-induced pore pressure response for hydration of a weightless material. Superposition was used to combine the self-weight consolidation and chemical shrinkage-induced pore pressure responses, allowing a qualitative assessment of potential pore pressure development in a CPB fill stope.

Anomalous pore pressure behaviour was observed during one-way consolidation of both CPB and uncemented tailings slurry specimens in the hydration cell. At the commencement of consolidation, excess pore pressures were observed to reduce, attempting to equilibrate with the applied back pressure as expected, but then with no variation in pressure or temperature, the pore pressure began to increase, or “rebound”. A range of physical and chemical testing was conducted to investigate potential causes. This included examination of the pore pressure distribution within the specimen, assessment of specimen permeability, investigation of osmotic pressure differences within equipment set-up, XRD, XRF, carbon and dissolved metal analyses.
(tailings material) and dissolved metals and cation and anion analyses (water). Ultimately it was hypothesised that the rebound may be generated by a rapid chemical precipitation. The rebound has the potential to limit the effective stress generation, by increasing pore pressure and countering the effect of consolidation.

The key outcomes from this research have highlighted the effect of curing temperature, consolidation behaviour and chemistry on the stress conditions of CPB and how, in turn, this affects material properties and behaviour.
DECLARATION FOR THeses CONTAINING PUBLISHED WORK AND/OR WORK PREPARED FOR PUBLICATION

The examination of the thesis is an examination of the work of the student. The work must have been substantially conducted by the student during enrolment in the degree.

Where the thesis includes work to which others have contributed, the thesis must include a statement that makes the student's contribution clear to the examiners. This may be in the form of a description of the precise contribution of the student to the work presented for examination and/or a statement of the percentage of the work that was done by the student.

In addition, in the case of co-authored publications included in the thesis, each author must give their signed permission for the work to be included. If signatures from all the authors cannot be obtained, the statement detailing the student's contribution to the work must be signed by the coordinating supervisor.

Please sign one of the statements below.

1. This thesis **does not contain** work that I have published, nor work under review for publication.

   Student Signature

   ![Signature]

2. This thesis contains **only sole-authored** work, some of which has been published and/or prepared for publication under sole authorship. The bibliographical details of the work and where it appears in the thesis are outlined below.

   Student Signature

   ![Signature]

3. This thesis contains published work and/or work prepared for publication, **some of which has been co-authored**. The bibliographical details of the work and where it appears in the thesis are outlined below. The student must attach to this declaration a statement for each publication that clarifies the contribution of the student to the work. This may be in the form of a description of the precise contributions of the student to the published work and/or a statement of percent contribution by the student. This statement must be signed by all authors. If signatures from all the authors cannot be obtained, the statement detailing the student's contribution to the published work must be signed by the coordinating supervisor.

   Student Signature

   ![Signature]

   Coordinating Supervisor Signature

   ![Signature]
# CONTENTS

<table>
<thead>
<tr>
<th>Section</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>ABSTRACT</td>
<td>III</td>
</tr>
<tr>
<td>DECLARATION</td>
<td>V</td>
</tr>
<tr>
<td>LIST OF FIGURES</td>
<td>X</td>
</tr>
<tr>
<td>LIST OF TABLES</td>
<td>XV</td>
</tr>
<tr>
<td>ACKNOWLEDGEMENTS</td>
<td>XVI</td>
</tr>
<tr>
<td>NOTATION</td>
<td>XVII</td>
</tr>
<tr>
<td>CHAPTER 1. INTRODUCTION</td>
<td>1</td>
</tr>
<tr>
<td>1.1. BACKGROUND</td>
<td>1</td>
</tr>
<tr>
<td>1.1.1. The backfilling process</td>
<td>1</td>
</tr>
<tr>
<td>1.1.2. CPB behaviour</td>
<td>4</td>
</tr>
<tr>
<td>1.1.3. Design of CPB</td>
<td>5</td>
</tr>
<tr>
<td>1.2. RESEARCH OBJECTIVES</td>
<td>6</td>
</tr>
<tr>
<td>1.3. THESIS ORGANISATION</td>
<td>7</td>
</tr>
<tr>
<td>CHAPTER 2. REVIEW OF LITERATURE</td>
<td>9</td>
</tr>
<tr>
<td>2.1. INTRODUCTION</td>
<td>9</td>
</tr>
<tr>
<td>2.2. THE CURING ENVIRONMENT</td>
<td>9</td>
</tr>
<tr>
<td>2.3. AN EFFECTIVE STRESS APPROACH TO CPB BEHAVIOUR</td>
<td>12</td>
</tr>
<tr>
<td>2.4. FACTORS AFFECTING HYDRATION</td>
<td>13</td>
</tr>
<tr>
<td>2.4.1. Physical properties</td>
<td>14</td>
</tr>
<tr>
<td>2.4.2. Chemistry</td>
<td>15</td>
</tr>
<tr>
<td>2.4.3. Temperature</td>
<td>15</td>
</tr>
<tr>
<td>2.5. SUMMARY</td>
<td>17</td>
</tr>
<tr>
<td>CHAPTER 3. MATERIALS AND METHODS</td>
<td>18</td>
</tr>
<tr>
<td>3.1. INTRODUCTION</td>
<td>18</td>
</tr>
<tr>
<td>3.2. MATERIALS</td>
<td>18</td>
</tr>
<tr>
<td>3.2.1. Tailings</td>
<td>19</td>
</tr>
<tr>
<td>3.2.2. Water</td>
<td>23</td>
</tr>
<tr>
<td>3.2.3. Binder</td>
<td>24</td>
</tr>
<tr>
<td>3.3. MIXES</td>
<td>25</td>
</tr>
<tr>
<td>3.4. EXPERIMENTAL METHODS</td>
<td>25</td>
</tr>
<tr>
<td>3.4.1. Specimen preparation</td>
<td>25</td>
</tr>
</tbody>
</table>
5.4.2. Effect of filler material on chemical shrinkage ................................................................. 83
5.4.3. Effect of salt on chemical shrinkage .................................................................................... 85
5.4.4. Modelling chemical shrinkage ........................................................................................... 87
5.4.5. Impact of volume change on a hydraulically isolated specimen ........................................ 92
5.4.6. Fitting model parameters to hydration cell data ............................................................... 94
5.4.7. Comparing ASTM and hydration cell test results ............................................................ 96
5.4.8. Impact of stiffness on chemical shrinkage ......................................................................... 97
5.4.9. Effect of permeability testing in hydration cell results ..................................................... 101
5.4.10. Potential issues with the ASTM test ................................................................................. 103
5.4.11. Discrepancy between ASTM and hydration cell chemical shrinkage values ................. 104
5.5. CONCLUSIONS ....................................................................................................................... 105

CHAPTER 6. INCORPORATING CHEMICAL SHRINKAGE INTO GIBSON’S SOLUTION. 107

6.1. INTRODUCTION ...................................................................................................................... 107
6.2. GIBSON’S SOLUTION ............................................................................................................. 108
6.3. CHEMICAL SHRINKAGE ....................................................................................................... 110
6.4. EXAMPLE APPLICATION ....................................................................................................... 116
6.5. CONCLUSION .......................................................................................................................... 118

CHAPTER 7. THE INFLUENCE OF CHEMISTRY ON CONSOLIDATION OF CEMENTING PASTE BACKFILL AND UNCEMENTED TAILINGS ........................................................................................................... 120

7.1. INTRODUCTION ...................................................................................................................... 120
7.1.1. Consolidation .................................................................................................................... 120
7.1.2. Consolidation of CPB ...................................................................................................... 124
7.2. EXPERIMENTAL TESTING PROGRAM .................................................................................... 126
7.3. EXPERIMENTAL RESULTS AND DISCUSSION ................................................................ 127
7.3.1. Consolidation testing ....................................................................................................... 127
7.3.2. Internal specimen stress conditions during consolidation ............................................... 130
7.3.3. Permeability of consolidating specimens ......................................................................... 136
7.3.4. Consolidation testing using an oedometer apparatus ..................................................... 139
7.3.5. Effects of osmotic suction on consolidation .................................................................... 141
7.3.6. Effects of physical constraint and material “reuse” on consolidation behaviour ............ 143
7.3.7. Values of c_v used in consolidation analyses ................................................................. 146
7.3.8. Potential temperature increase ....................................................................................... 147
7.3.9. Implications of chemical reactions on self-desiccation-induced effective stress development ................................................................. 148
7.3.10. Time dependent behaviour ............................................................................................ 151

7.4. MODELLING THE CONSOLIDATION BEHAVIOUR: POTENTIAL IMPACTS OF CHEMISTRY ON CONSOLIDATION EQUATIONS ......................................................................................................................... 153

7.5. CHEMICAL ANALYSIS .......................................................................................................... 155
7.5.1. Chemical comparison of specimens RL8 and RL9 ......................................................... 156
7.5.2. Precipitate potential ........................................................................................................ 157
7.6. IMPLICATION ON CHEMICAL SHRINKAGE MEASUREMENTS ............................................................. 159
7.7. CONCLUSIONS .......................................................................................................................... 159
7.8. RECOMMENDATIONS FOR FUTURE WORK ............................................................................... 161

CHAPTER 8. CONCLUSIONS ............................................................................................................ 163
8.1. CONCLUDING REMARKS ......................................................................................................... 163
8.2. MAIN OUTCOMES AND FUTURE RECOMMENDATIONS ......................................................... 165
  8.2.1. The influence of elevated curing temperatures ................................................................. 165
  8.2.2. Evaluation of chemical shrinkage ..................................................................................... 167
  8.2.3. Modification to Gibson’s solution to include self-desiccation ......................................... 168
  8.2.4. Chemical effects on consolidation behaviour ................................................................. 168
8.3. SUMMARY ............................................................................................................................... 170

REFERENCES ....................................................................................................................................... 171

APPENDIX A. CHEMICAL TESTING RESULTS ................................................................................ 177
# LIST OF FIGURES

<table>
<thead>
<tr>
<th>Figure</th>
<th>Description</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>FIGURE 1.1</td>
<td>Schematic showing typical stope-mining sequence with the use of CPB (from Helinski, 2007)</td>
<td>2</td>
</tr>
<tr>
<td>FIGURE 1.2</td>
<td>Typical stope backfilling situation with typical dimensions (from Helinski, 2007)</td>
<td>3</td>
</tr>
<tr>
<td>FIGURE 2.1</td>
<td><em>In situ</em> measurements of total stress and pore pressure (taken from Figure 7, Helinski et al., 2011b)</td>
<td>11</td>
</tr>
<tr>
<td>FIGURE 3.1</td>
<td>Tailings material particle size distribution</td>
<td>20</td>
</tr>
<tr>
<td>FIGURE 3.2</td>
<td>Hydration cell experimental set up</td>
<td>26</td>
</tr>
<tr>
<td>FIGURE 3.3</td>
<td>Chemical shrinkage experimental set up</td>
<td>29</td>
</tr>
<tr>
<td>FIGURE 3.4</td>
<td>Oedometer experimental set up</td>
<td>30</td>
</tr>
<tr>
<td>FIGURE 4.1</td>
<td><em>In situ</em> temperatures measured in a SNM stope</td>
<td>37</td>
</tr>
<tr>
<td>FIGURE 4.2</td>
<td><em>In situ</em> temperature measured in a RL stope</td>
<td>37</td>
</tr>
<tr>
<td>FIGURE 4.3</td>
<td>Locations of <em>in situ</em> temperature measurements in a RL stope</td>
<td>38</td>
</tr>
<tr>
<td>FIGURE 4.4</td>
<td>Temp/W model of (A) CPB-filled stope and (B) hydration cell specimen</td>
<td>40</td>
</tr>
<tr>
<td>FIGURE 4.5</td>
<td><em>In situ</em> temperature reduction due to heat dissipation in a stope versus a hydration cell</td>
<td>41</td>
</tr>
<tr>
<td>FIGURE 4.6</td>
<td>Semi-adiabatic curing apparatus by Ng et al. (2008)</td>
<td>42</td>
</tr>
<tr>
<td>FIGURE 4.7</td>
<td>Semi-adiabatic curing test set-up</td>
<td>43</td>
</tr>
<tr>
<td>FIGURE 4.8</td>
<td>Heat loss compensated semi-adiabatic curing test for OPC</td>
<td>44</td>
</tr>
<tr>
<td>FIGURE 4.9</td>
<td>Semi-adiabatic curing test – SNM 1.5% and 3% binder mixes</td>
<td>45</td>
</tr>
<tr>
<td>FIGURE 4.10</td>
<td>Semi-adiabatic curing test – KB 5% binder mix</td>
<td>45</td>
</tr>
<tr>
<td>FIGURE 4.11</td>
<td>Temp/W semi-adiabatic curing test – effect of insulation type (polystyrene, water, air or no insulation) on the mean volumetric specimen temperature (T&lt;sub&gt;s&lt;/sub&gt;)</td>
<td>46</td>
</tr>
<tr>
<td>FIGURE 4.12</td>
<td>Temp/W semi-adiabatic curing test modelling – effect of sample size (0.1x0.2m, 0.2x0.4m, 0.4x0.8m) on the mean volumetric specimen temperature (T&lt;sub&gt;s&lt;/sub&gt;)</td>
<td>47</td>
</tr>
<tr>
<td>FIGURE 4.13</td>
<td>Temp/W semi-adiabatic curing test modelling – effect of varying ambient temperature (20°C, 25°C, 32°C) on the mean volumetric specimen temperature (T&lt;sub&gt;s&lt;/sub&gt;)</td>
<td>47</td>
</tr>
<tr>
<td>FIGURE 4.14</td>
<td>Temperature control system</td>
<td>49</td>
</tr>
<tr>
<td>FIGURE 4.15</td>
<td>Temperature hydration cell apparatus</td>
<td>50</td>
</tr>
<tr>
<td>FIGURE 4.16</td>
<td>Specimen temperature probe</td>
<td>51</td>
</tr>
<tr>
<td>FIGURE 4.17</td>
<td>Example temperature measurements during heating of the hydration cell</td>
<td>51</td>
</tr>
<tr>
<td>FIGURE 4.18</td>
<td>Heater timing array</td>
<td>52</td>
</tr>
<tr>
<td>FIGURE 4.19</td>
<td>KB effective stress (P') measurements under varying curing conditions: (A) curing condition A - room temperature, no P' generation; (B) curing condition B - room temperature, P' generation</td>
<td>52</td>
</tr>
</tbody>
</table>
TEMPERATURE WITH $P'$ GENERATION; (c) CURING CONDITION C - ELEVATED TEMPERATURE, NO $P'$ GENERATION; (d) CURING CONDITION D – ELEVATED TEMPERATURE WITH $P'$ GENERATION. .......... 56

FIGURE 4.20 KB MEAN VOLUMETRIC SAMPLE TEMPERATURE ($T_s$) UNDER VARYING CURING CONDITIONS: (a) CURING CONDITION A - ROOM TEMPERATURE, NO $P'$ GENERATION; (b) CURING CONDITION C - ELEVATED TEMPERATURE, NO $P'$ GENERATION; (c) CURING CONDITION D – ELEVATED TEMPERATURE WITH $P'$ GENERATION. .. 57

FIGURE 4.21 RATE OF KB SPECIMEN STIFFNESS ($G_o$) GAIN UNDER VARYING CURING CONDITION: (A) CURING CONDITION A - ROOM TEMPERATURE, NO $P'$ GENERATION; (B) CURING CONDITION B - ROOM TEMPERATURE WITH $P'$ GENERATION; (C) CURING CONDITION C - ELEVATED TEMPERATURE, NO $P'$ GENERATION; (D) CURING CONDITION D – ELEVATED TEMPERATURE WITH $P'$ GENERATION. .......... 58

FIGURE 4.22 STRAIN-STRAIN CURVES FROM UCS TESTING OF KB SPECIMENS CURED UNDER VARYING CURING CONDITIONS: (A) CURING CONDITION A - ROOM TEMPERATURE, NO $P'$ GENERATION; (B) CURING CONDITION B - ROOM TEMPERATURE WITH $P'$ GENERATION; (C) CURING CONDITION C - ELEVATED TEMPERATURE, NO $P'$ GENERATION; (D) CURING CONDITION D – ELEVATED TEMPERATURE WITH $P'$ GENERATION. .......... 59

FIGURE 4.23 SNM EFFECTIVE STRESS ($P'$) MEASUREMENTS UNDER VARYING CURING CONDITIONS: (A) CURING CONDITION A - ROOM TEMPERATURE, NO $P'$ GENERATION; (B) CURING CONDITION B - ROOM TEMPERATURE WITH $P'$ GENERATION; (C) CURING CONDITION C - ELEVATED TEMPERATURE, NO $P'$ GENERATION; (D) CURING CONDITION D – ELEVATED TEMPERATURE WITH $P'$ GENERATION. .......... 60

FIGURE 4.24 SNM MEAN VOLUMETRIC SAMPLE TEMPERATURE ($T_s$) UNDER CURING CONDITION D - ELEVATED TEMPERATURE WITH $P'$ GENERATION. ...................................................................................... 61

FIGURE 4.25 RATE OF SNM SPECIMEN STIFFNESS ($G_o$) GAIN UNDER VARYING CURING CONDITION: (A) CURING CONDITION A - ROOM TEMPERATURE, NO $P'$ GENERATION; (B) CURING CONDITION B - ROOM TEMPERATURE WITH $P'$ GENERATION; (C) CURING CONDITION C - ELEVATED TEMPERATURE, NO $P'$ GENERATION; (D) CURING CONDITION D – ELEVATED TEMPERATURE WITH $P'$ GENERATION. .......... 62

FIGURE 4.26 STRAIN-STRAIN CURVES FROM UCS TESTING OF SNM SPECIMENS CURED UNDER VARYING CURING CONDITIONS: (A) CURING CONDITION A - ROOM TEMPERATURE, NO $P'$ GENERATION; (B) CURING CONDITION B - ROOM TEMPERATURE WITH $P'$ GENERATION; (C) CURING CONDITION C - ELEVATED TEMPERATURE, NO $P'$ GENERATION; (D) CURING CONDITION D – ELEVATED TEMPERATURE WITH $P'$ GENERATION. ...................................................................................... 63

FIGURE 4.27 COMPARISON OF EFFECTIVE STRESS ($P'$) GENERATION WITH TIME ($t$) FOR (A) KB AND (B) SNM FOR CURING CONDITION A - ROOM TEMPERATURE, NO $P'$ GENERATION; CURING CONDITION B - ROOM TEMPERATURE WITH $P'$ GENERATION; CURING CONDITION C - ELEVATED TEMPERATURE, NO $P'$ GENERATION; AND CURING CONDITION D – ELEVATED TEMPERATURE WITH $P'$ GENERATION. .......... 64

FIGURE 4.28 COMPARISON OF SMALL STRAIN STIFFNESS ($G_o$) GENERATION WITH TIME ($t$) FOR (A) KB AND (B) SNM FOR CURING CONDITION A - ROOM TEMPERATURE, NO $P'$ GENERATION; CURING CONDITION B - ROOM TEMPERATURE WITH $P'$ GENERATION; CURING CONDITION C - ELEVATED TEMPERATURE, NO $P'$ GENERATION; AND CURING CONDITION D – ELEVATED TEMPERATURE WITH $P'$ GENERATION. ...................................................................................... 66
FIGURE 4.29 COMPARISON OF PEAK UCS STRENGTH ($\sigma_u$) FOR (A) KB AND (B) SNM CURED UNDER CURING CONDITION A - ROOM TEMPERATURE, NO $P'$ GENERATION; CURING CONDITION B - ROOM TEMPERATURE WITH $P'$ GENERATION; CURING CONDITION C - ELEVATED TEMPERATURE, NO $P'$ GENERATION; AND CURING CONDITION D – ELEVATED TEMPERATURE WITH $P'$ GENERATION. .......... 67

FIGURE 4.30 COMPARISON OF MAXIMUM UCS STRESS AND ASSOCIATED AXIAL STRAIN FOR (A) KB AND (B) SNM CURED UNDER CURING CONDITION A - ROOM TEMPERATURE, NO $P'$ GENERATION; CURING CONDITION B - ROOM TEMPERATURE WITH $P'$ GENERATION; CURING CONDITION C - ELEVATED TEMPERATURE, NO $P'$ GENERATION; AND CURING CONDITION D – ELEVATED TEMPERATURE WITH $P'$ GENERATION. ......................................................................................................................... 68

FIGURE 5.1 HYDRAZATION CELL EXPERIMENTAL SET UP WITH STRAIN GAUGES ................................................. 74

FIGURE 5.2 MEASURED VOLUME CHANGE FROM CP ASTM TESTS ADJUSTED FOR EVAPORATION ...... 76

FIGURE 5.3 MEASURED VOLUME CHANGE FROM CPB ASTM TESTS ADJUSTED FOR EVAPORATION.... 76

FIGURE 5.4 CP ASTM TEST DATA NORMALISED BY MASS OF BINDER ................................................................. 78

FIGURE 5.5 CP ASTM TEST DATA NORMALISED BY VOLUME OF BINDER ............................................................. 78

FIGURE 5.6 CPB ASTM TEST DATA NORMALISED BY MASS OF BINDER ............................................................... 79

FIGURE 5.7 CPB ASTM TEST DATA NORMALISED BY VOLUME OF BINDER ............................................................ 79

FIGURE 5.8 BENDER ELEMENT MEASURED STIFFNESS, $K_s$ (MPA), MEASURED IN THE HYDRATION CELL 80

FIGURE 5.9 EFFECTIVE STRESS DEVELOPMENT, $P'$ (kPA), MEASURED IN THE HYDRATION CELL .......... 81

FIGURE 5.10 COMPARISON OF CP AND CPB NORMALISED CHEMICAL SHRINKAGE IN EQUIVALENT W/C RATIO SPECIMENS .................................................................................................................. 83

FIGURE 5.11 PARTICLE SIZE DISTRIBUTION OF LIMESTONE FILLER (BOUASKER ET AL., 2008) COMPARED WITH TAILINGS ........................................................................................................................ 84

FIGURE 5.12 COMPARISON OF SI CPB PROCESS WATER (TDS = 482 MG/L) VERSUS TAP WATER (TDS = 464 MG/L) CHEMICAL SHRINKAGE ........................................................................................................... 86

FIGURE 5.13 COMPARISON OF KB CPB PROCESS WATER (TDS = 210,000MG/L) VERSUS TAP WATER (TDS = 464MG/L) CHEMICAL SHRINKAGE ........................................................................................................... 86

FIGURE 5.14 COMPOSITION OF CEMENT PASTE BEFORE HYDRATION ................................................................. 87

FIGURE 5.15 DETERMINATION OF $E_u$ AND $K_u$ FOR ASTM CP TESTS .............................................................. 89

FIGURE 5.16 DETERMINATION OF $E_u$ AND $K_u$ FOR ASTM CPB TESTS ............................................................ 89

FIGURE 5.17 CHEMICALLY INDUCED STRAIN $\varepsilon_{sh\text{ASTM}}(T)$ CALCULATED FROM EQUATION 5.16 FOR ASTM CP TESTS .......................................................................................................................... 91

FIGURE 5.18 CHEMICALLY INDUCED STRAIN $\varepsilon_{sh\text{ASTM}}(T)$ CALCULATED FROM EQUATION 5.16 FOR ASTM CPB TESTS ........................................................................................................................ 92

FIGURE 5.19 HYDRATION MODEL, EQUATION 5.25, MATCHED TO EXPERIMENTAL BENDER STIFFNESS, $K_s$ ................................................................................................................................. 94

FIGURE 5.20 HYDRATION MODEL, EQUATION 5.16, MATCHED TO EXPERIMENTAL EFFECTIVE STRESS generation, $P'$ .................................................................................................................. 95

FIGURE 5.21 HYDRATION CELL CHEMICAL SHRINKAGE DEVELOPMENT, $\varepsilon_{sh\text{HYD}}(T)$, FROM EQUATION 5.16 ................................................................................................................................................. 96

FIGURE 5.22 EFFECT OF POISSON'S RATIO ON SI BULK STIFFNESS, $K_i$........................................................................ 98
FIGURE 7.9 LOCATIONS OF PORE PRESSURE MEASUREMENTS WITHIN HYDRATION CELL CONSOLIDATION SPECIMEN AT POINTS A, B, C AND D ................................................................. 131
FIGURE 7.10 EXCESS PORE PRESSURE DISTRIBUTION WITHIN SPECIMEN ................................................. 132
FIGURE 7.11 EXPERIMENTAL ISOCRONES OF EXCESS PORE PRESSURE DISTRIBUTION WITHIN A RL SPECIMEN AT T = 0, 10, 20 AND 30 HOURS COMPARED WITH THEORETICAL ISOCRONES ........ 133
FIGURE 7.12 THE THEORETICAL EFFECTIVE STRESS PROFILE (FROM ISOCRONES IN FIGURE 7.2) WITHIN A SPECIMEN CONSOLIDATING IN THE HYDRATION CELL ........................................... 135
FIGURE 7.13 EXCESS PORE PRESSURE DISSIPATION (U) AND VOLUMETRIC STRAIN (εV) DUE TO INCREMENTSAL STRESS APPLICATION THROUGH INCREMENTAL REDUCTIONS OF APPLIED BACK PRESSURE σb (SPECIMEN RL4) ........................................................................................................ 136
FIGURE 7.14 CONSOLIDATION PORE PRESSURE AND VOLUME CHANGE BEHAVIOUR PROFILES FOR SPECIMENS RL6 AND RL7 .................................................................................................. 137
FIGURE 7.15 < 75µm PSD FOR TOP AND BASE SLICES (EXTREMITIES) OF SPECIMEN RL6........ 138
FIGURE 7.16 < 75µm PSD FOR TOP AND BASE SLICES (EXTREMITIES) OF SPECIMEN RL7........ 139
FIGURE 7.17 VOID RATIO (ε) WITH VERTICAL EFFECTIVE STRESS VARIATION (σV OR p’) ON AN UNCEMENTED RL SPECIMEN USING AN OEDOMETER CELL .................................................. 140
FIGURE 7.18 VOID RATIO (ε) VARIATION AT 60kPa VIRGIN EFFECTIVE STRESS IN OEDOMETER CONSOLIDATION OF AN UNCEMENTED RL SPECIMEN ........................................................................... 140
FIGURE 7.19 CONSOLIDATION PORE PRESSURE PROFILE COMPARING EFFECTS OF WATER QUALITY ON CONSOLIDATION - UNCEMENTED RALEIGH SPECIMENS RL11 AND RL12 MIXED WITH PROCESS AND TAP WATER RESPECTIVELY ................................................................. 142
FIGURE 7.20 LIQUEFIED SPECIMEN RL5 CONSOLIDATION PROFILE ...................................................... 144
FIGURE 7.21 EFFECT OF SPECIMEN SUPPORT MOULD AND SOLIDS REUSE ON CONSOLIDATION PROFILE IN TESTS RL8 AND RL9 ........................................................................................................ 145
FIGURE 7.22 CLOSE UP OF FIGURE 7.21: 0 < τ < 18 HOURS ........................................................................... 146
FIGURE 7.23 RL SPECIMEN TEMPERATURE GENERATION ........................................................................ 147
FIGURE 7.24 RL15, 16 AND 17 SPECIMEN PREPARATION FLOW CHART ................................................ 148
FIGURE 7.25 RL CPB PORE PRESSURE REDUCTION .................................................................................. 149
FIGURE 7.26 RL CPB STIFFNESS ........................................................................................................ 150
FIGURE 7.27 RL CPB UCS ........................................................................................................ 150
FIGURE 7.28 SOURCE FUNCTION .................................................................................................. 154
FIGURE 7.29 CONSOLIDATION PROFILE WITH SOURCE TERM (Cp = 1, s = 0.2, dt = 0.05 AND K0 = 11) 154
LIST OF TABLES

Table 3.1 Tailings Specific Gravity ................................................................. 19
Table 3.2 Tailings Chemical Analysis - Semi-Quantitative XRF .................. 21
Table 3.3 Tailings Chemical Analysis - Semi-Quantitative XRD .................. 21
Table 3.4 Tailings Chemical Analysis - Organic and Inorganic Carbon ...... 22
Table 3.5 Tailings Chemical Analysis - Total Dissolved Metals ................. 22
Table 3.6 Water Chemical Analysis - Alkalinity, Electrical Conductivity, Hardness, pH and Total Dissolved Solids (TDS) .............................................................. 23
Table 3.7 Water Chemistry Analysis - Anions ............................................. 23
Table 3.8 Water Chemistry Analysis – Total Dissolved Metals ................. 24
Table 3.9 Binder Specific Gravity ................................................................. 24
Table 3.10 CPB Mix Properties ................................................................. 25
Table 4.1 Material Thermal Properties for Temp/W Modelling from McWilliam (2011) ... 39
Table 4.2 Testing Matrix of Experimental Curing Conditions for Temperature and Effective Stress Testing .................................................................................... 54
Table 4.3 Completed Temperature Hydration Cell Tests ............................ 55
Table 5.1 CPB Mix Properties ................................................................. 73
Table 5.2 CP Mix Properties ................................................................. 73
Table 5.3 Mass and Volume of Binder Used in ASTM Test Specimens ....... 77
Table 5.4 ASTM Test Model Parameters .................................................. 91
Table 5.5 Hydration Model Parameters $K_{\alpha}$, $\lambda$, $K_{\lambda}$, $K_{\nu}$, and $\xi_{\nu}$ for Hydration Cell Data ................................. 95
Table 5.6 Comparison of ASTM and Hydration Cell Strains and Equivalent Volume Change ................................................................. 96
Table 5.7 Required Stiffness, $K_{\alpha}$, Required to Match $\xi_{\nu,ASTM}$ and $\xi_{\nu,HYD}$ ................................................................. 97
Table 7.1 Consolidation Problem Boundary Conditions .......................... 123
Table 7.2 Consolidation Testing Mix Properties ........................................ 127
Table 7.3 Measured Permeabilities of Specimens RL6 and RL7 ............... 137
Table 7.4 Volume and Fines Fraction for RL6 and RL7 Fines PSD ......... 139
Table 7.5 $C_v$ Values from Consolidation Analyses of Uncemented Raleigh Specimens ..... 147
Table 7.6 Electrical Conductivity of Decant Water from Step#3 (Figure 7.24) in Test RL16 and RL17 ................................................................. 148
Table 7.7 Consolidation Summary .......................................................... 152
Table 7.8 Location of Chemical Testing Results ......................................... 155
This thesis would not have been successfully completed were it not for the involvement of a number of important people…

To my supervisors Professor Andy Fourie and Dr. James Doherty - Thank you for your expertise and guidance throughout the many questions and frustrations this research has produced. Your support has been invaluable. Professor Martin Fahey, thank you for planting the research seed in my mind and to Dr Matt Helinski, for the assistance and advice provided in the early days of this PhD.

Thank you to final year engineering student, Heather McWilliams for her assistance in the construction and calibration of experimental laboratory equipment developed as part of this research. Under my supervision, as her final year honours project, Heather conducted the initial TEMP/W modelling and initial experimental testing on the SNM tailings associated with the work presented in Chapter 4. Thank you to Professors David White, Antonio Carraro, and Carolyn Oldham for their technical expertise and insights with regard to the work presented in Chapter 7.

A special thanks to the soils and electronic laboratory and workshop staff, in particular Binaya Bhattarai, Usha Mani, Behnaz Abdollahzadeh, Claire Bearman, John Breen, Khin Seint, Shane De Catania, Neil McIntosh and Jim Waters. Your technical support and humorous banter has kept me sane.

The financial support provided an Australian Postgraduate Award, UWA Safety Net Top Up Scholarship and Externally-funded Ad-hoc Scholarship (Effective Stress Approach to Mine Backfill) is gratefully acknowledged.

Thank you to my colleagues and friends Lucile Quéau, Nathalie Boukpeti, Britta Bienen, Daniela Ciancio, Lisa Melvin, Cristina Vulpe, Susie Gourvenec, Yuxia Hu, Emma Leitner and Lavanya Avadiar. I am grateful for the friendship we have developed over the past four years. Thank you for the inspiration you have all provided.

To Pi Walker, thank you for the hours of chapter reading, and to Megan Surrette, you have been full of tireless encouragement from the very beginning. Thank you for being my personal cheer squad.

To Dan Jolley, your encouragement in the final months helped me across the line. Thank you for the support and fun you’ve provided during what would otherwise have been a very stressful time.

Finally, and most importantly, I would like to thank my Mother. Without your continual love, support and encouragement I would not be the person I am today. I cannot thank you enough for your sacrifices and my upbringing.
List of symbols

Roman

$\infty$  Infinity
$a$  Cross-sectional area
$B$  Skempton’s pore pressure parameter
$C$  Molar concentration
$C_c$  Cement content by % weight
$C_s$  Solids content by % weight
$C_{sand}$  Sand content by % weight
$c_v$  Coefficient of consolidation
$d$  Drainage path length
$e$  Void ratio
$e_o$  Initial void ratio
$E^*$  1D constrained stiffness parameter
$E_h$  Efficiency of hydration
$E_o$  Constrained 1D stiffness modulus
$g$  Gravity
$G_c$  Specific gravity of binder/cement
$G_{f, tails}$  Specific gravity of filler/tailings
$G_{f, sand}$  Specific gravity of sand
$G_t$  Equivalent specific gravity non-binder solids material
$G_o$  Small strain shear stiffness
$h$  Elevation head (Chapter 3)
$H(t)$  Height of fill in stope varying with time (Chapter 6)
$i$  Hydraulic gradient
$k$  Permeability
$K^*(t)$  Composite pore-fluid elastic stiffness
$K_i, K_s$  Bulk elastic stiffness
$K_{id}$  Initial specimen stiffness
$K_w$  Bulk stiffness of pore fluid
$L$  Specimen length
$M$  Bulk specimen mass
$M_c$  Mass of cement
$M_f$  Mass of filler/tailings
$M_w$  Mass of water
$m$  Fill rate
$m_c$  Mass of cement per unit volume
$m(t)$  Maturity function
$n$  Porosity
$p$  Total stress
$p'$  Effective stress
$q$  Flow rate
$q_u$  Peak unconfined compressive strength (UCS)
$R$  Molar gas constant
$S$  Source function used to model pore pressure rebound (Chapter 6)
$s$  Time at which source function is applied (Chapter 6)
$t$  Time (Chapter 4)
$t$  Time since fill commencement (Chapter 4, 6)
$t$  Time since the commencement of hydration cell/consolidation test (Chapter 5, 7)
$t'$  Time since the commencement of hydration (Chapter 6)
$T, T_v$  Consolidation time factor (Chapter 6, 7)
$T_a$  Ambient surrounding temperature
$T_G$  Adiabatic temperature
$T_k$  Temperature of fluid
$T_P$  Initial placing temperature
$T_s$  Surface specimen temperature, $T_s = T_v$
$T_v$  Volumetric mean specimen temperature (Chapter 4)
$u$  Pore pressure
$u_{mid}$  Internal specimen pore pressure
$U$  Normalised excess pore pressure
$\bar{u}$  Excess pore pressure
$\bar{u}_t$  Average excess pore pressure after time $t$
$\bar{u}_0$  Initial excess pore pressure
$u_b$  Pore pressure at base of specimen
<table>
<thead>
<tr>
<th>Symbol</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>$u_l$</td>
<td>Pore pressure at top of specimen</td>
</tr>
<tr>
<td>$V$</td>
<td>Pore fluid expelled due to consolidation</td>
</tr>
<tr>
<td>$V_c$</td>
<td>Volume of binder/cement</td>
</tr>
<tr>
<td>$V_s$</td>
<td>Shear wave signal arrival time (Chapter 3)</td>
</tr>
<tr>
<td>$V_s$</td>
<td>Volume of solid phase (Chapter 5)</td>
</tr>
<tr>
<td>$V_{sh}$</td>
<td>Chemical shrinkage volume</td>
</tr>
<tr>
<td>$V_t$</td>
<td>Total specimen volume</td>
</tr>
<tr>
<td>$V_w$</td>
<td>Volume of pore fluid</td>
</tr>
<tr>
<td>$w/c$</td>
<td>Ratio by mass of water to cement</td>
</tr>
<tr>
<td>$z$</td>
<td>Height above base of stope</td>
</tr>
<tr>
<td>$Z$</td>
<td>Drainage path ratio/normalised specimen length</td>
</tr>
</tbody>
</table>

**Greek**

<table>
<thead>
<tr>
<th>Symbol</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\zeta$</td>
<td>Internal integration coefficient (Fahey et al., 2010)</td>
</tr>
<tr>
<td>$\Delta$</td>
<td>(as prefix) used to denote change from initial or reference value</td>
</tr>
<tr>
<td>$\delta$</td>
<td>(as prefix) used to denote incremental change</td>
</tr>
<tr>
<td>$\varepsilon_a$</td>
<td>Axial strain</td>
</tr>
<tr>
<td>$\varepsilon_h$</td>
<td>Volumetric strain due to chemical shrinkage at full hydration (Chapter 5)</td>
</tr>
<tr>
<td>$\varepsilon_{sh}$</td>
<td>Volumetric strain due to chemical shrinkage developing with time</td>
</tr>
<tr>
<td>$\varepsilon_{sh,f}$</td>
<td>Volumetric strain due to chemical shrinkage at full hydration (Chapter 6)</td>
</tr>
<tr>
<td>$\varepsilon_V$</td>
<td>Volumetric strain</td>
</tr>
<tr>
<td>$\gamma$</td>
<td>Material bulk unit weight</td>
</tr>
<tr>
<td>$\gamma_{dry}$</td>
<td>Material dry unit weight</td>
</tr>
<tr>
<td>$\gamma_w$</td>
<td>Pore fluid unit weight</td>
</tr>
<tr>
<td>$\kappa^*$</td>
<td>Parameter governing pore pressure due to shrinkage strain</td>
</tr>
<tr>
<td>$\kappa_h$</td>
<td>Parameter governing rate of hydration</td>
</tr>
<tr>
<td>$\lambda$</td>
<td>Heat loss characteristic</td>
</tr>
<tr>
<td>$\lambda_{KS}$</td>
<td>Ratio of final stiffness after full hydration to initial stiffness</td>
</tr>
<tr>
<td>$\nu$</td>
<td>Poisson’s ratio</td>
</tr>
<tr>
<td>$\pi$</td>
<td>Osmotic pressure</td>
</tr>
<tr>
<td>$\rho$</td>
<td>Material density</td>
</tr>
<tr>
<td>$\rho_w$</td>
<td>Water density</td>
</tr>
<tr>
<td>$\sigma$</td>
<td>Total stress</td>
</tr>
<tr>
<td>$\sigma'$</td>
<td>Effective stress</td>
</tr>
<tr>
<td>$\sigma_V'$</td>
<td>Vertical effective stress measured in oedometer</td>
</tr>
</tbody>
</table>
\( \sigma_b \)  Back pressure
\( \sigma_c \)  Cell pressure

List of subscripts

\textit{ASTM}  Indicates parameter determined from ASTM test
\textit{Hyd}  Indicates parameter determined from hydration cell test

List of abbreviations

1D  One-dimensional
\textit{ASTM}  American Society for Testing and Materials
CPB  Cemented paste backfill
CP  Cement paste
dI  De-ionised water
EC  Electrical conductivity
GP  General purpose cement
KB  Kanowna Bell Gold Mine
LVDT  Linear variable differential transformer (displacement transducer)
OPC  Ordinary Portland cement
PPT  Pore pressure transducer
PSD  Particle size distribution
QA/QC  Quality control/quality assurance
RL  Raleigh Mine
S.D.  Standard deviation
SEM  Scanning electron microscopy
SI  St Ives Gold Mine
SNM  Savannah Nickel Mine
TDS  Total Dissolved Solids
TIC  Total inorganic carbon
TOC  Total organic carbon
TSF  Tailings storage facility
UCS  Unconfined compressive strength
UWA  University of Western Australia
XRD  X-ray Diffraction
XRF  X-ray fluorescence
1.1. Background

1.1.1. The backfilling process

The extraction of ore in underground mining results in the production of large, potentially unstable underground voids, or “stopes”. To support these underground excavations, columns or pillars of valuable material must be left behind to ensure local and regional geotechnical stability of the ground mass. However, leaving this valuable material underground decreases the financial return of the mine. Mine backfilling is a common technique used to fill excavated stopes, such that these pillars are no longer necessary and can instead be mined, increasing ore recovery without compromising on stability and thus safety. Figure 1.1 shows a schematic of a stope-mining operation, showing the interactions of the mining, processing and backfilling stages as they occur in a typical operation. The features of the operation most pertinent to the backfilling process, referred to by the letter indicated in Figure 1.1, are:

A  Ore extraction from stope.

B  Concentrator and thickener where ore is processed and full stream tailings produced directly for backfilling or storage in the tailings storage facility, or TSF (G).

C  Backfill plant where the fill components are mixed.

D  Delivery system transporting the fill from the backfill paste plant to the stope.

E  Backfill deposition into an excavated stope.

F  Blasting of a stope, exposing adjacent backfill.
Historically, backfilling has utilised waste rock or coarse tailings material, mixed with water and cement, or other binding agent, due to the complexities associated with the use of finer tailings fraction. However, the development of paste backfill, referred to as cemented paste backfill...
(CPB), has allowed successful utilisation of full-stream tailings, reducing the requirement for TSF-use without additional pre-processing of the tailings required. The addition of fines into the mix creates a paste-like material, reducing the potential for segregation of the backfill, as well as reducing the production of bleed/excess water during transportation, filling and curing (Potvin et al., 2005). This largely negates the need for underground backfill water management programs.

Figure 1.2 shows a schematic of a typical stope-filling scenario, where the CPB is deposited from the top of the stope, and confined by a constructed barricade across the opening or "draw point" at the base, through which the ore was originally removed.

![Figure 1.2 Typical stope backfilling situation with typical dimensions (from Helinski, 2007)](image)

With hydration of the cement, the CPB slurry becomes self-supporting, gaining stiffness and strength. When sufficient strength has developed, the secondary stope adjacent to the CPB mass may be mined with the resulting excavation supported by the hydrated CPB. It is crucial to understand when CPB will gain adequate strength, and the rate at which this occurs, so as to minimise the mining cycle without compromising the safety of the mining environment. Exposure of the CPB stope before the material is strong enough could result in significant safety issues should the material fail, potentially flowing through the underground workings, or a rock mass failure due to lack of CPB support. This may also be compounded by potential economic losses due to dilution of the ore being excavated. On the other hand, if exposure is delayed beyond the development of adequate material properties, this adds unnecessary time to the mining schedule, which can have significantly negative economic implications for the mining operation.
Historically, CPB design has been based on heuristics, resulting in either inefficient, conservative mix designs with a higher than necessary cement content, or the converse, whereby potentially weak materials may cause serious safety risks on exposure. An understanding of the material behaviour is required to achieve efficient mix designs for optimal CPB performance, optimising the cement content and associated costs, while minimising potential safety risks. The cost of CPB is highly dependent on the cement content. Due to the very large volumes of backfill involved, even a small reduction in cement/binder content can translate to a significant cost savings. Typical costs associated with the use of CPB in the Australian mining environment can range from $16/m³ to $65/m³ depending on the backfill plant design, cement content and the amount of backfill being produced (Andrews et al., 2010). This may represent 5-10% of overall mining costs, of which as much as 50% may be associated with the cost of the cement (Andrews et al., 2010).

1.1.2. CPB behaviour

The behaviour of CPB has been found to be largely controlled by two mechanisms; self-weight consolidation associated with drainage, and self-desiccation associated with the cement hydration process (Helinski et al., 2011a). Both of these processes are ongoing, such that material properties are continually changing as consolidation and hydration progress. Understanding the rate at which these mechanisms progress and most importantly their effect on material property development with time (i.e. strength, stiffness increase and permeability reduction), is critical for the successful use of CPB.

In addition to achieving adequate stability when a filled stope is exposed, containment prior to the development of self-supporting properties (i.e. during both filling and curing) is also critical. This is provided through the construction of containment barricades, as shown in Figure 1.2. These barricades are structurally sufficient to retain the solid portion of the CPB and can be made adequately permeable, via the insertion of drainage pipes, facilitating self-weight consolidation via drainage of pore fluid from the fill. Typically the rate at which the CPB is deposited into the stope (referred to as filling rate) may be sufficiently high, that in combination with low initial coefficient of consolidation of the fine-grained backfill, the filling of the stope may occur under undrained conditions. This may apply loading to the barricade equal to the self-weight of the overlying backfill, due to the hydrostatic conditions (Fahey et al., 2010). If there is no reduction in pore pressure, then the stress on the barricade does not reduce and the barricade must be designed to withstand this load. However, as soon as pore pressure dissipation commences then effective stress develops, reducing the loading on the barricade and promoting strength within the CPB. This process is often aided by the introduction of a rest period after initial CPB deposition; referred to as a plug pore (Helinski et al., 2011b; Thompson et al., 2011; Thompson et al., 2012). CPB is deposited into the stope until the fill height surpasses that of the brow of the barricade. The CPB is then left to cure for a period of time, typically 24 hours, after which filling again commenced. During the rest period, effective stress
is generated due to pore pressures reduction, which will help to reduce the total stress placed on the barricade when filling recommences.

The reduction in pore pressure occurs through a combination of conventional self-weight consolidation as well as cement hydration. The hydration of the cement induces a reduction in volume and thus reduction in pore pressure, similar to the self-desiccation mechanism in concrete (Grabinsky & Simms, 2006; Helinski et al., 2007a). Thus, an understanding of the consolidation and hydration process and how they interact during the filling and curing of a CPB filled stope has significant importance in management of stope backfilling.

1.1.3. Design of CPB

Laboratory testing of CPB is the most prominent aspect of both design and quality control/quality assurance (QA/QC) of CPB mixes (Cayouette, 2003; Revell, 2004). Such testing typically involves mixing and curing CPB with appropriate proportions of tailings, binder and water to produce the mix. The mix is then cast into small cylindrical moulds (approximately 50mm in diameter) that are then cured in a humid environment to prevent moisture loss from the specimen via evaporation. Specimen strength, measured via unconfined compressive strength (UCS), is then assessed at different curing times - often 7, 14 and 28 days. The achieved strengths are then translated to industry-developed rules-of-thumb specifying strength requirements based on the UCS, or specially developed CPB numerical models which predict strength and behaviour (Helinski et al., 2007b).

As such, laboratory curing and testing plays a crucial role in the CPB mix design, yet one of the most significant issues in the design process relates to this discrepancy between in situ and laboratory curing conditions. Numerous anecdotal stories and published studies (Cayouette, 2003; Revell, 2004; le Roux et al., 2005, Grabinsky & Bawden, 2007) have indicated that material cured in situ exhibits significantly greater strengths than equivalent mixes cured in a laboratory environment. Mix design based on laboratory testing requires calibration and correlation between actual behaviour in the stope and lab-cured specimen performance due to the empirical nature of the design procedure (Cayouette, 2003; Fall et al., 2010). The necessity for such empirical comparisons has been shown to relate to the difference in curing procedures, specifically due to the stress and temperature conditions in situ compared with a laboratory environment. Understanding the effect of these differences on CPB performance is a key focus in this thesis. In typical laboratory curing procedures, effective stress is not able to generate as would occur in the stope. This is due to the lack of applied total stress and subsequent reduction in pore pressures due to consolidation or self-desiccation. While the latter still occurs in a laboratory environment, it does not have the same effect as in stope curing. Underestimation of the ultimate properties, when the fill is exposed through mining of adjacent stopes, has significant safety and associated financial implications as previously suggested. Alternatively, over-conservatism in a design may increase turnaround times in backfilling and exposure of stopes. Understanding the rate at which material properties develop in the hydrating fill is thus crucial for the safe and economical operation of a mine.
The superior strength exhibited by \textit{in situ} cured CPB is attributed largely to effective stress generation (Helinski \textit{et al.}, 2011b; Thompson \textit{et al.}, 2011), the development of which is further enhanced by elevated curing temperatures. Helinski \textit{et al.}, (2007a) observed that the typical curing protocols in the laboratory did not allow for the generation of effective stresses in the same way that they occurred in the stope and developed a laboratory testing technique to assess the self-desiccation mechanism. Laboratory studies have also indicated that the elevated temperatures which occur underground compared with a laboratory environment (Fall \textit{et al.}, 2007), also significantly contribute to the increase \textit{in situ} strength (Fall \& Samb, 2009; Fall \textit{et al.}, 2010; Fall \textit{et al.}, 2007; Nasir \& Fall, 2009, 2010). The combined effect of effective stress generation and elevated temperature curing on CPB behaviour has not been quantified in the literature, despite their significant independent contributions to mechanical properties. A developed understanding of their collective impact on CPB material properties is crucial for efficient design of CPB mixes.

The laboratory protocol developed by Helinski and colleagues (Helinski \textit{et al.}, 2007a; Fahey \textit{et al.}, 2011) has offered significant improvement in the methodology of CPB mix design. However, the specialised equipment and time required for CPB testing may often be expensive and time consuming. The measurement of chemical shrinkage (the reduction in volume that occurs due to cement hydration) is routinely used in the design of cement pastes, mortars and concrete as the increase of chemical shrinkage provides an indication of hydration progress, which in turn can be correlated with changing material properties such as strength. This approach has not previously been used for CPB. However, it has the potential to provide a cheap and effective index test or screening tool to evaluate a range of different CPB mix recipes, prior to detailed laboratory assessment, assuming that the chemical shrinkage occurring in the specimen can be correlated with other CPB properties, such as stiffness increase or pore pressure reduction.

\textbf{1.2. Research objectives}

The results of \textit{in situ} studies (Thompson \textit{et al.}, 2011; Helinski \textit{et al.}, 2011b; Thompson \textit{et al.}, 2012); including coring and instrumentation of stopes during filling and curing, has provided valuable insight into the behaviour of \textit{in situ} CPB. However, such studies are very expensive and logistically challenging in an active mining environment. As such, laboratory testing still remains an integral part of the CPB design process. Without reliable laboratory testing techniques, empirical judgements must be made in the design process to relate the laboratory-measured properties to that of the \textit{in situ} material.

This thesis focuses on the use of experimental CPB element testing to examine and improve on laboratory testing techniques, specifically examining the coupled effects of stress and temperature on the development of stiffness and strength and the potential for chemical shrinkage testing to be used as an initial stage in the CPB mix design process. The experimental testing conducted in this thesis is focussed on the development of laboratory testing techniques to better replicate mechanisms critical to the \textit{in situ} curing process and
improve understanding of the behaviour and properties of very early age CPB (i.e. cementing, rather than cemented). Many “early ages” studies of CPB have tested material after 14 days of curing, instead, much of the experimental work conducted in this thesis occurs at 7 days or less. Understanding of the behaviour of CPB at this very early age is important for barricade design and construction, where understanding of the stress within the fill it retains may have significant implications for optimal barricade design.

The ultimate aim is to remove the inherent conservatism from the design process, providing valid scientific reasoning to justify potential cost saving via cement/binder reductions without compromising on safety. Specifically this thesis focuses on:

(i) building on the existing effective stress curing methodology (Fahey et al., 2011) by investigating the combined effect of effective stress and elevated temperature curing;

(ii) investigating the role of chemical shrinkage in the hydration of CPB and the potential for the measurement of this property to act as an index test for the ultimate design properties of CPB;

(iii) consideration of previously unexamined/unreported factors associated with self-weight consolidation which are subsequently established to be critical to the ultimate behaviour of CPB;

(iv) a qualitative relationship relating the self-weight consolidation pore pressures to those induced by self-desiccation, providing an analytical design tool with capabilities to predict barricade loading during CPB filling of a stope.

While no definitive design procedures or protocols are produced directly from this work, the research presented clearly highlights the importance of environmental factors as well as physical and chemical properties of mix constituents on the behaviour of CPB.

1.3. Thesis organisation

This thesis is organised into eight chapters and a single appendix. An outline of each individual chapter is presented here:

Chapter 2 presents an literature review which provides the reader with a general understanding of previous research relating to the development and use of CPB. At the beginning of each subsequent chapter, additional literature is reviewed as specifically relevant to the contents of that chapter.

Chapter 3 provides details of the various materials and methodologies utilised in the experimental laboratory testing components of this thesis. Physical properties and chemical composition of individual mix components (tailings, water and binder) are provided as well as details of the various CPB mixes studied. Descriptions and usage of standard experimental apparatuses are described here. Specific modifications are made to some of the apparatuses used in this research; such modifications are discussed in the relevant chapter.
Chapter 4 presents the development and testing of a hydration cell, modified to allow temperature control throughout laboratory curing of CPB specimens, replicating \textit{in situ} temperature conditions. The literature relating to the effects of temperature on cement hydration is presented and discussed in detail, highlighting the large contribution of temperature to the improvement of cemented material properties during hydration. Details of the development, calibration and testing of the temperature hydration cell are presented. The modified cell is then used to examine the individual and combined effects of effective stress and elevated temperature curing.

Chapter 5 examines the potential for chemical shrinkage measurements to be used as an index test in the design of CPB mixes. A hydration model is developed to relate pore pressure, small strain shear stiffness and chemical shrinkage. The ability to relate these properties through the model is then examined using the standard hydration cell test and ASTM C 1608-07 for the measurement of chemical shrinkage of hydraulic cement paste respectively.

Chapter 6 combines the effect of self-weight consolidation (from Gibson’s solution) and chemical shrinkage to develop a qualitative method of pore pressure determination in hydrating CPB.

Chapter 7 investigates the occurrence of unusual pore pressure behaviour during one-way consolidation of both CPB and uncemented tailings mixes. A range of experimental tests are conducted to examine potential physical mechanisms with potential to cause the anomalous observed consolidation pore pressure behaviour. The role of chemistry in the physical behaviour of the CPB and uncemented tailings mixes are also considered.

Chapter 8 discusses the main conclusions of this work and highlights avenues of further research.
2.1. Introduction

Mine backfilling techniques have been utilised for many years in a variety of different mines, comprising a large variety of different material including rock fill and hydraulic fill. In recent years, as the mining process has been optimised due to the development of new processes and equipment, increasing value is placed on ore recovery. This has also lead to changes in the properties of the mine waste produced. Cemented paste backfill (CPB) has developed in response to this demand, making use of full-stream mine tailings. CPB design has largely been based on industry developed rules-of-thumb, and as such there is significant room for optimisation in the design process. Curing conditions, specifically stress and temperature, have been observed to significantly affect the behaviour and due to the wide range of chemical compounds incorporated in a CPB mix due to different tailings or binder types and water quality, chemistry also plays a role.

CPB material does not behave like a cement paste or a concrete, yet the addition of binder, even in the small quantities that are used, modifies the behaviour of the material sufficiently that it no longer behaves like a soil, with material properties continually changing as the cement hydrates. As such, significant consideration and investigation has occurred into the behaviour of this fine-grained cemented material to understand how it behaves and how it can be used most effectively.

This chapter presents a summary of the current knowledge base with regard to the understanding of CPB behaviour and properties and the factors on which these depend. Particular consideration is given to the differences in curing conditions between an in situ stope and laboratory environment and specifically the role that this environment plays on the behaviour of the hydrated product. This is especially important given observed differences between in situ and laboratory cured material. An overview of the current techniques adopted to deal with these observed discrepancies in behaviour is provided and the areas where additional research may provide improved understand is highlighted.

2.2. The curing environment

The accepted industry definition of CPB states that 15% of the solid particles must be less than 20µm in diameter, no bleed water or segregation is produced during transportation or filling and the rheological behaviour can be described as non-newtonian (Potvin et al., 2005). A typical CPB mix is comprised of 70 - 80% solid material and typically 2 - 6% of that by weight is
cement/binder addition (Potvin et al., 2005). This percentage of solids within the mix allows the maximum volume of tailings to be utilised without compromising the mix rheology required for hydraulic transportation.

The design of CPB is typically conducted based on parametric studies to examine the cement and solids content necessary to achieve the mechanical properties required for adequate performance in an underground environment (Fahey et al., 2011). Such a study typically comprises laboratory mixing and curing of CPB followed by unconfined compressive strength (UCS) testing. The UCS is a crucial parameter in the subsequent design process, either through empirical design (Fall et al., 2010) or subsequent numerical modelling of the CPB behaviour (Nasir & Fall, 2010).

Many studies examining the performance of CPB have established that the CPB cured in situ exhibits better strength performance than an equivalent mix cured in a laboratory environment (Cayouette, 2003; Revell, 2004; le Roux et al., 2005). Revell (2004) compared UCS strength of laboratory prepared and in situ cored specimens; the latter recovered using a coring technique, and found that the in situ cored specimens exhibited almost three times the strength of the laboratory specimens. Given that accurate understanding of the in situ performance of CPB is the ultimate aim, laboratory testing is only conducted to characterise the strength of the material in the stope, Revell (2004) carried out additional testing with the aim of developing improved laboratory techniques such that the laboratory testing may provide a more accurate representation of the in situ material behaviour. The key findings were that significant “strength” was gained via fast strain rates when conducting UCS testing and that cubic specimens exhibited greater strength due to their 1:1 width to height ratio, compared with the more standard cylindrical specimens with a 1:2 width to length ratio. Perhaps most importantly, Revell (2004) observed that unsaturated fill exhibited larger strengths. In the case of the in situ core versus laboratory testing, poor storage of specimens post-coring and pre-testing reduced the saturation of the cored fill, artificially increasing the strength of the in situ material due to the generation of suctions. This has also been examined in the laboratory where specimens which underwent UCS testing in air achieved greater strengths than those tested submerged in a water bath, thereby preventing any suction development (Fahey et al., 2011). Similar behaviour was observed with saturated and unsaturated triaxial testing (le Roux et al., 2005). Evidence of air trapped within a stope-cured CPB fill, incorporated into the fill during mixing and deposition, has also indicated potential in situ strength may be gained from suctions developing in unsaturated fill (le Roux et al., 2005).

An in situ coring program conducted by le Roux et al., (2005), encountered difficulty in obtaining good in situ specimens, where only 48% core recovery was obtained from the drilling program and only five intact samples were obtained from additional Shelby tube sampling. This difficulty in obtaining good quality in situ sampled suggests that engineering judgement must be exercised when analysing the results of subsequent sample testing. Depending on the quality of the in situ sample recovery, as well as subsequent handling and storage, resulting strengths may be higher than that of the average material in the stope. Similarly for laboratory curing and
testing, which typically exhibit less variability than *in situ* results (Le Roux *et al.*, (2005) where the strengths obtained are easily reliant on specimen size and UCS testing conditions. While such techniques may account for some of the difference in strength between the different CPB, and these issues certainly should not be discounted, even with these differences, the *in situ* material is still sufficiently stronger to warrant further investigation.

To further understand the actual conditions under which CPB cures within a stope and better understand the difference in properties between *in situ* and laboratory cured material, a number of field instrumentation studies have been conducted (Belem *et al.*, 2004; Thompson *et al.*, 2009; Helinski *et al.*, 2011b; Thompson *et al.*, 2012). Such studies have included extensive instrumentation of stopes prior to filling. Total stress, temperature, negative pore pressure sensors and pore pressure transducers (PPT) allow real-time monitoring of the changes in conditions as the stope is filled and then as curing progresses. Where both pore pressures and total stress have been monitored, results of these studies have indicated that initially stope filling occurs under undrained conditions and thus the loading on the barricade, at its maximum, is hydrostatic (Thompson *et al.*, 2009; Helinski *et al.*, 2011b; Thompson *et al.*, 2012). As time and filling of the stope progresses, the pore pressure will begin to reduce compared with the total stress measurements as indicated in Figure 2.1. The effective stress generated as a result is determined by the difference between curves labelled $\sigma_v$ and $u$. The third curve presented, labelled “$\sigma_v$ no arching” will be discussed subsequently.

![Figure 2.1 In situ measurements of total stress and pore pressure (taken from Figure 7, Helinski *et al.*, 2011b)](image)

This reduction in pore pressure ($u$) with respect to total stress ($\sigma$) generates effective stress ($\sigma'$) within the material by
$\sigma' = \sigma_v - u$ \hspace{1cm} \textbf{Equation 2.1}

Helinski et al., (2007b) illustrated this mechanism, and its potential magnitude, in a laboratory environment through monitoring pore pressures within a hydrating CPB specimen. The specimen was cast into a sealed membrane in a modified permeability cell, known as a "hydration cell". A confining cell pressure of 850kPa was applied to the specimen, with a back pressure of 820kPa. The backpressure valve was then closed, hydraulically isolating the specimen within the cell, and the pore pressures monitored. As hydration progressed, the pore pressures were found to reduce significantly, from 820kPa to close to 0kPa after 265 hours. Helinski's hydration cell testing techniques features heavily in this research and the full details of its use and operation are presented in Chapter 3.

Temperature monitoring of stopes during fill and curing indicated that the temperature within the fill is often within the range of 30 to 50°C - higher than typical ambient laboratory temperatures (~23°C) at which laboratory-prepared CPB specimens are typically cured (Thompson et al., 2011, Thompson et al., 2012). Elevated temperature is well known to positively influence cement hydration reactions, increasing the rate and ultimate extent to which they occur (Klieger, 1958; Brooks & Al-Kaisi, 1990; Kim et al., 1998; Fall et al., 2010; Fall et al., 2007). This has been observed to significantly improve the mechanical properties of CPB compared with specimens cured at lower temperatures and thus also accounts for some of the strength/performance discrepancies between \textit{in situ} and laboratory CPB.

\textbf{2.3. An effective stress approach to CPB behaviour}

Helinski developed a framework and software, Minefill2D, within which the consolidation and hydration processes are fully coupled. The model was verified against field experiments (Helinski et al., 2011b) successfully validating the analytical approach. To successfully couple the two mechanisms, a 1D consolidation program “MinTaCo” – \textit{Mine Tailings Consolidation} (Seneviratne et al., 1996) was adapted to create Minefill2D which considers the changes to the consolidation behaviour due to cement hydration. This required taking into account the following:

- stiffness increase of the soil matrix as hydration occurs, as well as that caused by the self-weight compression;
- reducing permeability due to the reduction in pore connectivity resulting from the growth of cement hydration products in the material pores, as well as self-weight consolidation;
- net volume loss due to hydration, leading to the effect known as “self-desiccation”.

Self-desiccation occurs due to the larger volume of unhydrated cement components (cement and water) compared with the hydrated cement product. The hydration process acts like an internal “sink” removing water from the system (Grabinsky & Simms, 2006; Helinski et al., 2007a) The typically low permeabilities of some CPB material means than water is not able to move quickly through the fill to replenish the water volume lost with hydration and given the
increasing stiffness of the material, this small volume change can significantly reduce pore pressures, thereby generating an increase in effective stress. The lower the permeability of the CPB, the greater the generation of effective stress due to hydration. The governing hydration equations developed by Helinski and incorporated in Minefill2D are used to model the pore pressure reduction occurring due to the self-desiccation mechanism, based on a series of laboratory testing. Details of the development of these equations are discussed in Chapter 5.

Conversely, in low permeability materials, the effective stress generated by consolidation is small as it takes a longer time for the fluid to drain from the material, compared with higher permeability material. This is further compounded by decreases in permeability and increases in stiffness due to hydration. (Fahey et al., 2010) examined this through the application of Gibson’s consolidation solution (Gibson, 1958) to the backfilling of mine stopes. (Fahey et al., 2010) described CPB mixes such as that from Savannah Nickel Mine (SNM) for which the permeability was relatively high, allowing quick self-weight consolidation as consolidating fill, compared with CPB from the Kanowna Bell Mine (KB), which is slower to undergo conventional self-weight consolidation, as a non-consolidating fill. This terminology is also applied in this thesis. Thus, in summary, pore pressure reductions in a consolidating fill occur primarily through self-weight consolidation, while the self-desiccation mechanism is more prevalent in a non-consolidating fill. This highlights the inverse nature of these mechanisms and how they each influence the pore pressure. Chapter 6 details the modification of Gibson’s solution-weight consolidation, to include the self-desiccation effects in pore pressure, allowing a qualitative comparison of each mechanism’s effects on pore pressures.

Studies have shown that CPB cured under increasing effective stress (either due to self-weight consolidation, or the self-desiccation mechanism) results in increasing strength (Helinski et al., 2011a; Fahey et al., 2011) allowing the material to become self-supporting and reducing load applied to the containment barricade at the base of the stope. Barricade stress is further reduced through the occurrence of arching within the stope as the CPB develops shear strength and the vertical loading of the fill is partially transferred to the stope walls (Fahey et al., 2009; Fahey et al., 2010). Figure 2.1, shown previously, clearly indicates the difference in total stress within a stope both with and without the occurrence of arching. This phenomenon has also been observed in field instrumentation studies where orthogonally oriented total stress sensors have observed reduction in vertical load with an increase in horizontal stresses as effective stress generates (Thompson et al., 2009). Thus the impact of effective stress generation is two-fold. Not only does the reduction in pore pressure, which generates the effective stress, relieve direct pressure on the barricade, but it also allows arching to occur, which reduces the vertical stress within the stope and thus also the load applied to the barricade.

2.4. Factors affecting hydration

The role of self-weight consolidation in the behaviour of CPB has been discussed above and the mechanism and factors affecting consolidation are generally well understood so the remainder of this literature review will focus on the factors that affect hydration.
Hydration is highly susceptible to chemical and temperature changes as well as the physical characteristics of the fill. The chemistry of a CPB mix is highly complex due to the different chemicals introduced during the mining process; this compounds the often already complex geochemistry of the original ore. The water used in CPB may also have complex chemistry due to the common utilisation of process water, which in addition to being rich in chemicals from its use in mineral processing, can be hypersaline. The chemical complexity of a CPB mix is further increased by the hydration process, throughout which many different compounds are formed. The influence of temperature relates to the hydration/chemical reactions, where elevated temperatures increase the reaction rate and the ultimate extent to which reactions occur. The physical properties of the CPB from initial mixing, to filling of the stope, right through to exposure are continually changing due to the progression of hydration. Most of the changes due to hydration are completed after 28 days, but this may be affected by both physical (temperature and stress) and chemical conditions. As such, how these factors affect the generation of material properties is important to understand, hence the early age, cementing focus of this research.

Helinski’s effective stress approach demonstrates the significant influence that the rate of hydration has on the increase of effective stress and thus properties of CPB. As such an understanding of the factors that influence hydration are imperative to the understanding of CPB behaviour.

2.4.1. Physical properties

The cement content has a positive effect on the increase in strength and stiffness as intuitively expected (Helinski et al., 2007b). However, there is also considerable difference in hydration between different cement types (Cayouette, 2003) due to the varying binder chemistry.

The solids content of a fill also affects the hydration process with its ability to increase the strength of CPB for a given cement content, via an associated density increase (Cayouette, 2003; Rankine & Sivakugan, 2007). The increase in solids content also has the effect of reducing the water to cement ratio (by mass) of the mix, yielding the same effect as increasing the cement content (Revell, 2004). However, as the cement content increases, higher solids contents reduce the water available for hydration of the cement as well as potentially limiting the dispersion of cement within the mix due to reduced mixing ability, due to higher yield strength.

For a given cement content in a CPB containing well-graded tailings, a reduction in the tailings particle size (i.e. finer well-graded material) will also result in reducing the strength of the CPB due to a reduction in void space available for hydration product growth (Fall et al., 2005). The particle size of the cement is also important as smaller particles, have increased surface area with which hydration can occur, and thus impacts the ultimate CPB properties (Bouasker et al., 2008; Bentz, 2005).
2.4.2. Chemistry

The hydration process has been found to be influenced by chemistry of the overall mix (Benzaazoua et al., 2004; Fall & Benzaazoua, 2005). The use of alternative binders to replace or reduce the cement content, pozzolans such as slag or fly-ash, have been investigated as they are cheaper and found to positively affect strength in varying proportions (Cayouette, 2003; Benzaazoua et al., 2004; Revell, 2004; le Roux et al., 2005; Fall et al., 2010; Pokharel & Fall, 2011).

The presence of sulphides has been shown to significantly influence the behaviour, in particular the strength, of a hydrating CPB mix (Fall & Benzaazoua, 2005; Fall & Pokharel, 2010; Pokharel & Fall, 2011). Sulphide has a positive effect on the early age strength due to the reduction of the internal porosity, similar to the effect of increased solids content (Cayouette, 2003), where in addition to the cement hydration products, hydrated sulphide precipitates also fill the void spaces (Fall & Benzaazoua, 2005). That said, if the concentration of sulphides is too great, then their presence might limit the ability of the cement particles to hydrate, which would have a negative effect on the strength development. Beyond 28 days the same mechanism that aids in early age strength results in strength degradation. The sulphide products continue to expand causing damage to the CPB microstructure. High concentrations of sulphides tend to retard early age strength increases by the limiting the growth of cement hydration products. Given the use of CPB with sulphide rich tailings, this impact is important.

This discussion on the use of additives to the classical tailing-water-cement CPB highlights that significant changes can occur with regard to material strength and general development of properties through chemical variation. While the primary focus of this thesis is not related to the chemistry and reactions that occur when using CPB, some surprising results have been presented in Chapter 5 which highlight the importance of chemistry in the behaviour of CPB and thus it cannot be ignored.

2.4.3. Temperature

Curing temperature is also known to be highly influential in the development of final properties of a cementitious material. This is particularly well documented in cement and concrete literature (Klieger, 1958; Brooks & Al-Kaisi, 1990; Kim et al., 1998;). A range of laboratory, numerical and field studies conducted on CPB to examine the effect of curing temperature on the development of material properties have also found this to be true of CPB, indicating significant improvement in the mechanical properties at higher curing temperatures (Fall et al., 2007; Nasir & Fall, 2009; Nasir & Fall, 2010; Fall et al., 2010; Thompson et al., 2011, Thompson et al., 2012).

The effect of stress conditions on CPB material properties has been clearly established previously in this chapter (Section 2.3) and offers an explanation for the observed discrepancy between in situ and laboratory cured specimens. However, given the difference in temperature between a typical stope and laboratory environment, and the known effect of temperature on
hydration, thermal effects may also play a large role in the difference. In an underground environment the temperature is often significantly higher (potentially in excess of 50°C) than the laboratory temperature of 23°C\(^1\) due to geographical location and/or the thermal gradient increase with depth below the ground surface (Payne & Mitra, 2008).

In addition to this difference in ambient temperature, the heat contribution due to the exothermic nature of the cement hydration reactions must also be considered (Swaddiwudhipong et al., 2002). Laboratory cast and tested specimens are of such a small mass (typically 200 cm\(^3\)) compared with a full-scale stope deposits (typically 45,000 m\(^3\)) that any heat generated by these small specimens is dwarfed by the rate of heat dissipation. Conversely, in a stope, the rate of heat dissipation is slowed by the large mass such that losses are significantly smaller than the rate at which heat is generated by the exothermic hydration reactions, resulting in a net temperature increase (Fall et al., 2007).

CPB strength testing results (Fall et al., 2007, 2010) indicated that regardless of the binder employed, the rate of strength gain is increased by higher curing temperatures, while the magnitude of strength is dependent on the curing time. Increased curing temperatures were also found to effect the failure mode of cured CPB material (Fall et al., 2010). Samples cured at higher temperatures exhibited higher peak UCS strengths but limited post-peak strain, resulting in a quasi-brittle failure mode. The mode of failure is particularly relevant when considering the exposure stability of a CPB mass. Elevated curing temperatures are also known to increase pore structure fineness in CPB fill due to increased hydrate growth (Pokharel & Fall, 2011; Fall et al., 2010).

Understanding the affect curing temperature has on the progress of CPB hydration and subsequent property development is crucial in understanding the affect the curing environment (in situ versus laboratory) has on the final CPB product and thus how best to approach mix design. Given the positive influence of temperature on the hydration reactions, and the role of the hydration reactions in effective stress development, there would appear to be a compounding effect of elevated curing temperature and curing with effective stress generation, on the increase of CPB mechanical properties. While Helsinki’s effective stress approach and hydration cell testing protocol provides a vast improvement on prior design methods, the influence of temperature was not incorporated. Given the role temperature has on the rate of hydration and how this in turn affects the self-desiccation mechanism which is closely related to effective stress generation, it follows that elevated temperature has significant potential to enhance the effective stress conditions both in the stope, but also in Helsinki’s hydration cell apparatus. Further detailed discussion on the affects of temperature on CPB is presented in

\(^1\) There is a lack of explicit mention of curing temperatures in a mine site laboratory environment within the published literature. Based on the author’s understanding of processes occurring at mine sites referred to in this thesis, “typical” laboratory temperatures are assumed to be ~23°C. It is acknowledged that this may vary from site to site.
Chapter 4 with the aim of developing a temperature-controlled hydration cell to quantify the
contribution of elevated curing temperature to the self-desiccation mechanism.

2.5. Summary

This chapter has presented an overview of the current knowledge related to the design and use
of CPB, as well as highlighting the areas where knowledge gaps currently exist and how this
PhD research can improve understanding of CPB properties and behaviours.

The literature has shown the independent effects of effective stress generation and elevated
curing temperature, which promotes increased strength in CPB. However, the combined effect
of these conditions and their interaction has not been examined. Given the positive effect
temperature has on the hydration process, and the important role hydration plays in the
increase of effective stress in the stope, it is hypothesized that curing under elevated
temperature will increase the rate and eventual level of effective stress generation, ultimately
enhancing the mechanical properties of the CPB.

Further understanding of factors, both physical and chemical, affecting the chemical shrinkage,
and thus effective stress generation, are examined through tests measuring the volume of
chemical shrinkage. Helinski’s hydration model is then further developed to relate the volume
strain occurring in the hydration cell tests with that occurring in these chemical shrinkage tests.
The initial aim of this is to examine the potential for the chemical shrinkage test to act as a
screening test of CPB mixes by giving an initial indication of hydration properties, prior to the
more complex and time consuming hydration cell test.

The effect of complex chemistry on hydration has been well recorded in the literature; however,
there have been no reports of it affecting the consolidation behaviour of CPB. The
consolidation of both cemented (CPB) and uncemented tailings in this thesis highlights the
potential for chemical interactions and effects to modify this behaviour, which has implications
on the mechanical properties of CPB.
CHAPTER 3. MATERIALS AND METHODS

3.1. Introduction

A range of different materials and methodologies were examined and applied in the experimental work carried out in this thesis. This chapter summarises the physical and chemical characteristics of the various tailings, water and binder products used in CPB testing, in addition to information regarding the experimental testing apparatuses employed. The behaviour of a given mix is known to be affected by the physical and chemical properties of each individual constituent. As such, knowledge of each material type used in a mix is of importance. Mix design details for the range of CPB mixes examined in this thesis are also provided here.

Some of the experimental work provided in this thesis underwent a development phase, in particular the work presented in Chapter 4 and Chapter 6, such that techniques and equipment were modified as required throughout the testing program. While this chapter provides information on the standard experimental apparatuses and their operation, further explanation regarding any modifications (where required) are presented in the relevant chapters. The same applies for any variations in mix design.

To gain practical benefit and knowledge from this work, it is important that the CPB mixes investigated be accurate representations of the material used in practical applications (i.e. in an underground mining application). The backfill mix proportions examined in this thesis were taken from real mixes used in underground mining operations employing backfill methods. The tailings, binder and process water from these sites were transported from site to the UWA laboratory where the CPB mix was created and tested.

3.2. Materials

Information regarding the physical and chemical characteristics of tailings, water and binder components of the CPB mixes tested in this thesis is provided in this section, separated under the relevant material sub-headings. Chemical testing was carried out by external third parties and while only a summary of the results are presented in this chapter, the full report is included in Appendix A.
3.2.1. Tailings

The different tailings solid materials tested comprised the following list:

- Gold tailings from the Kanowna Belle mine located in the goldfields region of Western Australia (denoted as KB);
- Nickel tailings from the Savannah Nickel Mine located in the northern Kimberley region of Western Australia (denoted as SNM);
- Gold tailings from the Raleigh mine located in the goldfields region of Western Australia (denoted as RL);
- Gold tailings from the St Ives mine located in the goldfields region of Western Australia (denoted as SI);

The KB, SNM and SI tailings are millstream tailings, while the RL material is reclaimed from a tailings storage facility, where drying and oxidation has occurred.

The final RL solids mix comprises 70% by weight of tailings with the addition of 30% sand. All subsequent mentions of RL material refer to this tailings/sand mix. For comparison purposes in Chapter 6, a silica sand material is also examined.

The specific gravity ($G_f$) and particle size distribution (PSD) of each of the tailings materials (referred to as either tailings or filler material) are provided in Table 3.1 and Figure 3.1 respectively.

<table>
<thead>
<tr>
<th>Mix</th>
<th>$G_f$ tails</th>
<th>$G_f$ sand</th>
</tr>
</thead>
<tbody>
<tr>
<td>KB</td>
<td>2.72</td>
<td>-</td>
</tr>
<tr>
<td>SNM</td>
<td>3.20</td>
<td>-</td>
</tr>
<tr>
<td>RL</td>
<td>2.85</td>
<td>2.67</td>
</tr>
<tr>
<td>SI</td>
<td>2.80</td>
<td>-</td>
</tr>
<tr>
<td>Silica sand</td>
<td>-</td>
<td>2.70</td>
</tr>
</tbody>
</table>
Figure 3.1 Tailings material particle size distribution

The PSD for each material was determined using a sieve analysis (ASTM, 2007) for the coarse fraction (>75 μm) of particles. The finer fraction (<75 μm) was analysed using either hydrometer testing (ASTM, 2007) for the SNM and SI material, or laser scanning techniques (similar to those in White (2003) for the KB and RL materials. Slight discontinuities exist in the KB and RL PSD curves at the transition points between these two techniques. The location of the tailings PSD curves to the left of the “cross” labelled “minimum passing 20 μm for paste fill”, indicate that materials meet the general definition applied to paste backfill categorisation (Potvin et al., 2005).

Chemical composition of the various tailings materials (SNM, KB, RL tailings and RL sand) were examined using X-ray fluorescence (XRF), X-ray diffraction (XRD), organic and inorganic carbon and total dissolved metals analyses. A quantitative results summary for each of these tests is shown in Table 3.2, Table 3.3, Table 3.4 and Table 3.5 respectively. It should be noted that an empty cell present within any of the tables indicates a lack of chemical presence.

The full chemical analysis reports for the XRD (including diffraction plots), total dissolved metals and carbon analysis are provided in Appendix A.
### Table 3.2 Tailings chemical analysis - semi-quantitative XRF

<table>
<thead>
<tr>
<th>Units</th>
<th>SNM</th>
<th>KB</th>
<th>RL Tails</th>
<th>RL Sand</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al₂O₃</td>
<td>% wt.</td>
<td>11.1</td>
<td>13.1</td>
<td>14.0</td>
</tr>
<tr>
<td>SiO₂</td>
<td>% wt.</td>
<td>46.9</td>
<td>59.3</td>
<td>59.8</td>
</tr>
<tr>
<td>TiO₂</td>
<td>% wt.</td>
<td>0.578</td>
<td>0.378</td>
<td>0.64</td>
</tr>
<tr>
<td>Fe₂O₃</td>
<td>% wt.</td>
<td>28.7</td>
<td>2.93</td>
<td>7.8</td>
</tr>
<tr>
<td>MnO</td>
<td>% wt.</td>
<td>0.133</td>
<td>0.061</td>
<td>0.113</td>
</tr>
<tr>
<td>CaO</td>
<td>% wt.</td>
<td>2.79</td>
<td>4.85</td>
<td>3.85</td>
</tr>
<tr>
<td>K₂O</td>
<td>% wt.</td>
<td>1.61</td>
<td>2.9</td>
<td>1.86</td>
</tr>
<tr>
<td>MgO</td>
<td>% wt.</td>
<td>3.66</td>
<td>2.97</td>
<td>2.46</td>
</tr>
<tr>
<td>Na₂O</td>
<td>% wt.</td>
<td>1.23</td>
<td>4.6</td>
<td>1.87</td>
</tr>
<tr>
<td>P₂O₅</td>
<td>% wt.</td>
<td>0.029</td>
<td>0.168</td>
<td>0.099</td>
</tr>
<tr>
<td>SO₃</td>
<td>% wt.</td>
<td>11.7</td>
<td>0.88</td>
<td>2.64</td>
</tr>
</tbody>
</table>

### Table 3.3 Tailings chemical analysis - semi-quantitative XRD

<table>
<thead>
<tr>
<th>Units</th>
<th>SNM</th>
<th>KB</th>
<th>RL Tails</th>
<th>RL Sand</th>
</tr>
</thead>
<tbody>
<tr>
<td>Actinolite</td>
<td>% wt.</td>
<td>12.8</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Albite</td>
<td>% wt.</td>
<td>23.6</td>
<td>35.5</td>
<td>11.6</td>
</tr>
<tr>
<td>Biotite</td>
<td>% wt.</td>
<td>2.2</td>
<td>10.5</td>
<td></td>
</tr>
<tr>
<td>Muscovite</td>
<td>% wt.</td>
<td>14.2 *</td>
<td>20.2</td>
<td>1.5</td>
</tr>
<tr>
<td>Calcite</td>
<td>% wt.</td>
<td></td>
<td>1.6</td>
<td></td>
</tr>
<tr>
<td>Chalcopyrite</td>
<td>% wt.</td>
<td>0.5</td>
<td>8.5</td>
<td></td>
</tr>
<tr>
<td>Chlorite</td>
<td>% wt.</td>
<td>0.6</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Coquimbite</td>
<td>% wt.</td>
<td>0.1</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Cummingtonite</td>
<td>% wt.</td>
<td>5.3</td>
<td>3</td>
<td></td>
</tr>
<tr>
<td>Dolomite</td>
<td>% wt.</td>
<td>0</td>
<td>17.3</td>
<td></td>
</tr>
<tr>
<td>Hastingsite</td>
<td>% wt.</td>
<td></td>
<td>15.3</td>
<td></td>
</tr>
<tr>
<td>Kaolin</td>
<td>% wt.</td>
<td>2.2</td>
<td>0.4</td>
<td>8.9</td>
</tr>
<tr>
<td>Microcline</td>
<td>% wt.</td>
<td>19.4</td>
<td></td>
<td>7.1</td>
</tr>
<tr>
<td>Pyrite</td>
<td>% wt.</td>
<td></td>
<td></td>
<td>0.1</td>
</tr>
<tr>
<td>Pyrrhotite-5C</td>
<td>% wt.</td>
<td>6.6</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Quartz</td>
<td>% wt.</td>
<td>12.7</td>
<td>22.6</td>
<td>38.2</td>
</tr>
<tr>
<td>Halite</td>
<td>% wt.</td>
<td>2.9</td>
<td>2.5</td>
<td>0.4</td>
</tr>
<tr>
<td>Talc</td>
<td>% wt.</td>
<td>1</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

* Values are not as reliable as a large orientation correction was required.
Table 3.4 Tailings chemical analysis - organic and inorganic carbon

<table>
<thead>
<tr>
<th>Limit of Reporting</th>
<th>Unit</th>
<th>SNM</th>
<th>KB</th>
<th>RL Tails</th>
<th>RL Sand</th>
</tr>
</thead>
<tbody>
<tr>
<td>TIC</td>
<td>%wt.</td>
<td>&lt;0.05</td>
<td>1.53</td>
<td>0.57</td>
<td>&lt;0.05</td>
</tr>
<tr>
<td>TOC</td>
<td>%wt.</td>
<td>&lt;0.05</td>
<td>&lt;0.05</td>
<td>0.17</td>
<td>&lt;0.05</td>
</tr>
</tbody>
</table>

Table 3.5 Tailings chemical analysis - total dissolved metals

<table>
<thead>
<tr>
<th>Limit of Reporting</th>
<th>Units</th>
<th>SNM</th>
<th>KB</th>
<th>RL Tails</th>
<th>RL Sand</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al</td>
<td>mg/kg</td>
<td>18,800</td>
<td>2,850</td>
<td>15,700</td>
<td>1,920</td>
</tr>
<tr>
<td>Ca</td>
<td>mg/kg</td>
<td>8,900</td>
<td>30,000</td>
<td>22,000</td>
<td>49</td>
</tr>
<tr>
<td>Cu</td>
<td>mg/kg</td>
<td>140</td>
<td>23</td>
<td>91</td>
<td>3.4</td>
</tr>
<tr>
<td>Fe</td>
<td>mg/kg</td>
<td>130,000</td>
<td>16,000</td>
<td>46,000</td>
<td>1,500</td>
</tr>
<tr>
<td>K</td>
<td>mg/kg</td>
<td>7,100</td>
<td>1,300</td>
<td>2,500</td>
<td>14</td>
</tr>
<tr>
<td>Mg</td>
<td>mg/kg</td>
<td>11,000</td>
<td>16,000</td>
<td>13,000</td>
<td>190</td>
</tr>
<tr>
<td>Na</td>
<td>mg/kg</td>
<td>830</td>
<td>14,000</td>
<td>8,900</td>
<td>640</td>
</tr>
<tr>
<td>S</td>
<td>mg/kg</td>
<td>28,000</td>
<td>3,300</td>
<td>8,800</td>
<td>84</td>
</tr>
<tr>
<td>Si</td>
<td>mg/kg</td>
<td>1,200</td>
<td>410</td>
<td>290</td>
<td>570</td>
</tr>
<tr>
<td>Sr</td>
<td>mg/kg</td>
<td>24</td>
<td>250</td>
<td>80</td>
<td>0.9</td>
</tr>
</tbody>
</table>
3.2.2. Water

A range of different water types were employed in the mixes tested. Unless otherwise specified, the CPB mixes include water used in the actual backfilling process on site, termed process water. Often the process water had considerably higher mineral content than tap water as indicated in Table 3.6, where the total dissolved solids (TDS) measured in mg/L and electrical conductivity (EC) measured in units of millisiemens per metre (mS/m) of each water type is shown, in addition to the alkalinity, hardness (measured in units of mg/L) and pH.

Table 3.6 Water chemical analysis - alkalinity, electrical conductivity, hardness, pH and total dissolved solids (TDS)

<table>
<thead>
<tr>
<th></th>
<th>Limit of Reporting</th>
<th>Units</th>
<th>SNM</th>
<th>KB</th>
<th>RL</th>
<th>Tap</th>
</tr>
</thead>
<tbody>
<tr>
<td>Alkalinity</td>
<td>1</td>
<td>mg/L</td>
<td>&lt;1</td>
<td>52</td>
<td>83</td>
<td>-</td>
</tr>
<tr>
<td>Electrical conductivity</td>
<td>0.2</td>
<td>mS/m</td>
<td>429</td>
<td>21,000</td>
<td>17,500</td>
<td>-</td>
</tr>
<tr>
<td>Hardness</td>
<td>1</td>
<td>mg/L</td>
<td>1,700</td>
<td>24,000</td>
<td>34,000</td>
<td>-</td>
</tr>
<tr>
<td>pH</td>
<td>0.1</td>
<td>mg/L</td>
<td>7.4</td>
<td>7.4</td>
<td>7.4</td>
<td>-</td>
</tr>
<tr>
<td>TDS</td>
<td>1</td>
<td>mg/L</td>
<td>3,600</td>
<td>210,000</td>
<td>170,000</td>
<td>464</td>
</tr>
</tbody>
</table>

Additional water quality testing was also conducted to analyse the anion and total dissolved metal composition of KB, SNM and RL water, measured in units of mg/L. The results of this chemical analysis are presented in Table 3.7 and Table 3.8 respectively, while the full report is provided in Appendix A.

Table 3.7 Water chemistry analysis - anions

<table>
<thead>
<tr>
<th></th>
<th>Limit of Reporting</th>
<th>Units</th>
<th>SNM</th>
<th>KB</th>
<th>RL</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cl</td>
<td>1</td>
<td>mg/L</td>
<td>58</td>
<td>126,000</td>
<td>95,500</td>
</tr>
<tr>
<td>CO₃</td>
<td>1</td>
<td>mg/L</td>
<td>&lt;1</td>
<td>&lt;1</td>
<td>&lt;1</td>
</tr>
<tr>
<td>HCO₃</td>
<td>1</td>
<td>mg/L</td>
<td>&lt;1</td>
<td>63</td>
<td>102</td>
</tr>
<tr>
<td>NO₃</td>
<td>0.05</td>
<td>mg/L</td>
<td>&lt;0.05</td>
<td>64</td>
<td>3.4</td>
</tr>
<tr>
<td>SO₄</td>
<td>0.1</td>
<td>mg/L</td>
<td>2,530</td>
<td>3,730</td>
<td>15,200</td>
</tr>
</tbody>
</table>
Table 3.8 Water chemistry analysis – total dissolved metals

<table>
<thead>
<tr>
<th>Metal</th>
<th>Limit of Reporting</th>
<th>Units</th>
<th>SNM</th>
<th>KB</th>
<th>RL</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al</td>
<td>0.005</td>
<td>mg/L</td>
<td>0.067</td>
<td>&lt;0.025</td>
<td>&lt;0.025</td>
</tr>
<tr>
<td>As</td>
<td>0.001</td>
<td>mg/L</td>
<td>&lt;0.001</td>
<td>0.31</td>
<td>&lt;0.050</td>
</tr>
<tr>
<td>B</td>
<td>0.02</td>
<td>mg/L</td>
<td>&lt;0.02</td>
<td>0.62</td>
<td>6.7</td>
</tr>
<tr>
<td>Ba</td>
<td>0.002</td>
<td>mg/L</td>
<td>0.043</td>
<td>0.27</td>
<td>0.067</td>
</tr>
<tr>
<td>Ca</td>
<td>0.1</td>
<td>mg/L</td>
<td>488</td>
<td>3170</td>
<td>766</td>
</tr>
<tr>
<td>Cd</td>
<td>0.0001</td>
<td>mg/L</td>
<td>0.0001</td>
<td>0.077</td>
<td>&lt;0.0050</td>
</tr>
<tr>
<td>Co</td>
<td>0.005</td>
<td>mg/L</td>
<td>0.007</td>
<td>2.4</td>
<td>&lt;0.025</td>
</tr>
<tr>
<td>Cr</td>
<td>0.001</td>
<td>mg/L</td>
<td>&lt;0.001</td>
<td>&lt;0.005</td>
<td>&lt;0.005</td>
</tr>
<tr>
<td>Cu</td>
<td>0.002</td>
<td>mg/L</td>
<td>&lt;0.002</td>
<td>0.16</td>
<td>0.063</td>
</tr>
<tr>
<td>Fe</td>
<td>0.005</td>
<td>mg/L</td>
<td>5.1</td>
<td>2.4</td>
<td>&lt;0.025</td>
</tr>
<tr>
<td>K</td>
<td>0.1</td>
<td>mg/L</td>
<td>193</td>
<td>116</td>
<td>221</td>
</tr>
<tr>
<td>Mg</td>
<td>0.1</td>
<td>mg/L</td>
<td>128</td>
<td>3790</td>
<td>7830</td>
</tr>
<tr>
<td>Mn</td>
<td>0.001</td>
<td>mg/L</td>
<td>1.4</td>
<td>0.012</td>
<td>1.5</td>
</tr>
<tr>
<td>Mo</td>
<td>0.001</td>
<td>mg/L</td>
<td>&lt;0.001</td>
<td>0.1</td>
<td>&lt;0.050</td>
</tr>
<tr>
<td>Na</td>
<td>0.1</td>
<td>mg/L</td>
<td>244</td>
<td>72500</td>
<td>54200</td>
</tr>
<tr>
<td>Ni</td>
<td>0.001</td>
<td>mg/L</td>
<td>0.48</td>
<td>1.1</td>
<td>0.079</td>
</tr>
<tr>
<td>Pb</td>
<td>0.0001</td>
<td>mg/L</td>
<td>0.0001</td>
<td>0.075</td>
<td>0.056</td>
</tr>
<tr>
<td>V</td>
<td>0.005</td>
<td>mg/L</td>
<td>&lt;0.005</td>
<td>&lt;0.025</td>
<td>&lt;0.025</td>
</tr>
<tr>
<td>Zn</td>
<td>0.005</td>
<td>mg/L</td>
<td>0.046</td>
<td>0.063</td>
<td>0.37</td>
</tr>
</tbody>
</table>

3.2.3. Binder

The range of binder types employed included ordinary Portland cement (OPC), Minecem, a propriety binder especially developed for use with CPB, and Swan GP, a particular brand of ordinary Portland cement. The specific gravity \( (G_c) \) of each binder is provided in Table 3.9.

Table 3.9 Binder specific gravity

<table>
<thead>
<tr>
<th>Binder</th>
<th>( G_c )</th>
</tr>
</thead>
<tbody>
<tr>
<td>OPC</td>
<td>3.20</td>
</tr>
<tr>
<td>Swan GP</td>
<td>3.16</td>
</tr>
<tr>
<td>Minecem</td>
<td>3.20</td>
</tr>
</tbody>
</table>
3.3. Mixes

CPB mixes are characterised by their solids content \(C_s\), cement (or binder) content \(C_c\) and void ratio \(e\). The void ratio is defined as the ratio of volume of voids to volume of solids and solids content is defined as

\[ C_s = \frac{M_c + M_f}{M_f + M_c + M_w} \]

Equation 3.1

where \(M_c\) is the mass of cement, \(M_f\) is the mass of dry tailings, \(M_w\) is the mass of water contained in the mix. The cement content is usually specified in paste backfill as a ratio of dry mass of cement to the total dry mass of solids (i.e. cement + tailings).

\[ C_c = \frac{M_c}{M_f + M_c} \]

Equation 3.2

The cement content can also be expressed as the ratio by mass of water to cement, termed the water cement (\(w/c\)) ratio. These values for each mix are summarised in Table 3.10.

As the cement hydrates and the pore voids are filled with the hydrated gel product, the void ratio will reduce. Any void ratio value quoted refers to the initial void ratio prior to the commencement of hydration, denoted \(e_o\).

Table 3.10 CPB mix properties

<table>
<thead>
<tr>
<th>Material</th>
<th>Water</th>
<th>Binder</th>
<th>(w/c)</th>
<th>(C_c)</th>
<th>(C_s)</th>
<th>(C_{sand})</th>
<th>(e_o)</th>
</tr>
</thead>
<tbody>
<tr>
<td>KB</td>
<td>Process</td>
<td>OPC</td>
<td>6.00</td>
<td>5.0</td>
<td>78</td>
<td>0</td>
<td>0.80</td>
</tr>
<tr>
<td>SNM</td>
<td>Tap</td>
<td>Minecem</td>
<td>18.75</td>
<td>1.5</td>
<td>78</td>
<td>0</td>
<td>0.90</td>
</tr>
<tr>
<td>RL</td>
<td>Process</td>
<td>OPC</td>
<td>6.00</td>
<td>5.0</td>
<td>77</td>
<td>30</td>
<td>0.85</td>
</tr>
<tr>
<td>SI</td>
<td>Process</td>
<td>Swan GP</td>
<td>7.30</td>
<td>6.0</td>
<td>70</td>
<td>0</td>
<td>1.23</td>
</tr>
</tbody>
</table>

3.4. Experimental methods

Unless otherwise specified, all testing was carried out in a temperature-controlled laboratory at an ambient temperature of 23°C (+-2°C).

3.4.1. Specimen preparation

Paste specimens were prepared by combining the measured quantities of water, binder and tailings (plus sand for the RL mix) required to achieve the mix specifications detailed. The time at which first contact between cement and water occurs is recorded and used as the reference time, \(t = 0\).

Mixing was undertaken using a Hobart mixer as per standard industry practise for CPB QA/QC testing and as per the ASTM code for the measurement of chemical shrinkage of hydraulic cement paste (ASTM, 2009).
3.4.2. Hydration cell

The hydration cell was used to evaluate stiffness and effective stress generation in hydrating CPB, allowing the self-desiccation mechanism and development of effective stress of a CPB mix to be independently examined as discussed previously (Helinski et al., 2007a; Fahey et al., 2011). A schematic of this experimental setup is shown in Figure 3.2.

**Figure 3.2 Hydration cell experimental set up**

The slurry specimen was cast into a split-mould supported membrane as indicated in Figure 3.2. The specimen was then allowed to cure under undrained conditions over a seven day period with an applied cell pressure, \( \sigma_c \), of 400kPa. Due to the specimen’s slurry nature, \( \sigma_c \) is transferred to the specimen allowing pore pressures at both the top (\( u_t \)) and base (\( u_b \)) of the specimen to equal \( \sigma_c \), creating an initial stress condition of zero effective stress.

The initial slurry state of the specimen meant that the specimen was not able to be self-supporting. When casting the specimen into the membrane, the split mould was tightly closed to ensure a regular cylindrical specimen shape. Once the specimen has been cast and the top cap sealed into the membrane, the two halves of the mould were separated slightly to allow the applied cell pressure direct access to the specimen, while still offering adequate support to the specimen, preserving the cylindrical shape as the specimen hydrated.

The relationship between total, effective stress and pore pressure is provided here in Equation 3.3 using an adopted sign convention where the compressive mean effective stress (\( p' \)) and...
total stress \( (p) \) are negative, and a compressive pore pressure \( (u) \) is positive. This sign convention was adopted to facilitate implementation of a model, which will be developed in Chapter 5, into ABAQUS.

\[
p' = p + u \tag{Equation 3.3}
\]

In Chapter 7 an alternate notation and sign convention is adopted such that compressive mean effective stress \( (\sigma') \) positive and determined by

\[
\sigma' = \sigma - u \tag{Equation 3.4}
\]

where \( \sigma \) is the total mean stress (compressive expressed as positive) and compressive pore pressure \( (u) \) is negative.

In the context of the hydration cell set-up shown in Figure 3.2, \( u \) in Equation 3.3 represents the specimen pore pressure \( u_t \), and \( p \) represents the applied cell pressure \( \sigma_c \). As self-desiccation occurs, \( u_t \) reduces and \( p' \) increases indicating effective stress generation.

The pressure lines and pore pressure transducers (PPT) were de-aired using tap or deionised (DI) water, unless specified otherwise, to prevent corrosion of the equipment, if process water were used.

### 3.4.2.1. Small strain stiffness measurements

Bender elements (Dyvik & Olsen, 1991) were used in the hydration cell to calculate the small strain shear stiffness \( (G_o) \) using the material density \( (\rho) \) and the shear wave signal arrival time \( (V_s) \) (Helinski et al., 2007a) via

\[
G_o = \rho V_s^2 \tag{Equation 3.5}
\]

Small strain stiffness is used as an index for stiffness and hydration progression throughout hydration, rather than as a definitive value representative of actual in-stope conditions.

The small strain shear stiffness can then be converted to bulk stiffness \( (K_i) \)

\[
K_i = \frac{2(1+\nu)}{3(1-2\nu)} G_o \tag{Equation 3.6}
\]

where \( \nu \) is the Poisson’s ratio for the soil material. A value of \( \nu = 0.125 \) was used (Helinski et al., 2007a) unless otherwise specified.

### 3.4.2.2. Permeability

Permeability \( (k) \) of a specimen was determined within the hydration cell by measuring the flow volume generated by a pressure differential i.e. \( u_t - \sigma_b \) as indicated in Figure 3.2, across the length of the specimen such that
\[ k = \frac{q}{ai} \quad \text{Equation 3.7} \]

where \( q \) is the flow rate (m\(^3\)/s), \( a \) is the cross-sectional area of the specimen (m\(^2\)) and \( i \) is the hydraulic gradient (dimensionless). The hydraulic gradient was determined by

\[ i = \frac{\Delta h}{d} \quad \text{Equation 3.8} \]

where \( \Delta h \) is the change in head (m) over the drainage length \( d \) (m). \( \Delta h \) is determined by

\[ \Delta h = \frac{\mu - \sigma_h}{g} \quad \text{Equation 3.9} \]

where \( g = 9.81 \text{m/s}^2 \).

3.4.2.3. UCS testing

Uniaxial compressive strength (UCS) was determined at the completion of the seven day hydration cell test as per ASTM (2006). The specimen was submerged in a water bath and loaded at a constant strain rate of 0.1 mm/minute to determine the peak uniaxial compressive strength and post peak strain behaviour. The use of the water bath was intended to negate all suction-induced strength contributions as discussed by Fahey et al. (2011) and Helinski et al. (2011a).

3.4.2.4. Consolidation

The hydration cell was also used in this thesis to conduct consolidation testing of cemented and uncemented specimens. The details pertaining to this application are presented in Chapter 6.

3.4.3. Chemical shrinkage

Chemical shrinkage was measured using the ASTM Standard Test Method C1608-07 for Chemical Shrinkage of Hydraulic Cement Pastes, Procedure A (ASTM, 2009); this test methodology is referred to as the ‘ASTM test’. A known mass of cement-based material was placed in a plastic vial, forming a specimen \( \leq 10\text{mm} \) thick, with deionised (DI) water filling the remaining volume. The DI water was added gradually to prevent disturbance of the paste specimen; no mixing of the paste with this added water was observed. The vial was then sealed with a rubber stopper fitted with a calibrated capillary tube; this was inserted into the vial until an initial water level was visible in the tube. A drop of paraffin oil was placed at the top of the capillary tube to minimise evaporation.

A schematic diagram of the experimental set up is provided in Figure 3.3.
Figure 3.3 Chemical shrinkage experimental set up

The vial was kept in a temperature-controlled laboratory for the duration of the test (typically 30-40 days). As hydration progressed, the specimen consumed the available water, as indicated by a lowering of the water level in the capillary tube. This volume of consumed water is referred to as the chemical shrinkage. Each ASTM test was carried out in triplicate (as a minimum) and the total measured shrinkage in a test was limited by the size of the 0.6mL capillary tube. Further explanation and a definition of chemical shrinkage are provided in Chapter 5.

3.4.4. Oedometer consolidation test

An oedometer was used to examine the one dimensional (1D) consolidation behaviour of uncemented tailings mixes (equivalent to CPB mixes but without cement) as per ASTM (2004). Specific information regarding the mixes and applied stress levels during the tests are provided in the relevant chapter. The oedometer cell configuration (shown in Figure 3.4) was used to examine volume change response to consolidation by measuring the vertical displacement of a specimen, using an LVDT, during mechanical loading and unloading of a specimen.
3.4.5. Moisture content and density determination

At the completion of all tests the final specimen dimensions and weight were recorded. Specimens were then oven dried at 100°C for a minimum of 24 hours to determine the moisture content, allowing determination of bulk and dry specimen densities, $\gamma$ and $\gamma_d$ respectively, and void ratio, $e$.

3.5. Summary

This chapter has presented the materials, equipment and techniques employed in the experimental testing presented in this thesis. The range of material types and their associated properties are presented here, in addition to details of mix proportioning. Standard terminology regarding material properties and test parameters have also been presented and defined. Experimental testing procedures for specimen preparation; ASTM chemical shrinkage testing; hydration cell testing, including the use of bender elements, pore pressure measurements, UCS and permeability testing, consolidation testing in both the hydration cell and oedometer apparatuses as well as moisture content and density determination are also detailed.

Some of the experimental testing presented in this thesis has required specific and extensive equipment modification; in particular the research presented in Chapter 4 and Chapter 7. Where such modifications have occurred, or indeed any variation from the methodologies or material presented in this chapter, the changes made are described in detail where relevant.
4.1. Introduction

Cemented paste backfill (CPB) has been observed to achieve greater ultimate strength when cured in situ compared with equivalent mixes cured and tested in a laboratory environment (Revell, 2004; Fahey et al., 2011; le Roux et al., 2005). There are two significant differences between in situ and laboratory curing: material cured in situ in an underground stope generates effective stress due to both self-weight consolidation and the self-desiccation mechanism, in addition to the high humidity environment and higher temperatures due to the heat generated from exothermic hydration reactions which occur as the material cures. Effective stress and temperature increases do not typically occur in a laboratory-cured specimen due to the small specimen size cured under atmospheric conditions.

Details of the self-desiccation mechanism, and its role in effective stress generation, which enhances mechanical CPB properties has been explored in depth (Helinski et al., 2007a; Grabinsky & Simms, 2006; and Fahey et al., 2011) and previously discussed in Chapter 2. Helinski’s hydration cell apparatus allows curing specimens to generate effective stress in a laboratory environment, allowing the self-desiccation behaviour for a particular CPB mix to be examined.

Temperature is also known to play a critical role in the process of cement hydration of both cement pastes and concrete. Higher temperatures increase the rate and extent of hydration reactions, which in turn positively affects the strength development, compared with curing at lower temperatures. Numerical modelling and laboratory studies (Nasir & Fall, 2009; Nasir & Fall, 2010; Fall et al., 2010; Fall & Samb, 2006; Fall et al., 2007) as well as in situ data recovered from field studies (Thompson et al., 2011, Thompson et al., 2012) indicate these benefits also extend to CPB material, despite the lower cement content compared with cement pastes or concrete.

Traditional laboratory testing techniques used for design and quality control/quality assurance (QA/QC) of CPB mixes are usually carried out on small specimens, measuring approximately 50mm in diameter and 120mm in height, which are typically cast and cured in a laboratory environment with no applied stress and no temperature control. While Helinski’s hydration cell
provides the ability to cure specimens with effective stress generation, the existing set-up has no provision for in situ temperature replication.

This chapter expands on the brief temperature discussion provided in Chapter 2 and considers how heat develops during in situ curing of CPB and the implications this has on strength, compared with traditional laboratory curing. In situ temperature conditions are then applied in a laboratory environment through modification of Helinski’s hydration cell, allowing replication of in situ temperature conditions in addition to effective stress generation. Details of the development, calibration and initial testing of this temperature-controlled hydration cell are presented here, providing the first opportunity to comprehensively examine the combined effects of curing temperature and effective stress on CPB properties in a laboratory environment.

4.2. Effect of curing temperature on hydration

Heat generation during cement curing occurs due to the exothermic nature of the hydration reactions (Swaddiwudhipong et al., 2002). The increase in temperature due to this heat production has been found to significantly affect the rate and extent of hydration, both of which have large effects on the final hydrated material properties (Kim et al., 1998; Brooks & Al-Kaisi, 1990; Klieger, 1958).

The amount of heat produced during hydration is termed the ‘heat of hydration’ and can be determined experimentally in a number of ways (Morabito, 1998). However, adiabatic calorimetry is the only one of these techniques to allow both the total and rate of heat production to be determined (Gibbon et al., 1997). This technique measures the full amount of heat generated by the cementitious material as hydration progresses by preventing any heat exchange from the specimen with its surrounding environment. Adiabatic conditions are assumed to occur at depths greater than 0.5m in mass concrete (Gibbon et al., 1997), and similar conditions can be assumed for CPB within a stope where in situ temperature increases measured in hydrating CPB are referred to as adiabatic temperature rises.

Adiabatic calorimetry can be very expensive to carry out due to the high level of insulation and temperature feedback and control required to measure the heat generation and prevent its dissipation. To evaluate the effect of a particular temperature on hydration, isothermal testing may be carried out. Isothermal testing is where a constant temperature is maintained by allowing heat exchange between the system and the surrounding environment. Isothermal temperatures are much easier to maintain, as they can be controlled by an external heating (or cooling) source. Isothermal testing has been used to examine the effect of elevated temperatures in CPB hydration (Fall et al., 2007, 2010).
4.3. CPB hydration and curing temperature

Experimental and numerical studies carried out on CPB have demonstrated the effect of curing temperature on these chemical processes as they relate to the development of CPB mechanical properties. Specifically, the studies have examined the strength and deformation behaviour, along with microstructural properties and how these are influenced by binder type, thermal properties and environmental conditions, including stope size and shape, in conjunction with temperature.

4.3.1. Experimental studies

Laboratory studies have shown that elevated curing temperatures have a significant effect on final strength (represented by UCS and tensile strength tests) and the rate at which the strength develops, as indicated by UCS variations with time (Fall et al., 2010; Fall et al., 2007; Fall & Pokharel, 2010; Fall & Samb, 2008b). The strength of specimens containing higher binder content are also more positively influenced by higher curing temperatures than specimens with lower binder contents. The effect of temperature on early age CPB (<28 days) was examined by Fall et al., (2010) where the strength of specimens cured in a isothermal chamber at temperatures of 2°C, 20°C, 35°C and 50°C were compared at different ages. At elevated temperatures (35°C and 50°C) the strength gain was found to be greater than in specimens cured at 20°C. This was attributed to the increased hydration reaction rate at the elevated curing temperatures. However, as the material aged beyond 28 days, higher curing temperatures limited further strength development due to a lower diffusion rate of hydration products away from the binder particle, compared with the hydration reaction rate. This was also indicated by Fall et al., (2007) where the 90 day strength of a specimen cured at 35°C showed better mechanical performance than an equivalent specimen cured at 50°C. Reduced diffusion rates limit the movement of hydrate products away from cement particles and hinder the development of further product formation (Kjellsen et al., 1991).

Elevated curing temperatures were also found to influence the stress-strain relationship exhibited during UCS testing (Fall et al., 2010). Specimens cured at higher temperatures were found to fail at reduced peak strain levels in a quasi-brittle mode despite their higher ultimate strength; this was attributed to the increased precipitate formation due to the high reaction rates, limiting deformation potential (Fall et al., 2010). The pore structure of CPB cured at higher temperatures was also found to be finer due to increased hydrate growth within the pores (Pokharel & Fall, 2011; Fall et al., 2010). The effect of tailings particle size distribution (PSD) also plays a role, as higher percentage of fine, well graded tailings particles reduces the void space and thus the volume available for hydrate growth (Fall et al., 2005). This has a negative effect on specimen strength at higher curing temperatures (Fall et al., 2010).

The progression of the chemical hydration reactions and the ultimate properties achieved are highly dependent on the chemistry involved and the temperature at which these reactions occur.
This is evident in the considerable strength differences associated with different binder types and content as well as the use of additives in combination with different curing temperatures (Fall et al., 2007; Fall et al., 2010). As discussed in Chapter 2, the use of pozzolans such as fly ash or slag can positively influence the final material properties, in addition to the potential cost savings through reduction of the “regular” binder content. The pozzolanic reactions inherent in the slag material were observed to progress faster at earlier ages when cured under higher temperatures compared with ordinary Portland cement or fly ash reactions (Fall et al., 2007; Fall et al., 2010). These benefits achieved by the elevated curing temperature, with or without additives, were also found to be highly dependent on the presence of other chemicals within the curing environment, particularly sulphates (Fall & Pokharel, 2010; Pokharel & Fall, 2011). Lower binder percentages were also found to be more efficient at higher temperatures, compared with higher binder percentages. At laboratory temperatures of 20°C, increased binder content was found to typically increase the material strength; results from Fall et al., (2007) indicated greater UCS value for a specimen with 3.5% binder content cured at 35°C compared with a specimen of 6% binder content cured at 20°C.

These studies highlight the role curing temperature plays in strength development, and also provide evidence of the effects of chemical variations and the sensitivity of chemical reactions to elevated temperatures, and how these affect the mechanical properties of the curing material.

4.3.2. Numerical studies

The results of the above studies plus those from additional experimental and field studies (Khokholov & Kurilko, 2004; De Souza & Hewitt, 2005; and Williams et al., 2001) were used to validate a numerical model developed by Nasir & Fall (2009). This model was then used to examine the temperature generated in a CPB mass due to hydration reactions, with consideration of environmental factors on the heat generation including binder content, stope size, geometry and thermal properties of host rock mass and CPB. It was established that while larger CPB-filled stopes generated higher temperatures, the inclination angle of the stope and the thermal properties of the surrounding rock mass had negligible effect on heat generation. Higher binder content CPB mixes produced greater heat and faster filling rates minimised heat loss to the surrounding environment. The specific heat and thermal conductivity were both found to be important as they relate to the CPB’s ability to generate and retain heat.

To accurately model the thermal behaviour of CPB, the thermal properties of the material must be established. Célestin & Fall (2009) conducted a study to examine the effect of varying CPB mix properties on the thermal conductivity. Their findings indicated a significant effect of the tailings mineralogy and particle size on the thermal conductivity. Coarse, quartz-rich tailings were observed to have greater thermal conductivity, while variations in cement content and type showed minimal effect. Beyond 28 days there was little change in thermal conductivity. Higher curing temperatures were found to produce lower thermal conductivity in CPB material.
Nasir & Fall (2010) successfully coupled the effects of binder hydration with temperature and strength in a model predicting UCS strength for different mixes with varying cement content, initial temperature, stope size and rate of backfilling. However, this model did not consider the effects of consolidation and self-desiccation: two parameters that have been established as key in the development of strength (Helinski et al., 2007a; Fahey et al., 2011).

While the published literature clearly shows the significantly positive effects of elevated curing temperature on the development of CPB mechanical properties, nowhere has this effect been coupled with the known benefits of effective stress generation. To truly represent in situ conditions in a laboratory environment, these two effects must be considered in conjunction.

In situ monitoring studies which measured temperature and total stress within a CPB stope during periods of both filling and curing have been documented by Grabinsky & Thompson (2009), Grabinsky (2010) and Thompson et al. (2011). The total stress measured in a number of different stopes indicated a continuing rise in total stress when filling was halted. This rise appeared to correlate with the temperature increase. Based on this it appeared that the total stress increase was related to the thermal expansion of the solids due to the rising temperature (Thompson et al., 2011). This behaviour is the opposite of that expected after the completion of filling, as due to consolidation and/or self-desiccation of the mix, the pore pressures should initially be seen to reduce. This would be especially true if the self-desiccation mechanism was the primary means of pore pressure reduction, as the elevating temperatures will enhance the hydration process as discussed previously. However, depending on the permeability of the CPB fill, the flow of pore fluid may reduce the impact of self-desiccation. If thermal expansion was responsible for this total stress increase, it would need to be significant to overcome these other stress reducing mechanisms. Further investigation into the relationship between curing temperature and effective stress generation may offer further insight to these processes, although this is outside the scope of this thesis.

4.4. In situ versus laboratory curing temperatures

Heat generated in an underground mining environment may come from a variety of sources including backfill hydration, mine machinery, lighting or blasting as well as geographical location and geothermal gradient where temperatures rise with progressively deeper mining (Fall et al., 2010). In locations where permafrost conditions exist (i.e. northern Canada) or geological conditions where self-heating due to exothermic sulphide reactions may increase rock mass temperatures to the vicinity of 400°C (in rare circumstances) (Bernier & Li, 2003), consideration of extreme temperatures on CPB properties and behaviour must be considered. Studies conducted by Fall & Samb (2009) and Orejarena & Fall (2008) into the effects of very high temperatures on CPB hydration found that temperatures up to 200°C improved the strength. This was assumed to be due to the reduction in moisture content enhancing the stiffness of the cement gel or an increase in surface forces between gel particles (Fall & Samb, 2009).
Temperatures in excess of 200°C were found to have a detrimental effect on the mechanical properties of the material, attributed to thermal decomposition of hydrates, formation of dehydration induced cracks and microstructural cracking due to thermal incompatibility of tailings and cement particles. No information was provided as to how boiling of the pore fluid was prevented. Unsurprisingly, curing temperatures of 0°C reduced the ultimate strength and the rate at which the strength was developed; this was attributed to damage of the paste structure due to ice lens formation (Fall & Samb 2008a). These extreme temperatures are not encountered frequently, as indicated by the in situ data mentioned previously, which showed typical temperature increases up to 50°C (Grabinsky & Thompson, 2009; Thompson et al., 2011; Thompson et al., 2012). As such, the work in this chapter focuses on temperature in the range of 20-50°C.

Comparison of these described in situ thermal conditions with those encountered in many laboratory environments where temperatures can typically range from 20-23°C, highlights the difference in conditions between laboratory and mining environments. Due to the lower ambient temperatures and the small specimen sizes used in laboratory testing, any heat generated due to hydration is dissipated quicker than it can be produced, preventing any temperature rise. However, the size of the CPB mass, the insulation provided by the underground environment and the higher ambient temperatures present underground allow for significant heat generation by the exothermic hydration reactions associated with the hydration process.

These differences highlight the need for thermal control during laboratory specimen curing to more accurately replicate in situ conditions and produce specimens of similar properties to that of in situ cured material. The ability to replicate in situ thermal conditions, in addition to stress conditions (Fahey et al., 2012), in a laboratory environment offers the potential for enhanced safety and cost benefits in the design and use of CPB mixes.

4.4.1. In situ curing temperatures

Similar temperature increases to those measured by Grabinsky & Thompson (2009), Thompson et al. (2011) and Thompson et al. (2012) have been measured using similar in-stope monitoring at SNM and RL (M. Helinski, personal communication, 6 April, 2011) as shown in Figure 4.1 and Figure 4.2 respectively. Filling of the RL stope commenced at $t = 3.2$ hours and was completed at $t = 15.4$ hours. No information regarding filling times was provided for the SNM stope. However, it is assumed that filling commenced at approximately $t = 530$ hours; the duration of filling is also unknown.

The subsequent experimental work carried out in this chapter makes use of CPB material from SNM and KB. Details regarding these CPB mixes were provided in Chapter 3. Unfortunately no in situ temperature data was available from a KB stope. Given the similar mine type and location of the RL and KB mines in the Goldfields region of Western Australia, it could be assumed that the temperatures generated in each mine is likely to be similar. KB stopes, however, are typically much larger in size (~20m by 80m) compared with RL stopes (~6m by
10 m). An increase in bulk size can have a positive effect in generating higher temperatures due to reduced proximity from the thermal boundary where heat dissipates. As such the heat generated in the larger KB stopes may be greater than that measured in an RL stope. However, despite this, and in the absence of other KB specific data, the RL mine stope in situ temperature data was assumed to be similar to temperatures achieved in KB in situ cured CPB and thus considered here.

![Figure 4.1](image1.png)

**Figure 4.1** *In situ* temperatures measured in a SNM stope

![Figure 4.2](image2.png)

**Figure 4.2** *In situ* temperature measured in a RL stope

The physical locations for each temperature measurement taken within the RL stope are shown in Figure 4.3. The specific physical locations for the SNM temperature measurements were not available.
Figure 4.3 Locations of *in situ* temperature measurements in a RL stope

The temperature increase observed in the SNM stope is far more modest that that indicated in parts of the RL stope. This is explained by the use of 1.5% Minecem - a low-heat proprietary binder used in the SNM CPB mix, compared with 5% ordinary Portland cement (OPC) in the RL CPB mix.

An average rock mass temperature within the range of 23 - 32°C, with increase depth, is typical for underground mining environments in the Goldfields region (Payne & Mitra, 2008). As such, heat will most commonly dissipate from the hydrating CPB fill outwards, rather than absorbing heat from the surrounding rock mass.

So far in this chapter, the role of temperature in the hydration process and development of final CPB properties has been discussed and the magnitude of temperature increases in stopes considered. The following section uses temperature modelling to further compare *in situ* thermal conditions with those in the laboratory.

4.4.2. Modelling the difference between *in situ* and laboratory heat generation

Temp/W (Geostudio, 2007) was used to model the difference in CPB curing temperatures in a stope versus a hydration cell. The thermal properties for the various materials modelled are given in Table 4.1.
The basic assumed geometry of a CPB filled stope: 20m wide and 50m high, and a hydration cell specimen: 0.07m diameter and 0.16m in length were each modelled using plane strain and axisymmetric geometry respectively. This is shown in Figure 4.3 (a) and (b) respectively where only half of the geometry was considered. The CPB-filled stope is shown surrounded by rock mass and the hydration cell specimen surrounded by air inside the steel casing of the hydration cell.

Table 4.1 Material thermal properties for TEMP/W modelling from McWilliam (2011)

<table>
<thead>
<tr>
<th>Material</th>
<th>Unfrozen Thermal Conductivity [J/sec/m°C]</th>
<th>Unfrozen Volumetric Heat Capacity [J/m³°C]</th>
</tr>
</thead>
<tbody>
<tr>
<td>CPB</td>
<td>2.2</td>
<td>2.375E06</td>
</tr>
<tr>
<td>Rock</td>
<td>3.000</td>
<td>2.10E06</td>
</tr>
<tr>
<td>Air</td>
<td>0.025</td>
<td>1.00E03</td>
</tr>
<tr>
<td>Steel</td>
<td>45.30</td>
<td>3.92E06</td>
</tr>
<tr>
<td>Water</td>
<td>0.600</td>
<td>4.18E06</td>
</tr>
<tr>
<td>Styrofoam</td>
<td>0.040</td>
<td>0.12E06</td>
</tr>
</tbody>
</table>
Chapter 4  The Influence of Curing Temperature and Stress Conditions on Mechanical Properties of CPB

Figure 4.4 Temp/W model of (a) CPB-filled stope and (b) hydration cell specimen
A typical CPB heating curve was used to model the CPB temperature increase to a maximum of 32°C based on the numerical model developed by Nasir & Fall (2009) and their adopted material properties. This peak temperature is similar to that developed in the SNM stope, if an average of the two recorded data sets is considered (Figure 4.1). Assuming that the same heat is generated in both the stope and the hydration cell, the reduction in temperature in each scenario is compared in Figure 4.5. Over the 28-day period modelled, complete dissipation of the heat within the hydration cell specimen occurs, compared with only a 0.2°C drop in the CPB material in the stope.

The heating curve from Nasir & Fall (2010) was determined based on the adiabatic temperature increase due to heat generated by in situ CPB curing. In reality, any heat generated in the hydration cell specimen will immediately dissipate due to its small volume.

![Figure 4.5 CPB temperature reduction due to heat dissipation in a stope versus a hydration cell](image)

The role of elevated curing temperature on the development of desirable CPB properties and an understanding of the differences between in situ and laboratory curing has been defined and discussed. In situ monitoring of CPB stopes during filling and hydration have indicated the range of temperatures achieved during curing (Figure 4.1 and Figure 4.2). Is there a way to determine the temperature increase that can be expected in a stope prior to filling the stope? Being able to determine the expected conditions within a stope during CPB curing is key to accurately replicating in situ curing conditions and allowing safe, yet efficient and economic CPB design.
4.4.3. Determining temperature increases due to hydration

In the concrete industry, basic "adiabatic" tests can be carried out on site to determine the heat of hydration by measuring the temperature of material as it cures in an insulated box similar to that shown in Figure 4.6. However, given the difficulty of creating perfectly insulated conditions, these tests must be considered semi-adiabatic.

![Figure 4.6 Semi-adiabatic curing apparatus by Ng et al. (2008)](image)

A heat-loss compensation method was developed by Ng et al. (2008) to quantify the heat loss due to the semi-adiabatic nature of the test and thus determine the actual heat of hydration should hydration have occurred under perfect adiabatic conditions. The heat loss in the system is represented by the heat loss characteristic $\lambda$ and calculated based on the observed rate of actual measured volumetric mean temperature reduction ($\frac{\delta T_V}{\delta t}$) from within the specimen (averaged over the entire specimen volume), compared with the difference between the specimen surface temperature ($T_s$) and its surrounds ($T_A$) as shown by

$$\lambda = \frac{\frac{\delta T_V}{\delta t}}{T_s - T_A}$$

Equation 4.1

The higher the value of the heat loss characteristic, the faster heat loss occurs and thus the larger the error after heat loss compensation has been applied. Ng et al. (2008) recommend that an experimental test set-up should have a value of $\lambda$ less than $2.18 \times 10^{-6} \text{ s}^{-1}$. The adiabatic temperature ($T_{ad}$) can be calculated based on the adjustment of the measured temperature difference with the initial placing temperature ($T_p$) by the heat lost from the system, expressed as

$$T_{ad} = (T_V - T_p) + \lambda \int_0^t (T_s - T_A) \, \delta t$$

Equation 4.2
A similar experimental set-up to that shown in Figure 4.6 was created using a (0.5m by 0.4m by 0.3m) styrofoam box. This box, shown in Figure 4.7, was filled with spray foam insulation, allowing room for the casting of a 0.07m diameter and 0.13m high specimen in the centre. Two temperature sensors were used: one cast into the specimen to record $T_V$ and another placed outside the box to record $T_A$.

![Figure 4.7 Semi-adiabatic curing test set-up](image)

To test this experimental set-up, a volume of ordinary Portland cement (OPC) was cast into the box and temperatures measured during its hydration. For increased accuracy Ng et al. (2008) recommended that $T_V$ should be based on an average of multiple temperature readings recorded throughout the specimen. However, due to the small size of the specimen, temperature was only recorded in one specimen location and $T_S$ was assumed to be equal to $T_V$.

The results of the OPC semi-adiabatic test were then compensated using the method proposed by Ng et al. (2008) to determine maximum temperature generated by the OPC hydration. Two semi-adiabatic tests were conducted on OPC paste; both the measured and compensated temperatures are shown in Figure 4.8 along with published adiabatic testing data from Czernin (1980) and Puapansawat (2006), who also utilised OPC.
The heat loss characteristic determined for the experimental set-up was calculated as 6.0x10^-2 s^-1, a value significantly higher than the maximum value recommended by Ng et al. (2008), indicating that the insulation provided by the set-up was insufficient. Despite the large error, the compensated temperatures, determined via Equation 4.2, showed a good match with the published data determined by full adiabatic curing.

This heat loss compensation method was then applied to a CPB specimen to examine the adiabatic heat generation. Semi-adiabatic testing was conducted on the SNM and KB CPB mixes. No temperature increase was evident in the SNM test using either 1.5% or 3% Minecem binder as indicated in Figure 4.9. However, a slight effect of diurnal temperature variation within the temperature-controlled room (23°C ±1°C) was evident. The lack of observed temperature increase is presumably due to the low binder content and the specially designed low-heat chemistry of the binder.
Figure 4.9 Semi-adiabatic curing test – SNM 1.5% and 3% binder mixes

The KB CPB mix did achieve noticeable temperature increases. The uncompensated and compensated temperatures for the two semi-adiabatic tests conducted are shown in Figure 4.10.

Figure 4.10 Semi-adiabatic curing test – KB 5% binder mix

Despite the success of the semi-adiabatic testing on the KB mix compared with the SNM, the final adiabatic compensated temperature was not as large as expected given the in situ temperature measurements previously shown in Figure 4.1 and Figure 4.2. The SNM semi-adiabatic test did not register any temperature increase during hydration, despite a peak temperature of 36°C being recorded in the stope. Given that a temperature increase was evident in the KB semi-adiabatic testing, this would suggest that the real adiabatic temperature
would be much larger than the compensated value of ~28°C, assuming the temperatures within the CPB filled stopes reach values in excess of 40°C (Section 4.4.1).

The insulated box used in the semi-adiabatic testing was modelled using TEMP/W to examine possible methods to reduce heat dissipation, improving the thermal retention properties and potentially lowering the heat loss characteristic (λ) of the set-up. The heating curve from Nasir & Fall (2010), shown in Figure 4.5, was again used to heat the CPB specimen (numerically) inside the insulated box. The subsequent temperature reduction of the specimen due to heat dissipation was determined for a range of specimen sizes, different insulation material and surrounding ambient temperature as shown in Figure 4.11, Figure 4.12 and Figure 4.13 respectively.

The ambient temperature was maintained at 20°C and polystyrene was used to insulate a 0.1x0.2m specimen unless otherwise specified. The thermal properties for each of the different insulation types modelled are shown in Table 4.1.

![Graph showing the effect of insulation type on mean volumetric specimen temperature](image)

**Figure 4.11 TEMP/W semi-adiabatic curing test – effect of insulation type (polystyrene, water, air or no insulation) on the mean volumetric specimen temperature ($T_s$)**
The results of this modelling indicated that changing the insulation from polystyrene to air, raising the ambient temperature surrounding the experimental set-up and increasing the specimen size would all, in conjunction, reduce heat dissipation. However, a significant increase in specimen size and elevation of the ambient temperature of the laboratory environment are not feasible options, and the difference in temperature reduction between the currently used polystyrene insulation and air is very small. Thus none of these alterations to the experimental set-up were seriously considered for implementation.
Given the inability to determine the true adiabatic temperature of a CPB specimen using the heat-loss compensation method and semi-adiabatic testing, the choice for curing temperature for hydration cell specimens was based on in situ temperature measurements. The target specimen temperature for the SNM tests was chosen as 34°C based on the final equilibrium temperature evident in Figure 4.1. The choice of KB temperature was more difficult due to the absence of in situ temperature data and no clear results from the semi-adiabatic testing. Considering that a temperature increase was evident in the KB semi-adiabatic testing compared with no thermal response in the SNM semi-adiabatic testing and that significantly higher temperatures were recorded in the RL stope compared with the SNM stope, a target temperature of 44°C was chosen for the KB material.

4.5. Temperature hydration cell development

The results of the semi-adiabatic testing highlighted the difficulty in achieving perfect thermally insulated conditions. Given the inability of the fully insulated box to maintain heat within the specimen, the reduced insulation properties of the steel hydration will result in even greater heat loss. Therefore, an external source of heating was used to maintain temperature control of the specimen within the hydration cell.

To generate adiabatic curing conditions, any temperature increase experienced by the specimen must be solely due to exothermic hydration reactions. The role of a temperature control system is only to minimise or ideally prevent heat dissipation without actively heating the specimen. The isothermal curing of specimens (Fall et al., 2007, 2010) are not completely representative of true thermal conditions in a stope environment during CPB curing. While the temperature maintained in the thermal chamber may be the same as the final temperature achieved in a stope, the initial heating rate of the early age specimens required to reach the desired temperatures is not necessarily realistic compared with the potential rate at which the exothermic hydration reactions occur. The ultimate CPB properties are known to be highly dependent on the very early age curing temperatures (Fall et al., 2010) and so to accurately replicate in situ conditions, the temperature control system must allow heating of the specimens to their final curing temperature to occur in a similar timeframe to that experienced in situ. In the case of the in situ stope temperatures shown in Figure 4.1 and Figure 4.2 this occurs over a period of hours.

To allow a specimen to develop heat in the same way as would occur in situ, the thermal response of the system must be very quick and precise to adjust to the initially small incremental temperature increases to successfully prevent heat dissipation without adding additional heat to the specimen. This section of the chapter details the design, development and operation of a temperature control system integrated into the hydration cell experimental apparatus, previously described in Chapter 3, which allows replication of in situ thermal conditions for a curing CPB specimen.
4.5.1. The temperature control system

The external heat source was provided via two electrical heating mats, referred to as blankets, which were powered and controlled by a custom designed system. The main elements of this system are shown in Figure 4.14 and include:

- two heating blankets; one located around the outside of the hydration cell and the other within the void space surrounding the specimen inside the cell;
- three temperature sensors used to monitor the hydration cell case temperature from inside the cell, the air temperature within the cell and the internal specimen temperature; and
- a custom designed control system comprising a heater control unit and computer software to control the power supply to the heaters.

Figure 4.14 Temperature control system

Figure 4.15 shows how these elements are integrated into the existing hydration cell set-up.
Figure 4.15 Temperature hydration cell apparatus

The hydration cell was pressurised with air, allowing the internal heating blanket, as well as the case and air temperature sensors, to be used without waterproofing. The air also provides better thermal insulative properties due to its lower thermal conductivity compared with water (Table 4.1). Three different configurations were trialled for the specimen temperature sensor. Initially, the sensor was coated in plastic and cast into the specimen, but this was insufficient to keep out water when inundated with high pore pressures. Recovery of the sensor from the cemented specimen at the conclusion of the test was also unsuccessful, requiring a new sensor to be wired into the cell for each subsequent test. To improve waterproofing and allow easier recovery of the sensor, the sensor was encased in steel and inserted into the specimen through the base of the cell. However, due to its close proximity to the bender element, the steel casing of the probe attracted the bender signal, preventing the true stiffness of the specimen from being measured. This set-up was modified slightly to insert the steel encased sensor into the side of the specimen through a custom-made membrane sealed with O-rings. This successful configuration is shown in Figure 4.16.
4.5.2. Generating desired thermal curing conditions

Temperature control of the set-up was achieved via a feedback loop from the case temperature sensor to the control system, providing thermostatic control of the system by powering the heating blankets on and off to maintain the user-defined target temperature; there was no intermediate power setting for the heating blankets.

A time delay was found to exist between the application of heat via the heating blanket and when the heat manifests as a temperature increase in the specimen, as indicated in Figure 4.17.

After approximately 1 hour of heating, constant temperatures were reached and maintained in the air and case temperatures, yet the specimen temperature continued to increase, reaching a
constant value 3-4 hours later. It is due to this delay that the case temperature reading was used as the thermostat sensor of the system. Waiting until the specimen temperature reaches the desired level before powering off the heaters would introduce additional heat into the system and ultimately generate higher than desired temperatures within the specimen. To minimise this heating delay, and prevent overheating of the specimen beyond the desired temperature, a heater timing array, labelled as isothermal in Figure 4.18, was implemented. This array powers the heaters on and off for different durations (i.e. “c” seconds) depending on the difference in temperature (“delta degrees”) between the measured and target temperatures; the smaller the value of “delta degrees” the shorter the “T-on” period. The temperatures shown in Figure 4.17 were generated using the array values in Figure 4.18.

<table>
<thead>
<tr>
<th>Delta Degrees</th>
<th>Increasing Temp</th>
<th>Decreasing Temp</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>T-on</td>
<td>T-off</td>
</tr>
<tr>
<td>50</td>
<td>1</td>
<td>0.9</td>
</tr>
<tr>
<td>30</td>
<td>2</td>
<td>0.7</td>
</tr>
<tr>
<td>20</td>
<td>3</td>
<td>1.5</td>
</tr>
<tr>
<td>5</td>
<td>4</td>
<td>5</td>
</tr>
<tr>
<td>10</td>
<td>5</td>
<td>20</td>
</tr>
<tr>
<td>15</td>
<td>7</td>
<td>20</td>
</tr>
<tr>
<td>10</td>
<td>10</td>
<td>0</td>
</tr>
<tr>
<td>20</td>
<td>2</td>
<td>2</td>
</tr>
</tbody>
</table>

Figure 4.18 Heater timing array

The temperature gap between the specimen and its surrounds (see Figure 4.17) was shown to be too large, preventing the specimen generating its own heat; any heat that is generated would be immediately dissipated. This is clearly indicated by the constant temperature maintained in the specimen after the initial temperature increase. To improve the thermal response of the system and to reduce this temperature difference, small fans were installed inside the hydration cell to circulate the air, as well as implementing independent control of the internal and external heating blankets. The internal fans were found to have no effect on any of the measured temperatures, while independent control of each blanket proved too complex for the control system software. However, given the slow thermal response of the system, as indicated in the subsequent section (4.5.2.1), it is not anticipated that such modifications would adequately improve control over the thermal conditions of the system.
4.5.2.1. Adiabatic versus isothermal

Initially, it was intended to have the heating control system maintain the case temperature at a user-defined value below that of the specimen, using the heating array in Figure 4.18 labelled as “adiabatic”. Small intervals of “T-on/off” were used to allow closer control of the heat applied to the system; however, hydration cell tests conducted using this “adiabatic” heating were found to show no temperature increases, as no initial temperature increase was identified by the 0.5°C accuracy temperature sensors, such that the heating control system was not activated. An isothermal approach was then adopted as per the heating example in Figure 4.17 where the final curing temperature and the period over which the specimen was heated to reach this value were established as representative of that observed in situ.

To achieve the desired specimen curing temperature, a larger target temperature was chosen to account for the offset in temperatures discussed above. The case and air temperatures both overshot the target temperature. However, due to the delay in heat transfer, the specimen temperature wasn’t affected. Instead, the additional heat added into the system acted to slightly increase the rate of temperature rise of the specimen. Following heating, stable temperatures were achieved throughout the whole system (case, air and specimen), each separated by a constant temperature difference. These temperature offsets remained unchanged throughout the test, maintaining a constant temperature.

Given the heat transfer delays and temperature offsets, the target temperature and timing array values required to achieve a desired specimen temperature within a given time period had to be determined by extensive calibration testing. The results of the calibration process indicated target temperatures of 53°C and 40°C were required for KB and SNM testing to generate specimen temperatures of 44°C and 34°C respectively. The ultimate curing temperature of the specimen was not found to be sensitive to minor changes in the timing array.

It is this ability to heat the specimen during desired time period, in addition to coupling the elevated temperature curing with effective stress generation, that makes this experimental testing novel in its development, compared with previous CPB temperature testing work (Fall et al., 2010; Fall et al., 2007; Fall & Pokharel, 2010; Fall & Samb, 2008b).

4.6. Experimental testing program

The KB and SNM CPB mixes were chosen for testing in the temperature-controlled hydration cell because they represent the opposite ends of the spectrum with regard to consolidating (SNM) or non-consolidating (KB) behaviour (Fahey et al., 2010). Despite the previous discussion of in situ temperature measurements from the RL stope, no experimental testing was carried out with the RL material in this chapter.

To independently identify the effects of temperature and self-desiccation, or effective stress ($p'$) (as defined in Chapter 3), on the development of mechanical properties, specimens were cured under four different curing conditions, with varying stress and temperatures conditions. The
development of small strain stiffness \( (G_o) \) (also defined in Chapter 3) was monitored throughout the 7-day curing period and the 7-day UCS test results compared. This curing age is significantly younger than many of the experimental results published in the literature discussed previously (Section 4.3.1). However, given the focus of this research on the generation of material properties during the cementation process, this was considered an appropriate testing age. The four different curing conditions imposed were:

- room temperature, preventing \( p' \) generation (curing condition A);
- room temperature, allowing \( p' \) generation (curing condition B);
- elevated temperature, preventing \( p' \) generation (curing condition C); and
- elevated temperature, allowing \( p' \) generation (curing condition D).

These conditions are summarised in Table 4.2.

**Table 4.2 Testing matrix of experimental curing conditions for temperature and effective stress testing**

<table>
<thead>
<tr>
<th>Stress condition</th>
<th>No ( p' ) generation</th>
<th>With ( p' ) generation</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Temperature condition</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Room temperature</td>
<td>A</td>
<td>B</td>
</tr>
<tr>
<td>Elevated temperature</td>
<td>C</td>
<td>D</td>
</tr>
</tbody>
</table>

4.6.1. Stress and temperature conditions

A confining cell pressure, \( \sigma_c \), of 400kPa was applied and due to the slurry nature of the specimen, this pressure was transferred to the sample such that \( p' = 0 \) and \( u_i = \sigma_c \) as described for the standard hydration cell apparatus in Chapter 3.

To generate the stress conditions in curing conditions B and D, undrained conditions were maintained throughout the test to allow effective stress generation due to self-desiccation. In curing conditions A and C, measures were taken to prevent effective stress generation occurring. For the SNM tests this involved opening the top cap of the specimen to interface with the air-filled cell, forcing equilibrium between specimen pore pressure and the cell pressure. To prevent evaporation of the water phase from within the specimen due to the open top cap valve, a small open-ended tube filled with water was attached to the top cap opening; at the completion of testing the tube remained full with water.

Due to the high coefficient of consolidation \( (c_v) \) of the SNM material (consolidating fill), this approach was sufficient to dissipate any effective stress generation. However, this approach was insufficient for the low \( c_v \) KB fill (non-consolidating) where, instead, a back pressure \( (\sigma_b) \) was applied to the specimens to create \( p' = 5kPa \) to regulate the pore pressure. This minimal
effective stress was considered low enough to have the same effect as zero effective stress and would prevent liquefaction of the specimen if any minor calibration errors existed between the two pressure transducers. Although this was an improvement on the initial method, the applied back pressure was not completely successful at preventing effective stress conditions generating under curing conditions A and C. The effective stress plots for each curing condition of both SNM and KB mixes are shown in Section 4.7 and will be discussed later.

The process for choosing the curing temperatures for each material was outlined previously in Section 4.4. The elevated isothermal temperatures for KB and SNM specimens under curing conditions C and D were 44°C and 34°C respectively. The constant temperature room provided the 23°C curing temperature used for curing conditions A and B.

4.7. Results

This section of the chapter presents the results of the testing program, outlined in Table 4.2, for both KB and SNM CPB mixes. The results from each individual test including pore pressures, bender element stiffness, sample temperature and final UCS are presented here for both material type data sets. This section is divided into two parts; first the KB data is presented, followed by the SNM data. The results from all tests conducted under all curing conditions are plotted together, along with their mean value where appropriate. Table 4.3 indicates the number of hydration tests conducted under each curing condition. Detailed discussion of the results is reserved for Section 4.8 where the effect of different curing conditions on each CPB type is examined, as well as a comparison between the behaviour of the two different mixes.

<table>
<thead>
<tr>
<th>Material</th>
<th>Curing condition</th>
<th>No. tests</th>
</tr>
</thead>
<tbody>
<tr>
<td>KB</td>
<td>A</td>
<td>3</td>
</tr>
<tr>
<td></td>
<td>B</td>
<td>3</td>
</tr>
<tr>
<td></td>
<td>C</td>
<td>2</td>
</tr>
<tr>
<td></td>
<td>D</td>
<td>3</td>
</tr>
<tr>
<td>SNM</td>
<td>A</td>
<td>2</td>
</tr>
<tr>
<td></td>
<td>B</td>
<td>2</td>
</tr>
<tr>
<td></td>
<td>C</td>
<td>1</td>
</tr>
<tr>
<td></td>
<td>D</td>
<td>2</td>
</tr>
</tbody>
</table>

4.7.1. KB material temperature hydration cell test results

The KB test data for all curing conditions are presented here, showing the effective stress generation, internal specimen temperature, small strain stiffness and 7 day UCS stress-strain curve.
4.7.1.1. KB effective stress generation

The generation of effective stress due to self-desiccation for each curing condition A to D is shown in Figure 4.19 (a) to (d) respectively.

An average plot of the effective stress was not calculated for tests conducted under curing condition A due to problems with the effective stress data in test A1. The effective stress measured in A1 was based on the measurement of the applied back pressure rather than specimen pore pressures. Instead, A3 is considered representative of effective stress generation under curing conditions A.

The non-consolidating nature of the KB material made it difficult to maintain zero-effective stress during curing conditions A and C as indicated by Figure 4.19 (a) and (c). This issue will be discussed in detail subsequently. The tests conducted under curing condition B and D both generate effective stress as expected.
4.7.1.2. KB specimen temperature

The internal specimen temperatures throughout curing are shown in Figure 4.20 (a), (b) and (c) for curing conditions A, C and D.

Figure 4.20 KB mean volumetric sample temperature \(T_s\) under varying curing conditions: (a) curing condition A - room temperature, no \(p'\) generation; (b) curing condition C - elevated temperature, no \(p'\) generation; (c) curing condition D – elevated temperature with \(p'\) generation.

An issue with the specimen temperature probe prevented the recording of specimen temperature during testing of curing condition B. However, as the same temperature condition was applied in both curing conditions A and B, the average curing temperature for curing condition B is considered the same as that for curing condition A. The elevated “room temperature” reading observed in the A3 test (Figure 4.20 (a)) occurred due to the accidental activation of the temperature control system demanding a temperature of 25°C. As mentioned, the effective stress generated by this test was considered representative for this curing condition despite the slightly elevated temperature, as the difference in the temperatures between A1/A2 and A3 are minor compared with the elevated temperature of curing conditions C and D.
4.7.1.3. KB small strain stiffness development

The development of small strain stiffness in each hydration cell test is shown in Figure 4.21.

![KB A](image1)

![KB B](image2)

![KB C](image3)

![KB D](image4)

Figure 4.21 Rate of KB specimen stiffness \( (G_o) \) gain under varying curing condition: (a) curing condition A - room temperature, no \( p' \) generation; (b) curing condition B - room temperature with \( p' \) generation; (c) curing condition C - elevated temperature, no \( p' \) generation; (d) curing condition D – elevated temperature with \( p' \) generation.

4.7.1.4. KB UCS testing

The stress-strain curves from each UCS test are presented in Figure 4.22 (a), (b), (c) and (d) for KB curing conditions A, B, C and D respectively. Due to the difference in stress-strain behaviour of each test, an average of the entire stress-strain curve is not possible. Instead, to allow comparison between different curing conditions, the maximum stress and corresponding axial strain at which the peak stress occurs is averaged for each curing condition and marked with an ‘X’ as shown in Figure 4.22.
Figure 4.22 Strain-strain curves from UCS testing of KB specimens cured under varying curing conditions: (a) curing condition A - room temperature, no $p'$ generation; (b) curing condition B - room temperature with $p'$ generation; (c) curing condition C - elevated temperature, no $p'$ generation; (d) curing condition D - elevated temperature with $p'$ generation.

The minor difference in curing temperature for test A3 is reflected in greater stiffness and peak UCS results compared with tests A1 and A2. However, the slight benefit afforded to this A3 specimen compared with others cured under curing condition A, is shown to be relatively minor when compared with results of specimens cured at the elevated curing temperatures.

4.7.2. SNM material temperature hydration cell test results

Similarly for the SNM testing, the results are presented here for all curing conditions showing the effective stress generation, internal specimen temperature, small strain stiffness and 7 day UCS stress-strain curve.
4.7.2.1. SNM effective stress generation

The effective stress generated in each SNM specimen cured under curing conditions A, B, C and D are shown in Figure 4.23 (a), (b), (c) and (d) respectively.

![SNM A](image)

![SNM B](image)

![SNM C](image)

![SNM D](image)

Figure 4.23 SNM effective stress ($p'$) measurements under varying curing conditions: (a) curing condition A - room temperature, no $p'$ generation; (b) curing condition B - room temperature with $p'$ generation; (c) curing condition C - elevated temperature, no $p'$ generation; (d) curing condition D – elevated temperature with $p'$ generation.

Figure 4.23 (a) and (c) both indicate negative (rather than zero) effective stress values due to a slight difference in PPT calibration. These plots successfully indicate that the zero effective stress conditions for curing condition A and C have successfully been maintained throughout the test duration.

4.7.2.2. SNM specimen temperature

The only SNM tests which successfully recorded specimen temperature were those conducted under curing condition D, as shown in Figure 4.24.
Figure 4.24 SNM mean volumetric sample temperature ($T_s$) under curing condition D - elevated temperature with $p'$ generation.

Despite the absence of specimen temperature records for the SNM curing conditions A, B and C, the temperatures measured under the curing conditions A and B in the KB tests (Figure 4.20) were assumed to also represent the temperature in the equivalent SNM tests, with an average specimen temperature of 23°C. The elevated temperature for the SNM curing condition C was assumed to equal that of SNM curing condition D (Figure 4.24).
4.7.2.3. SNM small strain stiffness generation

The small strain stiffness generated by each specimen under each curing condition is shown in Figure 4.25 (a) to (d) for curing conditions A, B, C and D respectively.

![SNM A](image1)

![SNM B](image2)

![SNM C](image3)

![SNM D](image4)

**Figure 4.25** Rate of SNM specimen stiffness ($G_o$) gain under varying curing condition: (a) curing condition A - room temperature, no $p'$ generation; (b) curing condition B - room temperature with $p'$ generation; (c) curing condition C - elevated temperature, no $p'$ generation; (d) curing condition D – elevated temperature with $p'$ generation.
4.7.2.4. SNM UCS testing

The stress-strain curves from each UCS test are presented in Figure 4.26 (a) to (d) for SNM curing conditions A, B, C and D respectively.

![Stress-strain curves for SNM specimens](attachment:stress_strain_curves.png)

Figure 4.26 Strain-stress curves from UCS testing of SNM specimens cured under varying curing conditions: (a) curing condition A - room temperature, no $p'$ generation; (b) curing condition B - room temperature with $p'$ generation; (c) curing condition C - elevated temperature, no $p'$ generation; (d) curing condition D - elevated temperature with $p'$ generation.

The mean peak stress and corresponding mean axial strain is indicated by an ‘X’ for each curing condition as shown in Figure 4.26. The exception is curing condition C where only one test was conducted.
4.8. Discussion

This section compares the calculated mean results of each curing condition (this mean was presented previously with each individual test result) with other curing conditions of the same material, allowing comparison and discussion regarding the effect of temperature and effective stress on the material’s mechanical behaviour. The difference in behaviour between the two CPB mixes is then considered, with a discussion of the effect of temperature and effective stress of the consolidating SNM CPB compared with the non-consolidating KB CPB mix (Fahey et al., 2010).

4.8.1. Effect of temperature on mechanical properties

4.8.1.1. Comparison of effective stress for KB and SNM

The average effective stress plots from each stress condition, shown in Figure 4.19 and Figure 4.23, are plotted together in Figure 4.27 (a) and (b) for KB and SNM material respectively.

![Figure 4.27 Comparison of effective stress (p') generation with time (t) for (a) KB and (b) SNM for curing condition A - room temperature, no p' generation; curing condition B - room temperature with p' generation; curing condition C - elevated temperature, no p' generation; and curing condition D – elevated temperature with p' generation.](image)

Under curing conditions B and D, the magnitude of effective stress generated for both KB and SNM, is larger under a higher curing temperature i.e. p' generation is greater under curing condition D, compared with B. This is as expected, given the known effect of elevated curing temperatures on hydration progression, and that hydration progression generates the effective stress.

The difficulty in maintaining a zero effective stress condition in the KB material for curing conditions A and C is clearly shown by the ~20kPa p' maintained in curing condition A and the large effective stress generated in C. While the applied backpressure was not successful in preventing p' generation in the case of A, it was able to reduce the extent to which it developed.
given the difference between A and B. The effect of elevated curing temperature is again evident where $p'$ generation under C is significantly larger than that generated under A; however, it was not expected that the effective stress level of C would rival that of D, given the difference between $p'$ at room temperature (B) and $p'$ at an elevated temperature (D). Instead it was expected that the relative difference between A and C would remain the same as between B and D. Pore pressure was measured at the top end of the KB specimen, the opposite end of the specimen from where the backpressure was applied (see Figure 4.15). It is assumed that the pore pressure measured at this location is representative of the conditions throughout the specimen, while potentially, as will be shown in Chapter 7 Section 7.3.2, this may not be correct.

The difficulty in maintaining $p' = 0$ in the KB material is attributed to its categorisation as a non-consolidating fill (Fahey et al., 2010). Due to the low permeability of such a fill, the rate of pore fluid flow through the material matrix is reduced. In the case of the KB temperature hydration cell tests, the applied backpressure was expected to act to replenish the specimen’s pore fluid that was depleted by self-desiccation in order to prevent effective stress generation. Instead, the permeability of the KB fill is sufficiently low that the back pressure is not able to adequately replenish the fluid rate fast enough compared with the ‘use’ of water in the self-desiccation process and hence effective stress is inevitably generated. There were no such problems with the consolidating (higher permeability) SNM fill where $p' = 0$ was successfully maintained for all SNM tests under curing conditions A and C.

The effective stress generated in the KB specimens under each of the four curing conditions is considerably larger than the $p'$ achieved in the SNM set of tests. This is likely due to the lower binder content of 1.5% (by weight) for the SNM fill, compared with 5% for the KB fill, together with the lower relative permeability of the KB material, as discussed.

4.8.1.2. Comparison of small strain stiffness for KB and SNM

The average small strain stiffness plots from each stress condition, shown in Figure 4.21 and Figure 4.25, are plotted together in Figure 4.28 (a) and (b) for KB and SNM material respectively.
Chapter 4
The Influence of Curing Temperature and Stress Conditions on Mechanical Properties of CPB

(a) KB
(b) SNM

Figure 4.28 Comparison of small strain stiffness ($G_o$) generation with time ($t$) for (a) KB and (b) SNM for curing condition A - room temperature, no $p'$ generation; curing condition B - room temperature with $p'$ generation; curing condition C - elevated temperature, no $p'$ generation; and curing condition D – elevated temperature with $p'$ generation.

The SNM specimens cured under curing condition D show significantly greater stiffness development than specimens cured under A, B or C. It is not expected that the $G_o$ curve for C should be so low, given the effective stress generation demonstrated in Figure 4.27 (a) and the positive correlation between these two mechanical properties; there is no explanation that the author can provide for this result. Similarly there is no significant difference between the stiffness generation of A or B in the SNM tests. It must be repeated that the results presented in this chapter represent an average of two or three individual test results (with the exception of SNM curing condition C) and that the results shown here are reproducible.

The KB material shows greater stiffness development under both elevated temperature curing conditions C and D compared with the specimens cured at room temperature. Yet as remarked for the SNM, the expected correlation between $p'$ and $G_o$ is not evident, as the data for curing condition A shows greater $G_o$ values compared with B, despite the reverse with regard to $p'$. The magnitude of $G_o$ for curing condition D is comparable between the KB and SNM mixes, which is also contrary to the difference observed between $p'$ development in each material. The fact that this difference in expected $p'/G_o$ behaviour is evident in both SNM and KB material testing indicates that it is not simply related to the difficulties in maintaining specific stress condition (i.e. $p' = 0$) encountered in the KB tests.

4.8.1.3. UCS

To compare the 7-day specimen strength achieved by curing under each of the investigated curing conditions, the peak stresses indicated by the ‘X’ in Figure 4.22 and Figure 4.26 for KB and SNM respectively are plotted as $q_u$ in Figure 4.29 (a) and (b).
Chapter 4
The Influence of Curing Temperature and Stress Conditions on Mechanical Properties of CPB

Figure 4.29 Comparison of peak UCS strength ($q_u$) for (a) KB and (b) SNM cured under curing condition A - room temperature, no $p'$ generation; curing condition B - room temperature with $p'$ generation; curing condition C - elevated temperature, no $p'$ generation; and curing condition D - elevated temperature with $p'$ generation.

These results clearly indicate a trend of increasing strength with both effective stress and curing temperature, in addition to highlighting the positive interaction between these two variables.

The $q_u$ values, for the SNM CPB, show consistent increases in peak strength from $q_{uA} = \sim 140kPa$ to $q_{uD} = \sim 220kPa$. The results suggest that curing temperature provides a greater influence on specimen 7-day strength, than effective stress generation. This is indicated in the results where $q_{uC}$ is significantly larger than $q_{uB}$ for both KB and SNM CPB. However in the KB testing, unlike the SNM CPB, no further strength increase is afforded with the addition of effective stress generation, as seen by a comparison of $q_{uC}$ and $q_{uD}$. This is explained by a higher than expected strength from curing condition C, such that there is no significant difference between curing conditions C and D, given the previously discussed difficulties associated with a $p' = 0$ stress condition.

Considering the non-consolidating nature of the KB CPB, it was anticipated that KB would obtain the greatest benefits from curing under elevated temperatures due to its reliance on the self-desiccation mechanism for effective stress generation (due to slow self-weight consolidation resulting from low $c_v$) and the significant effect temperature has on this mechanism. This is validated by examining the percentage increase in strength between curing conditions, which is greater for KB compared with SNM. However, this is also related to the higher binder content of KB compared with SNM.

An indication of the comparative failure behaviour is given in Figure 4.30, where the axial strain level at which the peak strength $q_u$ is achieved, is plotted against $q_u$. 

- 67 -
Chapter 4  The Influence of Curing Temperature and Stress Conditions on Mechanical Properties of CPB

Figure 4.30 Comparison of maximum UCS stress and associated axial strain for (a) KB and (b) SNM cured under curing condition A - room temperature, no $p'$ generation; curing condition B - room temperature with $p'$ generation; curing condition C - elevated temperature, no $p'$ generation; and curing condition D – elevated temperature with $p'$ generation.

The peak UCS stresses ($q_u$) for the KB material fall within a 250kPa stress range over a corresponding strain range of <0.5%. Comparing this with the SNM UCS tests, where the peak UCS stress range is 80kPa over 2% strain, indicating increased ductility for the SNM material. The significantly larger strengths achieved by the KB specimens under all curing conditions compared with SNM, is almost certainly due to the higher binder content of the KB mix. The difference in strain may also relate to this, whereby the lower resulting strength allows for increased specimen ductility.

Surprisingly, if the full stress strain curve is examined for specimens cured under high temperatures (curing conditions C and D for either KB or SNM as shown in Figure 4.22 and Figure 4.26 respectively) there is no significant difference in post-peak failure mode, despite suggestions in the literature that curing at elevated temperatures should promote quasi-brittle failures (Fall et al., 2010).

4.9. Conclusions

This chapter presented the development, calibration and testing of a hydration cell successfully adapted to include temperature control. The hydration cell developed by Helinski et al. (2007a) was modified to include a temperature control system that allowed the hydration behaviour of CPB to be examined under both effective stress (due to self-desiccation) and elevated temperature curing. Such conditions are a closer representation of conditions in situ compared with traditional laboratory curing techniques.

The temperature-controlled hydration cell was then used to examine the effect of these combined stress and temperature conditions on the mechanical properties of CPB specimens.
KB and SNM specimens were cured in the modified hydration cell for a 7-day period under the following curing conditions: A - room temperature, no $p'$; B - room temperature with $p'$; C - elevated temperature, no $p'$; and D – elevated temperature with $p'$. Effective stress and stiffness development were measured throughout the hydration period and at the conclusion of the hydration cell test, the specimen strength was determined through UCS testing. All tests exhibited repeatability with results presented in this chapter representing an average of two or more tests (excluding SNM curing condition C).

Elevated curing temperatures were shown to promote effective stress generation and the combination of both conditions (D) were found to generate significantly greater stiffness compared with the curing conditions A, B and C. This effect was evident for both KB and SNM, however the different in SNM was more pronounced. The positive correlation between effective stress and stiffness was not clearly identified in this study, potentially relating to difficulties with maintaining specific stress conditions within the hydration cell.

The UCS strengths clearly identified an increasing trend in strength with the elevated curing temperature and effective stress generation. These results indicated that for both non-consolidating (KB) and consolidating (SNM) CPB, the effect of elevated curing temperature is more significant on the 7-day strength, compared with effective stress development. Yet effective stress generation still has a marked effect on the ultimate strength. No significant effects associated with temperature were observed on the post-peak UCS behaviour.

This suite of testing clearly indicates significant enhancement of laboratory-cured CPB strength with control over both temperature and stress conditions, compared with traditional curing techniques, or indeed even the use of Helinski’s original hydration cell which controls only stress conditions. The current laboratory testing techniques are shown to underestimate the strength of CPB compared with curing under conditions more representatives of those encountered in situ. The modified hydration cell provides a tool that can be used to better understand the effect of in situ stope conditions on CPB hydration in a laboratory environment, allowing for safer, more efficient and economical CPB design.

**4.10. Recommendations for future work**

Many studies have indicated superior strength properties associated with in situ cured CPB compared with laboratory curing. This strength difference has been independently attributed to effective stress curing (Helinski *et al.*, 2007a; Grabinsky & Simms, 2006) as well as elevated curing temperatures (Fall *et al.*, 2010; Fall & Samb, 2009; Fall *et al.*, 2010) which occurs in an underground environment. Until the undertaking of the experimental work presented in this chapter, no study had been conducted on the compounded effect of these parameters. While, the results presented here clearly highlight the significant contributions that both effective stress and curing temperature afford to strength development, no comparison was made to actual in situ cured material.
Chapter 4

The Influence of Curing Temperature and Stress Conditions on Mechanical Properties of CPB

It is recommended that full instrumentation (Grabinsky & Bawden, 2007) during filling and curing of a CPB stope, including pore pressure and temperature measurements, be carried out for comparison with the effective stress and temperature that is generated in the hydration cell. Ultimately coring of the cured CPB material can then be used to directly compare the UCS strength of the in situ cured material, as well as stress and temperature conditions, with an equivalent specimen prepared in the temperature-controlled hydration.

While the isothermal temperature conditions presented in the chapter allow for temperature increases similar to that expected in a stope, without full adiabatic calorimetry testing there is currently no alternative for determining the adiabatic temperature increase due to cement hydration. Initially it was aimed to incorporate adiabatic testing into the temperature-controlled hydration cell developed in this research. However, thermal control of the system proved too difficult to achieve within the time and budget constraints of this research.
5.1. Introduction

Strength and stiffness are two very important properties in the safe and efficient design of cemented paste backfill (CPB) mixes. The development rate of these properties are indicators of hydration progression and provide important information regarding the stability of CPB material when stopes are exposed - an imperative safety aspect of CPB usage.

Chemical shrinkage measurements are routinely used in the design and use of cement pastes (CP) and mortars as these measurements provide a reliable indication of hydration progression (Powers, 1935; Parrott et al., 1990; Bentz et al., 2008) and have also been shown to exhibit a quasi-linear relationship to the strength of the final cemented product (Geiker & Knudsen, 1982). This has led to the development of a standard test method ASTM C 1608-07 for the measurement of chemical shrinkage of hydraulic cement paste, referred to henceforth as the ASTM test. Helinski’s hydration cell work highlighted the relationship between the progression of hydration and consequential development of mechanical properties such as effective stress/strength and stiffness.

Given evidence of the relationships between chemical shrinkage and hydration progression and between hydration progression and strength, is there a correlation (as yet unexamined) between chemical shrinkage and the increase in mechanical properties of CPB? If such a relationship exists, the use of the ASTM test for the measurement of chemical shrinkage in hydrating CPB may provide a simple screening tool or index test for utilisation in both the mix design process and QA/QC.

This chapter presents the results of experimental ASTM chemical shrinkage and hydration cell tests carried out as detailed in Chapter 3. These results are analysed using a developed version of the hydration model developed by Helinski et al. (2007a) to relate effective stress and stiffness to chemical shrinkage by comparing volumetric strain generation in the hydration cell specimen with the ASTM-measured chemically induced shrinkage strain.

5.1.1. Chemical shrinkage

Chemical shrinkage is a measurement of the overall volume change of a cementitious material that occurs during hydration due to the reduced volume of the final hydrated products compared
with the unhydrated constituents (Powers, 1935; Tazawa et al., 1995). It is this volume change that drives the self-desiccation mechanism previously described in Chapter 2.

Chemical shrinkage may be measured in one of two ways: volumetric method, where the volume of water absorbed in the specimen gives a direct measurement of the volume change associated with chemical shrinkage, or examination of the change in buoyancy of a hydrating specimen submerged in a liquid (Sant et al., 2006; Lura et al., 2010). While the latter method is easy to automate, it is a more expensive option due to the requirement of a high precision balance. The former method has been adopted for chemical shrinkage measurements in this chapter, and a summary explanation of the procedural details of this chemical shrinkage ASTM test is provided in Chapter 3.

In addition to chemical shrinkage, autogenous shrinkage, a subset of chemical shrinkage, is also commonly measured providing information regarding the potential for cracking via the measurement of the bulk strain generated in a closed hydration system. Autogenous shrinkage only occurs with conventional self-desiccation, where no free water exists within the system and moisture migration is prevented from occurring (Tazawa et al., 1995; Sant et al., 2006). Self-desiccation typically occurs where the water to cement ratio (as defined in Chapter 3) is less than 0.4, (Geiker & Knudsen, 1982; Powers & Brownyard, 1947) although this number varies slightly for different cement types. Due to the very high $w/c$ ratio of CPB (e.g. $w/c > 5$) self-desiccation never truly occurs, and rather the self-desiccation refers only to the reduction in pore water pressure as described in Chapter 2. As such, autogenous shrinkage is not seen to occur in hydration of CPB. This brief discussion has only been presented here for the sake of completeness.

A range of chemical shrinkage tests are presented in this chapter which consider a range of different $w/c$ ratios and mix constituents. With a lack of relevant CPB chemical shrinkage data with which to compare the experimental results, chemical shrinkage measurements were also made on hydrating cement pastes (CP) and compared with studies by Bentz (2008) and Lura et al. (2010).

### 5.2. Experimental testing program

Detailed information regarding the experimental testing procedures adopted and materials tested in this chapter have been presented in Chapter 3. This section provides additional details of the individual mixes tested as well as the experimental equipment and techniques implemented specifically in this chapter.

#### 5.2.1. Mixes

The cementitious mixes are characterised by their cement content ($C_c$), or water to cement ratio ($w/c$), solids content ($C_s$), $w/c$ ratio and void ratio ($e$) as defined in Chapter 3. In the CP mixes, the only solid contained in the mix is cement, so $C_c$ is equivalent to $C_s$. The cement (or binder)
content can also be expressed as the ratio by mass of water to cement, termed the water cement (w/c) ratio.

While the main focus of this work is the standard CPB mixes presented in Chapter 3, testing on cement paste (CP) mixes was also conducted for comparison purposes. Additional CPB mixes, different to those presented in Chapter 3, were also examined to consider the effect of tailings addition and further understand the effect of w/c ratio through comparison with previously published test data for the more typical low w/c ratio CP mixes (Lura et al., 2010; Bentz et al., 2008). For clarity, the details of all CPB and CP mixes tested in this chapter are presented in Table 5.1 and Table 5.2 respectively. It should be noted that the KB and SNM mixes tested in this chapter used tap water, rather than their respective process waters.

Table 5.1 CPB mix properties

<table>
<thead>
<tr>
<th>Mix</th>
<th>Tailings</th>
<th>Binder</th>
<th>Water</th>
<th>$C_c$ [%]</th>
<th>$C_s$ [%]</th>
<th>w/c</th>
<th>$e_o$</th>
</tr>
</thead>
<tbody>
<tr>
<td>KB6</td>
<td>KB</td>
<td>OPC</td>
<td>Tap</td>
<td>4.76</td>
<td>77.8</td>
<td>6</td>
<td>0.8</td>
</tr>
<tr>
<td>KB4</td>
<td>KB</td>
<td>OPC</td>
<td>Tap</td>
<td>6.97</td>
<td>78.2</td>
<td>4</td>
<td>0.8</td>
</tr>
<tr>
<td>KB3</td>
<td>KB</td>
<td>OPC</td>
<td>Tap</td>
<td>9.09</td>
<td>78.6</td>
<td>3</td>
<td>0.8</td>
</tr>
<tr>
<td>KB</td>
<td>KB</td>
<td>OPC</td>
<td>Tap</td>
<td>5.0</td>
<td>77.8</td>
<td>6</td>
<td>0.8</td>
</tr>
<tr>
<td>KB T</td>
<td>KB</td>
<td>OPC</td>
<td>Tap</td>
<td>6.0</td>
<td>77.8</td>
<td>6</td>
<td>0.8</td>
</tr>
<tr>
<td>KB P</td>
<td>KB</td>
<td>OPC</td>
<td>Process</td>
<td>6.0</td>
<td>77.8</td>
<td>6</td>
<td>0.8</td>
</tr>
<tr>
<td>SI T</td>
<td>SI</td>
<td>Swan GP</td>
<td>Tap</td>
<td>6.0</td>
<td>70</td>
<td>7.3</td>
<td>1.23</td>
</tr>
<tr>
<td>SI P</td>
<td>SI</td>
<td>Swan GP</td>
<td>SI Process</td>
<td>6.0</td>
<td>70</td>
<td>7.3</td>
<td>1.23</td>
</tr>
<tr>
<td>SNM</td>
<td>SNM</td>
<td>Minecem</td>
<td>Tap</td>
<td>1.5</td>
<td>78</td>
<td>18.75</td>
<td>0.9</td>
</tr>
</tbody>
</table>

Table 5.2 CP mix properties

<table>
<thead>
<tr>
<th>Mix</th>
<th>Binder</th>
<th>Water</th>
<th>$C_c$ [%]</th>
<th>w/c</th>
</tr>
</thead>
<tbody>
<tr>
<td>CP4</td>
<td>OPC</td>
<td>Tap</td>
<td>20</td>
<td>4</td>
</tr>
<tr>
<td>CP3</td>
<td>OPC</td>
<td>Tap</td>
<td>25</td>
<td>3</td>
</tr>
<tr>
<td>Bentz</td>
<td>OPC</td>
<td>Tap</td>
<td>74</td>
<td>0.35</td>
</tr>
<tr>
<td>Lura</td>
<td>OPC</td>
<td>-</td>
<td>66</td>
<td>0.5</td>
</tr>
</tbody>
</table>

5.2.2. Hydration cell with strain gauges

In addition to the use of Helsinki’s hydration cell as described in Chapter 3, strain gauges were incorporated into the set-up for additional testing to directly measure the volume strain occurring
due to specimen hydration. The use of the strain gauges required the hydration cell set-up to be modified slightly, as shown in Figure 5.1.

![Strain gauge](image1)
![tapered rim](image2)
![Pronged internal strain gauge feet](image3)

**Figure 5.1 Hydration cell experimental set up with strain gauges**

Longitudinal slots were cut in the supporting mould to provide access for the strain gauge attachment while still supporting the slurry specimen as indicated in Figure 5.1a. The strain gauge feet (indicated in Figure 5.1a and Figure 5.1c) used to support the gauges were attached with super glue to both the inner and outer sides of the membrane. The pronged internal feet were cast into the specimen, allowing direct measurement of self-desiccation-induced displacements via the attached strain gauge. The inner top edge of the mould was tapered to allow unrestricted movement of the top cap as the specimen potentially shrank during hydration (Figure 5.1b). The tapering ensured the membrane remained free of any tension, which may otherwise resist the movement of the strain gauge feet cast into the specimen, potentially resulting in erroneous strain gauge readings.

The axial strain ($\varepsilon_a$) of the specimen was calculated as an average of the two strain gauge displacement readings ($\delta L_1$ and $\delta L_2$) over the length of specimen considered ($L$) given by

$$\varepsilon_a = \frac{\delta L_1 + \delta L_2}{2L} \quad \text{Equation 5.1}$$

The volumetric strain ($\varepsilon_V$) occurring in the specimen was calculated assuming isotropic conditions by

$$\varepsilon_V = 3\varepsilon_a \quad \text{Equation 5.2}$$
The stiffness of the specimen can then be determined using the standard elastic stress-strain relationship where the decrease in pore pressure $u$ is equivalent to the increase in effective stress $p'$, assuming a constant total stress as per Equation 3.3 in Chapter 3.

$$K_s = \frac{\delta u}{\delta \varepsilon}$$  \hspace{1cm} \text{Equation 5.3}

5.3. Experimental results

This section of the chapter presents the results of the ASTM chemical shrinkage and hydration cell tests. Detailed discussion of the results in this section is limited and will be presented in subsequent sections of the chapter. The measured data from each ASTM test is presented\(^2\), along with plots of the same data normalised by mass of volume of binder incorporated in the mix. The hydration cell test results are shown as plots of effective stress ($p'$) and stiffness ($G_s$) development with time. All CP and CPB mixes were tested using the ASTM test, while hydration cell testing was only carried out on the KB, SNM and SI P mixes.

5.3.1. ASTM chemical shrinkage test results

The data from the chemical shrinkage tests on CP and CPB specimens are shown in Figure 5.2 and Figure 5.3 respectively. Each ASTM test curve shown is based on a minimum of three tests, with the results presented as an average with one standard deviation (S.D.) error bars. The measured data has been adjusted to account for evaporation, which was found to occur at a rate of 0.0096 mL/day despite the use of paraffin oil to minimise evaporation as per the ASTM standard.

\(^2\) While the majority of the ASTM tests and all subsequent analysis is solely the work of the author, an estimated 40% of the ASTM test work was carried out by a visiting researcher, Dr Lucas Festugato in conjunction with those tests conducted by the author.
Typically the measured chemical shrinkage is normalised by the mass of cement contained in the mix to enable comparison between different mixes (Lura et al., 2010). In the CP test specimens, the mass of dry cement ($M_c$) is calculated by

$$M_c = \frac{M}{1 + \frac{w}{c}}$$  \hspace{1cm} \text{Equation 5.4}$$

where $M$ is the bulk mass of the specimen.
The inclusion of additional filler material, or tailings, in the CPB specimens requires the dry mass of cement to be calculated by

$$M_c = M_{Cc} C_s$$  \hspace{1cm} \text{Equation 5.5}$$

The volume of binder can then be determined by

$$V_c = \frac{M_c}{G_c \rho_w}$$  \hspace{1cm} \text{Equation 5.6}$$

where \(\rho_w\) is the density of water and \(G_c\) is the specific gravity of the binder.

The normalised CP and CPB ASTM data are shown in Figure 5.4 and Figure 5.6 respectively. The measured data has also been normalised by the volume of binder in the specimen as shown in Figure 5.5 and Figure 5.7. The average (minimum of three tests) mass and volume of binder per specimen used to normalise the measured data is presented in Table 5.3.

<table>
<thead>
<tr>
<th>Mix</th>
<th>(M_c) [g]</th>
<th>(V_c) [cm(^3)]</th>
</tr>
</thead>
<tbody>
<tr>
<td>KB6</td>
<td>0.5789</td>
<td>0.1809</td>
</tr>
<tr>
<td>KB4</td>
<td>0.8462</td>
<td>0.2644</td>
</tr>
<tr>
<td>KB3</td>
<td>1.4381</td>
<td>0.4494</td>
</tr>
<tr>
<td>KB</td>
<td>0.8454</td>
<td>0.2684</td>
</tr>
<tr>
<td>SI T</td>
<td>0.944</td>
<td>0.2987</td>
</tr>
<tr>
<td>SI P</td>
<td>0.9595</td>
<td>0.3037</td>
</tr>
<tr>
<td>SNM</td>
<td>0.262</td>
<td>0.0819</td>
</tr>
</tbody>
</table>

5.3.1.1. Normalised CP ASTM test results

The CP ASTM test results confirm the literature (Bentz et al., 2008; Lura et al., 2010; Powers, 1935) which states that specimens with larger \(w/c\) ratios yield greater normalised chemical shrinkage volume. Data from the published work of Lura et al. (2010) and Bentz (2008) is shown in Figure 5.4 as a comparison of low \(w/c\) ratio CP mixes (Table 5.2); only the normalised chemical shrinkage data was published showing volume change per mass of binder. The order of increasing normalised shrinkage volume reflects the increasing \(w/c\) ratios of each specimen; Bentz (\(w/c = 0.35\)), Lura (\(w/c = 0.5\)), CP3 (\(w/c = 3\)) and CP4 (\(w/c = 4\)).
Figure 5.4 CP ASTM test data normalised by mass of binder

The close comparison of CP3, and to a lesser extent CP4, in Figure 5.4 with the published data from Lura et al. (2010) and Bentz (2008) gives confidence in the ASTM procedure adopted in this work.

5.3.1.2. Normalised CPB ASTM test results

Comparing the difference between the CPB ASTM tests, the trend of increasing normalised chemical shrinkage with increasing w/c ratio is also demonstrated in Figure 5.6 and Figure 5.7. The SNM specimen has the highest w/c ratio of 18.75 and exhibits the largest normalised...
chemical shrinkage volume. This trend is also indicated in a comparison of the KB3, KB4 and KB6 results.

\[ \text{Figure 5.6 CPB ASTM test data normalised by mass of binder} \]

\[ \text{Figure 5.7 CPB ASTM test data normalised by volume of binder} \]

Despite this trend, a large variability in chemical shrinkage was measured for specimens with equivalent \( w/c \) ratios. The KB and SI P specimens both have similar \( w/c \) ratios of 6 and 7.3 respectively and thus would be expected to achieve similar volume change (Bentz et al., 2009), yet the chemical shrinkage occurring in the SI P specimen is considerably lower than the KB specimen. This difference may be accounted for by the chemical variations between the different tailings, water and cement combination, as such variations are known to affect the
hydration process (Bouasker et al., 2008; Pera, 1999). In addition to this, the KB and KB6 CPB mixes are identical in mix constituents and proportions, with both mixes having a w/c of 6 as shown in Table 5.1, yet the normalised chemical shrinkage volumes are considerably different, suggesting a degree of variability may be inherent in the ASTM testing protocol. Both of these ideas will be considered later in this chapter.

Comparison between the CP and CPB chemical shrinkage tests shows the latter undergoes much greater normalised chemical shrinkage. This is assumed to be due to the considerably greater w/c ratios in the CPB mixes compared with the typical w/c ratios of CP mixes (Lura et al., 2010; Bentz et al., 2009). However, the total and normalised chemical shrinkage observed in the CP3 and CP4 specimens (Figure 5.4 and Figure 5.5 respectively) are less than that achieved by the CPB tests for KB3 and KB4, which are of equal w/c ratio. The presence of tailings, also referred to as filler, in the CPB tests and the use of process water, is the most significant difference between these experiments and thus the presence of the filler particles and tailings/process water associated chemical variations must play a role in the development of chemical shrinkage. The effect of filler particles and w/c ratio on these differences will be discussed subsequently in Sections 5.4.1, 5.4.2 and 5.4.3.

5.3.2. Hydration cell testing results

The experimental data from the hydration cell tests are presented in Figure 5.8 and Figure 5.9. The $K_s$ data presented in Figure 5.8 is assumed to be equivalent to $K_i$ shown in Equation 3.6 in Chapter 3. The effective stress $p'$ generated in each CPB test is shown in Figure 5.9. The SI hydration test data is based on an individual test, while the results for the SNM and KB tests are presented as an average of three tests with one S.D. error bar as shown.

![Figure 5.8 Bender element measured stiffness, $K_s$ (MPa), measured in the hydration cell](image-url)
Chapter 5 Evaluation of Chemical Shrinkage in Cementing Paste
Backfill

The correlation between increasing $K_s$ and $p'$ is consistent in all hydration tests. It is interesting to note that there is no obvious correlation between the ASTM and hydration test results. In the ASTM tests (Figure 5.6) the SNM specimen achieved the largest normalised volume change, followed by KB, then the SI specimen which achieves the lowest normalised volume change. In the hydration cell tests, the KB specimen achieved the greatest $K_s$ and $p'$ generation (shown in Figure 5.8 and Figure 5.9 respectively), followed by SI and then the SNM specimen with the lowest value.

The irregular behaviour of the SI pore pressure is due to the undertaking of permeability testing at intervals during the hydration test. The potential impact of permeability testing on specimen pore pressures will be discussed later in Section 5.4.9.

5.4. Analysis and discussion

This section presents an analysis of the experimental data for both the ASTM and hydration cell tests. First, detailed consideration is given to the ASTM chemical shrinkage tests for both CP and CPB materials considering the effect of $w/c$ ratio, the presence of filler material and accuracy of the test method. Then, an equation for chemical shrinkage, based on the work of Powers & Brownyard (1947) and Helinski et al. (2007a), is presented and compared with the ASTM measured data. This is followed by the development of a model to determine the chemical shrinkage occurring in the hydration cell specimen based on pore pressure and stiffness data. The results of this model are then compared with the measured ASTM chemical shrinkage data.
5.4.1. Effect of w/c ratio on chemical shrinkage

The w/c ratio of cement paste is known to influence chemical shrinkage measurements (Powers, 1935; Bentz et al., 2008; Lura et al., 2010). When there is a high concentration of cement in a mix (low w/c ratio), there may be insufficient water for the cement reactions to complete fully (Powers, 1935) or reduced space available for cement gel growth. Larger w/c ratios provide more water for hydration reactions, increased absorption/volume change potential (Powers, 1935) and greater void space for hydration product growth. Chemical shrinkage is an indication of this empty pore volume (Bentz et al., 2008) and thus increases with w/c ratio. This is clearly evident if the experimental ASTM tests for both CP and CPB specimens (as seen in Figure 5.4 and Figure 5.6 respectively) are compared.

There are slight variations in chemical shrinkage between CPB specimens for similar w/c ratios (i.e. SI and KB). However, this can be attributed to chemical differences between the mixes i.e. due to particle fineness, cement and tailings chemistry, water salinity etc., which are known to affect hydration. The cement particle fineness is known to affect the volume of normalised chemical shrinkage occurring in the specimen (Bentz et al., 2008) where finer particles are more reactive due to their increased surface area and thus generate increased chemical shrinkage compared with that of coarser cement mixes. The tailings particle size also has an influence which will be considered later in Section 5.4.2.1. The chemistry of the mix is also important given the different chemical hydration products that are produced at different rates throughout the hydration process (Tazawa et al., 1995). The influence of chemistry and salinity will be further considered later in this chapter.

5.4.1.1. Accuracy of high w/c ratio chemical shrinkage test specimens

In pastes with low w/c ratios, capillary porosity may become disconnected, preventing water absorption (Powers, 1935) into the sample, producing potential errors in measurements, i.e. hydration may be hindered by lack of access to water. This may also occur if a specimen is too thick and the water is not able to penetrate the specimen (Sant et al., 2006). The chemical shrinkage ASTM standard (ASTM, 2009) prescribes a maximum 10 mm specimen thickness and a recommended minimum w/c = 0.4 to avoid this problem. It is stated that higher w/c ratios may produce significant bleeding of the cement paste, effectively reducing the w/c ratio as time progresses. Given the high w/c ratio present in the CPB mixes (w/c >7), this highlights a potential issue in the appropriate application of this test.

First considering the CP specimens (w/c = 3 and 4), this propensity for bleed water was clearly evident in the specimen preparation, where immediate consolidation of the cement was observed and visible bleed water evident. This would suggest that the actual w/c ratio at the start of test measurements was lower than the original design mix. Geiker & Knudsen (1982) quantified the difference in w/c ratio due to this effect, and found for a design w/c ratio of 0.8, the volume of bleed water produced reduced the w/c ratio by 5%; specimens at w/c = 0.4 showed insignificant difference.
The bleed water produced by the CP4 ASTM specimen within the first few minutes of specimen preparation was measured and found to reduce the actual \( w/c \) ratio from the design value of 4 to 1.24. This clearly demonstrates the lack of ability to test high \( w/c \) ratio CP specimens using the ASTM test. However, no consolidation or bleed water was observed at any stage of the CPB specimen preparation, despite the \( w/c \) ratios ranging from 3 to 18.75. Given the fine nature of the tailings material, their comparatively small values of specific gravity (when compared with typical binder specific gravity values) and slow rates of consolidation, it is reasonable that the behaviour of the tailings aggregate would dominate, suspending the cement in the slurry matrix, thus limiting the occurrence of bleed water. This potentially demonstrates an appropriateness of the use of this technique for CPB at their full range of \( w/c \) ratios.

5.4.2. Effect of filler material on chemical shrinkage

The CPB mixes were observed to achieve greater normalised chemical shrinkage volumes in the ASTM test, compared with the CP specimens. This may be attributed to the typically large differences in \( w/c \) ratio between the different mixes, particularly in the case of KB3, KB4 and CP3, CP4 mixes, where as discussed previously, the generation of bleed water in the CP specimens significantly reduced the actual \( w/c \) ratio of the mixes, below that of the design values. A comparison of the average data from each of these tests is shown in Figure 5.10.

![Figure 5.10 Comparison of CP and CPB normalised chemical shrinkage in equivalent w/c ratio specimens](image)

However, in addition to this, there is evidence in cement literature that the presence of a “filler” material acts to accelerate the rate and total achieved chemical shrinkage volume. This is due to the provision of additional nucleation sites for hydrate growth by the filler particles (Bouasker et al., 2008; Bentz, 2005). Bouasker et al. (2008) examined the effect of limestone filler inclusions on the hydration process and found that that some chemical processes were
accelerated due to the limestone’s presence as they allowed for better dispersion of cement particles within the mix. The added chemical variety introduced into the mix was also shown to accelerate hydration kinetics (Bouasker et al., 2008; Pera, 1999). Despite the influence of the filler material in increasing chemical shrinkage, Bouasker et al. (2008) found that this did not significantly influence the positive correlation found between hydration progression and chemical shrinkage (Powers, 1935; Parrott et al., 1990; Bentz et al., 2008).

The PSD for Bouasker’s filler limestone material and that of the tailings material (SI, KB, SNM) are plotted together in Figure 5.11 and show similar distributions. Given this similarity, presence of the tailings material may explain the difference between CP and CPB chemical shrinkage volumes for equivalent w/c ratio specimens, in addition to the effect of bleed water discussed previously. Given the chemical complexity of the tailings filler materials and the process water, it is likely that this also modifies the hydration reactions. This idea is examined further in Chapter 6.

![Figure 5.11 Particle size distribution of limestone filler (Bouasker et al., 2008) compared with tailings](image)

5.4.2.1. Effect of filler PSD on chemical shrinkage

Cement particle PSD has been noted to affect the hydration rate, with finer cement producing greater normalised chemical shrinkage volumes, as previously mentioned (Bentz et al., 2001). From this it is reasonable to consider that the filler material PSD may have a similar influence. If the normalised chemical shrinkage results for the KB and SI CPB mixes are compared with their respective PSDs, it is seen the finer KB mix (Figure 5.11) achieves a larger normalised volume change. This is assumed to be in addition to any differences that might exist due to the slight differences in w/c ratios.
5.4.3. Effect of salt on chemical shrinkage

Variation in water chemistry (e.g. salinity) is also known to affect the hydration behaviour (Benzaazoua et al., 2004). In CPB mixes, the process water typically has high salinity levels (although this may vary between mine operations) which has been observed to significantly vary CPB properties, in addition to further impacting potential interactions between the tailings and water phases.

Data published by Revell (2004) indicated saline water had a negative effect on early age UCS strength, compared with the same CPB mix prepared with tap water. However, at later stages in the hydration process (4 or 10 days in Revell’s work) the saline water acts to increase the strength of the CPB specimen. Revell (2004) hypothesised that some of the salt may fuse together after a volume of the water is incorporated in the hydration process, which would explain why the saline water has a positive effect on the long term strength compared with strength at early ages. This hypothesis was also supported by large quantities of halite crystals (commonly referred to as salt) identified in scanning electron microscopy (SEM) testing carried out on equivalent specimens to those strength tested (Revell, 2004).

The effect of salinity on the hydration process, as indicated by the development of chemical shrinkage, was examined by comparing four sets of ASTM test data: SI mixed with tap or process water, shown in Figure 5.12 and similar for KB in Figure 5.13. For each mix type (i.e. KB or SI) the mixes were identical (i.e. constant w/c ratio) apart from variation in water type. For the SI tests, the tap water specimen achieved greater normalised chemical shrinkage volumes (in both final magnitude and rate) than the process water, suggesting that even the very slight increased salt content of the process water had a limiting effect on hydration progression. This is contrary to the results of the KB ASTM tests where the process water specimen achieved greater chemical shrinkage than the tap water specimen. The initial rates of chemical shrinkage for the KB tests were observed to be similar for both water types.

The salinity of the SI process water (Table 3.6) is observed to be similar to that of tap water, which is considered low for process water. The process water used in the testing was obtained directly from the mine and has been referred to in this thesis by the received labelling. It is possible that an error in labelling may have occurred and that this water is actually potable water rather than process water.
Based on the conclusions drawn from Revell’s work, it would suggest that the KB tests are more reliable than the SI tests, given that both chemical shrinkage and strength development are correlated through hydration progression, and that the KB specimen mixed with process water achieved greater chemical shrinkage for $t > 24$ hours. The opposing trends exhibited in the KB
and SI testing may also relate to the large difference in salt content between the SI and KB (significantly higher in the KB process water), where increased water salinity enhances the chemical shrinkage in a similar fashion to Revell’s observed strength increase. This variation in trends may also highlight potential implications of chemical variations and reactions. As mentioned previously, this is considered further in Chapter 6.

5.4.4. Modelling chemical shrinkage

To evaluate the potential for the ASTM test to be used as a screening or index test, it is necessary to relate the chemical shrinkage that the test measures to the results of the hydration cell. This section of the chapter presents a hydration model relating chemical shrinkage to the stiffness and pore pressures measured in the hydration cell, thus allowing a direct comparison between the results of the two tests.

An unhydrated element of CPB is composed of water, tailings and cement as indicated in Figure 5.14. The total volume of the element is $V_t$, the volume of the water $V_w = n$ (assuming full saturation) and the volume of the solid phase is $V_s = 1 - n$, where $n$ is the porosity of the specimen. Porosity is expressed as a function of time, $n(t)$, to account for the reduction in specimen porosity with time due to the growth of cement gel particles as hydration progresses. Porosity is related to the void ratio by

$$n = \frac{e}{1 + e} \quad \text{Equation 5.7}$$

![Figure 5.14 Composition of cement paste before hydration](image)

As identified in the work of Helinski et al. (2007a), hydration involves the chemical combination of water and cement to produce a cement gel. However, the volume of cement gel formed at a given time is less than the volume of water consumed by the reaction, resulting in an overall
shrinkage volume, $V_{sh}(t)$. The volume of water consumed in the reaction can be considered to comprise two components - an amount converted to solid volume and an amount lost from the system ($V_{sh}$) as if removed by an internal sink (Helinski et al., 2007a). The volume is ‘lost’ from the system due to the higher density of the hydrate product, compared with water.

Powers & Brownyard (1947) found experimentally, that the total chemical shrinkage volume after full hydration ($V_{sh}(\infty)$) was given by

$$V_{sh}(\infty) = 0.064 M_c$$

Equation 5.8

where $M_c$ is the mass of cement. For mine tailings, however, the total volume of chemical shrinkage can be significantly influenced by the tailings mineralogy and mining process chemicals, therefore, Helinski et al. (2007a) presented Equation 5.8 as

$$V_{sh}(\infty) = E_n M_c$$

Equation 5.9

where $E_n$ is a parameter referred to as the ‘efficiency of hydration’ with typical units of cm$^3$/g, assuming that $M_c$ is expressed in grams (g) and $V_{sh}$ in cubic centimetres (cm$^3$). $E_n$ is determined experimentally for a given material (i.e. cement/tailings mix).

To express the chemical shrinkage volume as a function of time during hydration, Helinski et al. (2007a) multiplied Equation 5.9 by a maturity function $m(t)$ such that

$$V_{sh}(t) = E_n M_c m(t)$$

Equation 5.10

The maturity function is given by

$$m(t) = 1 - \exp(-\kappa_h t)$$

Equation 5.11

where $\kappa_h$ is a dimensionless parameter controlling the rate of hydration and $t$ is the time since commencement of hydration. This maturity function presented is a modification of that originally adopted by Helinski et al. (2007a).

The chemical shrinkage determined from Equation 5.10 can be compared with that measured in the ASTM test. If $E_n$ is expressed in cm$^3$/g and $M_c$ is expressed in g, then Equation 5.10 represents a volume change in cm$^3$ allowing the equation to be fitted to the experimental chemical shrinkage data. Figure 5.15 and Figure 5.16 show a comparison between Equation 5.10 and the ASTM data for CP and CPB specimens respectively. The $E_n$ and $\kappa_h$ values required to fit the equation are given in Table 5.4.
To allow the expression of the chemical shrinkage as a strain, a more meaningful parameter in geomechanics, consideration is given to the composition of a CPB element (Figure 5.14) where the relationship between each phase is such that

$$V_f + V_c = V_t - n$$

Equation 5.12

Equation 5.12 can then be related to mass via the specific gravities of each material.
\[ \frac{M_f}{G_f \rho_w} + \frac{M_c}{G_c \rho_w} = V_t - n \]

where \( G_f \) is the equivalent specific gravity for all non-binder solids.

Substituting \( M_f \) from Equation 3.2 for determination of \( C_c \) in Chapter 3, into Equation 5.13 and considering a unit volume (i.e. \( V_t = 1 \text{ cm}^3 \)) the mass of cement per unit volume, denoted as \( m_c \), in units of g/cm\(^3\), can be given by

\[ m_c = \frac{\rho_w C_c G_c G_f (1 - n)}{G_f C_c + (1 - C_c) G_c} \]

Equation 5.14

If the density of water is given as 1 g/cm\(^3\), and \( E_h \) in cm\(^3\)/g, then multiplying Equation 5.14 by \( E_h \) gives a dimensionless strain (\( \varepsilon_h \)) occurring after full hydration.

\[ \varepsilon_h = E_h \frac{\rho_w C_c G_c G_f (1 - n)}{G_f C_c + (1 - C_c) G_c} \]

Equation 5.15

The chemical volume strain as a function of time is then expressed by

\[ \varepsilon_{sh}(t) = \varepsilon_h \left(1 - \exp(-\kappa_h t)\right) \]

Equation 5.16

The subscript ‘sh’ in Equation 5.16 indicates the chemical shrinkage as it varies with time, compared with the subscript ‘h’ which denotes the value at the end of the hydration process.

The development of \( \varepsilon_{sh}(t) \) for each ASTM test was determined from the measured ASTM data, and shown in Figure 5.17 and Figure 5.18 for CP and CPB test respectively; the final \( \varepsilon_h \) values are presented in Table 5.4. The subscript ‘ASTM’ refers to the parameter value being determined based on measured ASTM data. These results will be discussed subsequently in Section (5.4.7).
Table 5.4 ASTM test model parameters

<table>
<thead>
<tr>
<th>Mix</th>
<th>$\kappa_{h,ASTM}$ [1/days]</th>
<th>$E_{h,ASTM}$ [cm$^3$/g]</th>
<th>$\varepsilon_{h,ASTM}$ [-]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Bentz</td>
<td>0.35</td>
<td>0.038</td>
<td>0.0229</td>
</tr>
<tr>
<td>Lura</td>
<td>0.35</td>
<td>0.050</td>
<td>0.0275</td>
</tr>
<tr>
<td>CP3</td>
<td>0.2</td>
<td>0.062</td>
<td>0.0143</td>
</tr>
<tr>
<td>CP4</td>
<td>0.25</td>
<td>0.095</td>
<td>0.018</td>
</tr>
<tr>
<td>KB3</td>
<td>0.3</td>
<td>0.15</td>
<td>0.0209</td>
</tr>
<tr>
<td>KB4</td>
<td>0.1</td>
<td>0.28</td>
<td>0.0298</td>
</tr>
<tr>
<td>KB6</td>
<td>0.35</td>
<td>0.225</td>
<td>0.0163</td>
</tr>
<tr>
<td>KB</td>
<td>0.2</td>
<td>0.51</td>
<td>0.0388</td>
</tr>
<tr>
<td>SI P</td>
<td>0.13</td>
<td>0.17</td>
<td>0.0129</td>
</tr>
<tr>
<td>SI T</td>
<td>0.13</td>
<td>0.32</td>
<td>0.0243</td>
</tr>
<tr>
<td>SNM</td>
<td>0.2</td>
<td>0.7</td>
<td>0.0177</td>
</tr>
</tbody>
</table>

Figure 5.17 Chemically induced strain $\varepsilon_{sh,ASTM}(t)$ calculated from Equation 5.16 for ASTM CP tests
Figure 5.18 Chemically induced strain $\varepsilon_{sh\, ASTM}(t)$ calculated from Equation 5.16 for ASTM CPB tests

5.4.5. Impact of volume change on a hydraulically isolated specimen

The volume change associated with chemical shrinkage also has a significant impact on a hydraulically isolated element; as volume change occurs, a fluid (liquid or gas) from an additional source must be made available to fill the resulting void. In the case of the ASTM tests, additional water is readily available to the specimen. However, if the element is hydraulically isolated as in the hydration cell situation, (i.e. no liquid or gas can enter), strain compatibility between the fluid and the solid matrix prevents a void from forming; instead, the fluid must expand and the soil skeleton compress to reduce the void to zero. This is the condition under which a specimen cures in a hydration cell. The total strain of the soil skeleton and the associated stress change in the element can be derived from a classical flexibility approach.

The volume change in the soil ($\delta V_s$) due to a compressive stress ($\delta \rho'$) is given by

$$\delta V_s(t) = -\frac{V_s(t)}{K_s(t)} \; \delta \rho'(t)$$  \hspace{2cm} Equation 5.17

where $K_s$ is the bulk modulus of the soil skeleton. The volume change of the pore fluid ($\delta V_w(t)$) due to a tensile stress ($\delta u(t)$) in the pore fluid is

$$\delta V_w(t) = -\frac{V_w(t)}{K_w} \; \delta u(t)$$  \hspace{2cm} Equation 5.18
where \( K_w \) is the bulk modulus of the pore fluid, assumed to be 2,000 MPa. As the compressive stress in the soil and the tensile stress in the fluid both tend to reduce the void, the compatibility condition that no void be formed in the soil element can be written as

\[
-\frac{V(t)}{K_s(t)} \delta p(t) - \frac{V_w(t)}{K_w} \delta u(t) + \delta V_{sh}(t) = 0
\]  
Equation 5.19

For a constant total stress \( \delta p'(t) = \delta u(t) \), which when substituted into Equation 5.19 and divided throughout by \( V_t(t) \) gives

\[
- \left( \frac{1}{K_s(t)} + \frac{n}{K_w} \right) \delta u(t) + \frac{\delta V_{sh}(t)}{V_t(t)} = 0
\]  
Equation 5.20

where the \( \delta V_{sh}(t)/V_t(t) \) term is the chemical strain within the system and thus equivalent to the \( \varepsilon_{sh} \) term from Equation 5.16. Rearranging Equation 5.10 gives the following relationship between changes in pore pressure and chemical volume strain such that

\[
\delta u(t) = K^*(t) \delta \varepsilon_{sh}(t)
\]  
Equation 5.21

where \( K^*(t) \) is the composite pore-fluid elastic stiffness, defined as

\[
K^*(t) = \frac{1}{\frac{1}{K_s(t)} + \frac{n}{K_w}}
\]  
Equation 5.22

The \( K_s(t) \) term will be defined subsequently.

While in actuality, \( n \) decreases with time due to the growth of cement gel within the voids, in this model the porosity has been assumed to remain constant. Despite the added influence of effective stress generation due to the hydraulic isolation of the specimen also acting to reduce the porosity, the influence of \( n \) within the model is limited. Stiffness is the only parameter affected by porosity, as shown in Equation 5.22. Examining this equation, \( n \) has little influence on the final value of \( K^* \) due to its division by the large value of \( K_w \).

Differentiating Equation 5.16 gives the incremental chemical volume strain throughout the hydration process. Substituting this into Equation 5.21 gives

\[
\delta u(t) = K^*(t) \varepsilon_n \kappa_n \exp(-\kappa_n t)
\]  
Equation 5.23

which can be integrated to determine the pore pressures generated with time, due to the chemical shrinkage occurring in a hydraulically isolated element.

\[
u(t) = \int K^*(t) \varepsilon_n \kappa_n \exp(-\kappa_n t) \, dt
\]  
Equation 5.24

The hydration of cement and growth of cement bonds results in considerable stiffness increase with time; from initial slurry to a self-supporting material. The following function is used to model the stiffness generation with hydration.
\[ K_s(t) = K_{si} \left( \lambda_{KS} \cdot (\lambda_{KS} - 1) \exp (-\kappa_h t) \right) \]  

Equation 5.25

where \( K_{si} \) is the initial specimen stiffness and \( \lambda_{KS} \) is the ratio of final stiffness after full hydration to initial stiffness.

Combining Equation 5.22, Equation 5.24 and Equation 5.25 then gives the full expression for pore pressure varying with time

\[ u(t) = \int \frac{1}{K_{si} \left( \lambda_{KS} - (\lambda_{KS} - 1) \exp (-\kappa_h t) \right) + n/K_w \delta_h \kappa_h \exp (-\kappa_h t)} dt \]  

Equation 5.26

5.4.6. Fitting model parameters to hydration cell data

The key parameters included in these Equations and required to model the hydration cell test are \( K_{si} \), \( \lambda_{KS} \), \( \kappa_h \), and \( \delta_h \). Indication of appropriate values for \( K_{si} \) and \( \lambda_{KS} \) can be determined directly from the measured bender element data, where \( K_{si} \) is the first recorded bender element signal equivalent stiffness, as determined from Equation 3.6 in Chapter 3, and \( \lambda_{KS} \) is the ratio of the first to the final bender stiffness value recorded over the 7-day hydration cell test. The value of \( \kappa_h \) is then varied to determine the best fit between the experimental \( K_s(t) \) data and Equation 5.25 as shown in Figure 5.19 and indicated by a ‘x’ or a solid line respectively.

![Figure 5.19 Hydration model, Equation 5.25, matched to experimental bender stiffness, Ks](image)

The parameter values required to match the experimental hydration stiffness data for each test are summarised in Table 5.5. The subscript \textit{hyd} refers to the parameter’s value being determined from hydration cell test data.
Table 5.5 Hydration model parameters $K_{si}$, $\lambda_{Ks}$, $\kappa_h$ and $\varepsilon_{h}$ for hydration cell data

<table>
<thead>
<tr>
<th>Mix</th>
<th>$K_{si \text{ hyd}}$ [kPa]</th>
<th>$\lambda_{Ks}$</th>
<th>$\kappa_{h \text{ hyd}}$ [1/days]</th>
<th>$\varepsilon_{h \text{ hyd}}$ [-]</th>
</tr>
</thead>
<tbody>
<tr>
<td>KB</td>
<td>1260</td>
<td>370</td>
<td>0.3</td>
<td>0.0005</td>
</tr>
<tr>
<td>SNM</td>
<td>400</td>
<td>410</td>
<td>0.2</td>
<td>0.0005</td>
</tr>
<tr>
<td>SI P</td>
<td>1100</td>
<td>250</td>
<td>0.13</td>
<td>0.0018</td>
</tr>
</tbody>
</table>

In the KB and SI tests, a bender signal was determined early in the test ($t < 2$ hours) giving a good estimate for the $K_{si}$ parameter. However, the first recorded bender stiffness for the SNM material was at approximately $t = 6$ hours, by which time a significant stiffness had already developed; in this case a lower $K_{si}$ was estimated.

The SI $\lambda_{Ks}$ parameter obtained directly from the bender data was too low to allow good matching of Equation 5.25 to the data. The duration of the hydration test was only 7 days while the hydration process continued on beyond that time, meaning that in reality the final stiffness of the SI parameter must be greater than indicated at the completion of the hydration cell test. The SNM and KB 7-day stiffness appear to be similar to that of the final stiffness at the completion of hydration.

The value of $\varepsilon_{h \text{ hyd}}$ is then varied to match Equation 5.16 (shown as a dotted line) to the experimental effective stress developed (shown as ‘x’) as shown in Figure 5.20. The required values of $\varepsilon_{h \text{ hyd}}$ are also shown in Table 5.5.

Figure 5.20 Hydration model, Equation 5.16, matched to experimental effective stress generation, $p'$
Having determined these parameters, the chemical shrinkage induced strain occurring throughout a hydration cell can then be determined using Equation 5.16. This strain is plotted in Figure 5.18 for each of the hydration cell tests.

![Figure 5.21 Hydration cell chemical shrinkage development, $\varepsilon_{sh\ hyd}(t)$, from Equation 5.16](image)

### 5.4.7. Comparing ASTM and hydration cell test results

The initial aim of this work was to establish if the ASTM chemical shrinkage tests may provide a simpler way to analyse a given CPB mix without the requirement of a full hydration cell test. If the chemical shrinkage strain from the hydration cell and ASTM tests are compared, either through the values of $\varepsilon_h$, as shown in Table 5.6 or the respective plots of $\varepsilon_{sh}(t)$ in Figure 5.18 and Figure 5.21, a large difference in strains is evident. The strains determined from the ASTM data are an order of magnitude (or more) larger than those determined from the hydration cell test and there is no correlation in the strain magnitudes for each material tested.

Based on these strains, the equivalent volume changes that would occur in a 30m high by 10m$^2$ stope are shown in Table 5.6. Volume changes suggested by the ASTM test results appear unrealistically high, while those based on the hydration cell results appear reasonable.

#### Table 5.6 Comparison of ASTM and hydration cell strains and equivalent volume change

<table>
<thead>
<tr>
<th>Mix</th>
<th>$\varepsilon_{h\ hyd}$ [-]</th>
<th>$\varepsilon_{h\ ASTM}$ [-]</th>
<th>Vol. change based on $\varepsilon_{h\ ASTM}$ [m$^3$]</th>
<th>Vol. change based on $\varepsilon_{h\ hyd}$ [m$^3$]</th>
</tr>
</thead>
<tbody>
<tr>
<td>KB</td>
<td>0.00055</td>
<td>0.0388</td>
<td>116.4</td>
<td>1.65</td>
</tr>
<tr>
<td>SNM</td>
<td>0.0004</td>
<td>0.0177</td>
<td>53.1</td>
<td>1.2</td>
</tr>
<tr>
<td>SI P</td>
<td>0.0018</td>
<td>0.0129</td>
<td>38.7</td>
<td>5.4</td>
</tr>
</tbody>
</table>
If the assumption that the ASTM test results are potentially unrealistic is put aside for a moment, other potential reasons for a lack of agreement between the two data sets are considered, including the stiffness measurements.

5.4.8. Impact of stiffness on chemical shrinkage

To investigate the accuracy the stiffness measurements determined by the bender elements, the various CPB specimen stiffness that was utilised in each analysis were further considered.

5.4.8.1. Back calculated stiffness required for $\varepsilon_{sh\,ASTM} = \varepsilon_{sh\,hyd}$

The hydration data was back analysed to determine the stiffness required to produce the volumetric strains measured in the ASTM tests. This was achieved by dividing the initial stiffness $K_{si\,hyd}$ by the ratio of $\varepsilon_{h\,ASTM}$ to $\varepsilon_{h\,hyd}$. The required initial stiffness $K_{si\,ASTM}$ is shown in Table 5.7 for each CPB material and compared with $K_{si\,hyd}$.

Table 5.7 Required stiffness, $K_{si\,required}$ required to match $\varepsilon_{sh\,ASTM}$ and $\varepsilon_{sh\,hyd}$

<table>
<thead>
<tr>
<th>Mix</th>
<th>$K_{si,ASTM}$ [kPa]</th>
<th>$K_{si,hyd}$ [kPa]</th>
</tr>
</thead>
<tbody>
<tr>
<td>KB</td>
<td>16</td>
<td>1260</td>
</tr>
<tr>
<td>SNM</td>
<td>9</td>
<td>400</td>
</tr>
<tr>
<td>SI P</td>
<td>153</td>
<td>1100</td>
</tr>
</tbody>
</table>

The required stiffness to generate a strain similar to that determined from the ASTM test is considerably lower than the initial stiffness measured in the hydration cell, with the latter based on an assumption of Poisson's ratio ($\nu$) as per Equation 3.6 in Chapter 3. This choice of Poisson's ratio is discussed further.

5.4.8.2. Influence of Poisson's ratio on CPB specimen stiffness

A Poisson's ratio ($\nu$) of 0.125 has been used in this work as it represents a typical “small strain” undrained Poisson’s ratio from within the full range of undrained values of $0.1 < \nu < 0.15$ (Helinski et al., 2007a). Galaa et al. (2011) commented that assuming a constant $\nu$ for the full range of hydration may be problematic as a slurry initially has $\nu$ close to 0.5, decreasing to a value in the range of that assumed by Helinski et al. (2007a) when the material becomes self-supporting and hydration nears completion.

The effect of Poisson’s ratio on bulk stiffness determined from the hydration data was examined by varying $\nu$ and comparing with the original $G_0$, as used in $K_{s\,hyd} (t)$, and the back calculated required stiffness function $K_{s\,ASTM} (t)$, denoted $K_{s\,required}$. This process is shown in Figure 5.22, Figure 5.23 and Figure 5.24 for SI, SNM and KB mixes respectively. It is evident in these figures that even at $\nu = 0.1$ the stiffness is still considerably greater than that required to achieve
the ASTM chemical shrinkage; thus the choice of $v$ has little impact in explaining the differences in strains encountered.

Figure 5.22 Effect of Poisson’s ratio on SI bulk stiffness, $K_i$

Figure 5.23 Effect of Poisson’s ratio on SNM bulk stiffness, $K_i$
5.4.8.3. Strain gauge measured stiffness

Measurement of specimen stiffness during hydration was also attempted via the use of strain gauges that were introduced to the experimental set-up to determine the volumetric strain, as per Section 5.2.2. Figure 5.25 shows an average of strain gauge measurements for three KB CPB hydration cell tests with one S.D. error bars.

The initial application of cell pressure was registered by the strain gauges as a large axial contraction due to the initial slurry nature of the specimen and potential uneven distribution of pressure initially, as the cell is pressurised. The strain gauge data was zeroed from this point and Figure 5.25 shows only subsequent volumetric strain. Over the 12 hours of strain measurements, an expansion of the specimen in clearly evident contrary to hydration theory where volume is known to be lost from the system. Using these strains to determine specimen stiffness would return obviously incorrect values of negative stiffness.

Figure 5.24 Effect of Poisson’s ratio on KB bulk stiffness, $K_i$
Figure 5.25 KB CPB strain gauge measured volumetric strain, $\varepsilon_V$

The strain gauges are very sensitive to movement of the specimen, as exemplified by the need to zero the strain gauge readings after the initial cell pressure application, and it is possible that additional specimen movements beyond the zeroing of the gauges may explain the specimen's apparent expansion. Consolidation of the specimen prior to the zeroing of the strain gauges may sufficiently enhance the specimen stiffness such that the accuracy of the strain gauge reading is also improved, allowing a more accurate observation of early age volume change.

An applied back pressure $\sigma_b = 340\text{kPa}$ was applied to the specimen to consolidate to an effective stress of $60\text{kPa}$ from the initial applied stress $\sigma_c = 400\text{kPa}$. However, unusual behaviour was observed when attempting to consolidate the KB CPB material as shown in Figure 5.26. The inset shown in Figure 5.26 shows a schematic representation of the experimental set-up.
Figure 5.26 Pore pressure behaviour in KB CPB consolidation test

It would be expected that with the application of a back pressure, \( \sigma_b \), to the specimen that the pore pressure, \( u_t \), would dissipate from the initial specimen stress condition applied by the cell pressure \( \sigma_c \), to match \( \sigma_b \). However, clearly this does not occur as indicated by the rebounding of the pore pressure at \( t \approx 1 \) hour. Significant investigation of this anomalous pore pressure response to consolidation is presented in Chapter 7, in addition to a detailed explanation of consolidation and the particular boundary conditions applied in the consolidation of specimens within the hydration cell. However, for the purposes of improved strain gauge measurements, consolidation was unsuccessful and further use of strain gauges was abandoned.

5.4.9. Effect of permeability testing in hydration cell results

Examination of the effective stress generation occurring in the hydration cell (see Figure 5.9) indicates greater variability and a steady rate of increase in the SI effective stress compared with other specimens. This difference is attributed to the permeability testing conducted throughout the SI hydration cell test. The permeability tests, as described in Chapter 3, were carried out with a 10kPa pressure differential applied for \( \approx 25 \) minutes over the length of the specimen at approximately 24 hour intervals. The dotted vertical lines in Figure 5.27 indicate these test periods. The solid line represents the measured pore pressure data, with large jumps in pore pressure evident at the times at which permeability testing was conducted, due to the applied pressure head. The dotted line shows the pore pressure adjusted to remove these pressure jumps and shows the overall cumulative effective stress increase; this is the data presented in Figure 5.9.
Figure 5.27 SI hydration cell permeability test periods and measured and adjusted effective stress data

The permeability values measured are not of interest in this chapter and thus have not been reported. However, the difference between effective stress generation in the SI test, where permeability testing was carried out and the KB and SNM hydration cell tests, where hydration pore pressures were uninterrupted, may have significant implications for the hydration model developed in this chapter. The permeability testing appears to affect the self-desiccation pore pressure reduction by disturbing the hydraulic isolation of the specimen and altering the progression of hydration. This potentially affects the ability of the hydration model to accurately represent the pore pressures in this test.

The effect of these permeability tests on the final hydrated product is difficult to gauge and there does not appear to be any obvious effect in the comparison of chemical strain measured in the ASTM or hydration cell for SI compared with KB and SNM (see Table 5.6), despite the obvious difference in effective stress generation. This difference between the adjusted SI pore pressure data and that of the SNM and KB tests suggests that the additional water introduced in the permeability testing has lasting/greater effect on the hydration of the CPB specimen, potentially altering the process of hydration. It is not considered that the reduced effective stress generation experienced by KB and SNM is due to an insufficient supply of water for full cement hydration due to the large w/c values (Powers & Brownyard, 1947), but another process as yet undetermined, which may relate to the difficulty in achieving pore pressure consolidation (Figure 5.26). If this is the case, then after the first permeability test when additional water is first added to the system, any measured permeabilities will not accurately represent the true permeability of the material as cured in situ (i.e. as it would hydrate without the additional water introduction).
It should also be noted that the magnitude of the KB pore pressure dissipation in the hydration cell test presented in this chapter, indicated by the $p'$ development shown in Figure 5.9, is considerably less than that achieved in the KB hydration cell testing conducted by (Helinski, 2007). The stress conditions under which Helinski’s KB specimen was hydrated were different to those applied in this work. Helinski’s aim was to determine the extent of pore pressure dissipation of a hydrating specimen. To do this, the effective stress experienced by the specimen was kept to a minimum by manually increasing the pore pressure within the specimen and then superposing the pore pressure measured between each pressure increase to determine the cumulative dissipation. This was observed to reach 800kPa dissipation over a ~10 day period (Helinski, 2007). However, in the KB test presented here, the specimen remained hydraulically isolated (undrained) for the entire test, achieving 100kPa of pore pressure dissipation.

The discussion regarding the behaviour in the SI hydration cell test with permeability testing may help to explain why the KB hydration cell tests carried out by Helinski (2007) were able to achieve such large pore pressure dissipation due to the continual addition of water with each “top-up” of pore pressure. Variation in chemical composition of the hydrating paste is also introduced by the additional fluid flushed through the specimen, which as mentioned previously in Section 5.3.1.2, may significantly affect specimen behaviour.

Permeability testing in the hydration cell introduces a large unknown into the system and the validity of the results from tests with permeability testing are questionable, particularly when modelled with the hydration model presented in this chapter. This must be considered when examining the lack of agreement between $\varepsilon_{sh\,ASTM}$ and $\varepsilon_{sh\,hyd}$.

5.4.10. Potential issues with the ASTM test

Instances of variability shown in the ASTM chemical shrinkage test results were discussed previously in relation to Figure 5.7, where significantly different chemical shrinkage was observed for KB and KB6 CPB specimen hydration, despite both specimens comprising identical mix constituents and $w/c$.

The success of the ASTM test requires firm contact between the rubber stopper and the plastic vial (Figure 3.3) as any movement of the rubber stopper will affect the water level in the capillary tube and hence the chemical shrinkage readings. The ASTM standard (ASTM, 2009) specifies the use of glass vials (which are destroyed during sample extraction) to reduce the potential for movement of the rubber stopper once it is inserted. However, due to cost constraints it was not possible to use glass vials in this research and instead plastic vials were utilised.

A drop in the capillary tube water level was occasionally observed in the early stages of chemical shrinkage testing and attributed to movement of the rubber stopper due to faster reductions in the water level compared with those that would occur due to hydration alone. However, these tests were easily identified and the data discounted from the final results presented. Despite this, sufficient confidence was gained in the testing protocol adopted, due to
the agreement of a minimum of three individual tests for each mix type, as well as the good comparison between the CP tests and published literature (Figure 5.4).

Another slight variation to the ASTM test experimental protocol was also made, where instead of a constant temperature water bath maintained at 23°C ± 0.5°C, the specimen vials were maintained in a temperature-controlled room at 23°C ± 2°C, resulting in a slightly larger temperature fluctuation range. Chemical shrinkage readings were made at a similar time each day so as to minimise any influence of diurnal temperature fluctuations. To confirm that this measure was sufficient to minimise temperature-related variations in chemical shrinkage readings, a number of ASTM tests were repeated (not shown here) making use of a water bath and no significant difference was observed in the results.

While the potential for variability may exist in the ASTM testing procedure, neither of these modifications to the standard testing procedure are considered realistic causes for the discrepancy between the ASTM and hydration cell test-determined chemical shrinkage.

5.4.11. Discrepancy between ASTM and hydration cell chemical shrinkage values

Consideration of the relationship between chemical shrinkage and pore pressure reduction suggests that any variation in pore pressure must either be directly affected by the chemical shrinkage, or another, alternate affect also influencing the chemical shrinkage. Observations relating to variability in pore pressure with the addition of permeability testing (Section 5.4.9), as well as the unexpected pore pressure behaviour observed during consolidation (Figure 5.26), highlight potential behaviour which may relate to an as-yet-undetermined mechanism affecting pore pressure behaviour, presumably by altering hydration behaviour and thus chemical shrinkage. The susceptibility of chemical shrinkage measurements to chemical variations in the mixes tested has also been highlighted (see Section 5.4.3) suggesting that chemical shrinkage cannot solely be explained in physical terms and that the chemical process must be considered. These observations alone are far from an adequate explanation regarding the discrepancy between chemical shrinkage determined in the hydration cell or measured from the ASTM tests. However, it certainly highlights the complexity of the hydration process and how this may manifest in physical properties and behaviours.

In this chapter, the ASTM test has been identified as potentially variable in its results as well as instinctually unrealistic in terms of the chemical shrinkage volumes it suggests. Additionally, the observed influence of additional water supplied into the initially hydraulically isolated system in the hydration cell coupled with the inability to consolidate a CPB specimen in the hydration cell also raises questions on the adequacies of hydration cell testing approach for determining chemical shrinkage. These ideas will be addressed further in Chapter 7 where investigation of the pore pressure rebound seen in Figure 5.26 is carried out and discussed.
5.5. Conclusions

This chapter presents the use of the ASTM Standard Test Method C1608-07 for Chemical Shrinkage of Hydraulic Cement Pastes (ASTM, 2009) and hydration cell experiments to examine chemical shrinkage occurring in hydrating cemented paste backfill.

The CPB chemical shrinkage results confirm trends evident in the concrete literature, which states that higher \( w/c \) ratios correlate with higher normalised chemical shrinkage. The addition of the tailings ‘filler’ material in the CPB acts to increase the normalised chemical shrinkage when compared with cement-only pastes of identical \( w/c \) ratio. Hypersaline process water was also observed to have a significant impact on the occurrence of normalised chemical shrinkage. However, no definitive conclusion regarding the exact effect could be reached based on the testing carried out.

A model was presented to determine chemical shrinkage volumes occurring in a hydration cell test specimen based on pore pressure and stiffness measurements. Comparison of this model with the ASTM test results indicates no correlation and significantly lower strains. The strains suggested by the ASTM test are shown to be unrealistically large when applied to a stope application.

To examine the discrepancy in strain magnitude between the two tests, the stiffness required to achieve agreement between the results was determined and found to be significantly lower than that measured in the hydration cell. The assumption of a constant Poisson’s ratio was examined and over a large range of \( \nu \) proved to be negligible in its effect. Strain gauges were unsuccessfully used to measure the actual operating stiffness of a hydration specimen due to their initial slurry state and high sensitivity of the gauges to large movement. Attempts to consolidate the specimen to an initial effective stress level from which the strain gauges could be zeroed were unsuccessful. The anomalous consolidation behaviour observed during this consolidation process was identified as a potentially key factor in understanding the discrepancy between the volume change determined in the ASTM tests compared with the hydration cell test. This will be considered in detail in Chapter 7 which examines the effect of chemistry on consolidation behaviour.

Permeability testing was observed to affect the pore pressure dissipation in the hydration cell through the introduction of a large unknown into the system which cannot be accounted for in the current hydration model. The exact influence of the additional water introduced into the system in the permeability testing, or indeed the reason behind the unsuccessful consolidation attempt is not understood, but it potentially indicates the presence of a different process/mechanism affecting either pore pressure in isolation (i.e. additional volume change separate to the chemical shrinkage) or which modifies the hydration process itself.

The ASTM tests are generally accepted to be a direct measure of the volume change occurring due to the chemical processes as hydration occurs and are independent of the physical
conditions under which hydration occurs. However, the ASTM results presented in this chapter are shown to be inconsistent with the chemical shrinkage determined from hydration cell results. Based on the findings in this chapter, the ASTM chemical shrinkage test cannot be correlated to the hydration cell test and thus is not appropriate for use as an index test.

The issues raised in this chapter regarding unexpected pore pressure responses in the hydration cell tests are potentially implicated in this disparity between chemical shrinkage determination testing and are further investigated in Chapter 7.
CHAPTER 6. INCORPORATING CHEMICAL SHRINKAGE INTO GIBSON’S SOLUTION

6.1. Introduction

The dissipation of pore pressure that develops within a CPB-filled stope during deposition has a direct impact on the effective stress development, which in turn is linked to the stiffness and strength of the material (Helinski et al., 2007). These two properties are key considerations in the exposure stability of a backfilled stope and as well the load applied to barricades during the filling and curing period as discussed in Chapter 2. It is therefore important to develop an understanding of the factors that influence pore pressure during and after filling.

Gibson (1958) developed a one-dimensional solution to evaluate the pore pressures generated during the consolidation of accreting sedimentation. Fahey et al. (2010) applied Gibson’s solution to a mine backfilling scenario. One significant limitation of this application is its inability to account for chemical shrinkage. In addition to this significant limitation, the solution also does not consider the potential for (i) arching to occur within the stope (Fahey et al., 2009); (ii) lateral flow due to the 1D restriction; or (iii) variation in material properties occurring with CPB hydration, including variations in the coefficient of consolidation ($c_v$).

The effect of chemical shrinkage on the behaviour of CPB was examined in Chapter 5 along with a model considering the effect of self-desiccation induced by chemical shrinkage, on the pore pressures within the hydrating fill. The results of Chapter 5 indicate a significant effect of hydration on the pore pressure which for some fills i.e. non-consolidating, rival the reductions induced by conventional consolidation as modelled by Gibson’s solution.

This chapter provides a numerical solution in the form of a series of dimensionless charts that allow chemical shrinkage to be incorporated into Gibson's solution. While these charts may not provide a quantitative ability to predict pore pressure in a mine stope due to the factors listed previously, they do demonstrate a qualitative link between the groups of parameters that will affect pore pressures, such as the coefficient of consolidation, chemical volume shrinkage, rate of filling and hydration, shrinkage volume and stiffness. An example using an actual backfilling scenario of a KB stope is used to demonstrate the impact of hydration on pore pressures compared with self-weight consolidation alone.
6.2. Gibson’s Solution

Gibson’s solution was developed for a range of different boundary conditions, assuming a constant permeability $k$, a constant constrained modulus $E_0$ and therefore a constant coefficient of consolation $c_v$ where

$$c_v = \frac{E_0 k}{\gamma_w}$$

Equation 6.1

and $\gamma_w$ is the unit weight of the pore fluid.

Fahey et al. (2010) identified the drained base and constant filling rate scenario to be most relevant to the deposition of CPB in an excavated stope. A schematic of this scenario is provided in Figure 6.1.

**Figure 6.1 Boundary conditions of CPB deposition in a stope**

The height of the CPB fill ($H(t)$) is determined based on a constant filling rate ($m$) and the time since fill commencement ($t$) such that

$$H(t) = mt$$

Equation 6.2

The base of the stope and top fill surface are assumed to be fully drained such that pore pressure ($u$) is zero ($u = 0$) at $z = 0$ and $z = H(t)$, where $z$ is a vertical co-ordinate measuring the height above the base of the stope.
Using these boundary conditions, Gibson (1958) presented the following expression for excess pore pressure.

\[
\begin{align*}
  u(z,t) &= -\gamma z (1 + m z^2 cv) + \gamma m \frac{m z^2}{2cv} \exp\left(\frac{z^2}{4cvT}\right) \int_0^\infty \xi^2 \coth\frac{m \xi}{2cv} \sinh\frac{z \xi}{2cv} \exp\left(-\frac{\xi^2}{4cvT}\right) d\xi \\
  \text{Equation 6.3}
\end{align*}
\]

where \( \gamma \) is the unit weight of the backfill and \( \xi \) is an internal integration coefficient. The solution to this equation was presented in terms of a dimensionless time factor \( T \).

\[
T = \frac{m^2 t}{cv} = \frac{mH(t)}{cv}
\]

\text{Equation 6.4}

This solution can then be plotted in its dimensionless form, as shown in Figure 6.2, indicating the pore pressure distribution throughout the height of the fill at various dimensionless fill times.

![Figure 6.2 Isochrones of pore pressure from Gibson's solution](image)

Self-weight consolidation was also modelled numerically using the ABAQUS finite element software package (ABAQUS, 2011), by creating a one-dimensional model composed of separate layers; initially all layers were deactivated. A staged analysis was then conducted by activating layers from the bottom upward at a rate corresponding to a given fill rate \( m \), as indicated in Figure 6.3. Self-weight loading was applied to each layer, with zero pore pressure boundary conditions prescribed to the bottom of the model and to the top of active upper layer (i.e. \( u = 0 \) at \( z = 0 \) and \( z = H(t) \)). Results from the ABAQUS analysis are also shown in Figure 6.2, for a model filled in 20 layers and demonstrate an excellent match to Gibson's solution.
Chapter 6  
Incorporating Chemical Shrinkage into Gibson’s Solution

6.3. Chemical Shrinkage

In Chapter 5, a detailed explanation of chemical shrinkage was provided and an equation proposed for modelling the volume strain induced by the chemical shrinkage ($\varepsilon_{sh}$) with time. This equation is repeated here as Equation 6.5.

$$\varepsilon_{sh} (t') = \varepsilon_{sh f} (1 - \exp(-\kappa_h t'))$$  \hspace{1cm} \text{Equation 6.5}

where $\varepsilon_{sh f}$ is the chemical volume strain after full hydration, $\kappa_h$ is a parameter controlling the rate of hydration, with units of $t'^{-1}$, and $t'$ is the time since commencement of hydration. Note, the $t'$ here is the same as the $t$ used in Chapter 5. This equation is plotted in dimensionless form as shown in Figure 6.4.

Figure 6.3  ABAQUS staged analysis for modelling self-weight consolidation

However, consolidation is not the only process occurring in a CPB filled stope that affects the pore pressures. The hydration of the CPB is occurring in conjunction with the self-weight consolidation and inducing chemical shrinkage which also has a significant effect on pore pressures within the hydrating material (Helinski et al., 2007).

fill rate, $m$

fill rate, $m$

$u = 0$

$u = 0$

$u = 0$

$u = 0$

Layer 1

$H_1$

$H_2$

$H_i$

...
Figure 6.4 Normalised chemical shrinkage against dimensionless time

Using Equation 5.3 from Chapter 5 with the 1D stiffness parameter $E^*$, an incremental volume strain $\delta \varepsilon_{sh}(t')$ can be generated by a change in pore pressure $(\delta u(t'))$ given by

$$\delta u(t') = E^* \delta \varepsilon_{sh}(t')$$

Equation 6.6

where

$$E^* = \frac{1}{1/E_0 + n/K_w}$$

Equation 6.7

and where $n$ is the porosity of the material expressed as a function of time due to porosity reduction with cement gel growth, and $K_w$ is the bulk stiffness of the pore fluid, assumed to be water (2,000 MPa).

If the hydration of the backfill commences at the time it is deposited, then the hydration time ($t'$) at a particular location ($z$) can be related to the actual fill time ($t$) and the fill rate ($m$) through the following equation

$$t' = t - \frac{z}{m}$$

Equation 6.8

Doherty & Muir Wood (2013) showed for a one-dimensional problem with shrinkage strain as in Equation 6.5, that the pore pressure is governed by the dimensionless parameter

$$\kappa^* = \frac{\kappa_h H^2}{C_v}$$

Equation 6.9

where $H$ is the length of the drainage path. It can be shown, by substituting Equation 6.1 and Equation 6.4 into Equation 6.9, that
Therefore the fill time \( t \) and hydration rate \( \kappa_h \) provide a link between the term controlling the self-weight consolidation \( T \) and the term controlling the pore pressure due to chemical shrinkage \( \kappa^* \). The hydration profile with depth is also controlled by the actual fill time \( t \) and the rate of hydration \( \kappa_h \). For example, it can be shown that at \( z = 0 \) for \( \kappa_h t = 0.22, 0.51, 0.92, 1.62, 2.3 \) and \( 4.61 \), the material has achieved 20, 40, 60, 80, 90 or 99 percent hydration respectively; while for \( \kappa_h t = 5.76, 7.67, 11.51, 23.03, 46.1 \) and 92.1 the material achieves 99 percent hydration at \( z/H = 0.2, 0.4, 0.6, 0.8, 0.9 \) and 0.99, respectively. These hydration profiles are illustrated in Figure 6.5.

Figure 6.5 Isochrones of shrinkage with depth for various dimensionless fill times \( \kappa_h t \)

In the absence of an analytical solution for the hydration pore pressures, ABAQUS was employed to numerically model the pore pressure distribution in hydrating fill. Similarly to the self-weight analysis, the chemical shrinkage was examined in a staged analysis by simulating the filling of the stope in layers; however, the fill was assumed to be weightless to allow independent observation of shrinkage effects on the pore pressures.

The results of this analysis are presented in a series of charts, similar to Gibson's solution, allowing determination of pore pressure \( u \) at varying heights within the deposited fill, at different dimensionless fill times within the hydration process \( \kappa_h t \) for given values of \( m, c_v, E^* \) and \( \varepsilon_{shf} \). These charts, are presented in Figure 6.6 and Figure 6.7 for \( \kappa_h t = 0.22, 0.51, 0.92, 1.61, 2.30, 4.61, 5.76, 7.67, 11.51, 23.03, 46.1, 92.1 \).
Figure 6.6 Isochrones of normalised pore pressure due to chemical shrinkage at various dimensionless fill times
Figure 6.7 Isochrones of normalised pore pressure due to chemical shrinkage at various dimensionless fill times
The charts in Figure 6.6 and Figure 6.7 provide insight into the influence of fill time ($t$) and hydration rate ($\kappa_{ht}$) have on shrinkage-induced pore pressures. The shapes of the isochrones vary as a function of $\kappa_{ht}$ because hydration profiles also vary with $\kappa_{ht}$, as shown in Figure 6.5. For low values of $\kappa_{ht}$ (slow rate of hydration compared with fill time), the magnitude of shrinkage-induced pore pressure is relatively low. It can be seen that the shrinkage-induced pore pressure increases up to $\kappa_{ht} = 11.5$ and then reduces with a further increase in $\kappa_{ht}$. This is clearly shown in Figure 6.8, where the normalised pore pressure ($u/E^*\varepsilon_{shf}$) at $z/H=0.5$, for various $T$ values, is plotted against the normalised fill time ($\kappa_{ht}$).

![Figure 6.8 Isochrones of normalised pore pressure due to chemical shrinkage at various dimensionless fill times](image)

The optimum fill times required to achieve the maximum shrinkage-induced pore pressure at $z/H = 0.5$, range from $2.3 < \kappa_{ht} < 11.5$, beyond which the magnitude of shrinkage-induced pore pressure reduces. Note, $\kappa_{ht} = 5$ indicates total curing time as indicated in Figure 6.4. This suggests that the optimum fill time ranges between 0.9 of the total curing time (i.e. $t$ when $\varepsilon_{shf}(t)/\varepsilon_{shf} = 1$), which is often assumed to be 28 days, and twice the total curing time. This timeframe is significantly greater than typical backfilling times, such that filling times may not be able to take advantage of the peak negative pore pressures occurring due to chemical shrinkage.

It has previously been shown that $c_v$ controls not only the pore pressures in conventional self-weight consolidation (Gibson, 1958), but also the hydration process through the term $\kappa^*$ (Doherty & Muir Wood, 2013). This relationship between the two processes makes it possible to superpose the self-weight and hydration pore pressure profiles, combining them into a single curve allowing the determination of the pore pressure in a consolidating, hydrating CPB stope.
Chapter 6  Incorporating Chemical Shrinkage into Gibson’s Solution

The influence of the parameter \( T \) on the magnitude of the shrinkage pore pressures has also been established. When considering self-weight consolidation, large values of \( T \) produce positive large pore pressures as indicated in Figure 6.2. In the hydration process, for large values of \( T \), higher negative pore pressures are also achieved due to the progression of hydration and associated shrinkage with time. This is such that large \( T \) values illicit opposite pore pressures responses from each of the two mechanisms. This effect on the combined pore pressure response will be further examined in the following example application.

6.4. Example Application

To determine the pore pressures from both self-weight consolidation and chemical shrinkage, curves from Gibson’s chart can be superposed with an appropriate curve from either Figure 6.6 or Figure 6.7. To assess the superposition technique, pore pressure distributions determined separately for each mechanism (self-weight consolidation and chemical shrinkage) are superposed and then compared with a model that includes fully-coupled consolidation and shrinkage. An example of this process is provided here.

The parameters adopted for this application are derived from the real KB stope filling data and back analysis presented by Helinski et al. (2011b). However, in this chapter, both stiffness and permeability are assumed to remain constant throughout filling. A 10m fill scenario was considered with CPB deposition occurring at a rate of 0.3 m/hr (or 7.2 m/day) for material with a coefficient of consolidation at deposition of 6 m²/day, i.e. \( H = 10 \text{m}, m = 0.3 \text{m/day}, c_v = 6 \text{m}^2/\text{day} \) and \( t = 33.3 \text{hours} \) giving \( T = 12 \) (from Equation 6.4). The hydration process is characterised by \( \kappa_h = 8.3 \times 10^{-3} \text{hours}^{-1}, \varepsilon_{shf} = 0.0015 \) and \( E^* = 43,000 \text{ kPa} \) (Doherty, 2013), giving \( \kappa_{ht} = 0.25 \).

The individual pore pressures generated from independent ABAQUS analyse considering either self-weight consolidation or shrinkage in isolation are each presented in Figure 6.9. Using the principle of superposition, the combined pore pressure distribution is also shown and compares well with the results of a third analysis combining both shrinkage and self-weight. It can be seen that the sum of the shrinkage only and the self-weight only is identical to a model that incorporates both mechanisms. This clearly validates the superposition technique.
Figure 6.9 ABAQUS analysis of pore pressure due to self-weight consolidation (self-weight only), chemical shrinkage (shrinkage only), coupled self-weight and shrinkage (coupled) and superposition of self-weight and shrinkage (superposition) for $T = 12, \kappa_h t = 0.25$ and $E^*\varepsilon_{sh} t = 72.19$

The individual pore pressure charts presented in this chapter (Figure 6.4 and Figure 6.6 or Figure 6.7 for self-weight and shrinkage respectively) do not show isochrones for $T = 12$ and $\kappa_h t = 0.25$. However, to highlight the use of the charts presented in this chapter and their practically in the absence of a fully-coupled ABAQUS analysis, the upper and lower bound of pore pressure ranges are shown in Figure 6.10 by self-weight consolidation isochrones for $T = 8$ and $T = 16$ and shrinkage isochrones for $T = 8$ and $T = 32$ at $\kappa_h t = 0.22$. 
Chapter 6  Incorporating Chemical Shrinkage into Gibson’s Solution

Figure 6.10 ABAQUS analysis of pore pressure due to self-weight consolidation (self- weight), chemical shrinkage (shrinkage), coupled self-weight and shrinkage (coupled) and superposition of self-weight and shrinkage (superposition) for \( T = 12, \kappa_{\text{h}} t = 0.25 \) and \( E^*e_{\text{sh}} f = 72.19 \) with analytical analysis of “upper” and “lower” bound \( T = 8 \) and \( T = 16 \) for self-weight consolidation and \( T = 8 \) and \( T = 32 \) at \( \kappa_{\text{h}} t = 0.22 \) for shrinkage.

The self-weight and shrinkage lines (in black) generated from the individual ABAQUS analyses, together with the superposition and fully-coupled analysis curves are repeated from Figure 6.9. These curves are then compared with the upper and lower bounds represented by \( T = 8 \) and 16 for self-weight consolidation and \( T = 8 \) and 32 for chemical shrinkage, each obtained from Figure 6.4 and Figure 6.6 or Figure 6.7 respectively. A slight discrepancy exists between the numerical chemical shrinkage pore pressure and those from the analytical solution. This is due to the use of \( \kappa_{\text{h}} t = 0.22 \) in the analytical curves rather than the actual value of \( \kappa_{\text{h}} t = 0.25 \). Despite this difference the analytical curves still provide a good indication of the pore pressure response to chemical shrinkage.

While the charts generated in this chapter don’t necessarily provide a quantitative assessment of the pore pressures present within a stope during filling, they are able to provide an indicative, qualitative assessment of the pore pressure behaviour due to self-weight consolidation and chemical shrinkage, both independently and in combination.

6.5. Conclusion

This chapter presented a series of numerically generated dimensionless charts for the shrinkage induced pore pressures within a weightless material in a one-dimensional column increasing with thickness over time. Curves from these charts can be combined with curves
from Gibson’s consolidation solution to incorporate the effects of chemical shrinkage and self-weight consolidation on the pore pressure in accreting sedimentation. This was achieved through the identification of a link between the standard term controlling self-weight consolidation, \( T \), and the term controlling pore pressure response to hydration, \( \kappa^* \) and a suitable form of normalised shrinkage induced pore pressure, \( u/E^*\varepsilon_{sh. f} \).

The overall pore pressure response of a hydrating and consolidating fill increasing in thickness with time can then be determined using superposition to combine the pore pressure associated with self-weight consolidation and chemical shrinkage. This approach was successfully validated through an example comparing the superposed pore pressure with that determined from a fully-coupled ABAQUS analysis that included both chemical shrinkage and self-weight mechanisms.
7.1. Introduction

The experimental work presented in Chapter 5 (Figure 5.26) indicated an unusual pore pressure response to the applied consolidation pressure. Given the connection between conventional consolidation and self-desiccation (Helinski et al., 2007a; Fahey et al., 2010; Fahey et al., 2011), and the relationship between pore pressure reduction and chemical shrinkage, it was hypothesised that this anomalous pore pressure response may be related to the discrepancy between the measured chemical shrinkage in the ASTM test compared with that determined through the hydration cell test. As such, further investigation into the consolidation behaviour of CPB is warranted.

This chapter details a testing program that sought to identify the cause of the observed anomalous consolidation behaviour. The program involved consolidation testing on both uncemented tailings and CPB specimens to observe pore pressure behaviour under a range of different conditions to determine the cause of the behaviour, and also test how this unusual behaviour influences the self-desiccation pore pressure reductions and potential effects on the physical properties of CPB. The ultimate finding of the work presented in this chapter indicates potential reaction(s) between tailings solids and process water as the most likely cause for the anomalous pore pressure behaviour during consolidation.

7.1.1. Consolidation

Consolidation is the volume reduction that occurs due to drainage of a loaded, saturated soil. Upon loading a saturated soil volume, the total applied stress is initially carried by the stiff water phase. Gradually, as water flows from the specimen due to an applied pressure difference, the stress is transferred to the solid phase until pore pressure equilibrium is achieved. Thus the rate of volume reduction is dependent on both the soil’s compressibility and ability to conduct pore fluid. The diffusion equation governing one-dimensional consolidation as proposed by Terzaghi (1943) is given as

\[
\frac{\partial^2 u}{\partial z^2} = \frac{1}{c_v} \frac{\partial u}{\partial t}
\]

Equation 7.1
where \( c_v \) is the coefficient of consolidation with units of length\(^2\)/time, \( \bar{u} \) is the excess pore pressure, \( z \) is the axial distance along the soil volume and \( t \) is the time since commencement of consolidation.

To solve Equation 7.1 the following non-dimensional factors are determined:

- **Excess pore pressure** \( U \)
  
  \[
  U = \frac{\bar{u}_t}{\bar{u}_0}
  \]

  **Equation 7.2**

  where \( \bar{u}_0 \) is the initial average excess pore pressure throughout the specimen and \( \bar{u}_t \) is the average excess pore pressure after time \( t \).

- **Time factor** \( T_v \)
  
  \[
  T_v = \frac{c_v t}{d^2}
  \]

  **Equation 7.3**

  where \( d \) is the length of the drainage path.

- **Drainage path ratio** \( Z \)
  
  \[
  Z = \frac{z}{d}
  \]

  **Equation 7.4**

  where \( z \) represents any location along the specimen length \( d \).

Substituting these factors into Equation 7.1 (Whitlow, 2001) gives

\[
\frac{\delta^2 U}{\delta z^2} = \frac{\delta U}{\delta T_v}
\]

**Equation 7.5**

Consolidation testing of CPB was undertaken in the hydration cell experimental setup as detailed in Chapter 3, Section 3.4.2. The operation of this hydration cell was modified to allow consolidation to occur. A schematic of this experimental setup as modified from that detailed in Chapter 3 is shown in Figure 7.1.
Prior to the commencement of consolidation, the specimen was completely undrained and a confining pressure ($\sigma_c$) applied to the specimen throughout the cell. Due to the slurry nature of the specimen, this pressure was transferred into the specimen allowing pore pressures at both the top ($u_t$) and base ($u_b$) of the specimen (and indeed throughout the specimen) to equal $\sigma_c$, such that the initial boundary condition is $u_t\text{ initial} = u_b\text{ initial} = \sigma_c$. Consolidation commenced with the opening of the back pressure valve at the base of the specimen, exposing the base of the specimen to an external back pressure ($\sigma_b$) which is lower than $\sigma_c$. Drainage at the top of the specimen remained closed.

This pressure differential between $\sigma_b$ and $\sigma_c$ generates a hydraulic gradient along the drainage path ($d$) causing pore fluid to flow from the base of the specimen; this flow volume is denoted $V$ in Figure 7.1. Immediately at the commencement of consolidation $u_b = \sigma_b$ and $u_t = \sigma_c$, equating to an initial excess pore pressure $\tilde{u}_0 = u_t - u_b$. As the pore fluid drains from the specimen, the internal pore pressures reach an equilibrium, such that at the end of consolidation $u_t = u_b = \sigma_b$. These boundary conditions are summarised in Table 7.1.
Table 7.1 Consolidation problem boundary conditions

<table>
<thead>
<tr>
<th>Time Period</th>
<th>Boundary conditions</th>
</tr>
</thead>
<tbody>
<tr>
<td>Initially</td>
<td>( u_t = u_b = \sigma_c )</td>
</tr>
<tr>
<td>Start of consolidation</td>
<td>( u_t = \sigma_c; u_b = \sigma_b )</td>
</tr>
<tr>
<td>End of consolidation</td>
<td>( u_t = u_b = \sigma_b )</td>
</tr>
</tbody>
</table>

Using MATLAB (R2012a) to solve for Equation 7.5 with these boundary conditions, a series of isochrones are produced showing the excess pore pressure \( (U) \) along the normalised length of the specimen \( (Z) \) for various values of dimensionless time \( (T_v) \) in Figure 7.2. The excess pore pressure dissipation at a particular location can also be observed as shown in Figure 7.3.

![Figure 7.2 Isochrones of normalised excess pore pressure \( (U) \) with normalised height, \( (Z) \) for different dimensionless time factors \( (T_v) \)](image-url)
The $c_v$ value can be determined by matching the MATLAB model to the observed response.

### 7.1.2. Consolidation of CPB

Consolidation of CPB was first examined in this work using the KB CPB mix when experimenting with the use of strain gauges as discussed in Chapter 5, Section 5.2.2. The initial consolidation was carried out with $\sigma_c = u_t = 400\text{kPa}$, and $\sigma_b = 340\text{kPa}$ as indicated in Figure 7.4. The initial pore pressure behaved as expected, i.e. dissipating towards the applied backpressure $\sigma_b$. However, with no variation to the applied pressures or ambient temperature, $u_t$ suddenly increased, or “rebounded”, at $t = 1$ hour (where $t = 0$ is the start of consolidation), recovering 17kPa of the previously dissipated pore pressure. Subsequent $\sigma_c$ increases (at $t = 30$ hours and $t = 42$ hours) were applied to examine the response of $u_t$ which initially responded with a matching pressure increase, indicating working order of the transducers, no blockages in the system and full specimen saturation as indicted by $B = 1$ (Skempton, 1954) for both of these pressure increments. Similar initial consolidation-like $u_t$ reductions were observed after these $\sigma_c$ increases with a subsequent increase in $u_t$ at $t = 43$ hours, one hour after the cell pressure was increased to 500kPa.

![Figure 7.3 Excess pore pressure dissipation ($U$) with dimensionless time factor ($T_v$) at normalised height ($Z$) along the specimen](image)

The $c_v$ value can be determined by matching the MATLAB model to the observed response.
It was expected that the pore pressure would dissipate, reducing to match that of the applied back pressure, $\sigma_b$ shown in Figure 7.4 as the dotted line. Instead, the measured behaviour is shown by the solid line, $u_t$. The areas indicated within the circles are shown in closer detail in Figure 7.5 and Figure 7.6 for $t < 10$ hours and $t > 40$ hours respectively.
Chapter 7 The Influence of Chemistry on Consolidation of Cementing Paste Backfill and Uncemented Tailings

Figure 7.6 KB CPB consolidation profile: close up of $t > 40$ hours

It is necessary to clearly state that during this pore pressure ‘rebound’, the pore pressure gradient still existed within the specimen via the application of $\sigma_b$. However, despite this, the pore pressure measured at the top of the specimen ($u_t$) does not dissipate, seemingly violating the requirement for pressure equilibration within the system.

The remainder of this chapter investigates this anomalous behaviour. First, the consolidation behaviour of different mix types, including cemented and uncemented mixes of a range of different tailings materials were examined to establish repeatability of the observed behaviour. Then, different possible explanations for the observed behaviour were examined, focusing on the stress conditions applied to the specimen and their effect on consolidation, as well as the potential effect of physical and chemical changes in material properties.

7.2. Experimental testing program

The experimental testing program includes permeability, oedometer, standard hydration cell and UCS tests as described in the relevant sections of Chapter 3. The hydration cell was also used for consolidation testing as briefly described earlier in this chapter. Detailed information regarding each individual material, specimen preparation techniques and mix proportioning was also provided in Chapter 3. It should be noted that the bender elements were not utilised during consolidation testing.

The materials tested in the experimental work presented in this chapter include KB and RL CPB as well as uncemented specimens comprising SNM and RL tailings or silica sand with water. Each mix tested is summarised in Table 7.2.
### Table 7.2 Consolidation testing mix properties

<table>
<thead>
<tr>
<th>Material</th>
<th>Water</th>
<th>Binder</th>
<th>C&lt;sub&gt;c&lt;/sub&gt; [%]</th>
<th>C&lt;sub&gt;s&lt;/sub&gt; [%]</th>
<th>C&lt;sub&gt;sand&lt;/sub&gt; [%]</th>
<th>e&lt;sub&gt;o&lt;/sub&gt;</th>
</tr>
</thead>
<tbody>
<tr>
<td>KB CPB Process OPC</td>
<td>5</td>
<td>77.88</td>
<td>0</td>
<td>0.8</td>
<td></td>
<td></td>
</tr>
<tr>
<td>RL CPB Process Minecem</td>
<td>5</td>
<td>76.8</td>
<td>30</td>
<td>0.85</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Uncemented SNM Process</td>
<td>-</td>
<td>-</td>
<td>78</td>
<td>0</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Uncemented RL Process</td>
<td>-</td>
<td>-</td>
<td>74.8</td>
<td>0.85</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Silica sand Tap</td>
<td>-</td>
<td>-</td>
<td>86.7</td>
<td>100</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Uncemented KB Process</td>
<td>-</td>
<td>-</td>
<td>77.88</td>
<td>0</td>
<td>0.8</td>
<td></td>
</tr>
</tbody>
</table>

#### 7.3. Experimental results and discussion

This section outlines the experimental processes followed and results obtained while investigating the anomalous consolidation behaviour exhibited in Figure 7.4. First, the results of consolidation tests carried out on a range of different mix types (including cemented and uncemented) are presented, establishing repeatability of the pore pressure rebound. Detailed consideration is then given to the stress conditions imposed on an uncemented specimen during consolidation and the subsequent effects on the behaviour of the soil, including the potential for failure of the specimen, variation of particle size distribution throughout the specimen, changes in permeability and the influence of osmotic suction. The chemical composition of each mix is also considered and it is hypothesised that the observed behaviour is affected by chemical interactions between the solid and liquid phases of a mix, and it is this reaction(s) that generate the pore pressure rebound observed. CPB hydration cell tests were then conducted to investigate this hypothesised effect on the self-desiccation-induced pore pressure reductions. A summary of the results from the consolidating testing program, accompanied with a brief description of each test is presented in Table 7.7 at the conclusion of this section (Section 7.3).

#### 7.3.1. Consolidation testing

The pore pressure response throughout consolidation, carried out in the hydration cell, is presented as the normalised excess pore pressure $U$ (Equation 7.2) against actual time $t$, allowing examination of potential time-dependent behaviour. The $C_c$ values associated with the relevant uncemented specimen consolidation analyses are shown in Section 7.3.7, Table 7.5 of this chapter.

**7.3.1.1. Pore pressure response to consolidation**

The consolidation behaviour of KB and RL CPB as well as uncemented sand, SNM and RL mixes were examined and compared as shown in Figure 7.7. The applied stress conditions for consolidation were $\sigma_c = u_{i\text{ initial}} = 400\text{kPa}$ and $\sigma_b = 340\text{kPa}$ with $B = 1$, with the exception of the sand specimen where $\sigma_c = 400\text{kPa}$, $u_{i\text{ initial}} = 344\text{kPa}$ and $\sigma_b = 260\text{kPa}$ and $B = 0.85$. The full
consolidation of the sand specimen is shown by the immediate excess pore pressure dissipation from \( U = 1 \) to \( U = 0 \) indicating no problems with the equipment or techniques adopted.

**Figure 7.7 Excess pore pressure dissipation (\( U \)) versus time (\( t \)) for consolidation of KB CPB, RL CPB, sand, uncemented SNM and RL mixes**

Comparison of the consolidation responses in Figure 7.7 shows considerable variability between different materials, yet only the silica sand specimen undergoes full consolidation without subsequent pore pressure rebound. The KB CPB curve shows the normalised pore pressure response previously shown in Figure 7.4.

An obvious difference in the time at which consolidation commences in cemented and uncemented specimens is evident. The KB and RL CPB specimens begin dissipating excess pore pressure much quicker than the uncemented RL and SNM mixes, despite the application of \( \sigma_b \) at the same time, \( t = 0 \). This is likely due to the self-desiccation process commencing as the cement begins hydrating. Greater pore pressure rebounds are also achieved in the CPB specimens, compared with the uncemented mix.

The initial rate of excess pore pressure dissipation for uncemented RL is particularly slow, with 10 hours required to dissipate 10% \( U \), after which the rate increases, likely due to the increasing stiffness, dissipating an additional 40% \( U \) in a period of 2.5 hours. This is followed by a rebound in pore pressure, recovering half of the excess pore pressure previously dissipated. By comparison, the RL CPB specimen, with the addition of cement, displays a cyclical variation of pore pressure, eventually tending back toward \( U = 1 \). This extreme variation in \( U \), which achieves almost complete recovery of dissipated pore pressure, was only evident in the RL CPB specimen. This ‘cyclical’ variation of the RL CPB pore pressure dissipation compared with
Chapter 7  The Influence of Chemistry on Consolidation of Cementing Paste Backfill and Uncemented Tailings

the uncemented specimens is perhaps due to the self-desiccation mechanism acting to reduce the pore pressures, counteracting the “rebound mechanism”. This idea will be considered in further detail in Section 7.3.9.

The initial consolidation of the SNM specimen is similar to that of the uncemented RL, yet the SNM material experiences very rapid consolidation and almost immediate pore pressure rebound just prior to $t = 2.5$ hours.

The most obvious difference between all these tests is the chemistry of the materials involved. The silica sand mixed with tap water has a relatively simple chemical composition compared with the variety of tailings, process water and cement combinations of the other mixes. A significant difference in the PSD of the tailings and the silica sand also exists (Figure 3.1).

7.3.1.2. Volume change response

In addition to the pore pressure measurements, the volume change occurring in the RL tailings specimen during consolidation was determined through measurement of the expelled pore fluid, $V$ (see Figure 7.1). Figure 7.8 shows the normalised pore pressure (from Figure 7.7) plotted with total volumetric strain ($\varepsilon_V$) where the volumetric strain is calculated by

$$\varepsilon_V = \frac{dV}{V_T}$$

Equation 7.6

where $dV$ is the change in volume since the start of the test ($t = 0$) and $V_T$ is the total specimen volume at $t = 0$.

Figure 7.8  Excess pore pressure dissipation ($U$) and volumetric strain ($\varepsilon_V$) with time ($t$) for uncemented Raleigh specimen, RL3
Despite the pore pressure increase observed at $t = 13$ hours, pore fluid continues to drain from the specimen, approaching zero at the completion of the test (although not necessarily becoming zero). This significant reduction in flow indicates equilibrium between $\sigma_b$ and $u_t$, marking the end of consolidation despite the pore pressure gradient. However, minor flow may continue to drain from the specimen, during any period where a pressure gradient is applied. The rate of volume expulsion continues to decrease smoothly over the time period where the pore pressure rebound occurs.

7.3.2. Internal specimen stress conditions during consolidation

The pore pressure distribution within the specimen was assumed to match closely to that of the analytical solution presented in Figure 7.2. The pore pressure profile within the specimen was determined based on the measured value of $u_t$ at a single location, the top of the specimen, and the applied back pressure $\sigma_b$ at the base. However, given the anomalous pore pressure behaviour exhibited during consolidation, further investigation to confirm the distribution within a specimen was conducted in this section of the chapter. The pressure gradient generated in a specimen due to the variation in pore pressure is the driving factor in consolidation and thus understanding how this varies throughout the specimen is key to understanding the consolidation behaviour.

Typically two-way drainage is used where possible when consolidating as, due to the decreased drainage path length ($d/2$), compared with one-way consolidation, the time-to-full-consolidation is reduced by a factor of 4 as evident in Equation 7.3. Pore pressures are not typically measured within the two-way drained specimen (e.g. typical triaxial tests) and consolidation is assumed to be complete when fluid flow from the specimen is no longer observed (i.e. $\Delta V = 0$ in Figure 7.1). Consolidation via one-way drainage, however, allows observation of the pore pressure dissipation at one end of the specimen ($u_t$) and $\Delta V$ at the single drainage boundary (see Figure 7.1). It is possible that this observed rebound in pore pressure after consolidation commencement has not been observed previously due to the common application of two-way drainage. This will be discussed in further detail subsequently.

At the instant the bottom drainage valve is opened (i.e. $\sigma_b$ is applied to the specimen) an infinite hydraulic gradient is generated across the drainage boundary of the specimen as indicated by Equation 3.8 in Chapter 3. Throughout consolidation, the hydraulic gradient reduces as the pore pressure within the specimen dissipates, equilibrating (normally) with $\sigma_b$. Two-way drainage generates higher hydraulic gradients compared with one-way drainage due to the shorter drainage path lengths. The dissipation of excess pore pressure measured in RL3 at $u_t$ (i.e. $Z = 1$), shown in Figure 7.8, does not match that exhibited by the analytical solution in Figure 7.2. To further investigate this difference and understand the cause behind the pore pressure increase during consolidation, further testing was carried out to determine the pore pressure distribution profile at other locations ($1 < Z < 0$) within a specimen, to identify a
location, if any, where the pore pressure rebound does not occur and instead the pore pressure dissipates as per the analytical solution.

7.3.2.1. Internal pore pressure distribution

In addition to the measurements of $\sigma_c$, $\sigma_b$ and $u_t$, indicated in Figure 7.1 there is also provision for the measurement of pore pressures ($u_{mid}$) at different internal locations along the length of the specimen, as indicated by points B and C in Figure 7.9. This is achieved with a PPT connected to a small tube inserted into the specimen through the top cap. The end of the tube was covered with filter paper to prevent any solid material blocking the tube. The length of the tube was adjusted to measure pore pressure at varying locations within the specimen. The apparatus was limited to a maximum of three pore pressure transducers, measuring $u_t$ at point A, $u_b$ at point D and $u_{mid}$ at points B and C. To examine the pore pressure at these multiple locations within the specimen it was necessary to conduct more than one consolidation test. Two identical tests were carried out varying the location of the $u_{mid}$ transducer with $\sigma_c = u_{t\text{ initial}} = 400 \text{kPa}$ and $\sigma_b = 340 \text{kPa}$.

Figure 7.9 Locations of pore pressure measurements within hydration cell consolidation specimen at points A, B, C and D

The first test (RL13) measured pore pressure at points A, B and D and the second test (RL14) at points A, C and D. The degree of excess pore pressure, U, is plotted against time for...
specimen location A, B and C as shown in Figure 7.10. The pore pressure at D is the applied back pressure $\sigma_b$. The pore pressure measurements at point A for both tests are shown to allow comparison between the consolidation responses generated in each test.

Figure 7.10 Excess pore pressure distribution within specimen

Initially, when $t < 10$ hours, the rate of excess pore pressure dissipation is greater at points B (dotted line) and C (dashed line) compared with point A (solid lines), as consistent with the isochrones in Figure 7.3. At $t = \sim 10$ hours, the pore pressure at point A begins to rebound while consolidation continues at points B and C. The pore pressure at point A recovers approximately 10% of its initial 50% $U$ dissipation, equilibrating at $U = 60\%$ for the remainder of the test. The pore pressure response at points B and C are very similar until $t = 23$ hours where the pore pressure at point B rebounds, recovering more than 40% $U$ to match the pore pressure at point A. No pore pressure rebound was observed at point C where 90% consolidation (i.e. to 10% $U$) occurred and was maintained until the test was terminated.

Further information on the consolidation behaviour at other locations within the specimen may be inferred based on the presented responses. At a location between point A and B (i.e. $1 < Z < 0.33$) it is anticipated that a pore pressure response would plot between the responses at A and B, as per the consolidation solution represented by the isochrones in Figure 7.3. Any pore pressure rebound observed at such location would be anticipated to occur at a time between the A and B rebounds, proportional (not necessary linearly) to the distance between these two points.

Isochrones of excess pore pressure dissipation at $t = 0$, 10, 20 and 30 hours for the three different values of $Z$ (= 0.17, 0.33 and 1) are shown in Figure 7.11. Comparison of these isochrones with the theoretical curve, originally shown in Figure 7.2 and reproduced in Figure
7.11 as the dashed lines, clearly highlights the variation in excess pore pressure dissipation within the specimen. The T values for each of the theoretical isochrones shown do not compare directly with the experimental isochrones and merely indicated the typical variation of $U$ along the length $Z$ for variation in time; $T$ and $t$ relate through Equation 7.3.

No change in the experimental excess pore pressure is observed at the top of the specimen beyond $t = 10$ hours. The values of $U$ at $Z = 1$ are taken as an average of the two equivalent $Z = 1$ curves from Figure 7.10 and the slight variation observed is attributed to experimental scatter. Despite this lack of $U$ dissipation at $Z = 1$, the pore pressures at the other specimen locations showed a further dissipation for increased time (i.e. $t = 10, 20$ hours), as expected, indicated by the theoretical curves. Between $t = 20$ and $t = 30$ hours, the “rebound” mechanism occurs preventing any change in $U$ for $Z < 0.2$ and increasing the pore pressures within the specimen, such that the isochrone for $t = 20$ reverses direction.

![Figure 7.11 Experimental isochrones of excess pore pressure distribution within a RL specimen at $t = 0, 10, 20$ and $30$ hours compared with theoretical isochrones](image)

Between points B and C (i.e. $0.33 < Z < 0.17$) the existence of a “boundary” is postulated, separating the pore pressure rebound response observed at point B, with the smooth consolidation observed at point C. This is such that at a location on the point B side of this “boundary”, a pore pressure rebound might occur, increasing the pore pressure to match the response observed at points A and B. On the point C side of the “boundary”, a location might show pore pressures converging to the pore pressure behaviour at point C. If the specimen thickness is small enough, such that the entire specimen lies on the point C side of the boundary, then this rebound may be prevented from occurring. However, as previously noted, it is interesting to observe that even at point C the excess pore pressure is not fully dissipated (i.e.
for $Z = 0.17, \ U \neq 0$) and that over a period of 10 hours no change in $U$ occurs as indicated by the isochrones for $t = 20$ and $t = 30$ hours in Figure 7.11.

Having examined the pore pressure distribution within the specimen at three different locations (points A, B and C) attention was refocused on the effects of this stress distribution on consolidation behaviour and potential causes for the observed pore pressure increases.

7.3.2.2. Incremental effective stress increase

In an investigation of liquefaction-induced excess pore pressure generation, Okada et al. (2004) observed large changes in permeability before and after shear failure of a specimen in undrained ring shear tests. Failure was found to produce a concentration of fines surrounding the shear plane either due to fines migration from other areas of the specimen, or due to grain crushing induced by shearing. This concentrated presence of fines was found to reduce the permeability of the specimen and, along with shear failure, facilitate an excess pore pressure increase by reducing the rate of pore pressure dissipation away from the shear zone.

The idea of shear failure within the consolidation testing carried out here is not relevant as no shear stresses are applied to the specimen to induce such a failure. However, the influence of the stress gradient developed along the length of the specimen due to the one-way drainage warrants closer examination.

Figure 7.12 shows a theoretical effective stress profile generated at the commencement of consolidation. Initially a constant level of zero effective stress ($\sigma'$) is present throughout the entire specimen. When $\sigma_b$ is applied at the base of the specimen, the pore pressure at this boundary ($u_b$) reduces to match this value generating effective stress such that $\sigma' = \sigma_c - u_b$; while at the top of the specimen the effective stress is still zero, as $\sigma_c = u_t$. 
Figure 7.12 The theoretical effective stress profile (from isochrones in Figure 7.2) within a specimen consolidating in the hydration cell

Obviously this theoretical effective stress profile is different to what was actually observed in the specimen, as indicated by the reducing pore pressures in Figure 7.10, that relate to effective stress via Equation 3.3 or Equation 3.5. In the previous consolidation testing, shown in Figure 7.7, Figure 7.8, and Figure 7.10, initial stress conditions were applied to produce $\sigma' = 60\text{kPa}$ at the base of the specimen. The greater the effective stress level, the higher the hydraulic gradient and thus the faster the pore fluid flow through the specimen as per Equation 3.7, Equation 3.8 and Equation 3.9 in Chapter 3.

To examine the effect of a reduced hydraulic gradient on the consolidation pore pressure behaviour, initial stress conditions $\sigma_c = u_{i\text{ initial}} = 410\text{kPa}$ and $\sigma_b = 390\text{kPa}$ were applied to a RL specimen (RL4). The higher $\sigma_b$ value reduces the initial $\sigma'$ at the specimen base, allowing the effect of a reduced hydraulic gradient on the pore pressure rebound to be observed. The value of $\sigma_b$ was then reduced in 10kPa increments as indicated in Figure 7.13 and the pore pressure consolidation response at point A (Figure 7.9) observed.
The first stress stage showed no pore pressure rebound during the 22 hour test period, so after $t = 22$ hours and 40% $U$ dissipation, $\sigma_b$ was reduced to 380kPa. The change in stress conditions increased the rate of volume strain and the degree of excess pore pressure dissipation increased (i.e. from 60% $U$ to 70% $U$), yet the rate at which that latter occurred was unchanged. After two hours of further consolidation with $\sigma_b = 380kPa$, the pore pressure began to rise, eventually recovering all the previously dissipated pore pressure, indicating that the gradual increase in hydraulic gradient does not prevent the rebound. Further reduction of $\sigma_b$ to 370kPa again increased the rate of $\varepsilon_V$, but $U$ remained unaffected.

7.3.3. Permeability of consolidating specimens

The excess pore pressure increases observed by Okada et al. (2004) were accompanied by changes in specimen permeability of 3 to 4 orders of magnitude. Further consolidation tests were therefore carried out, coupled with permeability testing, to observe if the RL pore pressure rebound also coincided with a change in permeability.

Permeability was examined prior to the commencement of consolidation and again at the completion of consolidation, after the pore pressure rebound was observed. Permeability was determined as per Equation 3.7 in Chapter 3, with a 20kPa differential pressure applied across the specimen (i.e. $\sigma_b - u_i = 20$). Initially 10kPa was applied, however, this was insufficient to produce measurable flow through the specimen. An RL specimen (RL6) was consolidated with...
\[ \sigma_c = \sigma_{\text{initial}} = 400\text{kPa} \text{ and } \sigma_0 = 340\text{kPa} \text{ with the resulting response shown in Figure 7.14. An additional RL tailings specimen (RL7) was consolidated under the same conditions but the test was allowed to continue over a longer duration, beyond the pore pressure rebound to examine the effect of pore pressure recovery on the permeability. The data from RL7 is also shown in Figure 7.14.}

\[ k_1 \text{ at } t = 0 \text{ hours} \]
\[ k_2 \text{ at } t = 15.3 \text{ hours for RL6 and } t = 42.7 \text{ hours for RL7, as indicated in Figure 7.14. These permeabilities are presented in Table 7.3 with a clear order of magnitude difference between initial and final permeabilities.}

<table>
<thead>
<tr>
<th>Test</th>
<th>( k_1 ) [m/s]</th>
<th>( k_2 ) [m/s]</th>
</tr>
</thead>
<tbody>
<tr>
<td>RL6</td>
<td>( 1.3 \times 10^{-9} )</td>
<td>( 1.76 \times 10^{-10} )</td>
</tr>
<tr>
<td>RL7</td>
<td>( 2.3 \times 10^{-9} )</td>
<td>(&lt; k_2 \text{ RL6})</td>
</tr>
</tbody>
</table>

The value of \( k_2 \) for RL7 could not be determined due to inadequate generation of flow under 20kPa applied differential pressure. This indicates a significant reduction in permeability due to
the additional pore pressure recovery achieved, indicating a final permeability lower than the $k_2$ measured in RL6.

This large reduction in final permeability is of particular interest considering that this pore pressure rebound has seemingly been unobserved previously. In a triaxial test for example, where two-way drainage is typically employed, little time is wasted between the completion of consolidation (as indicated by volume flow from the specimen), closing the back pressure valves and the commencement of shearing. This would provide insufficient time, due to the low permeability, for pore pressure to equilibrate through the specimen and be identified by pore pressure measurements if consolidation is incomplete or pore pressure has rebounded.

### 7.3.3.1. Fines migration within a consolidating specimen

The pore fluid flow induced by consolidation has the potential to cause a migration of fines within the specimen. While a reduction in flow due to a reduced effective stress gradient was not found to prevent a pore pressure rebound (Figure 7.13), it is possible that the reduced flow may have still have been sufficient to facilitate the migration of fines. If significant fines migration were to occur, a difference in fines content should be clearly evident at the extremities of the specimen, whereby fines may migrate from the top of the specimen carried by the induced pore fluid flow, to the base of the specimen, accumulating near the drainage boundary.

To examine this potential, a 3 - 4mm slice was removed from the top and base of both the RL6 and RL7 specimens and the fines (< 75μm) percentage in each slice compared. The material in each slice passing through a 75μm sieve was analysed using the laser particle sizing technique. The final PSDs are shown in Figure 7.15 and Figure 7.16 for specimens RL6 and RL7 respectively. The total slice volume and corresponding percentage fines is provided in Table 7.4.

![Figure 7.15 < 75μm PSD for top and base slices (extremities) of specimen RL6](image)
Despite the differences between $k_1$ and $k_2$ for both RL6 and RL7 specimens, there is no significant difference between the fines PSD at the specimen extremities. In RL7, the top of the specimen actually shows finer material than at the base; however the difference is not significant. It is possible that any variation in fines is more isolated, yet visual inspection of the specimens revealed no obvious differences.

### 7.3.4. Consolidation testing using an oedometer apparatus

Oedometer testing was conducted, as per Chapter 3, Section 3.4.4, to investigate the potential for creep or volume change due to expansive soils in the RL tailings material; either of these behaviours may affect consolidation behaviour.

Loading increments of 10, 20, 60, 100, 200, 500, and 1000kPa were applied to a RL specimen with the loading sequence halted for 48 hours at 60kPa to observe creep or volume expansion. The specimen was then unloaded to 200kPa and then 10kPa. The compression and swelling curve for this test is shown in Figure 7.17 with the variation of specimen void ratio ($\varepsilon$) throughout the halted period shown in Figure 7.18.
Chapter 7 The Influence of Chemistry on Consolidation of Cementing Paste Backfill and Uncemented Tailings

Figure 7.17 Void ratio ($e$) with vertical effective stress variation ($\sigma'_v$ or $p'$) on an uncemented RL specimen using an oedometer cell

The 60kPa load was maintained over the 48-hour period as this stress represents the same consolidation stress applied to the hydration cell specimen where the pore pressure rebound was observed. During this time period the vertical displacement of the specimen was monitored and indicated negligible volume change, either expansive or contractive, after the initial volume reduction due to the applied load. It should be noted that there was evidence of very slight diurnal effects.

Figure 7.18 Void ratio ($e$) variation at 60kPa virgin effective stress in oedometer consolidation of an uncemented RL specimen

The oedometer apparatus does not allow for pore pressure measurements, so no comment can be made in this regard. The primary conclusion is therefore that neither creep nor expansion of the specimen is responsible for the pore pressure ‘rebound’ during consolidation.
7.3.5. Effects of osmotic suction on consolidation

Consolidation is also known to be affected by osmotic pressures inherent in soils with concentrated solutes contained in the pore fluids. Osmotic flow occurs as a solvent passes from a lower to higher concentrated solution and if flow is restricted then a pressure difference will be generated, equal to the osmotic pressure difference between the two solutions. The osmotic pressure is related to the salt content (proportional to TDS) of the pore fluid in a soil and will vary due to alteration in TDS concentration. This can have an effect on the mechanical properties of the soil, including volume change and strength (Fredlund et al., 2012, Barbour & Fredlund, 1989).

The osmotic pressure ($\pi$ in kPa) can be approximated by the van’t Hoff equation:

$$\pi = R T_k C$$  \hspace{1cm} \text{Equation 7.7}

where $R$ is the molar gas constant (8.314 J/mol K), $T_k$ is the temperature of the fluid (K), and $C$ is the sum of the molar concentrations in solution (mol/L) (Fredlund et al., 2012). The concentration is determined from the TDS reading and the molecular weight of the solute.

In a clay soil, osmotic pressure may induce a volume change known as osmotic consolidation due to the nature and structure of the clay minerals, such that the presence of salt can change the inter-particle repulsive stresses which act to suppress the double layer, thus reducing the void ratio (Barbour & Fredlund, 1989). Due to the low clay mineral content in the materials used in this research (see Table 3.3 in Chapter 3), osmotic consolidation is not considered to be likely, yet there is potential for osmotically induced consolidation.

Osmotically induced consolidation occurs due to the osmotic flow of pore fluid out of a soil in response to an osmotic pressure difference (Barbour & Fredlund, 1989). This ultimately achieves an increase in effective stress, similar to conventional consolidation. Due to the high TDS process water present in the material pores, and the low TSD tap water used in the pressure lines and transducers of the experimental equipment, there is significant potential for osmotically induced consolidation to occur in the experimental testing carried out in this chapter.

At the drainage boundary of the specimen, the process water pore fluid interfaces with the fresh water in the pressure lines, potentially producing an osmotic pressure gradient with the potential to generate osmotic flow between the different water types. Using Equation 7.7 to determine the osmotic pressure associated with the Raleigh process water and the tap water (see Table 3.6 in Chapter 3 for the respective TDS readings) values of 5,500kPa and 18kPa respectively were calculated, indicating a maximum theoretical difference in osmotic pressures of 5MPa or more. These values only provide an indication of the osmotic pressures, as the assumption of sodium chloride (NaCl) as the only solute in each water type is unrealistic. The water chemistry testing carried out and presented in Chapter 3 can be used to determine the exact solution composition. However, for the purpose of this calculation and discussion a solution of NaCl was assumed.
This 5MPa pressure difference would generate additional pore fluid flow out of the specimen, further enhancing pore pressure reduction in addition to conventional consolidation. Clearly the potential for osmotic pressure to be involved with the pore-pressure rebound mechanism is unlikely. Regardless, osmotic effects on the consolidation behaviour were examined by repeating a RL consolidation test (RL11) with RL process water filling the equipment pressure lines and transducers, in addition to the specimen’s pores. The stress conditions were the same as for previous consolidation tests: $\sigma_c = u_t = 400kPa$ and $\sigma_b = 340kPa$. The consolidation response of the RL11 specimen is shown in Figure 7.19 and shows a very smooth curve achieving full consolidation until a pore pressure rebound occurs at about 18 hours after consolidation commenced. This rebound completely recovered all previously dissipated pore pressure.

![Figure 7.19](image)

**Figure 7.19** Consolidation pore pressure profile comparing effects of water quality on consolidation - uncemented Raleigh specimens RL11 and RL12 mixed with process and tap water respectively.

This behaviour is compared with another RL consolidation test (RL12) with identical stress conditions but employing tap water (rather than process) in the specimen mix and pressure lines. The consolidation response of RL12 is also shown in Figure 7.19 and is not as smooth compared with RL11, yet still achieves full consolidation before the pore pressure rebound which also fully recovers the previously dissipated $U$. These were the first tests where full consolidation prior to the pore pressure rebound was observed. The pore pressure response post-rebound is also very smooth compared with previous tests. If osmotically induced consolidation was in fact happening previously due to the two different types of water used within the system, then this would act to enhance consolidation. It is interesting that complete
pore pressure reduction occurs in a system where the potential for osmotic pressure effects to occur were removed, illustrating that there was indeed some value in completing this check.

There is also a difference in the time at which the pore pressure rebound occurs in each test; the process water-only (RL11) test experiences the pore pressure rebound 5 hours after the tap water-only (RL12) test rebound. These rebounds also coincide with a slight increase in $\varepsilon$, that has not been observed in any of the previous tests where $\Delta V$ was measured. The only difference between these tests is the difference in water chemistry, where RL11, which uses high TDS process water, will have a greater salt content than the RL12 tests which used tap water.

The permeability at the completion of RL11 was also determined and found to be $1.1 \times 10^{-10}$ m/s for an applied differential pressure of 70 kPa. Initially, differential pressures of 20 kPa, 30 kPa, 40 kPa and 50 kPa were applied to the specimen, but proved insufficient to generate any flow. This measured permeability is similar to that observed for RL6 (Table 7.3), but was measured from flow induced by a considerably larger pressure differential. No permeability was measured for the RL12 test.

These tests indicate that osmotic pressure is not responsible for the pore pressure rebound, as despite removing potential osmotic pressure gradients, the pore pressure rebound still occurs. However, removing the osmotic gradients by using only one water type within the experimental system clearly had an effect on the consolidation behaviour as indicated by the following:

- Differences between RL11 and RL12;
- Complete and smooth excess pore pressure dissipations for the first time in this testing program; and
- Significantly lower permeability of RL11 compared to previous permeabilities (i.e. RL6) measured in tests employing different water types.

Despite these observations, it is not possible to definitively determine if the difference is related to osmotic pressure differences or instead subtle variations in chemistry due to the difference between the process and tap water.

7.3.6. Effects of physical constraint and material “reuse” on consolidation behaviour

The physical constraint applied by the mould in the hydration cell set up (see Figure 3.2 in Chapter 3) was identified as another possible influence on the observed consolidation behaviour. If the mould were to limit the application of cell pressure to the specimen, this would have the potential to generate non-homogeneous stress conditions within the specimen. Re-equilibration of stresses as consolidation occurs may then “fail” the structure of the specimen, potentially generating excess pore pressure in a similar manner to that observed by Okada et al. (2004). To examine this theory, another uncemented RL specimen (RL8) was consolidated without the mould. The same stress conditions were applied as for previous consolidation tests: $\sigma_c = u_l = 400$ kPa and $\sigma_b = 340$ kPa.
At this point in the experimental testing program there was concern regarding the amount of RL tailings material remaining for subsequent testing, so the solid material (tailings and sand) from a previously consolidated specimen (RL5) was reused. The RL5 test displayed the usual anomalous pore pressure rebound. However, due to an incorrect calibration of one of the pore pressure transducers the specimen was liquefied, causing significantly lower density and higher void ratio of the final specimen compared with other RL consolidation tests. This prevented direct comparison of RL5 with the other tests. The plots of $u_t$, $\sigma_c$, $\sigma_b$ and $\varepsilon_v$ throughout the RL5 test are shown in Figure 7.20. The pore pressure has not been normalised to $U$ and instead is shown as the direct pore pressure reading, similar to Figure 7.4.

Figure 7.20 Liquefied specimen RL5 consolidation profile

Permeability testing using a 10kPa pressure differential was carried out at the beginning and end of the RL5 consolidation test. The initial specimen permeability $k_1$ was measured at $4.74 \times 10^{-9}$ m/s, during which 8.8 cm$^3$ was flushed through the specimen. The final specimen permeability $k_2$ was measured at $3.96 \times 10^{-9}$ m/s, flushing 6.7 cm$^3$ of water through the specimen. Considering the pore volume of the consolidated specimen was calculated to be 188.54 cm$^3$, the volume of water that passed through the specimen during each of the permeability tests represent only ~3% of the total pore volume.

After oven drying, the solid material from the RL5 specimen was reconstituted with the appropriate volume of process water (as dictated by the mix requirements in Table 5.1) and consolidated without the support of a mould in the consolidation test RL8. Observing the resulting consolidation response in Figure 7.21, full dissipation of excess pore pressures was achieved with no subsequent pore pressure increase, i.e. behaviour was similar to the silica sand test, which is the usual expected response. To examine reproducibility of this desired
consolidation result, an additional consolidation test (RL9) was conducted. “New” RL tailings material (i.e. not reconstituted solids from a previous consolidation test) was used in RL9 that was also conducted without the support of the mould. The resulting consolidation response is also shown in Figure 7.21, where a pore pressure rebound is evident after the initial partial consolidation.

Comparison of these two results established that the full consolidation observed in RL8 was related to the reuse of solids and not the use of the mould. Whatever effect is generating the pore pressure increase was not present in the reused solids test (RL8), despite a pore pressure increase during the first consolidation event of the material (RL5) and subsequent mould-less consolidation (RL9).

Figure 7.21 Effect of specimen support mould and solids reuse on consolidation profile in tests RL8 and RL9
Chapter 7 The Influence of Chemistry on Consolidation of Cementing Paste Backfill and Uncemented Tailings

- 146 -

Figure 7.22 close up of Figure 7.21: 0 < t < 18 hours

At the end of consolidation, specimens were susceptible to deformation due to their soft state, despite consolidation. However, after oven drying, the material was very stiff and could no longer be deformed by hand; indeed, breaking of the specimen with any implement became quite difficult. Added water was readily absorbed into the specimen producing a deformable specimen, suggesting that either suction or water-soluble cementation/precipitate effect is a large contributor to the post-drying specimen strength.

Precipitation during consolidation offers a potential explanation for the pore pressure rebound. The complex chemical composition of both the various tailings material and process waters (see Chapter 3 Sections 3.2.1 and 3.2.2) provides potential for chemical reactions to occur between the mix constituents. The previous consolidation (RL5) of the solid material used in RL8 may have allowed potential chemical reactions to reach completion and when the material was re-used, the precipitate products were dissolved by the added water and insufficient reagents were available for further reactions, resulting in subsequent “normal” pore pressure dissipation during consolidation. In RL9, however, the material was interacting for the first time (as in RL5) and thus the assumed chemical reaction resulted in a pore pressure rebound.

7.3.7. Values of $c_v$ used in consolidation analyses

The $c_v$ values resulting from the consolidation analyses of uncemented Raleigh specimens RL13 and RL14 (Figure 7.10), RL6 and RL7 (Figure 7.14), RL11 and RL12 (Figure 7.19), and RL8 and RL9 (Figure 5.21) are presented in Table 7.5.
Table 7.5 \( c_v \) values from consolidation analyses of uncemented Raleigh specimens

<table>
<thead>
<tr>
<th>Specimen</th>
<th>( c_v ) [( \text{m}^2/\text{yr} )]</th>
</tr>
</thead>
<tbody>
<tr>
<td>RL13</td>
<td>13</td>
</tr>
<tr>
<td>RL14</td>
<td>9</td>
</tr>
<tr>
<td>RL6</td>
<td>20</td>
</tr>
<tr>
<td>RL7</td>
<td>14</td>
</tr>
<tr>
<td>RL11</td>
<td>13</td>
</tr>
<tr>
<td>RL12</td>
<td>13</td>
</tr>
<tr>
<td>RL8</td>
<td>8</td>
</tr>
<tr>
<td>RL9</td>
<td>17</td>
</tr>
</tbody>
</table>

7.3.8. Potential temperature increase

Heat generation from any chemical reaction may help to identify the chemical process(es) occurring, or indeed the generation of any heat itself may be sufficient to induce a pore pressure increase through thermal expansion of the solid phase. To check for heat generation, an RL tailings specimen was placed in an insulated box with remote temperature sensors used to monitor the internal specimen temperature over a 68-hour period. Three temperature sensors were used; located in the top and bottom halves of the specimen and inside the lid of the insulated box. The temperatures recorded by these sensors are shown in Figure 7.23 and indicate only diurnal changes in temperature occurring. This is not to imply the absence of any chemical reactions, only that any such reaction does not generate significant heat.

Figure 7.23 RL specimen temperature generation
7.3.9. Implications of chemical reactions on self-desiccation-induced effective stress development

Given the roles and connections between consolidation and self-desiccation in the development of strength in CPB (Helinski et al., 2007a; Fahey et al., 2010; Fahey et al., 2011), there is a great potential for these observed pore pressure rebounds to affect the pore pressure reductions induced by the self-desiccation mechanism in hydrating CPB. The hydration cell, in its original function as described in Chapter 3, Section 3.4.2, was used to cure three RL CPB specimens each prepared in a different manner, to examine differences in the 7-day mechanical properties, for consideration of the early age, cementing behaviour.

A series of RL hydration cell tests were conducted on RL CPB (with a cement content of 5%), where the solids (minus cement) of each mix were “washed” to remove/dilute the chemical reagents prior to consolidation and the effect on self-desiccation pore pressure reduction observed. The specimen preparation techniques followed for each test are outlined in the flow charts presented in Figure 7.24.

**Table 7.6 Electrical conductivity of decant water from Step#3 (Figure 7.24) in test RL16 and RL17**

<table>
<thead>
<tr>
<th>Water</th>
<th>EC [mS/cm]</th>
<th>pH</th>
</tr>
</thead>
<tbody>
<tr>
<td>RL Solids + DI (RL16)</td>
<td>746</td>
<td>6.14</td>
</tr>
<tr>
<td>Uncem. RL Mix + DI (RL17)</td>
<td>1,516</td>
<td>5.93</td>
</tr>
<tr>
<td>RL Process Water</td>
<td>17,500</td>
<td>7.4</td>
</tr>
</tbody>
</table>
Chapter 7 The Influence of Chemistry on Consolidation of Cementing Paste Backfill and Uncemented Tailings

The standard procedure for specimen preparation and subsequent hydration cell testing (see Chapter 3) was followed for the RL15 hydration cell test (indicated in Figure 7.25 as curve “15”). The pore pressure reductions due to self-desiccation that occurred in this test occur at a relatively steady, smooth rate. The mix proportions prepared for this test was identical to that used for the RL CPB test shown in Figure 7.7. In the test shown in Figure 7.7, both consolidation and hydration are occurring simultaneously and given this, it would be expected that at the completion of consolidation (i.e. when \( U = 0 \)) pore pressures would continue to reduce due to the self-desiccation. This is obviously not the case as \( U = 0 \) is never achieved and the cyclical pore pressure behaviour appears to result due to competition between the rebound mechanism to restore the initial excess pore pressure, and the self-desiccation mechanism acting to reduce pore pressures as occurs in Figure 7.25.

It then follows that formation of a chemical precipitate(s) could limit the pore pressure reduction produced due to self-desiccation, and thus limit the development of strength and stiffness of the hydrating material. The re-use of solids in RL8 produced a typical consolidation pore pressure response. Whether this occurred specifically due to the “flushing” during the permeability tests in RL5, or an exhaustion of a chemical reaction produced by the initial combination mix ingredient chemistry, evaporation during oven drying or by some other means, the pore pressure rebound mechanism was no longer present.

The results of the two hydration cell tests, RL16 and RL17, are compared with the initial RL15 hydration cell test in Figure 7.25, Figure 7.26 and Figure 7.27 for pore pressure reduction (\( u_t \)) - related to effective stress generation via Equation 3.3 (or Equation 3.5), small strain stiffness development (\( G_s \)) and stress-strain UCS curve respectively. Chapter 3 outlines the experimental determination of these parameters.

![Figure 7.25 RL CPB pore pressure reduction](image)

*Figure 7.25 RL CPB pore pressure reduction*
It was anticipated that if the (assumed) chemical mechanism behind the consolidation pore pressure rebound could be removed, then larger pore pressure reductions during hydration might be observed, such that pore pressure reduction, strength and stiffness would all increase with reducing EC measurements (Table 7.6) of the decant water i.e. RL17, RL16 and RL15. However, there is no indication that the washing of the specimen solids is sufficient to remove the pore pressure rebound (as occurred in RL8). Consolidation tests should have first been conducted on the specimen to confirm the effect of washing of the solids, prior to the hydration cell tests.

It also appears that the dilution and evaporation processes involved in these tests have significantly affected the self-desiccation mechanism, given the different pore pressure responses between the three tests, for identical cement content. This clearly indicates that the effective stress/strength development of CPB is dependent on other factors such as chemical composition/concentration rather than simply the process cement hydration and/or that the cement hydration process is also controlled or at the very least influenced by these factors.
7.3.10. Time dependent behaviour

It has been previously mentioned in this chapter that no reports have been identified in the literature to indicate that the anomalous pore pressure response to consolidation observed in this work has been reported by others. This may either be because it hasn’t occurred or simply because it has not been observed, for reasons explained in the previous discussion regarding two versus one-way drainage. The internal specimen pore pressure distribution shown in Figure 7.10 indicated a potential drainage path length “boundary” within the specimen where for lengths shorter than the “boundary”, no pore pressure rebound was observed to occur. Assuming that the mechanism generating the pore pressure rebound is chemical in nature, the length of the drainage path alone cannot be an influence as the length is uncoupled from the chemistry. However, the length of the drainage path may be related through its effect on the hydraulic gradient and/or the time to consolidation.

In Figure 7.13 it was shown that reducing the initial hydraulic gradient applied to the specimen at the commencement of consolidation did not prevent the pore pressure rebound from occurring. The large hydraulic gradient induced by a shorter drainage path (Equation 3.8 in Chapter 3) could however be sufficient to overcome the rebound mechanism. However, if the mechanism is indeed chemically related, the pressure gradients generated are not of a significant magnitude to affect the chemical processes. A shorter drainage path also increases the time to consolidation, so it is possible that for longer length specimens (such as those considered here) the consolidation timeframe may interact with the chemical timeframe. Given that the pore pressure rebound is consistently observed to occur in the hydration cell consolidation specimens around \( t = 10 \) to 15 hours, if the specimen was to fully consolidate prior to that time period (i.e. shorter drainage path length) then the pore pressure rebound may not be observed.

Ultimately, however there are too many variables in the consolidation tests presented in this chapter to adequately validate any further this potential explanation.
<table>
<thead>
<tr>
<th>Mix Description</th>
<th>$u_{\text{initial}}$ [kPa]</th>
<th>$\sigma_b$ [kPa]</th>
<th>$k_1$ [m/s]</th>
<th>$k_2$ [m/s]</th>
<th>Excess $U$</th>
<th>Figure No.</th>
</tr>
</thead>
<tbody>
<tr>
<td>KB CPB CPB consolidation</td>
<td>400</td>
<td>340</td>
<td>0.6</td>
<td>-</td>
<td>-</td>
<td>Figure 7.7</td>
</tr>
<tr>
<td>RL CPB CPB consolidation</td>
<td>400</td>
<td>340</td>
<td>0.6</td>
<td>-</td>
<td>-</td>
<td>Figure 7.7</td>
</tr>
<tr>
<td>RL3 Uncemented consolidation</td>
<td>400</td>
<td>340</td>
<td>0.5</td>
<td>-</td>
<td>-</td>
<td>Figure 7.7</td>
</tr>
<tr>
<td>SNM Uncemented consolidation</td>
<td>400</td>
<td>340</td>
<td>0.66</td>
<td>-</td>
<td>-</td>
<td>Figure 7.7</td>
</tr>
<tr>
<td>RL3/14 Pore pressure distribution along consolidating specimen length (A, B, C)</td>
<td>400</td>
<td>340</td>
<td>varies</td>
<td>-</td>
<td>-</td>
<td>Figure 7.7</td>
</tr>
<tr>
<td>RL6 $k$ tests before and after &quot;rebound&quot; (@ 20kPa), fines PSD at specimen extremes</td>
<td>400</td>
<td>340</td>
<td>0.1</td>
<td>-</td>
<td>-</td>
<td>Figure 7.7</td>
</tr>
<tr>
<td>RL7 $k$ tests before and after &quot;rebound&quot; (@ 20kPa), fines PSD at specimen extremes</td>
<td>400</td>
<td>340</td>
<td>0.15</td>
<td>-</td>
<td>-</td>
<td>Figure 7.7</td>
</tr>
<tr>
<td>RL8 No mould, &quot;fresh&quot; solids, pore pressure &quot;rebound&quot;</td>
<td>400</td>
<td>340</td>
<td>0.15</td>
<td>-</td>
<td>-</td>
<td>Figure 7.7</td>
</tr>
<tr>
<td>RL9 No mould, &quot;fresh&quot; solids, pore pressure &quot;rebound&quot;</td>
<td>400</td>
<td>340</td>
<td>0.15</td>
<td>-</td>
<td>-</td>
<td>Figure 7.7</td>
</tr>
<tr>
<td>RL11 Process water in specimen and pressure lines; $k_2$ determined @70kPa</td>
<td>400</td>
<td>340</td>
<td>0</td>
<td>1.10x10^{-10}</td>
<td>-</td>
<td>Figure 7.7</td>
</tr>
<tr>
<td>RL12 Tap water in specimen and pressure lines</td>
<td>400</td>
<td>340</td>
<td>0</td>
<td>-</td>
<td>-</td>
<td>Figure 7.7</td>
</tr>
<tr>
<td>RL13 Liquefied specimen, $\sigma_c$ = 382kPa, $u_{\text{initial}}$ = 390kPa</td>
<td>390</td>
<td>344</td>
<td>-</td>
<td>4.74x10^{-9}</td>
<td>3.96x10^{-9}</td>
<td>Figure 7.7</td>
</tr>
<tr>
<td>RL14 Incremental consolidation stress application</td>
<td>410</td>
<td>varies</td>
<td>0.6</td>
<td>1.3x10^{-9}</td>
<td>-</td>
<td>Figure 7.7</td>
</tr>
<tr>
<td>RL15 &quot;typical&quot; CPB hydration pore pressure reduction</td>
<td>400</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>Figure 7.7</td>
</tr>
<tr>
<td>RL16 CPB hydration of washed solids</td>
<td>400</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>Figure 7.7</td>
</tr>
<tr>
<td>RL17 CPB hydration of washed uncemented mix</td>
<td>400</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>Figure 7.7</td>
</tr>
<tr>
<td>RL18 CPB hydration of washed mix</td>
<td>400</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>Figure 7.7</td>
</tr>
<tr>
<td>RL19 CPB hydration of washed mix</td>
<td>400</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>Figure 7.7</td>
</tr>
<tr>
<td>RL20 CPB hydration of washed mix</td>
<td>400</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>Figure 7.7</td>
</tr>
<tr>
<td>RL21 CPB hydration of washed mix</td>
<td>400</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>Figure 7.7</td>
</tr>
<tr>
<td>RL22 CPB hydration of washed mix</td>
<td>400</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>Figure 7.7</td>
</tr>
<tr>
<td>RL23 CPB hydration of washed mix</td>
<td>400</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>Figure 7.7</td>
</tr>
<tr>
<td>RL24 CPB hydration of washed mix</td>
<td>400</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>Figure 7.7</td>
</tr>
<tr>
<td>RL25 CPB hydration of washed mix</td>
<td>400</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>Figure 7.7</td>
</tr>
<tr>
<td>RL26 CPB hydration of washed mix</td>
<td>400</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>Figure 7.7</td>
</tr>
<tr>
<td>RL27 CPB hydration of washed mix</td>
<td>400</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>Figure 7.7</td>
</tr>
<tr>
<td>RL28 CPB hydration of washed mix</td>
<td>400</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>Figure 7.7</td>
</tr>
<tr>
<td>RL29 CPB hydration of washed mix</td>
<td>400</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>Figure 7.7</td>
</tr>
<tr>
<td>RL30 CPB hydration of washed mix</td>
<td>400</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>Figure 7.7</td>
</tr>
</tbody>
</table>
7.4. Modelling the consolidation behaviour: potential impacts of chemistry on consolidation equations

The preceding results and discussion presented in this chapter are sufficient to argue that the pore pressure rebound occurs as a result of chemical interaction/conditions and potential precipitation. The physical effect of a precipitate formation is speculated to manifest as an increase in volume within the specimen. This section considers the effect of such an increase on the pore pressure response during consolidation by modifying the consolidation equation presented in Equation 7.1 to include an additional term. This additional term is referred to as the source function and is used to introduce a volume increase over a specified time period during consolidation, thereby allowing the subsequent pore pressure response to be examined. Further discussion regarding the details of the chemical mechanism generating the rebound is discussed subsequently in Section (7.5).

The modified consolidation equation is shown in Equation 7.8

\[ S + cv \frac{\partial^2 \bar{u}}{\partial z^2} + \frac{\partial \bar{u}}{\partial t} = 0 \]

Equation 7.8

where \( S \) is the source function given by

\[ S = [K_\varepsilon \langle t - s \rangle^0] - [K_\varepsilon \langle t - (s + dt) \rangle^0] \]

Equation 7.9

where \( K_\varepsilon \) is the stress associated with the volume increase, \( s \) and \( dt \) are the time and duration respectively of/over which the source function is introduced. The Macaulay discontinuity function, also known as the unit step function or Heaviside function, is used to generate the pore pressure increase in the source function \( S \) as shown in Figure 7.28.
Chapter 7 The Influence of Chemistry on Consolidation of Cementing Paste Backfill and Uncemented Tailings

Figure 7.28 Source function

Plotting of Equation 7.8 is shown in Figure 7.29 and indicates the physical effect of this volume increase at a given time \( t \), on the consolidation pore pressure behaviour. Comparison of Figure 7.29 with the experimental data presented previously (i.e. Figure 7.8) indicates the ability to model this pore pressure rebound mechanism with this modified consolidation equation.

Figure 7.29 Consolidation profile with source term \((c_v = 1, s = 0.2, dt = 0.05 \text{ and } K_e= 11)\)

Following the source-function-controlled pore pressure increase, consolidation continues as per the Terzaghi’s consolidation equation, while experimental testing has indicated that the
mechanism generating this pore pressure response reduces the permeability of the material, limiting further consolidation. This was demonstrated by the consolidating pore pressure profiles of RL6 and RL7 (Figure 7.14) and the measured permeabilities before and after the pore pressure rebound (Table 7.3). While Figure 7.29 does not show the actual pore pressure response for the RL consolidation case considered, the ability to physically model the influence of a chemical precipitate on pore pressure demonstrates the plausibly of this mechanism.

7.5. Chemical analysis

A suite of chemical testing was conducted on the tailings and process water of the KB, SNM and RL mixes as presented in Chapter 3. This testing included measurements of alkalinity, electrical conductivity, hardness, pH and TDS as well as detection of dissolved anions and metals for each of the process waters. Semi-quantitative XRD and XRF analyses were conducted on the tailings solids, as well as carbon levels (both organic and inorganic) and detection of a range of different metals. Additional XRD testing was conducted on the RL8 and RL9 specimens to compare chemical composition and potentially identify any chemical precipitate(s) that may responsible for the pore pressure rebound evident in RL8, and not in RL9. Table 7.8 indicates the relevant locations in Chapter 3, where summary tables of these results can be found. The full reports for the water chemical analysis, as well as the XRD (including diffraction plots), total dissolved metals and carbon analysis for the tailings solids are provided in Appendix A.

Table 7.8 Location of chemical testing results

<table>
<thead>
<tr>
<th>Test</th>
<th>Testing result summary location in Chapter 3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tailings - XRF</td>
<td>Table 3.2</td>
</tr>
<tr>
<td>Tailings - XRD</td>
<td>Table 3.3</td>
</tr>
<tr>
<td>Tailings - total organic and inorganic carbon</td>
<td>Table 3.4</td>
</tr>
<tr>
<td>Tailings - total dissolved metals</td>
<td>Table 3.5</td>
</tr>
<tr>
<td>Water - alkalinity, EC, hardness, pH and TDS</td>
<td>Table 3.6</td>
</tr>
<tr>
<td>Water – anions</td>
<td>Table 3.7</td>
</tr>
<tr>
<td>Water - total dissolved metals</td>
<td>Table 3.8</td>
</tr>
</tbody>
</table>

Chemical analysis of the solid material showed no significant amounts of swelling clays, with only a trace concentration of smectite identified in the Raleigh sand material (Table 3.3).
Chapter 7  
The Influence of Chemistry on Consolidation of Cementing Paste Backfill and Uncemented Tailings

7.5.1. Chemical comparison of specimens RL8 and RL9

The results of the semi-quantitative XRD analysis conducted on the RL8 and RL9 specimens (Table 3.3) indicated similar chemical composition with the exception of the percentage of the mineral halite, also known as sodium chloride or more commonly as salt, contained in each specimen. The RL8 specimen was found to contain more than twice the percentage of halite than that contained in RL9.

An additional point of note regarding the RL8 and RL9 XRD results is the observation of an unidentified peak in the diffraction spectra at 6.01 Å in both RL8 and RL9 specimens (see Appendix A). This peak may indicate mcguinnessite, a mineral belonging to the rosacite family. However, this is considered unlikely and given that it is observed in both specimens, its presence is unlikely to be related to any difference in consolidation behaviour between the specimens. Given the significant difference in the percentage of halite present in the RL8 and RL9 specimens, it is necessary to consider this as a potential factor in the difference between specimen consolidation behaviour.

7.5.1.1. Potential effects of salt

Oven drying of a moist specimen results in evaporation of the pore fluid from within the specimen and causes dissolved salts (used as the general term here to indicate an ionic compound) to precipitate on the surface of the specimen. The resulting dried specimen will exhibit an increase in the salt content compared with the initial solids due to the compounds left behind in the water evaporation process. This may explain the higher halite content observed in RL8, compared with RL9, due to the reconstituted nature of the RL8 specimen. It is possible that the “normal” consolidation exhibited by RL8 relates to this difference in halite content, or indeed any other difference in salt content, observed between the specimens.

Hydration and crystal growth of salt minerals has been found to generate pressure (Mortensen, 1933) which can lead to flaking and mechanical breakage of mineral grains in fresh rock (Bradley et al., 1978, Bartrum, 1936; Mustoe, 1982). A similar phenomenon has also been observed in concrete structures (Winkler & Wilhelm, 1970). Hydration of salt crystals causes the crystals to expand into the larger pores, as they initially have insufficient chemical energy to fill the smaller pores. As expansion occurs, pressures within the pores increases until the mineral’s bond rupture threshold is reached and salt crystal growth can be promoted in smaller pores (Trenhaile, 1987; Wellman & Wilson, 1965). This pressure generated by the hydration of

\[ \text{3 The influence of oven drying specimens at } 100\degree \text{C was considered negligible. While high temperatures may impact the structural integrity of the minerals, heating to temperatures in excess of } 500\degree \text{C is required to drive off organic material, which is more volatile compared with other minerals. Heating to } 100\degree \text{C only reduces hydration spheres around solid particles and removes the liquid phase, leaving dissolved minerals behind as precipitates.} \]

---

- 156 -
Chapter 7 The Influence of Chemistry on Consolidation of Cementing Paste Backfill and Uncemented Tailings

Salt minerals may offer an explanation for the pore pressure rebound observed in the experimental work of this chapter.

Given the large chemical variation between the materials tested (gold and nickel tailings from different geographical locations and mining processes) it would be a large coincidence if the same chemical reaction was responsible for the anomalous consolidation behaviour in each material. Instead, the presence of halite, a common mineral, offers a credible explanation, particularly given the use of process water (high TDS) within each of the mixes tested.

Revisiting Figure 7.21 which showed the consolidation pore pressure response of RL8 and RL9, it is observed that the RL8 specimen containing the higher halite content successfully dissipated all excess pore pressure during consolidation and maintained its consolidated pore pressure level without any subsequent rebounds. Comparatively, the RL9 specimen with the lower halite content displayed a pore pressure rebound shortly after full consolidation. If salt hydration (of halite or potentially other minerals) as explained above is responsible, then it would be expected that the specimen with the higher salt content would experience the pore pressure rebound. This is contrary to the two RL consolidation tests conducted here.

The potential effect of the halite on the consolidation process is also evident in the RL11 and RL12 consolidation tests which use process and tap water respectively (Figure 7.19). While both specimens experienced a pore pressure rebound after full consolidation, which recovered all the previously dissipated excess pore pressure, there is an offset in the time at which the rebounds occurred. The RL12 specimen mixed with tap water rebounded first at $t = 14$ hours, followed by the RL11 process water specimen, approximately 5 hours later. Due to the increased salt content in the RL process water compared with tap water (indicated by the TDS and EC readings in Chapter 3, Table 3.6) the RL11 specimen would most certainly contain a higher salt content compared with RL12. This would intuitively suggest, based on the salt hydration hypothesis, that the higher salt content specimen should show a pore pressure rebound within a shorter time period compared with the equivalent RL12 specimen with a lower salt content. However, again, the results shown in Figure 7.19 indicate the contrary, with the lower salt content specimen RL12 “rebounding” before RL11.

While this discussion is based on only a single set of tests (RL8 and RL9), the observations relating to these tests also appear valid for the RL11 and RL12 tests. To confirm this indicated trend further testing is required. However, the results presented here highlight a potentially increased complexity in the process involved beyond the salt hydration discussion provided. Additional complexity is also suggested by the lack of halite evident in the SNM tailings material (Table 3.3) yet the SNM CPB mix still showed evidence of the pore pressure rebound (Figure 7.7).

7.5.2. Precipitate potential

It was previously stated that any process which occurs to generate the consolidation pore pressure rebound and subsequent permeability reduction, must be a relatively common reaction
given the presence of this mechanism across the range of tailings types and geographical locations presented in this chapter. This said however, there must be some common combination of chemicals across the three material sets (RL, SNM and KB) with the potential to react.

The chemistry associated with KB and RL tailings and process water are observed to be similar as they are both gold tailings, while the SNM nickel tailings and process water chemistry are vastly different. No similarities between chemical composition of the SNM and the KB/RL tailings were evident in the XRF or carbon analysis test results (Table 3.2 and Table 3.4 respectively) and only albite, muscovite (from the XRD in Table 3.3) and aluminium (Al), magnesium (Mg) (from the analysis of metals in Table 3.5) were observed in similar quantities across the three tailings types. Analysis of the process waters also indicated no similarities between SNM and KB/RL with regard to alkalinity, EC, hardness, pH and TDS readings (Table 3.6). Examination and comparison of the cations and anions indicated in each of the process waters (Table 3.7 and Table 3.8 respectively) shows only Ba^+ and SO_4^- ions occurring in similar quantities (i.e. same order of magnitude) in the SNM, KB and RL water. This creates the potential for the formation of barium sulphate (BaSO_4).

Barium sulphate is known to be commonly used in the mining process to facilitate in TDS reduction during processing, due to its ability to be rapidly precipitated (Maree et al., 2004; Hlabela et al., 2007). This barium sulphate precipitation hypothesis is consistent with the idea of a sudden pore pressure increase like those observed in tests presented in this chapter, which requires a rapid reaction rate, considerably faster than the rate of consolidation to overcome the drained conditions imposed on the specimen.

The XRD analysis only detects minerals with crystalline structures which develop over a longer timeframe than the duration of the consolidation tests conducted. Given the short time frame over which the “rebound” occurs and the lack of variation in crystalline products identified in the XRD results of the RL9 specimen compared with the RL8 (no rebound), the BaSO_4 precipitate hypothesis is not contradicted. The effects of sulphate on the strength development of CPB have been examined and found to have a significant influence (Fall & Benzaazoua, 2005; Fall & Pokharel, 2010; Pokharel & Fall, 2011). However, there is no mention in the literature of other possible implications on CPB or tailings slurry behaviour due to the presence of sulphate and certainly no evidence of such an effect on the pore pressure response during consolidation, as shown here, due to either a sulphate or indeed any other cause. That said, significantly more testing must be carried out for full confirmation of the hypothesis. Such testing is significantly outside the scope of this thesis and the author’s area of expertise; however, some recommendations for further work are made in Section 7.8.
7.6. Implication on chemical shrinkage measurements

The work presented in Chapter 5 showed a large discrepancy between the ASTM measured chemical shrinkage and that determined from the hydration cell data via the hydration model proposed. No definitive explanation could be developed for the difference between the shrinkage measurements. However, the anomalous pore pressure behaviour investigated in this chapter has been shown to relate to the complex chemistry involved and given the relationship between pore pressure and chemical shrinkage developed in Chapter 5, may offer an explanation for the variation in chemical shrinkage. This chapter has highlighted the complexity of the chemistry involved in the use of tailings and process water; the chemical situation is further complicated by the addition of cement binder.

While the potential reasons for the chemical shrinkage differences discussed in Chapter 5 are still valid and potentially provide a partial explanation for the discrepancy in chemical shrinkage determination techniques, the implications of the chemical mechanism must be fully considered. To do this, the exact cause and conditions of the mechanism must be fully understood and further chemical shrinkage hydration testing would be required to determine if such a chemical mechanism is indeed partially (or fully) responsible for the difference in $\varepsilon_{sh \, hyd}$ and $\varepsilon_{sh \, ASTM}$.

7.7. Conclusions

This chapter detailed the experimental process followed while investigating the anomalous pore pressure behaviour originally exhibited during consolidation of a KB CPB mix in Chapter 5. The work presented here has exhausted a significant number of avenues in the search for a physical explanation for the mechanism generating this pore pressure rebound. The experiments have focused on uncemented tailings mixes for the simplicity afforded by the removal of the self-desiccation mechanism.

Sufficient repetition of consolidation tests, where the pore pressure rebound was observed, substantiates this mechanism as a true phenomenon, which has been shown to occur in a range of cemented and uncemented CPB/tailings mixes, with the exception of a silica sand mix where consolidation occurred as expected. The obvious difference between the silica sand and the other material was the complexity of the chemistry involved.

Uncemented RL tailings were selected for further testing to investigate this phenomenon. While the pore pressure rebound suggested consolidation was not occurring, the volume of pore fluid drained from the specimen during consolidation plateaued, signifying consolidation completion. A significant reduction in permeability was also observed to be associated with the pore pressure rebound and the greater the rebound (i.e. more pore pressure recovered), the greater the reduction in specimen permeability. The permeability reduction was shown to be unrelated to the specimen particle size distribution, as indicated by no evidence of particle migration between the specimen extremities during consolidation.
Examination of the stress distribution with time along the length of the consolidating specimen indicated that the drainage path length plays a role in this anomalous consolidation pore pressure behaviour. As the drainage path length reduced, the time from the start of consolidation to the occurrence of the rebound increased, until a critical drainage path length was reached where the pore pressure maintained its consolidated state. This is attributed to the reduced time to consolidation for a shorter drainage path length, compared with the relatively steady 10 to 15 hours required for the rebound to develop, i.e. a lack of interaction between the consolidation and chemical-reaction timeframes.

The effect of osmotic pressure variations on the consolidation pore pressure behaviour was examined via the variation of water (via salinity) in the equipment pressure lines. Osmotically-induced consolidation was indicated to facilitate pore pressure dissipation during consolidation. The results of tests utilising only one source of water throughout the system (within both pressure lines and specimen) showed different consolidation pore pressure behaviour, where, for the first time, full consolidation occurred prior to the pore pressure rebound. In addition the pore pressure response throughout these tests was observed to be significantly smoother in comparison with previous tests where different water types were employed.

Consolidation of a reused specimen (i.e. where prior consolidation and oven drying occurred before additional water was added, then followed by subsequent consolidation) did not develop the pore pressure rebound. From this it was hypothesised that the pore pressure rebound was generated by a chemical reaction forming a precipitate, inducing an increase in volume within the specimen, which manifests as a pore pressure increase. It is possible that the pore pressure rebound did not occur upon subsequent reuse of the material as the reaction of relevance had been exhausted. The change in permeability is consistent with the idea of precipitate formation, as this would reduce the pore volume and hence the permeability.

Examination of the XRD chemical testing data showed no significant difference between the reused specimen (no pore pressure rebound) and an equivalent first-use specimen (with pore pressure rebound) apart from an increase in percentage quantity of halite in the reuse specimen. This difference was attributed to the precipitation of minerals from the initial consolidation process water left behind when the specimen was oven dried prior to the ‘re-use’ of the material in a subsequent consolidation test. A difference between specimens mixed with tap or process water was observed, where the process water specimen pore pressure rebound occurred earlier than the tap water specimen. This was also attributed to the salt content, increased by the minerals contained in the process water compared with chemical composition of tap water.

The potential for salt hydration to generate the observed rebound was discussed. However, due to the comparatively reduced halite content in the SNM material compared with the other material – all of which experienced the pore pressure rebound - further consideration was given to the chemical composition. Examination and comparison of the cations and anions present indicated in each of the process waters indicated only barium and sulphate occurring in similar
quantities in the SNM, KB and RL water creating the potential for the formation of barium sulphate (BaSO₄). This supports the hypothesis of rapid mineral precipitation generating the pore pressure rebound. Investigations to confirm and further investigate this hypothesis require significantly greater chemical consideration and analyses, which is beyond the scope of this thesis. The work presented in this chapter clearly highlights the important role and effect chemical composition of both CPB and uncemented tailings may have on its physical behaviour, and that this cannot be underestimated.

7.8. Recommendations for future work

To identify and fully understand the chemical mechanism hypothesised to influence the consolidation pore pressure behaviour witnessed in this chapter, significant further chemical analysis of the individual materials involved is required and further consolidation testing must be carried out in a controlled manner to minimise chemical variability.

The chemical test results presented in Chapter 3 (see location summary in Table 7.8) provide information regarding reagents that may be involved in any potential chemical reactions. To confirm the potential for BaSO₄ precipitation and identify any additional potential products which may form, chemical modelling should be undertaken. Understanding the proportions of each physical mix constituent involved and thus the chemical reagents, as well as the physical conditions (i.e. temperature and pressure), a chemical modelling package (such as Visual Minteq or PHREEQC) would allow evaluations of potential reactions and resulting products. Once the full range of potential reactions are identified, a hypothesis may be developed and tested using a variety of chemical and physical experimental procedures.

Given the observed effects of chemical composition on the consolidation behaviour, a range of physically equivalent consolidation tests should be conducted on a range of different tailings and/or CPB mixes, in a chemically controlled manner to limit the introduction of chemical variation between tests. In addition to the final consolidated specimen, the water expelled from the specimen during consolidation should be collected for chemical analysis. The results, compared with the chemical analysis of the individual mix constituents, may allow for isolation of the full extent of chemical processes occurring and provide further insight into the controlling reaction generating the pore pressure rebound. A combination of sequential extraction and XRD testing could then be used to determine the full chemical composition of the consolidated specimen.

A full understanding of chemical processes and interactions occurring during consolidation and hydration of CPB mixes, and how these may manifest in physical behaviour and properties, has a large potential for influence over CPB mechanical behaviour, including strength increase and pore pressure reduction (i.e. \( \sigma' \) generation). With an understanding of the chemical processes involved, manipulation of the chemical reactions generated (i.e. through variation of water type,
or the use of additives to change chemical composition) may allow for enhanced control of CPB hydration to gain more favourable *in situ* properties.
CHAPTER 8. CONCLUSIONS

8.1. Concluding remarks

The key outcomes from this research have highlighted the effect of curing temperature, consolidation behaviour and chemistry on the stress conditions of CPB and how, in turn, this affects material properties and behaviour. It has been well established that elevated curing temperatures increase the reaction rate and the extent of cement hydration, ultimately affecting the mechanical properties of the fill. Similarly, different chemical compositions of cementitious material have also been shown to also affect hydration and the final mechanical properties. Understanding these effects and their impacts on CPB behaviour is crucial, given the difference in laboratory curing conditions under which design and QA/QC testing is carried out and the in situ curing environment of a stope, as well as the complex chemical nature of the tailings and process water used in most backfilling operations.

The implications of temperature and chemical composition on the ultimate strength, from a chemical reaction perspective, are relatively well documented in literature regarding cementitious material and CPB cured and tested in a laboratory environment. However, little consideration has been afforded to the effect of temperature and chemistry on the stress development of in situ cured material. The effective stress approach developed by Helinski and colleagues (Helinski, 2007; Helinski et al., 2007a; Fahey et al., 2011 and Helinski et al., 2011b) highlighted the significant effect that curing under effective stress, resulting from the self-desiccation mechanism, has on improving the mechanical properties of CPB. The role of temperature has been examined in this thesis in the context of experimental laboratory element testing of CPB, focusing on the effects on the self-desiccation mechanism and the effect on early age material strength.

CPB has been observed to achieve greater ultimate strengths when cured in situ compared with material cured in a laboratory environment (Cayouette, 2003; Revell, 2004; le Roux et al., 2005). In addition to the improvement in material properties afforded by curing under effective stress (Helinski et al., 2007b), elevated curing temperatures are also known to improve properties of CPB (Fall et al., 2010, 2007). In a stope, elevated temperatures are achieved due to the exothermic reactions of cement hydration and the large confined mass of CPB in a stope that results in very slow heat dissipation rates. This elevated temperature, in addition to the effective stress generation due to self-desiccation, is credited with the increased strength of in situ cured CPB compared with equivalent specimens cured in a laboratory. The development of a temperature-controlled hydration cell has allowed the combined effect of elevated curing temperature and effective stress generation on CPB to be examined in a laboratory.
environment. The results have indicated the potential for the curing temperature to play an increased role on the mechanical properties for both consolidating and non-consolidating CPB, compared with curing under effective stress in isolation.

Anomalous pore pressure behaviour observed during standard one-way consolidation testing was found to be significantly impacted by the chemical composition and/or interactions of the CPB constituents (i.e. tailings, water and binder). The pore pressure "rebound", acting in conjunction with the self-desiccation mechanism suggests the potential to limit pore pressure reduction experienced, thus reducing the extent of effective stress generation during curing. This would appear to have an ultimately detrimental effect on the developing material properties. This phenomenon has not been previously documented in the literature and an extensive experimental testing program was conducted to investigate potential causes.

Given the complexity, time and associated expense of the effective stress testing protocols developed by Fahey et al. (2011) and utilised in this research, other techniques, both experimental and analytical, were examined. Chemical shrinkage testing, using the ASTM method (ASTM, 2009), as utilised in cement and concrete industries, was applied to CPB with the aim of developing an index test for the comparison of different mixes. Chemical shrinkage volumes are known to correlate with the progression of hydration, and thus with increases in mechanical properties such as strength and stiffness. To correlate measured shrinkage volumes with the hydration cell test results, particularly the pore pressure behaviour (i.e. effective stress generation), an extended version of Helinski’s hydration model (Helinski, 2007) was applied. However, this was unsuccessful with no consistent behavioural trends observed; this was attributed to the unquantified effects of potential chemical interactions. Despite this, the results of the independent chemical shrinkage testing still provided useful indications regarding the comparative differences in mix proportions, materials and chemical effects.

Gibson’s consolidation solution (Gibson, 1958) was also modified to incorporate the effects of self-desiccation in addition to the self-weight consolidation problem for which it was originally developed. The combined pore pressures were determined from the superposition of the positive pore pressure generated from self-weight consolidation (Gibson, 1958) with the negative pore pressure generated by the self-desiccation mechanism. This approach was successfully validated by comparison with a fully coupled consolidation and hydration ABAQUS analysis. This approach allowed for a qualitative assessment of the pore pressure which may be expected in a CPB filled stope throughout a given fill and curing time period, based on initial CPB material properties. Understanding this pore pressure behaviour provides insight into mechanical behaviour, as has been demonstrated in both the literature and the temperature hydration cell testing presented in this thesis.

Ultimately this research has highlighted the importance of environmental effects on the behaviour of CPB. Neglecting temperature effects may produce an underestimation of CPB material properties and behaviour as they generate in a stope, leading to unnecessary conservatism and associated expense in the design of CPB, or alternatively potential safety implications if empirical design factors are utilised. Similarly, without understanding the full
effects and implications of chemical compositions/interactions on material properties and behaviour, elements of uncertainty remain in the design and use of the CPB product.

8.2. Main outcomes and future recommendations

This section presents a detailed summary of the final conclusions drawn from each area of research presented in this thesis. In addition, recommendations for future research in these areas are also provided. This section contains only brief discussion, as detailed discussions have been included in each of the relevant chapters.

8.2.1. The influence of elevated curing temperatures

8.2.1.1. The temperature-controlled hydration cell

Successful development of a temperature-controlled hydration cell was detailed. This cell allowed the effects of elevated curing temperature and effective stress generation on the mechanical properties and behaviour of cementing paste backfill to be examined, both independently and in combination. The apparatus was modified from that developed by Helinski (2007) to allow for isothermal curing conditions in addition to effective stress generation. The temperature control system allowed for controlled temperature increase of the specimen to occur at different rates, replicating the generation of temperature in a hydrating CPB-filled stope. Attempts were made to develop the equipment further to allow adiabatic testing conditions. Such conditions would allow independent heat generation by the specimen as a result of the exothermic cement hydration reactions, thus simulating the temperature increase of CPB curing in a stope. Additional heat supplied by the temperature control system would prevent dissipation of the cement-generated heat without adding additional thermal energy. However, the thermal properties of the developed set-up were insufficient to prevent dissipation of heat generated by the small specimen without significant contribution to the heat load. Replication of in situ temperatures, instead, occurred with the use of a heater timing array and a target peak temperature, based on in situ temperature measurements. Recommended further work includes improvement of the thermal control of the hydration cell to allow for adiabatic testing. Such improvements may include utilisation of different materials for hydration cell construction, or the use of a temperature-controlled thermal chamber allowing accurate thermal control of the entire set-up. This would remove the reliance on in situ testing to establish target curing temperatures and rates, allowing independent assessment of potential in situ curing temperatures.

8.2.1.2. Effect of temperature and effective stress on CPB mechanical properties

The temperature-controlled hydration cell was used to assess the role of both effective stress and elevated temperature curing on the mechanical properties of CPB specimens. Two different CPB mixes (KB and SNM) were hydrated in the apparatus for a 7-day period, under four different curing conditions. These curing conditions comprised: (A) no effective stress generation at room temperature; (B) effective stress generation at room temperature; (C) no
effective stress generation at an elevated temperature; and (D) effective stress generation at an elevated temperature. These four conditions allowed a direct comparison of the effects of effective stress generation and elevated temperature both individually and in combination. Different elevated curing temperatures were chosen for each of the mix types, specifically 34°C and 44°C for SNM and KB respectively, based on in situ temperature data and semi-adiabatic laboratory testing. All test results were observed to be repeatable, with the results presented as an average of two or more tests (with the exception of SNM curing condition C, where only one test was completed).

Pore pressure and stiffness were measured for each test throughout the 7-day hydration period. The elevated curing temperature was found to promote effective stress generation and the combination of both conditions (curing condition D) were found to increase the stiffness generated compared with curing conditions A, B and C. The condition of zero effective stress was not successfully maintained in the KB specimens, due to the non-consolidating nature of the material (low permeability). This difficulty in controlling the pore pressures within the specimens may relate to the anomalous pore pressure behaviour identified in the consolidation testing (Chapter 7), which indicated a potential counteraction of the self-desiccation mechanism. Further temperature-controlled hydration cell testing of the zero effective stress generation conditions should be conducted once an understanding of the processes and mechanisms responsible for the anomalous pore pressure consolidation behaviour have been determined. This will allow a fully independent analysis of the effects of curing temperature, compared with effective stress generation to be conducted. However, despite this, the results of the 7-day UCS testing clearly indicated an increasing trend in the strength with elevated temperature and stress generation. The testing indicated in both materials that the effect of elevated temperature on the early age strength is more significant than effective stress generation. This work has clearly indicated the importance of the curing environment on the ultimate mechanical properties and behaviour of CPB.

8.2.1.3. Recommendations for in situ CPB analysis for validation of laboratory results

In situ stope instrumentation (specifically temperature and pore pressures measurements) and testing of cored in situ cured CPB material should be carried out for comparison and validation of the curing conditions applied in the temperature-controlled hydration cell (particularly the final curing temperature and rate of heat generation) compared with those occurring in situ. Comparison of the ultimate strengths will give an indication of the reproducibility of in situ conditions in the temperature-controlled hydration cell. However, the material will not be directly comparable given the differences associated with the self-weight consolidation occurring in situ, which does not occur in a hydration cell. This suite of data, along with laboratory consolidation testing, will provide data sufficient for further interpretation of CPB behaviour using numerical methods (Helinski et al., 2007b; Doherty & Muir Wood, 2013).
8.2.2. Evaluation of chemical shrinkage

The ASTM test procedure confirmed similar hydration behavioural trends for CPB as for cement paste (CP), initially validating the use of the ASTM test for use with CPB. Specifically, the volume of chemical shrinkage, normalised by either mass or volume of binder, was found to increase with increasing w/c ratio, similar to CP behaviour. The measured volume was found to be significantly greater for CPB mixes compared with equivalent w/c ratio CP mixes, which was attributed to the increased number of nucleation sites provided by the tailings solids for cement hydration to occur. Hyper-saline water was also observed to affect the chemical shrinkage. However, the evidence was conflicting regarding which produced the larger chemical shrinkage volume. The results in Chapter 7 highlighted the importance of chemistry on the hydration process, which was shown to relate to other CPB behaviours and properties, such as consolidation, permeability and effective stress generation.

The primary purpose of the ASTM chemical shrinkage testing was to assess its ability to act as an index/screening test for different CPB mixes prior to more detailed assessment of these mixes via the hydration cell test. The success of such an approach relies on the ability to link the chemical shrinkage measurements with the hydration cell data. Successful correlation of the ASTM and hydration cell tests would provide a useful screening tool to assess the suitability of different CPB mixes, particularly helpful when considering the effects of alternate binders (e.g. fly ash, slag, ground glass etc. with cement) on the overall mix behaviour. However, comparison of the volumetric strains showed no clear correlation between the two data sets, with the ASTM results indicating strains more than an order of magnitude larger than those determined from the hydration cell test. The volume reduction associated with these strains suggests that those indicated by the ASTM tests are unrealistically large, suggesting a 4% volume reduction in a typical stope curing situation. However, given the good comparison between the ASTM CP tests and published data, as well as recognised trends evident in the results, no immediate explanation could be suggested based on the ASTM testing. Instead further investigation into the hydration cell testing was conducted.

Stiffness is a key parameter in the determination of strain and given the dependence of the bulk stiffness determination in the hydration cell on the assumed value of Poisson’s ratio, the effect of different values within the typical range was investigated. However, this did not account for the large difference in strains, so alternate stiffness measurement techniques were also examined. Strain gauges were introduced into the hydration cell setup to offer a direct measurement of the strain occurring due to self-desiccation, allowing an indirect assessment of the stiffness. However, difficulty was encountered in obtaining initial, early age strain measurements due to sensitivity of the gauge and the initial slurry nature of the specimen.

Consolidation of the specimen was attempted to increase stiffness without waiting for hydration-related stiffness generation; the aim being to improve the strain gauge measurements. However, consolidation was not observed to be achieved based on the pore pressure response. Initially the pore pressures indicated a typical consolidation response, with excess pore
pressure reducing to equilibrate with the applied back pressure, but then, with no variation in pressure or temperatures, the pore pressure began to increase, or “rebound”. This anomalous rebound was observed in multiple consolidation attempts. The lack of success with specimen consolidation resulted in the abandonment of the strain gauges for stiffness measurements. Thus, based on the experimental test work conducted, the ASTM tests were unable to be adequately related to the hydration cell test.

Future test work should be conducted to confirm the magnitude of stiffness and effective stress generation in light of the anomalous pore pressure response to consolidation that was observed. Similarly, in the temperature-controlled hydration cell work, the effective stress determined in the hydration cell may be influenced by the mechanism responsible for the anomalous consolidation pore pressure response. This will, in turn, influence the development of stiffness during hydration. Any potential variation in the effective stress generation in the hydration cell may account for differences between the ASTM chemical shrinkage and hydration cell determined strains. Successful correlation of chemical shrinkage measurements and effective stress generation may further validate both techniques and allow for use of the ASTM chemical shrinkage test as an effective index test for the initial characterisation and design of CPB mixes.

8.2.3. Modification to Gibson’s solution to include self-desiccation

Successful adaptation of Gibson’s consolidation solution to incorporate the effects of self-desiccation on the pore pressure response was presented. A series of design charts were developed to determine the chemical shrinkage-induced pore pressure response for hydration of a weightless material. The ability to superpose the shrinkage-induced pore pressure response with Gibson’s self-weight consolidation response, due to an established relationship between the respective governing parameters, was also indicated. This allows a qualitative assessment of potential pore pressure generation within a stope during curing, where both consolidation and hydration is occurring.

8.2.4. Chemical effects on consolidation behaviour

A significant number of experimental avenues were explored in the search for a physical explanation for the anomalous pore pressure behaviour observed during one-way consolidation. In the consolidation tests where the anomalous behaviour was observed, initially the pore pressure behaved as expected, dissipating toward the applied backpressure. However, without any variation in pressure conditions, the pore pressure was suddenly observed to increase or “rebound”, in most cases recovering almost all the previously dissipated pore pressure. Ultimately, experimental consolidation and chemical testing indicated that a chemical reaction, potentially forming a precipitate may be responsible for the anomalous behaviour.

Initially, the phenomenon was observed during the consolidation of CPB specimens, yet it was also observed in the consolidation of uncemented tailings slurries. Testing of the latter was focused on due to the simplicity afforded by the lack of self-desiccation. Sufficient repetitions of
testing of both uncemented and cemented tailing slurries, including RL, KB and SNM material, were conducted to confirm the pore pressure rebound as a true phenomenon. The exception was the silica sand specimen that consolidated as expected. The most significant difference between the tailings specimens and the sand specimen was the complex chemistry associated with the tailings and process water combination, compared with inert silica sand and tap water. Despite the pore pressure rebound suggesting that consolidation had not occurred, the volume of pore fluid expelled from the specimens due to the applied pressure difference suggested that consolidation was occurring.

A significant reduction in permeability was observed to coincide with the pore pressure rebound; the greater the rebound magnitude, the greater the reduction in permeability. This reduction in permeability was determined to be unrelated to particle migration within the specimen, as determined via particle size distribution analysis of the extremities of a post-consolidated specimen.

Investigation of the pore pressure distribution with time along the length of the specimen indicated that the drainage path length played a role in the anomalous rebound behaviour. It appeared that when a critical drainage path length was reached, the pore pressure rebound was not observed. A shorter drainage path length equates to a shorter time to consolidation and, given the consistent occurrence of the rebound between 10 to 15 hours after the commencement of consolidation, this shorter time to consolidation may prevent interaction between consolidation and chemical reaction timeframe in thin specimens.

Osmotic pressure variation due to differences in water quality between water contained in the specimen (process) and in the equipment pressure lines (tap) was also considered as potentially impacting the overall specimen pore pressure behaviour. While osmotically induced consolidation was indicated to facilitate the pore pressure dissipation during consolidation, (opposite in effect to the generation of the rebound) the use of a single water type throughout the system varied the pore pressure response, where for the first time, full consolidation (i.e. $U = 0$) occurred prior to the pore pressure rebound. The time at which the rebound occurred varied depending on the type of water contained in the system; the tap water-only specimen exhibited a pore pressure rebound 5 hours prior to the process water-only specimen.

Consolidation of a RL specimen reconstituted from the dried solids of a previous consolidation test was the only instance where the pore pressure rebound was not observed. This “normal” consolidation behaviour could not be replicated with “new” tailings (i.e. previously unconsolidated). An XRD analysis of the two specimens (with and without rebound) indicated increased halite content in the reused specimen. Yet this was not considered an adequate explanation for the rebound given the lack of halite present in the SNM material, which also exhibited the anomalous consolidation pore pressure behaviour. Further chemical testing was conducted on the tailings and water used in the various mixes, including XRD, XRF, organic and inorganic carbon content and total dissolved metals of the tailings material and total dissolved metals, anions and cations of the water. Examination of anions and cations present in each of the process waters indicated that only barium ($Ba^+$) and sulphate ($SO_4^{2-}$) ions occurred
in similar quantities in water that was utilised in all mixes exhibiting the rebound. This creates potential for generation of barium sulphate (BaSO$_4$) which is known to readily precipitate (Maree et al., 2004; Hlabela et al., 2007), supporting the hypothesis of rapid mineral precipitation generating the pore pressure rebound. The rebound has the potential to limit the effective stress generation, by reducing pore pressure reductions that occur in consolidating, hydrating specimens. While the effects of sulphates on mechanical strength have been examined previously (Fall & Benzaazoua, 2005; Fall & Pokharel, 2010; Pokharel & Fall, 2011), their potential effects on the physical process of consolidation and self-desiccation have not been previously observed.

Significantly more experimental work is required to confirm this hypothesis; this is beyond the scope of this thesis. The recommended first steps in continuing this investigation is the use of the chemical testing results presented in this thesis to examine reagents that may be involved in potential chemical reactions, using chemical modelling procedures. The presence of any potentially identified reactions should then be confirmed using chemical and physical experimental procedures. This should include repetition of consolidating testing (similar to the procedures presented in this research) on a range of different CPB mixes (i.e. varying tailings and water types) under conditions where chemical differences are incrementally introduced. Such testing should include chemical analysis of any water expelled during consolidation. Comparison of the water chemistry with a chemical analysis of the individual mix constituents, in addition to sequential extract and XRD testing may be used to determine any reactions involved in the generation of the pore pressure rebound.

As identified in the above sections of this chapter, understanding this pore pressure rebound phenomenon is the first step required before other further work can be conducted on the issues and questions raised in this research. This work highlights the potential effects of chemical composition and interactions on the behaviour of mine tailings, and indeed CPB. Understanding the potential chemical processes involved and how these may manifest in physical properties and behaviour may allow for enhanced control of CPB hydration via chemical manipulation to achieve more favourable in situ CPB properties and behaviour.

8.3. Summary

The focus of this research was to further understand the behaviour of cementing paste backfill, during early ages, within the effective stress framework. The effect of temperature and stress conditions, in addition to chemical composition, on CPB curing has been highlighted and indicated to significantly influence the behaviour of early age CPB. A previously unreported phenomenon was observed in the pore pressure response to consolidation of CPB, and uncemented tailings slurry equivalent mixes. This was attributed to the chemistry of the water and its interactions within the consolidating mixes. Such behaviour was discussed in regard to its potential to influence the self-desiccation behaviour of CPB and thus the generation of mechanical properties during early ages, and ultimately the final properties of the cured CPB.
REFERENCES


APPENDIX A. CHEMICAL TESTING RESULTS

A.1. *Semi Quantitative XRD Report (CSIRO)*
Semi Quantitative XRD Report

1. Sample Preparation:
The samples received were the loosely consolidated sands and tailings ranging in colour from off white to dark brown. Sub samples weighing approximately 3g were crushed using an agate mortar and pestle and back loaded into a standard X-Ray Diffraction (XRD) sample holder for semi-quantitative analysis.

2. Experimental Method:
Data was collected using a Bruker D4 XRD fitted with a Cu tube and Fe filter, and a Lynxeye position sensitive detector, using the following settings:
- Random oriented samples:
- 2θ range = 5–90°, Step size = 0.03°, Divergence slit = 1 Degree

4. Discussion:
Comparison between samples RL5-8 and RL9 show that the main difference is that RL5-8 has more Halite while RL9 has more dolomite. There is also a peak with a d-space of 6.014Å that is still unaccounted. The relay sand sample is composed mostly of quartz with minor amounts of clay however there are broad low angle peaks indicating that smectite (expanding clays) or other poorly ordered or amorphous material (such as organic material) may be present. The Cannington sample also has broad low angle peaks which may be due to smectite or other poorly ordered or amorphous material.

5. Diffraction Data files:
Diffraction data files in the original binary format (with the file extension .raw) are available on request. Images of the diffraction patterns with markers lines for the main minerals are shown below.
Semi Quantitative XRD Report

6. Semi-quantitative results:
The semi-quantitative results are based on the XRD pattern obtained from a "randomly" orientated sub sample.

The amounts indicated are a guide only and are based on rough Rietveld refinements of the diffraction patterns from the "randomly" orientated samples and previous work of this nature; however the variations in clay properties, sample preparation, and the degree of orientation in the “random” orientated samples will all affect the peak heights and the estimates of the amount present.

<table>
<thead>
<tr>
<th></th>
<th>Cannington</th>
<th>KB</th>
<th>Lanfranchi</th>
<th>rayleigh sand</th>
<th>rayleigh tails</th>
<th>RL5-B</th>
<th>RL3</th>
<th>SNM*</th>
</tr>
</thead>
<tbody>
<tr>
<td>Actinolite</td>
<td></td>
<td>2.9</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>12.8</td>
</tr>
<tr>
<td>NaCa feldspar (Albite)</td>
<td>4.6</td>
<td>35.5</td>
<td>20.6</td>
<td>11.6</td>
<td>3</td>
<td>2.6</td>
<td>23.6</td>
<td></td>
</tr>
<tr>
<td>Mica (Biotite)</td>
<td>1.2</td>
<td>18.6</td>
<td>10.5</td>
<td>5.6</td>
<td>6.7</td>
<td>2.2</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Mica (Muscovite)</td>
<td>2.7</td>
<td>20.2</td>
<td>1.5</td>
<td>14.3</td>
<td>13.8</td>
<td>14.2*</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Calcite</td>
<td></td>
<td>1.6</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Chalcopyrite</td>
<td>0.8</td>
<td>0.5</td>
<td>2</td>
<td>8.5</td>
<td>1.2</td>
<td>1.3</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Chlorite</td>
<td>3.1</td>
<td>0.6</td>
<td>4.4</td>
<td>7.3</td>
<td>6.4</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Coquimbite</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>0.1</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Cummingntonite</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>5.3</td>
<td></td>
</tr>
<tr>
<td>Dolomite</td>
<td>0.7</td>
<td>17.3</td>
<td>12.3</td>
<td>1.1</td>
<td>2</td>
<td>0</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Fluorite</td>
<td>9.8</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Hornblende (Hastingsite)</td>
<td>7.9</td>
<td></td>
<td>15.3</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Kaolin</td>
<td>7.9</td>
<td>0.4</td>
<td>11.6</td>
<td>8.9</td>
<td>18.5</td>
<td>16</td>
<td>2.2</td>
<td></td>
</tr>
<tr>
<td>Magnetite</td>
<td>6.7</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Microcline</td>
<td>10.3</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>19.4</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Pyrite</td>
<td></td>
<td>0.1</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Pyrrhotite-SC</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>6.6</td>
</tr>
<tr>
<td>Quartz</td>
<td>35</td>
<td>22.6</td>
<td>22.3</td>
<td>90.7</td>
<td>38.2</td>
<td>42.7</td>
<td>48.6</td>
<td>12.7</td>
</tr>
<tr>
<td>Sadelite</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Halite</td>
<td>2.1</td>
<td>2.9</td>
<td>1.3</td>
<td>0.4</td>
<td>2.5</td>
<td>6.1</td>
<td>2.5</td>
<td></td>
</tr>
<tr>
<td>Talc</td>
<td>2.9</td>
<td></td>
<td>4</td>
<td></td>
<td></td>
<td></td>
<td>1</td>
<td></td>
</tr>
<tr>
<td>Tremolite</td>
<td>4.4</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Grand Total</td>
<td>100.1</td>
<td>100</td>
<td>100</td>
<td>100</td>
<td>99.9</td>
<td>99.3</td>
<td>99.3</td>
<td>100.1</td>
</tr>
</tbody>
</table>

- Values are not as reliable as a large orientation correction was required.
A.2. Report of Examination (ChemCentre)
### Appendix A

Chemical Testing Results

---

**Report on:** 11 samples received on 10/09/2013

<table>
<thead>
<tr>
<th>LAB ID</th>
<th>Material</th>
<th>Client ID and Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>13ED491 / 001</td>
<td>water</td>
<td>SNM</td>
</tr>
<tr>
<td>13ED491 / 002</td>
<td>water</td>
<td>Lan Franchi</td>
</tr>
<tr>
<td>13ED491 / 003</td>
<td>water</td>
<td>KB</td>
</tr>
<tr>
<td>13ED491 / 004</td>
<td>water</td>
<td>Cannington</td>
</tr>
<tr>
<td>13ED491 / 005</td>
<td>water</td>
<td>RL</td>
</tr>
<tr>
<td>13ED491 / 006</td>
<td>soil</td>
<td>SNM</td>
</tr>
<tr>
<td>13ED491 / 007</td>
<td>soil</td>
<td>Lan Franchi</td>
</tr>
<tr>
<td>13ED491 / 008</td>
<td>soil</td>
<td>KB</td>
</tr>
<tr>
<td>13ED491 / 009</td>
<td>soil</td>
<td>Cannington</td>
</tr>
<tr>
<td>13ED491 / 010</td>
<td>soil</td>
<td>RL Plains</td>
</tr>
<tr>
<td>13ED491 / 011</td>
<td>soil</td>
<td>RL Sand</td>
</tr>
</tbody>
</table>

---

<table>
<thead>
<tr>
<th>Analyte</th>
<th>Method</th>
<th>Unit 1</th>
<th>Unit 2</th>
<th>Unit 3</th>
<th>Unit 4</th>
</tr>
</thead>
<tbody>
<tr>
<td>Alkalinity, total</td>
<td>IALKIWATI</td>
<td>mg/L</td>
<td>&lt;1</td>
<td>280</td>
<td>52</td>
</tr>
<tr>
<td>Aluminium</td>
<td>IMETWOCNP</td>
<td>mg/L</td>
<td>0.087</td>
<td>0.034</td>
<td>&lt;0.025</td>
</tr>
<tr>
<td>Arsenic</td>
<td>IMETWOCNS</td>
<td>mg/L</td>
<td>&lt;0.001</td>
<td>&lt;0.050</td>
<td>0.31</td>
</tr>
<tr>
<td>Barium</td>
<td>IMETWOCNP</td>
<td>mg/L</td>
<td>0.043</td>
<td>0.068</td>
<td>0.27</td>
</tr>
<tr>
<td>Bicarbonate</td>
<td>IALKIWATI</td>
<td>mg/L</td>
<td>&lt;1</td>
<td>317</td>
<td>83</td>
</tr>
<tr>
<td>Boron</td>
<td>IMETWOCNP</td>
<td>mg/L</td>
<td>&lt;0.52</td>
<td>8.0</td>
<td>0.02</td>
</tr>
<tr>
<td>Calcium</td>
<td>IMETWOCNS</td>
<td>mg/L</td>
<td>0.0001</td>
<td>&lt;0.0020</td>
<td>0.077</td>
</tr>
<tr>
<td>Calcium</td>
<td>IMETWOCNS</td>
<td>mg/L</td>
<td>488</td>
<td>823</td>
<td>3170</td>
</tr>
<tr>
<td>Carbonate</td>
<td>IALKIWATI</td>
<td>mg/L</td>
<td>&lt;1</td>
<td>&lt;1</td>
<td>&lt;1</td>
</tr>
<tr>
<td>Chloride</td>
<td>ICLWICDA</td>
<td>mg/L</td>
<td>68</td>
<td>67720</td>
<td>124000</td>
</tr>
<tr>
<td>Chromium</td>
<td>IMETWOCNP</td>
<td>mg/L</td>
<td>&lt;0.001</td>
<td>&lt;0.005</td>
<td>&lt;0.005</td>
</tr>
<tr>
<td>Cobalt</td>
<td>IMETWOCNP</td>
<td>mg/L</td>
<td>0.007</td>
<td>&lt;0.025</td>
<td>2.4</td>
</tr>
<tr>
<td>Copper</td>
<td>IMETWOCNP</td>
<td>mg/L</td>
<td>&lt;0.002</td>
<td>0.026</td>
<td>0.18</td>
</tr>
<tr>
<td>Electrical Conductivity</td>
<td>IECWSE</td>
<td>mS/m</td>
<td>420</td>
<td>14200</td>
<td>21000</td>
</tr>
<tr>
<td>Hardness, total</td>
<td>HTOTZWACA</td>
<td>mg/L</td>
<td>1700</td>
<td>27000</td>
<td>24000</td>
</tr>
<tr>
<td>Iron</td>
<td>IMETWOCNP</td>
<td>mg/L</td>
<td>5.1</td>
<td>0.15</td>
<td>2.4</td>
</tr>
<tr>
<td>Lead</td>
<td>IMETWOCNS</td>
<td>mg/L</td>
<td>0.0001</td>
<td>0.76</td>
<td>0.075</td>
</tr>
<tr>
<td>Magnesium</td>
<td>IMETWOCNP</td>
<td>mg/L</td>
<td>126</td>
<td>6080</td>
<td>3790</td>
</tr>
<tr>
<td>Manganese</td>
<td>IMETWOCNP</td>
<td>mg/L</td>
<td>1.4</td>
<td>0.93</td>
<td>0.012</td>
</tr>
<tr>
<td>Molybdenum</td>
<td>IMETWOCNS</td>
<td>mg/L</td>
<td>&lt;0.001</td>
<td>0.50</td>
<td>0.10</td>
</tr>
<tr>
<td>Nickel</td>
<td>IMETWOCNP</td>
<td>mg/L</td>
<td>0.49</td>
<td>1.1</td>
<td>1.1</td>
</tr>
</tbody>
</table>

---

Page 1 of 4
## Appendix A: Chemical Testing Results

<table>
<thead>
<tr>
<th>LAB ID</th>
<th>001</th>
<th>002</th>
<th>003</th>
<th>004</th>
</tr>
</thead>
<tbody>
<tr>
<td>Client ID</td>
<td>SNM</td>
<td>Lan Fracchi</td>
<td>KB</td>
<td>Cannington</td>
</tr>
<tr>
<td>Sampled Date</td>
<td>12/06/2013</td>
<td>12/06/2013</td>
<td>12/06/2013</td>
<td>12/06/2013</td>
</tr>
<tr>
<td>Analyte</td>
<td>Method</td>
<td>Unit</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Nitrate</td>
<td>INTA1WFIA</td>
<td>mg/L</td>
<td>&lt;0.05</td>
<td>0.44</td>
</tr>
<tr>
<td>pH</td>
<td>IPHIWAGE</td>
<td>4.7</td>
<td>7.7</td>
<td>7.4</td>
</tr>
<tr>
<td>Potassium</td>
<td>IMET1WICCP</td>
<td>mg/L</td>
<td>103</td>
<td>460</td>
</tr>
<tr>
<td>Sodium</td>
<td>IMET1WICCP</td>
<td>mg/L</td>
<td>244</td>
<td>37200</td>
</tr>
<tr>
<td>Sulphate (from S)</td>
<td>IMET1WICCP</td>
<td>mg/L</td>
<td>2930</td>
<td>11000</td>
</tr>
<tr>
<td>TDS by summation</td>
<td>InTDS_Sum</td>
<td>mg/L</td>
<td>3800</td>
<td>120000</td>
</tr>
<tr>
<td>Vanadium</td>
<td>IMET1WICCP</td>
<td>mg/L</td>
<td>&lt;0.005</td>
<td>&lt;0.026</td>
</tr>
<tr>
<td>Zinc</td>
<td>IMET1WICCP</td>
<td>mg/L</td>
<td>0.046</td>
<td>0.84</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>LAB ID</th>
<th>005</th>
<th>006</th>
</tr>
</thead>
<tbody>
<tr>
<td>Client ID</td>
<td>RL</td>
<td>SNM</td>
</tr>
<tr>
<td>Sampled Date</td>
<td>12/06/2013</td>
<td>12/06/2013</td>
</tr>
<tr>
<td>Analyte</td>
<td>Method</td>
<td>Unit</td>
</tr>
<tr>
<td>Alkalinity, total</td>
<td>IALK1WATI</td>
<td>mg/L</td>
</tr>
<tr>
<td>Aluminium</td>
<td>IMET1WICCP</td>
<td>mg/L</td>
</tr>
<tr>
<td>Arsenic</td>
<td>IMET1WICMS</td>
<td>mg/L</td>
</tr>
<tr>
<td>Barium</td>
<td>IMET1WICCP</td>
<td>mg/L</td>
</tr>
<tr>
<td>Bicarbonate</td>
<td>IALK1WATI</td>
<td>mg/L</td>
</tr>
<tr>
<td>Boron</td>
<td>IMET1WICCP</td>
<td>mg/L</td>
</tr>
<tr>
<td>Cadmium</td>
<td>IMET1WICMS</td>
<td>mg/L</td>
</tr>
<tr>
<td>Calcium</td>
<td>IMET1WICCP</td>
<td>mg/L</td>
</tr>
<tr>
<td>Carbonate</td>
<td>IALK1WATI</td>
<td>mg/L</td>
</tr>
<tr>
<td>Chloride</td>
<td>ICO1WCDA</td>
<td>mg/L</td>
</tr>
<tr>
<td>Chromium</td>
<td>IMET1WICCP</td>
<td>mg/L</td>
</tr>
<tr>
<td>Cobalt</td>
<td>IMET1WICCP</td>
<td>mg/L</td>
</tr>
<tr>
<td>Copper</td>
<td>IMET1WICCP</td>
<td>mg/L</td>
</tr>
<tr>
<td>Electrical Conductivity</td>
<td>IEC1WZSE</td>
<td>mS/m</td>
</tr>
<tr>
<td>Hardness, total</td>
<td>HTG1WYACA</td>
<td>mg/L</td>
</tr>
<tr>
<td>Iron</td>
<td>IMET1WICCP</td>
<td>mg/L</td>
</tr>
<tr>
<td>Lead</td>
<td>IMET1WICMS</td>
<td>mg/L</td>
</tr>
<tr>
<td>Magnesium</td>
<td>IMET1WICCP</td>
<td>mg/L</td>
</tr>
<tr>
<td>Manganese</td>
<td>IMET1WICCP</td>
<td>mg/L</td>
</tr>
<tr>
<td>Molybdenum</td>
<td>IMET1WICMS</td>
<td>mg/L</td>
</tr>
<tr>
<td>Nickel</td>
<td>IMET1WICMS</td>
<td>mg/L</td>
</tr>
<tr>
<td>Nitrate</td>
<td>INTA1WFIA</td>
<td>mg/L</td>
</tr>
<tr>
<td>pH</td>
<td>IPHIWAGE</td>
<td>7.4</td>
</tr>
<tr>
<td>Potassium</td>
<td>IMET1WICCP</td>
<td>mg/L</td>
</tr>
<tr>
<td>Sodium</td>
<td>IMET1WICCP</td>
<td>mg/L</td>
</tr>
<tr>
<td>Sulphate (from S)</td>
<td>IMET1WICCP</td>
<td>mg/L</td>
</tr>
<tr>
<td>TDS by summation</td>
<td>InTDS_Sum</td>
<td>mg/L</td>
</tr>
<tr>
<td>Vanadium</td>
<td>IMET1WICCP</td>
<td>mg/L</td>
</tr>
<tr>
<td>Zinc</td>
<td>IMET1WICCP</td>
<td>mg/L</td>
</tr>
<tr>
<td>Aluminium</td>
<td>IMET2SAICP</td>
<td>mg/kg</td>
</tr>
<tr>
<td>Calcium</td>
<td>IMET2SAICP</td>
<td>mg/kg</td>
</tr>
<tr>
<td>Copper</td>
<td>IMET2SAICP</td>
<td>mg/kg</td>
</tr>
<tr>
<td>Iron</td>
<td>IMET2SAICP</td>
<td>mg/kg</td>
</tr>
<tr>
<td>Magnesium</td>
<td>IMET2SAICP</td>
<td>mg/kg</td>
</tr>
<tr>
<td>Potassium</td>
<td>IMET2SAICP</td>
<td>mg/kg</td>
</tr>
</tbody>
</table>

13ED491
### Appendix A Chemical Testing Results

<table>
<thead>
<tr>
<th>LAB ID</th>
<th>Client ID</th>
<th>Sampled Date</th>
<th>Method</th>
<th>Unit</th>
<th>Date 1</th>
<th>Date 2</th>
<th>Date 3</th>
<th>Date 4</th>
</tr>
</thead>
<tbody>
<tr>
<td>005</td>
<td>SNM</td>
<td>12/09/2013</td>
<td>LM725A/ICP</td>
<td>mg/kg</td>
<td>1200</td>
<td>168</td>
<td>410</td>
<td></td>
</tr>
<tr>
<td>007</td>
<td>Lan Frash</td>
<td>12/09/2013</td>
<td>LM725A/ICP</td>
<td>mg/kg</td>
<td>330</td>
<td>70</td>
<td>250</td>
<td></td>
</tr>
<tr>
<td>008</td>
<td>Heb</td>
<td>12/09/2013</td>
<td>LM725A/ICP</td>
<td>mg/kg</td>
<td>28000</td>
<td>32000</td>
<td>3300</td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Analyte</th>
<th>Method</th>
<th>Unit</th>
<th>Date 1</th>
<th>Date 2</th>
<th>Date 3</th>
<th>Date 4</th>
</tr>
</thead>
<tbody>
<tr>
<td>Total Inorganic Carbon (comb)</td>
<td>%</td>
<td>&lt;0.05</td>
<td>1.38</td>
<td>1.53</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Total Organic Carbon (comb)</td>
<td>%</td>
<td>0.05</td>
<td>0.05</td>
<td>&lt;0.05</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>LAB ID</th>
<th>Client ID</th>
<th>Sampled Date</th>
<th>Method</th>
<th>Unit</th>
<th>Date 1</th>
<th>Date 2</th>
<th>Date 3</th>
<th>Date 4</th>
</tr>
</thead>
<tbody>
<tr>
<td>008</td>
<td>Canning</td>
<td>12/09/2013</td>
<td>LM725A/ICP</td>
<td>mg/kg</td>
<td>8180</td>
<td>15700</td>
<td>1620</td>
<td></td>
</tr>
<tr>
<td>010</td>
<td>RL Tails</td>
<td>12/09/2013</td>
<td>LM725A/ICP</td>
<td>mg/kg</td>
<td>46000</td>
<td>22000</td>
<td>49</td>
<td></td>
</tr>
<tr>
<td>011</td>
<td>RL Sand</td>
<td>12/09/2013</td>
<td>LM725A/ICP</td>
<td>mg/kg</td>
<td>200</td>
<td>3.4</td>
<td>10000</td>
<td></td>
</tr>
<tr>
<td>Aluminum</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>10000</td>
<td>13000</td>
<td>160</td>
<td></td>
</tr>
<tr>
<td>Calcium</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>2540</td>
<td>2500</td>
<td>14</td>
<td></td>
</tr>
<tr>
<td>Copper</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>60</td>
<td>59</td>
<td>640</td>
<td></td>
</tr>
<tr>
<td>Iron</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>4600</td>
<td>13000</td>
<td>160</td>
<td></td>
</tr>
<tr>
<td>Magnesium</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>4600</td>
<td>13000</td>
<td>160</td>
<td></td>
</tr>
<tr>
<td>Potassium</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>200</td>
<td>3.4</td>
<td>10000</td>
<td></td>
</tr>
<tr>
<td>Silicon</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>300</td>
<td>9000</td>
<td>640</td>
<td></td>
</tr>
<tr>
<td>Strontium</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>22</td>
<td>80</td>
<td>6.9</td>
<td></td>
</tr>
<tr>
<td>Sulfur</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>7600</td>
<td>8800</td>
<td>84</td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Analyte</th>
<th>Method</th>
<th>Unit</th>
<th>Date 1</th>
<th>Date 2</th>
<th>Date 3</th>
<th>Date 4</th>
</tr>
</thead>
<tbody>
<tr>
<td>Total Inorganic Carbon (comb)</td>
<td>%</td>
<td>0.38</td>
<td>0.57</td>
<td>&lt;0.05</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Total Organic Carbon (comb)</td>
<td>%</td>
<td>&lt;0.05</td>
<td>0.17</td>
<td>&lt;0.05</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

### Method Description

- **Comb**: Carbon and Sulfur in soils by combustion, in-house method 507.
- **ICLS1WAT1**: Alkalinity (as CaCO3) and constituents by acid titration (APHA 350 B).
- **ICLS1W12DA**: Cationic analysis by DAA (Discrete Atomic Absorptometry), APHA and in-house methods.
- **ICLS1W12SE**: Electrical conductivity in water compensated to 25°C (APHA 2501B).
- **ICLS1W12WA**: Total Hardness as mg/L CaCO3 by calculation from calcium and magnesium (APHA 2540 B).
- **ICLS1W12CP**: Total dissolved metals by ICPAES (APHA 3120).
- **ICLS1W12MS**: Total dissolved metals by ICPAES (APHA 3120).
- **ICLS1W12AI**: Acid digestable metals (dry wt basis) by digestion and ICPAES (USEPA 3051A modification).
- **ICLS1W12FS**: Nitrite plus Nitrate in water by FIA expressed as Nitrate (APHA 4500N-NO3-I).
- **ICLS1W12FS**: pH in water by pH meter (APHA 4500H-P).
- **ICLS1W12FS**: Total Dissolved Solids (TDS) by summation of major ions.

*These results apply only to the sample(s) as received. Unless arrangements are made to the contrary, these samples will be disposed of after 30 days of the issue of this report. This report may only be reproduced in full.*