Heap leaching is a method for extracting metals from ores and is particularly applicable to low grade deposits due to its low operating and capital costs. The main drawback of heap leaching is the lower recoveries than more traditional techniques, such as flotation followed by smelting. The major contributing factors are inefficiencies in the liquid addition and mass transport. This thesis describes a combined experimental and theoretical modelling approach for better understanding the behaviour of these complex multiphase systems. The first major finding was that these systems exhibit hysteresis in the liquid holdup as the flow rate is varied. This means that the commonly used approach of directly correlating the flow rate and holdup is not entirely appropriate. A novel experimental and analysis procedure was used to demonstrate that the main reason for the hysteresis was an increase in the number of flow paths as the liquid flow was increased, but no subsequent decrease in the flow paths as the flow is decreased.

This work developed a theoretical liquid holdup model to describe the flow behaviour in between the particles by performing liquid flow experiments in a non-porous model glass bead system and slightly porous ore system in a way that it accounts for liquid content hysteresis in both systems. The experimental results showed the effect of inter- and intra-particle porosity on heap flow behaviour. If the inter-particle liquid content is separated from the liquid held within the ore particles, it was shown that both systems follow a similar square relationship between the two model parameters and the only difference is the pre-factor in the flow model. This demonstrated the importance of separating these two contributions to the liquid holdup when trying to make accurate predictions.

The transient flow behaviour of the packed bed systems was described both experimentally and using simulations in order to both validate the flow models and to study the behaviour during start-up and shutdown.

The salt tracer tests and Positron Emission Particle Tracking (PEPT) are independent techniques to measure the dispersion behaviour in packed beds and column leaching. It was shown that the results from these two methods are very similar, but that the PEPT results also provide more details as to the mechanisms involved.
DECLARATION

I declare that no portion of the work referred to in this thesis has been submitted in support of an application for another degree or qualification at this or any other university or institute of learning.

I. M. SAMAN K. ILANKOON

October 2012
# TABLE OF CONTENTS

ABSTRACT.......................................................................................................................... II  
DECLARATION...................................................................................................................... III  
LIST OF FIGURES ............................................................................................................... X  
LIST OF TABLES ................................................................................................................ XVIII  
ACKNOWLEDGEMENTS .................................................................................................... XIX  

CHAPTER 1 ......................................................................................................................... 1  
INTRODUCTION .................................................................................................................. 1  
  1.1. Motivation .................................................................................................................. 1  
  1.2. Organisation of the Thesis ......................................................................................... 3  

CHAPTER 2 ......................................................................................................................... 6  
LITERATURE REVIEW ...................................................................................................... 6  
  2.1. Introduction .............................................................................................................. 6  
  2.2. Minerals Processing ................................................................................................. 7  
  2.3. Heap Leaching ........................................................................................................ 8  
    2.3.1. Dump Leaching .................................................................................................. 8  
    2.3.2. Operational Procedure of Heap Leaching ......................................................... 9  
    2.3.3. Ore Preparation ............................................................................................... 9  
    2.3.4. Heap Construction .......................................................................................... 11  
    2.3.5. Solution Application ....................................................................................... 11  
    2.3.6. Pregnant Solution Collection ......................................................................... 12  
  2.4. Systems Designed to Study Heap Leaching ............................................................ 12  
  2.5. Modelling of Heap Leaching ................................................................................... 13  
    2.5.1. Particle Level Models ....................................................................................... 14  
    2.5.2. Heap Level Models ......................................................................................... 15  
  2.6. Analogous Systems to Heap Leaching ................................................................... 17  
    2.6.1. Trickle Bed Reactors ....................................................................................... 18  
    2.6.2. Ground Water Flow ....................................................................................... 19
<table>
<thead>
<tr>
<th>Section</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>2.7. Fluid Flow in Packed Beds and Heaps</td>
<td>21</td>
</tr>
<tr>
<td>2.7.1. Droplet and Rivulet Flow</td>
<td>21</td>
</tr>
<tr>
<td>2.7.2. Heap Flow Behaviour</td>
<td>22</td>
</tr>
<tr>
<td>2.7.3. Liquid Holdup in Packed Beds and Heaps</td>
<td>24</td>
</tr>
<tr>
<td>2.7.4. Liquid Holdup Models</td>
<td>25</td>
</tr>
<tr>
<td>2.8. Non-homogeneous Flow Features in Packed Beds and Heaps</td>
<td>27</td>
</tr>
<tr>
<td>2.8.1. Wall Flow</td>
<td>27</td>
</tr>
<tr>
<td>2.8.2. Uneven Solution Distribution</td>
<td>29</td>
</tr>
<tr>
<td>2.8.3. Causes of Uneven Liquid Distribution</td>
<td>30</td>
</tr>
<tr>
<td>2.8.3.1. Channelling</td>
<td>30</td>
</tr>
<tr>
<td>2.8.3.2. Locally Saturated Regions</td>
<td>31</td>
</tr>
<tr>
<td>2.8.3.3. Effects of Compaction and Consolidation of Ore</td>
<td>31</td>
</tr>
<tr>
<td>2.9. Liquid Dispersion and Mass Transport in Packed Beds and Heaps</td>
<td>31</td>
</tr>
<tr>
<td>2.9.1. Hydrodynamic Dispersion</td>
<td>32</td>
</tr>
<tr>
<td>2.9.1.1. Axial and Radial Dispersion</td>
<td>33</td>
</tr>
<tr>
<td>2.9.1.2. Peclet Number</td>
<td>33</td>
</tr>
<tr>
<td>2.9.2. Residence Time Distribution Models</td>
<td>34</td>
</tr>
<tr>
<td>2.9.2.1. Advection-Dispersion Equation (ADE)</td>
<td>35</td>
</tr>
<tr>
<td>2.9.2.2. Mobile-Immobile Model (MIM)</td>
<td>37</td>
</tr>
<tr>
<td>2.9.2.3. Mixing Flow Models</td>
<td>39</td>
</tr>
<tr>
<td>2.10. Liquid Distribution in Packed Beds and Heaps</td>
<td>39</td>
</tr>
<tr>
<td>2.10.1. Models of Solution Distribution</td>
<td>41</td>
</tr>
<tr>
<td>2.10.1.1. Random Walk</td>
<td>41</td>
</tr>
<tr>
<td>2.10.1.2. Diffusion Theory</td>
<td>42</td>
</tr>
<tr>
<td>2.10.1.3. Rivulet Model</td>
<td>43</td>
</tr>
<tr>
<td>2.11. Measuring Particle Motion using PEPT</td>
<td>44</td>
</tr>
<tr>
<td>2.12. Summary</td>
<td>45</td>
</tr>
</tbody>
</table>
CHAPTER 3

EXPERIMENTAL METHOD WITH PACKED BEDS

3.1. Introduction ................................................................. 48
3.2. 1-D Experimental Rig.................................................... 49
  3.2.1. Empty Column....................................................... 50
    3.2.1.1. Flanged Perspex Cylinders .................................. 50
    3.2.1.2. Bottom Plate for the Packed Columns....................... 51
  3.2.2. Load Cell and Data Logger ...................................... 53
  3.2.3. Liquid Distributor .................................................. 55
    3.2.3.1. Even Distribution of Water at the Top of the Packed Bed 55
    3.2.3.2. Number of Drip Points ...................................... 57
    3.2.3.3. Mounting of the Liquid Distributor.......................... 58
  3.2.4. Packing Media ...................................................... 58
    3.2.4.1. Glass Beads .................................................... 58
    3.2.4.2. Copper Ore Particles ........................................ 59
    3.2.4.3. Voidage and Porosity ........................................ 61
    3.2.4.4. Limitations of This Study ................................... 62
  3.3. Experimental Conditions ........................................... 63
  3.4. Liquid Holdup Measurements with Non-Porous Particles ....... 64
    3.4.1. Steady State Liquid Holdup .................................... 64
      3.4.1.1. Gravimetric Method ....................................... 65
      3.4.1.2. Drainage Method .......................................... 65
      3.4.1.3. Accuracy of the Measurement Techniques ............... 66
    3.4.2. Residual Liquid Holdup ........................................ 67
  3.5. Liquid Holdup Measurements with Porous Ore Particles ....... 68
  3.6. Hydrodynamic Dispersion Measurements .......................... 69
    3.6.1. Tracer Type and Injection ..................................... 69
    3.6.2. Measurement of the Effluent Tracer Concentration ......... 70
    3.6.3. Data Acquisition System ...................................... 74
    3.6.4. Method of Salt Tracer Tests .................................. 74
  3.7. Summary ...................................................................... 75
# Table of Contents

**CHAPTER 4**

HYSTERESIS AND INTER-PARTICLE FLOW MODEL WITH THE MODEL SYSTEM .................................. 77

4.1. Introduction .................................................................................................................. 77
4.2. Steady State Liquid Holdup ....................................................................................... 78
4.3. Liquid Holdup Hysteresis ......................................................................................... 80
4.4. Understanding of Liquid Holdup Hysteresis ............................................................ 86
4.5. Theoretical Modelling of Flow Paths ........................................................................ 87
   4.5.1. Observations of Rivulet Flow ........................................................................... 87
   4.5.2. Residual Liquid Holdup of the Model System .................................................. 89
   4.5.3. Explanation of Liquid Holdup Hysteresis ......................................................... 92
   4.5.4. Inter-Particle Flow Model .................................................................................. 94
       4.5.4.1. Effect of Particle Size ................................................................................ 100
       4.5.4.2. Estimating the Effect of the Contact Angle ............................................... 101
       4.5.4.3. Effect of the Rivulet Orientation .................................................................. 105
       4.5.4.4. Size and Volume of the Residual Liquid Connections ................................ 106
4.6. Flow Behaviour in Poly-Dispersed Model System ..................................................... 107
4.7. Summary .................................................................................................................... 110

**CHAPTER 5**

LIQUID HOLDUP RESULTS FOR THE ORE SYSTEM ................................................................. 111

5.1. Introduction ................................................................................................................ 111
5.2. Liquid Holdup of the Ore Bed ................................................................................... 112
   5.2.1. Liquid Holdup Hysteresis .................................................................................. 115
5.3. Steady and Residual Liquid Contents ....................................................................... 115
   5.3.1. Steady State External Liquid Holdup .................................................................. 117
   5.3.2. Residual Liquid Holdup ..................................................................................... 118
5.4. Modelling of Flow Paths .......................................................................................... 123
   5.4.1. Validity of the Inter-Particle Flow Model ......................................................... 125
5.5. Results of the Poly-Dispersed Ore Beds ................................................................... 127
5.6. Comparison of the Model and the Ore Systems ......................................................... 130
   5.6.1. Pre-Factor .......................................................................................................... 132
   5.6.2. Residual Liquid Holdup ..................................................................................... 133
5.7. Summary .................................................................................................................... 135
# Table of Contents

## CHAPTER 6

**TRANSIENT BEHAVIOUR IN THE MODEL GLASS BEAD AND ORE SYSTEMS**

6.1. Introduction ................................................................. 137
6.2. Transient Changes of Liquid Holdup .................................... 138
6.3. Movement of the Wetting Front for the Model Glass Bead System .................. 139
6.4. Simulation of Liquid Drainage for the Ore System ......................... 144
6.5. Summary .......................................................................... 153

## CHAPTER 7

**HYDRODYNAMIC DISPERSION ANALYSIS** ........................................ 154

7.1. Introduction .......................................................................... 154
7.2. Residence Time Distribution Analysis .......................................... 155
7.3. Numerical Solution of Mobile-Immobile Model .............................. 158
7.4. Analysis of Axial Dispersion Coefficient ...................................... 162
7.5. Mobile and Static Liquid Fraction ............................................. 167
    7.5.1. Static versus Residual Liquid Holdup .................................... 170
7.6. Mass Transfer Coefficient ...................................................... 171
7.7. Summary .......................................................................... 173

## CHAPTER 8

**POSITRON EMISSION PARTICLE TRACKING (PEPT) IN PACKED BEDS** ......... 175

8.1. Introduction .......................................................................... 175
8.2. Positron Emission Particle Tracking (PEPT) .................................... 176
    8.2.1. Basics of PEPT ............................................................ 176
    8.2.2. Applications of PEPT .................................................... 178
8.3. PEPT Trajectories in Packed Beds of Particles ............................... 179
8.4. Experimental Method for PEPT ................................................ 180
8.5. PEPT Data Analysis .............................................................. 186
    8.5.1. Time Weighting Function ................................................. 188
    8.5.2. Smoothed Pept Trajectory ............................................... 193
8.6. Dispersion Coefficients from the Smoothed PEPT Data .................... 194
    8.6.1. Comparison of Axial Dispersion with Salt Tracer Method .......... 198
8.7. Summary .......................................................................... 199
<table>
<thead>
<tr>
<th>Chapter</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>9.1. Conclusions</td>
<td>201</td>
</tr>
<tr>
<td>9.1.1. Liquid Holdup Hysteresis</td>
<td>201</td>
</tr>
<tr>
<td>9.1.2. Inter-Particle Flow Model</td>
<td>202</td>
</tr>
<tr>
<td>9.1.3. Effect of Particle Porosity</td>
<td>202</td>
</tr>
<tr>
<td>9.1.4. Transient Flow Behaviour</td>
<td>203</td>
</tr>
<tr>
<td>9.1.5. Hydrodynamic Dispersion Measurements</td>
<td>204</td>
</tr>
<tr>
<td>9.2. Future Work</td>
<td>204</td>
</tr>
<tr>
<td>9.2.1. Improved Modelling and Simulation of Industrial Heap Leaching</td>
<td>205</td>
</tr>
<tr>
<td>9.2.2. More Realistic Particles and Packings</td>
<td>205</td>
</tr>
<tr>
<td>9.2.3. Residual and Intra-Particle Liquid Holdup</td>
<td>206</td>
</tr>
<tr>
<td>9.2.4. 2-D Liquid Flow Behaviour</td>
<td>206</td>
</tr>
<tr>
<td>9.2.5. PEPT Technique to Map The Flow Behaviour in Packed Beds</td>
<td>206</td>
</tr>
</tbody>
</table>

REFERENCES | 208

NOMENCLATURE | 222

ABBREVIATIONS | 229

APPENDICES | 230
LIST OF FIGURES

Figure 2.1: The relationship of the particle size and grade of the mined ore on various leaching operations (Dresher, 2004). ................................................................. 8

Figure 2.2: The operations of a heap leaching plant. Figure shows the initial and subsequent operations of copper heap leaching (GE Infrastructure, http://www.gewater.com/pdf/Bulletin968EN.pdf). ................................................................. 10

Figure 2.3: Shrinking core model describing the dissolution of oxide copper ore. Figure shows the leached rim and the unreacted core. The concentration gradients of copper and the reagent (i.e. A in this figure) are also given (Bartlett, 1992a). ......................................................... 15

Figure 2.4: One dimensional heap level model by dividing the heap into square columns (Roman et al., 1974). .............................................................................................. 16

Figure 2.5: Macro level (left) and micro level (right) flow features of the packed beds (Ng and Chu, 1987; Maiti et al., 2006). ................................................................. 21

Figure 2.6: The solution flow channels through the rock particles of a heap (Petersen and Petrie, 2010). ........................................................................................................ 23

Figure 2.7: The particle segregation during the heap construction (Yusuf, 1984: p. 12). ...... 30

Figure 2.8: Schematic representation of the advection-dispersion model. ......................... 36

Figure 2.9: Schematic representation of the mobile-immobile model (de Andrade Lima, 2006). .................................................................................................................. 38

Figure 2.10: Schematic representation of the mixing flow model during step change of the tracer concentration (Bouffard and West-Sells, 2009). ......................................................... 39

Figure 2.11: Tracer particle trajectory within the field of view of the positron camera (Cole, 2011). ...................................................................................................................... 45

Figure 3.1: 1-D column leaching rig used in this study. .......................................................... 49

Figure 3.2: 300 mm (left) and 500 mm (right) cylindrical Perspex sections. ......................... 50

Figure 3.3: Design and the main components of the bottom plate including the drainage pathways and the O-ring. ................................................................. 51

Figure 3.4: Perspex and steel bottom plates are shown separately. ....................................... 52

Figure 3.5: The bottom plate attached to the 300 mm Perspex column with the stud sections to mount the column to the metal frame. ...................................................... 52
Figure 3.6: The top section of the experimental rig. The safety nuts and the load cell are shown. ................................. 53

Figure 3.7: Schematic representation of the data acquisition system to record the load cell data. ................................................................. 54

Figure 3.8: The calibration curve of the load cell and data logger system ................................................................. 55

Figure 3.9: The tube network used in the liquid distributor. ................................................................. 56

Figure 3.10: Vertically mounted drip points (left) and the total liquid distributor of 32 drip points mounted over the column (right). .................................................................................. 56

Figure 3.11: Glass beads of different sizes that used in this experimental study to make the model system .................................................................................. 59

Figure 3.12: Particle size distribution of the original copper ore sample. ................................. 60

Figure 3.13: Copper ore particles of selected narrow size ranges used in this study. ................ 61

Figure 3.14: Experimental set-up to measure the drained liquid from the column using an electronic balance .................................................................................. 65

Figure 3.15: Holdup variation of drainage and gravimetric methods for all the particle sizes of the model system. The packed bed was initially dry and superficial velocity was 0.0075 mm/s. a) 2 mm b) 10 mm c) 14 mm d) 18 mm. ................................................................. 67

Figure 3.16: Determination of steady and residual liquid holdup values of the model system in 500 mm column packed with 2 mm particles .................................................................................. 68

Figure 3.17: The scaled effluent tracer conductivity curves for different tracer volumes injected at the inlet of the column. The 300 mm column packed with 18 mm particles was used and the liquid superficial velocity was 0.06 mm/s .................................................................................. 70

Figure 3.18: Residence time distribution curves using different small reservoirs at the outlet of the column. The 300 mm column packed with 18 mm particles was used and the liquid superficial velocity was 0.03 mm/s .................................................................................. 71

Figure 3.19: Schematic representation of the small reservoir with parameters to perform conductivity corrections. .................................................................................. 72

Figure 3.20: Comparison of the corrected and measured RTD curves for 300 mm packed bed of 18 mm particles at superficial velocities of 0.03 mm/s (left) and 0.12 mm/s (right). ................................. 73

Figure 3.21: The components of the data acquisition system used to record the effluent tracer conductivity .................................................................................. 74

Figure 3.22: Effluent conductivity measuring system at the bottom of the packed bed ............ 75
Figure 4.1: Total liquid holdup for all the particle sizes in 300 mm column using the dry bed start-up condition. ................................................................. 78

Figure 4.2: Total liquid holdup for all the particle sizes in 300 mm column using the wet bed start-up condition. ................................................................. 79

Figure 4.3: Comparison of dry bed and wet bed liquid holdups in the model system for all the particle sizes in 300 mm column. ................................................................. 79

Figure 4.4: Increasing and decreasing arms of flow rate and first cycle of liquid holdup measurements using 18 mm particles in 300 mm column. ................................................................. 80

Figure 4.5: Dry bed liquid holdup hysteresis for all the particle sizes in 300 mm column. a) 2 mm b) 10 mm c) 14 mm d) 18 mm. ................................................................. 81

Figure 4.6: Wet bed liquid holdup hysteresis for all the particle sizes in 300 mm column. a) 2 mm b) 10 mm c) 14 mm d) 18 mm. ................................................................. 82

Figure 4.7: Dry bed liquid holdup variation in the first three cycles of operation in 300 mm bed (C1, C2 and C3 indicate first, second and third cycles). a) 14 mm particles b) 18 mm particles................................................................. 83

Figure 4.8: Wet bed liquid holdup variation in first three cycles of operation in 300 mm bed (C1, C2 and C3 indicate first, second and third cycles). a) 2 mm particles b) 18 mm particles.... 84

Figure 4.9: The effect of the maximum flow rate on liquid holdup hysteresis behaviour. The 300 mm dry packed bed with dry 18 mm beads was wetted as listed in Table 4.1................. 85

Figure 4.10: Initial liquid introduction into the top surface of the packed bed. ....................... 88

Figure 4.11: Random flow behaviour of rivulets through liquid filled connections between the 18 mm particles. ........................................................................ 88

Figure 4.12: Rivulet flow behaviour through the 18 mm particles of the packed bed. ............. 89

Figure 4.13: Steady and residual liquid holdup during the increasing and decreasing flow rates in 500 mm column packed with 2 mm particles. The superficial velocities in mm/s are shown................................................................. 90

Figure 4.14: Residual holdup variation of all the particle sizes in 500 mm dry packed bed for both increasing and decreasing arms of flow. a) 2 mm b) 10 mm c) 14 mm d) 18 mm. ............ 91

Figure 4.15: The variation of $v_\ast$ against $\theta^*$ in the increasing and decreasing arms of flow for all the particle sizes in 500 mm packed bed. a) 2 mm b) 10 mm c) 14 mm d) 18 mm............ 94

Figure 4.16: Schematic representation of the flow behaviour of a rivulet. The liquid reservoirs and the flowing liquid are shown. ................................................................. 95
Figure 4.17: The relationship between the relative flow rate ($v^*_s$) and the additional liquid content of that rivulet ($\theta^* - 1$) for dry bed start-up condition of the model system. .................. 97

Figure 4.18: The relationship between the relative flow rate ($v^*_s$) and the additional liquid content of that rivulet ($\theta^* - 1$) for wet bed start-up condition of the model system. .................. 98

Figure 4.19: The relationship between the relative flow rate ($v^*_s$) and the additional liquid content of that rivulet ($\theta^* - 1$) for all the particle sizes of the model system in both dry and wet bed start-up conditions. .......................................................................................................................... 99

Figure 4.20: The pre-factor of the inter-particle flow model as a function of the particle size of the mono-dispersed packed bed on the dry bed start-up. .......................................................... 101

Figure 4.21: Schematic representation of rivulet flow behaviour with tortuous flow paths between the two liquid filled connections. ...................................................................................... 102

Figure 4.22: Schematic representation of the cross-section of the flowing rivulet over the surface of the glass bead. ................................................................................................................. 104

Figure 4.23: The dimensionless drag coefficient as a function of the contact angle of the rivulets ($k_T = 1$ and $g^* = g$). ............................................................................................................. 105

Figure 4.24: The variation of the parameter, $A_r/C_{drag}$, against the particle size of the model system for different packed bed heights ........................................................................................................... 107

Figure 4.25: The relationship between $v^*_s$ and $\theta^* - 1$ for dry bed start-up condition using the poly-dispersed beds of non-porous glass beads. All the mixtures were prepared by mixing equal weights from each particle size. ........................................................................................................... 109

Figure 5.1: The initial liquid flow behaviour through dry ore particles in 300 mm column. The particle size range of the ore particles was 8-11.2 mm. ......................................................................................... 112

Figure 5.2: Comparison of liquid contents between the model and ore systems in 300 mm column which were started dry. ............................................................................................................... 113

Figure 5.3: Contribution of internal pore volume on the total liquid holdup in the ore system. 114

Figure 5.4: Liquid holdup hysteresis of the initially dry ore particles in 300 mm column. ....... 115

Figure 5.5: Variation of the liquid holdup during the drainage in the 300 mm column at superficial velocity of 0.12 mm/s containing 10 mm glass beads (left) and 8-11.2 mm ore particles (right). ......................................................................................................................... 119

Figure 5.6: Comparison between the drainage velocity from glass beads and ore particles in 300 mm column. Initial liquid addition rate was 0.12 mm/s................................................................. 120
Figure 5.7: Drainage velocity for the 300 mm column containing 8-11.2 mm ore particles at superficial velocity of 0.12 mm/s (log-normal axes). ........................................................................................................ 120

Figure 5.8: Actual and estimated liquid holdup for 8-11.2 mm ore particles during the drainage in 300 mm column at a superficial velocity of 0.12 mm/s. ......................................................... 122

Figure 5.9: End drainage velocity of different ore fractions at different superficial velocities in the 300 mm packed bed. ........................................................................................................ 123

Figure 5.10: Liquid holdup hysteresis of pore saturated externally dried ore particles in 300 mm packed bed. ........................................................................................................ 124

Figure 5.11: Variation of $v_s^*$ against $\theta^*$ for the increasing and decreasing arms of the flow for different size ranges of ore particles in 300 mm column. a) 4-8 mm b) 13.2-16 mm c) 20-26.5 mm d) 37.5-45 mm. ........................................................................................................ 125

Figure 5.12: The relationship between the relative flow rate ($v_s^*$) and the additional liquid content of that rivulet ($\theta^* - 1$) for all the size fractions of ore particles. ..................................... 126

Figure 5.13: The relationship between $v_s^*$ and $\theta^* - 1$ for the binary mixture of 8-11.2 mm and 16-20 mm. The same relationship for the individual size fractions is also shown........... 128

Figure 5.14: The relationship between $v_s^*$ and $\theta^* - 1$ for the binary mixture of 11.2-13.2 mm and 20-26.5 mm. The same relationship for the individual size fractions is also shown. .. 129

Figure 5.15: The relationship between $v_s^*$ and $\theta^* - 1$ for the binary mixture of 13.2-16 mm and 26.5-31.5 mm. The same relationship for the individual size fractions is also shown........... 129

Figure 5.16: The relationship between $v_s^*$ and $\theta^* - 1$ for the binary mixture of 16-20 mm and 31.5-37.5 mm. The same relationship for the individual size fractions is also shown....... 130

Figure 5.17: The relationship between the relative flow rate ($v_s^*$) and the additional liquid content of that rivulet ($\theta^* - 1$) for both model and ore systems. The model system was at dry bed start-up condition. The ore particles were initially saturated and then externally dried.................................................................................................................................. 132

Figure 5.18: Comparison of the pre-factor ($K$) in the model and the ore systems.................. 133

Figure 5.19: Variation of the residual liquid holdup as a function of geometric mean size of the different ore size fractions in 300 mm column. ................................................................. 134

Figure 5.20: Variation of the residual liquid holdup as a function of geometric mean size of the ore size fractions and binary mixtures in 300 mm column. ................................................................. 134

Figure 6.1: Schematic illustration of liquid holdup variation in the periodic operation (Liu et al., 2009). ........................................................................................................ 139
Figure 6.2: Schematic illustration of the mechanisms within a standing wave. .......................... 140

Figure 6.3: Schematic illustration of standing waves for different column lengths.................. 141

Figure 6.4: Drained weight of water for 10 mm particles in different column lengths for a superficial velocity of 0.0075 mm/s. a) as a function of time b) as a function of time shifted relative to 800 mm column.................................................................................................................. 142

Figure 6.5: Drained weight of water for 14 mm particles in different column lengths for a superficial velocity of 0.0075 mm/s. a) as a function of time b) as a function of time shifted relative to 800 mm column.................................................................................................................. 143

Figure 6.6: Simulated and experimental drainage curves for different superficial velocities with 8-11.2 mm ore particles in 300 mm column (solid line is simulated, diamonds are the experimental results). a) 0.12 mm/s b) 0.03 mm/s.................................................................................................................. 147

Figure 6.7: Simulated and experimental liquid contents during the drainage for different superficial velocities with 8-11.2 mm ore particles in 300 mm column. a) 0.12 mm/s b) 0.03 mm/s.................................................................................................................. 148

Figure 6.8: External liquid content of 8-11.2 mm ore particles in 300 mm column as a function of column height for the superficial velocities of: a) 0.12 mm/s b) 0.03 mm/s. ........ 150

Figure 6.9: External liquid content of 8-11.2 mm ore particles in 300 mm column as a function of column height for the superficial velocities of: a) 0.015 mm/s b) 0.0075 mm/s. ... 151

Figure 6.10: Simulated and experimental drainage curves with 8-11.2 mm ore particles in 300 mm column for the superficial velocities of: a) 0.015 mm/s b) 0.0075 mm/s. ................ 152

Figure 7.1: Effluent tracer conductivity variation with time in 300 mm column packed with 18 mm glass beads.................................................................................................................. 155

Figure 7.2: Normalised tracer response curves for 300 mm column packed with 18 mm glass beads.................................................................................................................. 156

Figure 7.3: Cumulative RTD curves for 300 mm column packed with 18 mm glass beads....... 157

Figure 7.4: The mobile-immobile model fitted to the experimental data of 18 mm particles at a superficial velocity of 0.015 mm/s in dry bed operation using a 300 mm column. ........... 161

Figure 7.5: The mobile-immobile model fitted to the experimental data of 18 mm particles at a superficial velocity of 0.03 mm/s in dry bed operation using a 300 mm column. ............. 161

Figure 7.6: Liquid holdup values measured gravimetrically during the salt tracer tests for the first cycle of operation in dry and wet bed start-up conditions using 18 mm particles in 300 mm column. .................................................................................................................. 163
Figure 7.7: Mobile liquid velocities at each superficial liquid velocity based on the liquid contents in Figure 7.6................................................................. 164

Figure 7.8: Variation of axial dispersion coefficient in 300 mm column packed with 18 mm glass beads as a function of: a) superficial liquid velocity b) mobile region liquid velocity. ...... 165

Figure 7.9: Variation of axial dispersion coefficient in 300 mm column packed with 10 mm glass beads as a function of: a) superficial liquid velocity b) mobile region liquid velocity. ...... 166

Figure 7.10: Mobile liquid holdup (a) and mobile liquid fraction as a percentage (b), in 300 mm column packed with 14 mm glass beads................................................................. 168

Figure 7.11: Static liquid holdup variation for both dry and wet bed start-up conditions in 300 mm column packed with 14 mm glass beads................................................................. 169

Figure 7.12: Relationship between the residual and static liquid contents of 18 mm particles in 300 mm column for both dry and wet bed start-up conditions................................. 170

Figure 7.13: Mass transfer coefficient between the mobile and immobile regions as a function of superficial liquid velocity a) 14 mm b) 10 mm glass beads in 300 mm column. ...... 172

Figure 8.1: Detected line of responses (LoRs) of the tracer particle and the corrupted events with PEPT (Cole, 2011).................................................................. 177

Figure 8.2: Final position of the tracer particle using the triangulation algorithm (Cole, 2011).178

Figure 8.3: PEPT camera located in iThemba Labs, South Africa. The 300 mm column packed with 18 mm particles is shown inside the positron camera................................. 180

Figure 8.4: Empty 300 mm Perspex column with bottom plate inside the PEPT gantry. ......... 181

Figure 8.5: Schematic representation of the PEPT coordinate system used in this work relative to the empty Perspex column inside the PEPT gantry. ................................. 182

Figure 8.6: Empty column (300 mm) mapping inside the active area of the positron camera.. 184

Figure 8.7: The 300 mm packed bed with 18 mm particles and mounted liquid distributor inside the PEPT gantry. ................................................................. 184

Figure 8.8: Images of the tracer particle. The top image shows the scale of the tracer particle. ................................................................. 185

Figure 8.9: High frequency noise measurements when the tracer particle was stationary in the collection tray. The standard deviations of the y and z data are 850 µm and 450 µm respectively ................................................................. 187

Figure 8.10: The kernel for a weighting function composed of cubic splines for interpolating and smoothing the original PEPT data (Cole, 2010c: p. 119). ................................................................. 188
Figure 8.11: High frequency noise measurements and the smoothed coordinates when the tracer particle was stationary in the collection tray (black line illustrates the deviations from the average tracer particle position for the smoothed PEPT data). The standard deviations of the smoothed $y$ and $z$ data are 360 µm and 200 µm respectively. .............................................. 191

Figure 8.12: Graphs of the $y$, $x$, $z$ positions of the tracer particle measured with PEPT as a function of time during a period of 4.5 seconds (only a part of a trajectory to show noise removal). The top graph is the vertical ($y$) direction and the middle and the bottom graphs are horizontal $x$ and $z$ directions respectively. The original data is shown on left and the smoothed data with a time weighting kernel width of 200 milliseconds is on the right............. 192

Figure 8.13: The PEPT tracer trajectories of the tracer particles through a 300 mm column packed with 18 mm glass beads (blue-450 µm, red-420 µm, magenta-380 µm, black-370 µm). .................................................................................................................................................... 193

Figure 8.14: The effect of the time base on the calculated axial dispersion coefficient........ 194

Figure 8.15: Graph showing the $y$ coordinates of the tracer descent within the packed bed. Superficial velocity of the liquid is 0.12 mm/s................................................................. 196

Figure 8.16: Smoothed mobile tracer trajectories in $y$-direction downwards through the 18 mm glass beads in 300 mm column after removing the stationary location data............ 197

Figure 8.17: Variation of the axial and radial dispersion coefficients for 18 mm glass beads in 300 mm column based on the PEPT location data of the tracer particle......................... 197

Figure 8.18: Comparison of the axial dispersion coefficients for 18 mm glass beads in 300 mm column calculated using the RTD and PEPT methods. Figure also shows radial dispersion coefficients with PEPT............................................................... 198
# LIST OF TABLES

Table 2.1: The operational parameters in trickle bed reactors. ................................................................. 18
Table 2.2: Liquid holdup correlations for column leaching and trickle bed reactors. ................................. 26
Table 2.3: Variation of liquid holdup with experimental variables of the system. ................................. 27
Table 2.4: The ratio of $d_R/d_p$ values to minimise the wall effect ......................................................... 28
Table 2.5: Axial dispersion correlations for column leaching and trickle bed reactors. ......................... 34
Table 2.6: Experimental studies using advection-dispersion model to calculate the axial dispersion coefficient ........................................................................................................................................... 36
Table 3.1: Resolution and accuracy of the connected system. ......................................................................... 54
Table 3.2: Comparison of the number of drip points per square metre for this study and Ter Veer et al. (1980). .............................................................................................................................................. 57
Table 3.3: Experimentally determined voidage and porosity values of the packed beds. ....................... 62
Table 3.4: Summary of the experimental conditions in this study ................................................................. 64
Table 4.1: Set of flow rates and the sequence of flow rate during the experimental run. ......................... 85
Table 4.2: Actual slopes of the lines between $v_s^*$ and $\theta^* - 1$ in Figure 4.19 for all the particle sizes and different column lengths of the model system in both dry and wet bed start-up conditions .............................................................................................................................................. 100
Table 4.3: Pre-factor of the mono-dispersed and poly-dispersed model systems. .................................. 109
Table 5.1: Comparison of the breakthrough times for the same particle size in the model and ore systems. ................................................................................................................................................. 117
Table 5.2: Comparison of the breakthrough times for all the particle sizes in the model and ore systems. ................................................................................................................................................. 118
Table 5.3: Actual slopes of the lines between $v_s^*$ and $\theta^* - 1$ in Figure 5.12 for all the particle sizes in the ore system in 300 mm column. The ore particles were initially saturated and externally dried before placed in the 300 mm column. ........................................................................................................................................... 127
Table 5.4: Bond numbers of the model and the ore systems using particle size as the characteristic length scale ................................................................................................................................................. 131
Table 7.1: Calculated $t_R$ values from both liquid holdup measurements and RTD curves. ........... 158
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1.1. MOTIVATION

Mined high grade metallic ores can be treated easily by using conventional mineral processing techniques. As an example, many mines perform milling followed by agitation leaching for high grade gold ores or for base metals, milling followed by flotation and then smelting. The situation is dramatically different for low grade ores, because it is not possible to treat these ores economically using the same processes. The high capital and operating cost associated with milling operations means that the operator will not recover sufficient value from low grade sources.

Decreasing availability of high grade mineral resources and increasing mineral consumption are forcing engineers to mine lower grade deposits. This needs to be done with economic and environmental requirements for reduced energy consumption. Heap leaching is a hydrometallurgical process that can potentially achieve this. The Run-of-Mine (ROM) ore or the crushed ore is piled to construct the heaps and the leaching solution is applied at the top. Finally, the pregnant solution enriched with dissolved metals is collected from the bottom for the extraction of valuable metals.

In addition, small ore reserves favour the use of heap leaching, while milling plants are favourable to deal with the large reserves where the capital outlay can be justified. Thus mines with low grade ores or relatively limited ore reserves tend to utilise heap leaching because it is the only mineral processing technique available to treat them (Bartlett, 1992a). The overburden rock volumes which contain very low metal grade have been discarded as waste over many years of the life of mine. These rock volumes contain millions of tons of
valuable metal and the extraction of metals from these waste rocks is now desired. Therefore, treating of mine overburden is another application of leaching (often referred to as dump leaching).

Despite its suitability for low cost processing of low grade ores, its efficiency is less than that of more traditional methods such as froth flotation followed by smelting. Adverse flow behaviour within the heaps is a major factor leading to the lower extraction and thus knowledge of the flow behaviour within heaps is vital to improve the efficiency. Due to the low liquid addition rates in the heap leaching, the flow is unsaturated and it is complex. However, the hydrodynamics of column and heap leaching has not been investigated extensively compared with the reaction kinetics of heap leaching (de Andrade Lima, 2006). Therefore, a sound knowledge of the hydrodynamics within the heaps is one of the key areas to design, scale up and optimise the efficiency of the process.

The first step in this project is to design and build an experimental rig that would allow the liquid holdup in a packed column to be accurately measured as the liquid addition is varied. Several authors have pointed out the existence of liquid holdup and pressure drop hysteresis in trickle bed reactors (eg. Kan and Greenfield, 1978, 1979; Levec *et al.*, 1986; Levec *et al.*, 1988; Lazzaroni *et al.*, 1989, Maiti *et al.*, 2006; Maiti *et al.*, 2008) and the heap leaching systems are analogous enough for hysteresis in the fluid flow to be anticipated despite the significant differences between the two systems. The second aim of the project is to investigate the liquid holdup hysteresis in column leaching through a novel experimental methodology, followed by theoretical modelling to explain the dominant mechanisms and predict the behaviour.

The porosity of the packed particles has two significantly different length scales, which are the pore spaces within the particles and the inter-particle spaces between the particles (i.e. interstitial space). Therefore, this distinct separation of length scales implies that liquid content within the particles will not have the same effect on liquid flow as the liquid holdup between the particles and must thus be considered separately. The third aim of this study is to experimentally investigate and model the effect of the particle porosity on the overall flow through a heap by comparing the behaviour of a bed consisting of non-porous glass beads with a system consisting of similar sized ore particles.
Hydrodynamic dispersion is a major concern for the mass transport within a heap. This is because it will influence the transport of both reagent species and the dissolved metal species. In this project the dispersion behaviour was studied using salt tracers.

There is still debate as to the mechanisms at work during the mass transfer of solutes and the hydrodynamic dispersion. The main reason is that the flow behaviour has been inferred from dispersion measurements obtained by measuring the spread of a soluble tracer such as a salt or a dye. Positron Emission Particle Tracking (PEPT) is a novel technique to investigate the flow patterns in the opaque systems and it has found several applications including mineral processing techniques such as froth flotation and tumbling mills. Finally, in this work the hydrodynamic dispersion coefficient in an unsaturated packed bed is obtained by monitoring the motion of a neutrally buoyant tracer particle as it moves through the bed. The tracer particle motion obtained from PEPT technique was used to determine the dispersion coefficients in the packed beds. The values are compared to that obtained by measuring hydrodynamic dispersion using a salt tracer.

The achievement of these goals will provide a much better understanding of the underlying flow mechanisms in column and heap leaching. The liquid holdup model developed from the experimental results of this work can be incorporated into future computational models that can be used to describe overall performance of the heap and thus be used to improve and optimise the design and operation of these systems.

1.2. ORGANISATION OF THE THESIS

The thesis is organised as follows. Chapter 2 is a review of literature relevant to the work carried out in this study. The significance of the heap leaching process is explained, together with the fundamentals of the technique. As this study focuses on the fluid flow behaviour, the systems used to study heap hydrodynamics are explained. This is followed by an explanation of current particle and heap level models that describe the heap performance. In addition, some analogous systems to heap leaching are cited. In order to describe the fluid flow, the dominant liquid flow features are identified, followed by a summary of previous liquid holdup studies and models. Finally, the mass transfer and the hydrodynamic dispersion mechanisms are presented together with the relevant models.
Chapter 3 presents a description of the 1-D column leaching rig commissioned for this work. It includes cylindrical Perspex columns of 300 mm and 500 mm in length, a bottom plate, a load cell and a data logger system and a liquid distributor. In addition, the packing media used in this experimental study are described and those are non-porous glass beads and narrow size range of copper ore particles. The steady state and residual liquid holdup measurement techniques are described for both systems, followed by a description of the methodology of the hydrodynamic dispersion experiments using a salt tracer.

In Chapter 4, the results of steady state liquid holdup tests for the model system are presented and the existence of liquid holdup hysteresis behaviour is shown for different particle sizes. By following a novel methodology to determine the steady state and residual liquid holdups, the liquid holdup hysteresis behaviour is explained. In addition, the derivation of the theoretical inter-particle flow model is given to obtain a relationship between the flow rate and the liquid content of the rivulets, followed by an analysis of the liquid holdup results according to the model.

Chapter 5 is an analysis of the liquid holdup results in the ore system and it also includes a comparison between the model and ore systems. The applicability of the inter-particle flow model in the porous ore system is investigated with a novel experimental method, which was used to measure the steady state and residual liquid contents. The experimental results of the ore bed are described according to the flow model similar to the model system. This is followed by a comparison of the results between the two systems.

In Chapter 6, the transient liquid holdup behaviour is studied. In particular, the initial wetting of bed when the liquid addition is first turned on and the final drying when the liquid addition is turned off is studied. A simplified transient model is used to describe this behaviour.

Chapter 7 presents the results of the hydrodynamic dispersion experiments conducted in this study. It includes an analysis of the accuracy of the resultant residence time distribution (RTD) curves. The mobile-immobile model equations are described, which were used to numerically solve the RTD curves in order to determine the axial dispersion coefficients. In addition, this chapter includes a description of the characterisation of the dispersion coefficients with the appropriate characteristic velocity in the packed bed.
Chapter 1: Introduction

Chapter 8 gives the results of tracking tracer particles with PEPT. The analysis of the tracer particle trajectories is described using PEPT location data, followed by a description of the high frequency noise removal using a smoothing function. The calculation of both the axial and radial dispersion coefficients is presented using the smoothed location data. In addition, the axial dispersion coefficients given by PEPT are compared with the same given by salt tracer tests in Chapter 7.

Chapter 9 is the final chapter of this work and presents conclusions and future work.

The appendix presents the experimental data, the detailed derivations of the equations used in the analysis of the results and the details of the journal publications developed during this research. One has been peer-reviewed and published. Two papers have been submitted for peer review.


CHAPTER 2

LITERATURE REVIEW

2.1. INTRODUCTION

The objective of this study is the investigation of the hydrodynamics of heap leaching, which is one of the hydrometallurgical techniques used to extract metals from low grade ores. However, despite the acknowledged importance of hydrodynamics to heap flow behaviour, there have been few studies in this area. This study aims to provide insights into heap flow behaviour through the use of laboratory scale experiments.

This chapter will introduce the various operational stages of heap leaching before giving a detailed description of the current understanding of the underlying flow mechanics involved. There are also a wide range of systems of different scale used to study heap hydrodynamics and these will also be described. The modelling of heap leaching is an important method used to investigate phenomena that occur at an industrial scale. The fundamental heap leach models, which have been developed on both particle and heap scales, will be discussed. The similarities between the flow behaviour of this system and that of trickle bed reactors and ground water flow can also be used for better understanding of heap flow mechanics and hence, they will be treated as analogous systems.

The main features of the understanding of heap hydrodynamics are fluid flow, liquid dispersion and mass transport in packed beds and heaps. The fluid flow behaviour will be described with respect to the flow features found in beds of particles, which are of a similar size range to those employed in industrial heap leaching. This study will also describe important aspects of liquid holdup. Finally, the principles of hydrodynamic dispersion and mass transfer will be presented together with the relevant models from the literature in
order to explain the significance of the techniques that can be used to describe heap hydrodynamics.

2.2. MINERALS PROCESSING
Mineral consumption is growing significantly as world population is growing faster than at any time in history. In addition, the global standard of living is increasing and will affect future mineral demand more than the growing population (Kesler, 2007). As an example, copper and copper alloy products are used mainly in building construction, electric and electronic products, transportation, consumer and general products and industrial machinery and equipment. The consumption of copper was projected to increase by 1.5% in 2011 (Edelstein, 2012). Global mine production of copper was 15.9 million metric tons in 2010 and 16.1 million metric tons in 2011 (Edelstein, 2012). These figures highlight the necessity of mineral production to deal with the growing consumer demand.

Generally, global mineral reserves are enough to fulfil the consumer demand of minerals for the next 50 years. However, mining and minerals processing engineers must produce about 60% higher mineral demand than today if mineral demand increases at the current rate (about 1% annually) (Kesler, 2007). This target is not easy to supply as the grade of identified global mineral reserves is declining and this implies that these lower grade ores need to be processed efficiently.

Although various mineral processing techniques are used to extract desired metals from Run-of-Mine (ROM) material while discarding waste or gangue minerals, it is important to remember that there are a few techniques that are profitable for low grade ores. Heap leaching is one such technique available to economically treat low grade ores. Typically, mines with relatively limited low grade ore reserves tend to utilise heap leaching as it is the only available method for profitable extraction (Bartlett, 1992a). Heap leaching accounts for around 15% of world’s gold and copper production (Bouffard and West-Sells, 2009). This figure is increasing year on year and demonstrates the importance of this in mineral extraction process. However, the recovery using heap leaching is relatively lower than separation techniques such as flotation (50-60% in heap leaching compared to 85% in froth flotation) and an increase in performance of heap leaching is crucial in the mining and mineral processing industry.
2.3. HEAP LEACHING
Leaching as a method for extracting metals from ores can be carried out in many different ways and can take place in tanks, vats, heaps, dumps and columns depending on the size distribution of ore particles (Figure 2.1) (see Appendix A for agitated tank leaching and vat leaching). However, in the conventional heap leaching process the crushed and often agglomerated ore particles are stacked on a sloped impermeable layer (i.e. leach pad) and, on top of the heap, a leaching solution is applied, which gradually percolates through the ore particles. The drained solution is collected off the impermeable layer and is known as the pregnant solution. This solution is then processed to recover the valuable metals (Comba and McGill, 1992; de Andrade Lima, 2004).

The heap leaching process has been used for many years as an effective method to recover metals such as gold, silver, copper, zinc, nickel, cobalt and uranium and also salts such as potassium nitrate (de Andrade Lima, 2004; Mellado et al., 2009).

2.3.1. DUMP LEACHING
The mine overburden or waste from mine operations often contains very low amounts of recoverable minerals. Dump leaching is employed on this material (Figure 2.1) to extract possible value despite the unsuitability of traditional methods to treat it (Bartlett, 1992a; Wu et al., 2007).

![Figure 2.1: The relationship of the particle size and grade of the mined ore on various leaching operations (Dresher, 2004).](image-url)
From an operational point of view, dump leaching is similar to heap leaching, but the two processes are widely different in their geometries, stacking techniques, solution management procedures and control over the particle size. Unlike heap leaching, when performing dump leaching, the ore is taken directly from the mine prior to any size reduction process or pre-treatment (i.e. crushing or agglomeration) (Yusuf, 1984; Bouffard and Dixon, 2001).

2.3.2. OPERATIONAL PROCEDURE OF HEAP LEACHING
The heap leaching process is simple in principle and complex in practice due to the many factors which affect the performance. These include ore characteristics, chemical phenomena and liquid flow characteristics. Process control of heap leaching is difficult due to large mass of rocks involved, the long leaching time (Yusuf, 1984) and the large delay between making operational changes and the system’s response.

From an operational point of view, heap leaching is composed of two stages known as the extraction stage and the separation stage (Bartlett, 1992a). The extraction stage includes ore preparation, heap construction, leaching reagents application and solution collection. The subsequent separation stage involves metal separation processes from the resultant solution (i.e. pregnant solution). For base metals this usually involves solvent extraction followed by electro-winning. Figure 2.2 shows these operational stages for copper heap leaching.

2.3.3. ORE PREPERATION
Ore preparation operations may depend on several factors such as type, quantity and grade of ores, operational conditions and project economics. The typical materials stacked in heap leach piles are crushed or crushed and agglomerated ore (Bouffard and West-Sells, 2009). Some mines treat ROM ore using heap leaching and, in such cases, the ore heap has a substantially variable particle size distribution which ranges from large boulders to very fine particles. This adversely affects the metal extraction efficiency due to the profile heterogeneity of the heap. In such cases ore blending (i.e. grade control before crushing and agglomeration), crushing and agglomeration is usually carried out to reduce this effect (Bartlett, 1992a).
The typical size range of ore particles employed in industrial heap leaching is -20 mm or -25 mm after the size reduction of ROM ore. Generally, this can be achieved using a closed circuit two stage crushing operation but it requires high operational costs (Bartlett, 1992a). The presence of fines (i.e. smaller than 74 µm or 200 mesh) with crushed particles in a heap is problematic as it reduces permeability and causes non-uniform liquid flow through the heap.

![Figure 2.2](http://www.gewater.com/pdf/Bulletin968EN.pdf)

**Figure 2.2:** The operations of a heap leaching plant. Figure shows the initial and subsequent operations of copper heap leaching (GE Infrastructure, [http://www.gewater.com/pdf/Bulletin968EN.pdf](http://www.gewater.com/pdf/Bulletin968EN.pdf)).

The proportion of fines in a heap is reduced by agglomeration. Typically a crushed ore mixture will be rotated in drum into which the leaching solution (see Figure 2.2) and/or commercial binders such as cement is added (Bartlett, 1992a; Bouffard, 2008; Bouffard and West-Sells, 2009). Fines stick onto the crushed particles to produce an ore mixture of relatively narrow size distribution compared to the original mixture of the crushed ore. The production of agglomerates of uniform size is important as it leaves the largest proportion of air voids when the heap is constructed. This will be a decisive factor, especially in copper
sulphide leaching, where oxygen concentration is rate controlling parameter of the leaching reaction (Bouffard, 2008).

### 2.3.4. HEAP CONSTRUCTION

Heap construction is a massive engineering task, with heaps extending from a fraction of a square kilometre to a few square kilometres in surface area and up to 100 metres in height (Decker and Tyler, 1999a). The height and construction method of the heap will depend upon factors such as whether the material is likely to segregate or become compacted, both of which can result in low liquid permeability and therefore solution flow problems (Yusuf, 1984). The first stage of the heap construction is the leach pad construction, which stops downward solution percolation through the heap into the ground water. This is desirable as the leach solutions can contain acids or cyanide as well as leached heavy metals, all of which are potentially damaging to the environment. Loss of leach solution into the ground water would also result in the loss of product.

Heap construction is carried out by dumping from haul trucks (Figure 2.2), stacking with a front end loader and mechanical stacking using conveyor belts (Bartlett, 1992a). It differs slightly according to the ore to be used. When prepared ore is being used and when relatively level ground is available, leach pads are reused several times. But for ROM ore, successive layers of ore are stacked onto one another to construct the heap (each layer being referred to as a lift). In this case the leach pads are also the permanent disposal site for the material after the leaching period (Bartlett, 1992a).

### 2.3.5. SOLUTION APPLICATION

After the construction of the heap, leaching is performed by applying the leaching solution (i.e. chemical reagents) at the top (Figure 2.2). The solution distribution devices are usually constructed of plastic to minimize cost and prevent corrosion. A detailed description of solution distribution devices is given by Bartlett (1992a).

The applied solution trickles through the particles, simultaneously reacting with the ore as it does so (Sanchez-Chacon and Lapidus, 1997). Thus, solution irrigation should be used to ensure proper interaction between the reagents and ore. The leaching solution should only occupy part of the interstitial voids with the remainder occupied by air, which is important as many of the leaching reactions require oxygen (Bouffard and West-Sells, 2009). The liquid
content should also be low enough to allow for the replenishment of the air. Air motion is typically convective, being caused by the heating from exothermic reactions as well as wind motion over the heap. In some operations air is also blown into the heap.

In order to prevent saturation, the rate of liquid addition in heap leaching is very low and typical values are between $10^{-6}$-$10^{-5}$ m/s, which are superficial liquid addition rates based on the cross-sectional area of the entire ore bed (Roman and Olsen, 1974; Roman et al., 1974; Murr, 1979; Murr et al., 1981; Smith, 2002; Cariaga et al., 2003; de Andrade Lima, 2006). Other important parameters include the irrigation time, which will vary according to several factors, probably the most important of which is ore type. The duration of solution application for oxidized gold ores is about 60 days but it is about 500 days for copper sulphide ores due to the vast differences in reaction kinetics (Bouffard and West-Sells, 2009).

The solution enriched with metals, which is known as the “pregnant solution” or “pregnant liquor” is collected from the bottom of the heap (Figure 2.2). In acid leaching, the metal extraction stage will regenerate some of the acid which, together with fresh makeup acid, is returned to the heap. The fluids which are returned to the heap are known as “barren solutions” (Yusuf, 1984; Bartlett, 1992a).

### 2.3.6. PREGNANT SOLUTION COLLECTION

To accommodate pregnant and barren solutions large solution collection ponds are necessary (see Figure 2.2). The pregnant solution ponds have a capacity of order of few thousand cubic meters whereas the barren solution pond’s capacity is order of few hundred cubic meters (US EPA Report, 1992). Solution collections ponds are also built using an impermeable bottom layer to ensure proper solution collection without any leakages and to prevent environmental contamination. The capacities of these ponds are determined by considering rainfall, annual spring snow melt, extended shutdown of the metal separation plant and possible emergencies (Bartlett, 1992a).

### 2.4. SYSTEMS DESIGNED TO STUDY HEAP LEACHING

Heap leaching has been studied at a wide range of different scales from laboratory scale columns to cribs and silos, having the height of the anticipated heap, up to pilot scale and full scale heaps. This section describes the systems used to provide preliminary data about
heap flow behaviour and leaching reactions at different scales before industrial heap leaching is practiced.

After initial tests of leachability using milled ore in agitated or bottle roll tests, the next stage in testing usually involves columns. Column tests usually involve tens to hundreds of kilograms of crushed ore placed in columns tens of centimetres wide and up to a few metres tall (Bartlett, 1992a). Column leaching tests are often used to determine optimum leaching conditions. These include leaching reagent consumption, maximum recovery of mineral ores and the rate of recovery in the initial stage of heap leaching. However, the column leaching tests often have lower bulk density for the ore and are subject to wall channelling, both of which can lead to significantly different recovery values compared to the operational heaps. The data obtained from column leaching is usually not sufficient to make informed decisions regarding an industrial scale heap leaching operation. It is therefore necessary to conduct larger scale tests as well (Roman, 1977; Kappes, 1981; Murr et al., 1981; de Andrade Lima, 2004, 2006; Bouffard and West-Sells, 2009).

Cribs and silos are usually of a similar height to an industrial heap and a few metres in diameter and will thus contain a few hundred tons of ore (Murr, 1979; Murr et al., 1981). Unconfined pilot heaps carry 1000 to 100,000 tons of ore. These large scale heaps are expensive to operate, but most closely mimic the behaviour of a full scale heap (Bartlett, 1992a; Bouffard and West-Sells, 2009).

Typically, few weeks are enough to obtain reliable experimental data through laboratory scale column leaching tests but large scale column leaching experiments (eg. Murr, 1979; Murr et al., 1981) and pilot heaps are usually operated for several months or few years while carrying out series of experiments.

2.5. MODELLING OF HEAP LEACHING

Based on the experimental studies of heap leaching in columns or at the pilot scale, recovery of metals can be modelled in two stages: the particle level and the heap level. Particle level models describe the dissolution of mineral grains in the ore particles through the reactions with leaching reagents, whereas the heap level models are used to describe the transport of dissolved products through the heap. The subsequent sections briefly summarise relevant models that cover these two scales of modelling.
2.5.1. PARTICLE LEVEL MODELS

When the mineral dissolution kinetics are fast and the mineral grains are disseminated uniformly throughout the particle, the rate of leaching of mineral grains is diffusion controlled. The diffusion equation (2.1) can be used to describe the leaching behaviour assuming that particles are quasi-spherical.

\[
\varepsilon \frac{\partial C}{\partial t} = \frac{D_0 \varepsilon}{\tau_l} \left( \frac{\partial^2 C}{\partial r_p^2} + \left( \frac{2}{r_p} \right) \frac{\partial C}{\partial r_p} \right)
\]

(2.1)

where \( \varepsilon \) is the rock porosity, \( D_0 \) is the diffusivity, \( \tau_l \) is the average tortuosity of open pore paths in the ore particle, \( C \) is the concentration of diffusing species in solution and \( r_p \) is the radius of the rock particle (Bartlett, 1992a).

The solution of the diffusion equation (Crank, 1956) is given in equation 2.2 to determine the fractional extraction, \( F_{t,r_p} \) (Bartlett, 1992a). The cyanide dissolution of submicron gold particles can be reasonably modelled using this equation (Bartlett, 1992a).

\[
F_{t,r_p} = 1 - \frac{6}{\pi^2} \sum_{n=1}^{\infty} \frac{1}{n^2} \exp \left( - \frac{D_{eff} n^2 \pi^2 t}{r_p^2} \right)
\]

(2.2)

where \( D_{eff} \) is the effective diffusivity given by \( D_0 \varepsilon / \tau_l \), \( r_p \) is the radius of the rock particle, \( t \) is time and \( n \) is the index of the infinite series.

Roman et al. (1974) studied the rate limiting mechanisms for oxide copper ore leaching and reported that the diffusion of reactant into the particle is rate controlling of the leaching reaction rather than the reaction kinetics of ore particles. When the mineral dissolution kinetics are fast, especially in copper oxide leaching, the diffusion controlled reaction can be modelled with the widely used shrinking core model (Ishida and Wen, 1968; Bartlett, 1992a, 1992b, 1997). According to the shrinking core model, the dissolution progresses inward through the rock particle with an expanding shell of leached material surrounding the unreacted copper oxide mineral (Figure 2.3). Many investigators (eg. Roman and Olsen, 1974; Roman et al., 1974; Bartlett, 1992a, 1992b) have simulated the fractional extraction of oxide copper ore using this model assuming that leaching is a quasi-steady state process and rock particles are spherical.

The other kind of particle level leaching models assumes the mineral dissolution is relatively slow together with slow diffusion of reagents. These are referred to as mixed kinetics modes.
and they do not illustrate a sharp discontinuity between the fully leached rim and the unleached core, which can be seen in the shrinking core model. The mixed kinetics models were developed primarily to describe the leaching behaviour of primary sulphide ores (Bartlett, 1992a). Some of the models are non-steady state mixed kinetics models while others are pseudo-steady state mixed kinetics forms (Bartlett, 1992a). A detailed description of these is given by Bartlett (1992a, 1992b).

Dixon (1992) presented a particle level model similar to the non-steady state mixing sulphide leaching model described by Bartlett (1992a, 1992b). The model was based on the unsteady state continuity equation for a reagent species in spherical co-ordinates. However, the rate of dissolution of mineral grains was expressed using a variable order rate constant (i.e. a power law relationship with the exponent as a fitting parameter).

![Shrinking core model](image)

**Figure 2.3**: Shrinking core model describing the dissolution of oxide copper ore. Figure shows the leached rim and the unreacted core. The concentration gradients of copper and the reagent (i.e. A in this figure) are also given (Bartlett, 1992a).

### 2.5.2. HEAP LEVEL MODELS

The particle level models are typically coupled with solute transport to simulate the overall column leaching behaviour and industrial heap performance using heap level models.

Roman and co-workers (Roman and Olsen, 1974; Roman et al., 1974) have described the transport of dissolved species through the heap by dividing the heap into square columns
(see Figure 2.4). de Andrade Lima (2004) has developed a similar model by dividing the heap into planar elements instead of columns. This approach to heap modelling reduces the flow behaviour to a one dimensional system. Both models assumed plug flow behaviour and neglected horizontal flow. However, the actual heap fluid flow deviates significantly from plug flow (experimental demonstrated by Bouffard and West-Sells, 2009). Therefore, the horizontal flow distributions such as radial dispersion and stagnant zones present in the heap are important variables required to accurately model the heap flow.

The heap level model derived by Dixon (1992) for an isothermal heap also assumed unsteady state plug flow behaviour. All physical parameters within the heap are assumed to be uniform and the model is capable of simulating the leaching process of one or more solid reactants contained within the porous ore particles. It also accounts for particles with a distribution of sizes.

Figure 2.4: One dimensional heap level model by dividing the heap into square columns (Roman et al., 1974).

The modelling of heap leaching process presented by Petersen and Dixon (2002) combines a reaction-diffusion model with separate models for equilibrium reactions in solution, chemical and biological reactions, heat transport and gas flow mechanisms. The complex overall process of heap leaching was considered as number of sub-processes operating at different levels from individual mineral grains of ore particles (i.e. micro scale) to heap level (i.e. macro scale) to develop the model, which is known as the HeapSim model (Dixon, 2003; Dixon and Petersen, 2003, 2004). The main sub-processes in the HeapSim model are grain scale, particle scale, cluster scale and heap scale. The cluster scale is the combination of several ore particles, in which the model combines gas uptake, bacterial kinetics and intra-
and inter-particle diffusion. In addition, the transport mechanisms of leaching solution, heat and gas are taken into account to represent the full heap scale (Petersen and Dixon, 2007).

The complex HeapSim model has been applied for copper leaching from chalcopyrite and pyrite and zinc leaching from marmatite and pyrite, which is combination of ZnS and FeS. More importantly, the model has been calibrated by Petersen and Dixon (2007) based on the large scale (0.15 m diameter and 6 m height) zinc column leaching experimental data and then it was validated using pilot scale heap test results. It was shown that the significant difference in mineralogy during model calibration and model validation affects the validity of a direct comparison. However, the HeapSim model simulated the measured data very closely in the pilot scale zinc leaching process. Carrying out a sensitivity analysis, it was found that the most important design parameters were heap height, rate of solution application, acid concentration of the feed leaching reagent, drip emitter spacing during the solution application and the temperature of the applied solution (Petersen and Dixon, 2007).

Heap leaching modelling has also been carried out based on Richard’s equation, which is a description of fluid flow in particle beds often used in ground water flow and oil reservoir modelling. Cross et al. (2006) used Richard’s equation to describe the unsaturated liquid flow using the relations of liquid content and hydraulic conductivity defined by van Genuchten (1980). The liquid flow was coupled to the solute transfer within the heap using liquid content and liquid flux. The computational model also included gas transport and heat transfer through the heap. Finally, the heap model was used to simulate the leaching behaviour of pilot scale columns and full scale heaps with the parameters found from columns composed of copper and oxide gold ore.

2.6. ANALOGOUS SYSTEMS TO HEAP LEACHING

Flow through packed beds and porous media has been studied in chemical engineering, petroleum engineering and hydrology. In chemical engineering applications, trickle bed reactors are similar to the operation of column leaching. The flow of ground water and contamination of ground water from waste disposal sites also has a number of similarities to heap leaching. The hydrodynamics of trickle bed reactors and ground water flow can be used to understand the hydrodynamics of heap leaching and could be considered as the analogous systems to column and heap leaching (Roman and Bhappu, 1993). While these
systems resemble heap leaching and can provide some insights, there are also important differences in terms of, for instance, particle size and liquid and gas flow rates.

### 2.6.1. TRICKLE BED REACTORS

Trickle bed reactors (TBRs) are fixed packed beds filled with catalysts or reactant solids, with high liquid flow rates and high pressure gas flow through the particles (Luciani et al., 2002). Industrially important reactions take place between the liquid and dissolved gas on the interior surface of the catalyst (Maiti et al., 2008). TBRs have widespread industrial applications in petroleum, petrochemical and bio-chemical industries, industrial wastewater treatment and agriculture and in the pharmaceutical industry (Specchia and Baldi, 1977; Kan and Greenfield, 1978, 1979; Tosun, 1984; Fu and Tan, 1996; Saroha et al., 1998; Luciani et al., 2002; Maiti et al., 2008).

There is less data available on the hydrodynamics of heaps compared to the extensive studies that have been carried out for trickle bed reactors. A number of significant differences between heap leaching and TBRs such as irregular porous ore particles compared to regular shape catalytic particles, very slow gravitational flow and stagnant gas flow compared to moderate to high liquid and gas flow rates, and atmospheric pressures compared to moderate to high pressures limit the applicability of the hydrodynamics of TBR’s to column or heap leaching (Bouffard and Dixon, 2001; de Andrade Lima, 2006). However the dimensionless numbers used to characterise their flow behaviour can provide important and useful information for heap leaching hydrodynamics (de Andrade Lima, 2006). Several investigators (eg. Satterfield, 1975; Schwartz et al., 1976b; Fu and Tan, 1996; Al-Dahhan and Highfill, 1999; de Andrade Lima, 2006) have highlighted the typical operational conditions of TBRs. Table 2.1 gives a summary of these parameters.

<table>
<thead>
<tr>
<th>Parameter in TBRs</th>
<th>Values</th>
</tr>
</thead>
<tbody>
<tr>
<td>Particle size</td>
<td>0.8-3.2 mm (porous catalyst particles)</td>
</tr>
<tr>
<td>Pressure of the reactor</td>
<td>From atmospheric pressure to high pressures of up to 20 or 30 MPa</td>
</tr>
<tr>
<td>Liquid flow rate</td>
<td>0.1-10 kg/m² s</td>
</tr>
<tr>
<td>Gas flow rate</td>
<td>$10^{-4}$-0.1 kg/m² s</td>
</tr>
<tr>
<td>Fluid flow direction</td>
<td>co-current down flow/up flow and counter-current flow</td>
</tr>
</tbody>
</table>
The two main flow regimes in TBRs are low interaction and high interaction. The low interaction regime or trickle flow occurs at low liquid superficial velocities (up to 0.005 m/s) and low gas superficial velocities (up to 3 m/s). Under these conditions a continuous gas phase exists and liquid flows through the particles as rivulets and films (Holub et al., 1993; Saroha et al., 1998; Toye et al., 1998). The trickle flow is the one most analogous to the flow regime found in heap leaching and it is worthwhile to study the trickle bed hydrodynamics to describe the flow patterns in heap leaching (see section 2.7).

2.6.2. GROUND WATER FLOW
Significant progress has been made in recent years in modelling flow behaviour in heterogeneous soils (Decker and Tyler, 1999b). The generalization of Darcy’s law for unsaturated porous media was performed by Richards (1931) because it was originally derived for saturated flow. Richard’s equation was derived by combining Darcy’s law with the continuity equation for capillary flow (Richards, 1931). It describes the capillary flow through unsaturated porous media such as soil and clay and the flow through variably saturated porous media (Cross et al., 2006):

\[
\hat{q} = K_c(\theta) \nabla H
\]  

(2.3)

\[
\nabla \hat{q} + \frac{\partial \theta}{\partial t} = 0
\]  

(2.4)

where \( \hat{q} \) is the Darcy flux, \( \theta \) is the moisture content, \( H \) is the pressure head, \( K_c \) is the unsaturated hydraulic conductivity which is a function of \( \theta \). Soil scientists have defined a water pressure head in the water-air system relative to the atmospheric pressure, which is negative and mathematically equal to the negative of the capillary pressure head (Morel-Seytoux, 1973). If moisture content \( \theta \) is a single valued continuous function of the capillary potential and \( B \) is the capillary capacity of the medium, Richard’s equation for the capillary flow can be expressed as follows (Richards, 1931):

\[
B \frac{\partial H}{\partial t} = \frac{\partial}{\partial z} \left[ K_c(H) \frac{\partial H}{\partial z} \right] - \frac{\partial K_c(H)}{\partial z}
\]  

(2.5)

where \( z \) is the vertical co-ordinate of the system.

Equation 2.5 has been used by many researchers to describe the fluid flow in soils and heap leaching systems (e.g. Morel-Seytoux, 1973; Nielsen et al., 1986; Decker, 1996; Decker and Tyler, 1999b; Cross et al., 2006). The accuracy of Richard’s equation depends on field
measured parameters such as $K_c(H)$ and $H(\theta)$, which are non-linear functions. These are difficult to measure and the error introduced in measuring them could affect the flow described by Richard’s equation (Schaffer, 1997).

The applicability of Richard’s equation to describe the unsaturated flow is questionable in the presence of relatively impermeable soil layers and in structured soils which contain relatively large and more or less continuous voids or macro-pores. It also ignores the soil matrix and assumes the role of the air phase is negligible (Nielsen et al., 1986). Richard’s equation also does not consider dispersion effects in the liquid motion and ignores anisotropy and hysteresis. Therefore two phase air-water flow equations have been developed by the generalization of Darcy’s law to describe ground water flow. These flow equations were originally applied in petroleum engineering flow simulations (Green et al., 1970; Morel-Seytoux, 1973).

Other than the fluid flow, much attention has focussed on describing the solute transport through soil and analogous solute transport phenomenon taking place in heap and dump leaching. The solute transfer processes in the soil are important in the context of water supply, environmental protection and agriculture (Coats and Smith, 1964; Matsubayashi et al., 1997). This behaviour in porous media has been described by means of dispersion coefficients. The residence time distribution (RTD) curves after injecting salt or dye tracers into the soil column is fitted using the advection-dispersion model and/or the mobile-immobile model (see section 2.9) to quantify dispersion. Similar residence time distribution studies using tracers have been performed in heap, dump and column leaching (Armstrong et al., 1971; Murr, 1979; de Andrade Lima, 2006).

Hysteresis is a phenomenon occurring when the behaviour of a system depends not only on the current input conditions, but also on the system history. Many authors have pointed out the existence of pressure drop and liquid holdup hysteresis in trickle bed reactors (eg. Kan and Greenfield, 1978, 1979; Levec et al., 1986; Levec et al., 1988; Lazzaroni et al., 1989; Maiti et al., 2006; Maiti et al., 2008). Also the effect has been observed on unsaturated hydraulic conductivity curve and soil water retention curve in ground water flow systems (eg. Richards, 1931; Nielsen et al., 1986). Heaps typically have much lower liquid flow rates than those found in trickle bed reactors and usually little or no gas flow. However, the systems are analogous enough to expect hysteresis in the fluid flow in heap leaching.
It is also worthwhile to describe the fluid flow in packed bed and heaps by analysing both direct and analogous systems to heap leaching.

2.7. FLUID FLOW IN PACKED BEDS AND HEAPS

In this section, existing models for the fluid flow and liquid holdup behaviour in heaps and other packed beds are examined. Before this is done, the small scale fluid flow structures and features will be discussed as understanding them is a key step in modelling and predicting the overall fluid flow behaviour.

2.7.1. DROPLET AND RIVULET FLOW

Many researchers (eg. Jesser and Elgin, 1943; Roman and Bhappu, 1993; Maiti et al., 2006, Maiti et al., 2008) have illustrated the different micro (particle level) and macro level flow regimes through the particles of packed beds. Figure 2.5 shows on the left macro level flow textures whereas on the right it illustrates particle level flow features.

Liu et al. (1997) performed liquid flow experiments through the particles of the packed bed using video picture analysis. Results indicated two main flow patterns, were the droplet and the rivulet flow.

The discrete droplet flow occurs at very low flow rates. Gravity is responsible for creating discrete droplets by a continuous break up and reform mechanism, since continuous droplet
flow is not possible at very low liquid flow rates. When a liquid drop breaks up on collision with solid particles, a liquid fraction remains on the particle surface as inertia of the liquid fraction is not sufficient to overcome the friction created by the surface of the solid particle. The movement of the liquid fraction starts when by the next large droplet which can coalesce with the remaining liquid on top of the solid particle surface (Liu et al., 1997). This is the mechanism of the droplet flow through the particles. The size distribution of droplets is a function of liquid properties and packing characteristics.

The discrete droplet flow would transform into continuous droplet flow when the flow rate gradually increases. Then liquid flows continuously from one contact point between the particles to the next contact point in the form of very fine liquid streams called rivulets. Jesser and Elgin (1943) performed liquid flow experiments through 12.7-25.4 mm diameter particles, which is relevant to the typical size range of particles employed in heap leaching. The study concluded that for a mass flow rate of less than 2 kg/m² s, the liquid flows as fine rivulets.

2.7.2. HEAP FLOW BEHAVIOUR

Heaps exhibit unsaturated flow behaviour through the semi-consolidated porous ore particles of wide size distribution. A typical heap has particles that are mainly in the size range of millimetres to a few centimetres. This is significant as the Bond number goes from significantly below to significantly above 1 over this range of particle sizes. The Bond number (Bo) is the ratio of gravity to capillary forces (\( Bo = \rho gd_p^2/\gamma \), where \( \gamma \) is the surface tension, \( \rho \) is the density of the liquid, \( d_p \) is the particle size and \( g \) is gravitational acceleration). It indicates that the flow through these heaps is in a transition region between capillary and gravity dominated flow.

The flow characteristics depend on factors such as the construction procedures of the heap, the application of the leach solution, the size of the heap and the size distribution and the porosity of the rock particles (Roman, 1977; Murr et al., 1981; Yusuf, 1984). As the leaching solution trickles through the ore particles the flow channels are developed and Figure 2.6 shows this phenomenon.

This flow behaviour through the rock particles is known as bulk advective flow and it typically occurs as small rivulets between the contact points of particles. Other than the
advection, diffusion of reagents into and out from the particles occurs due to the porosity of the ore particles (Yusuf, 1984; Decker and Tyler, 1999a; Petersen and Petrie, 2000).

This implies that the applied leaching solution occupies the space between the interconnected particles and also that fluid is present in pore spaces within the individual ore particles. Thus, the porosity of the heap has two distinct length scales, namely that of the channels between the particles (i.e. interstitial space) and within the particles (i.e. intra-particle space). The interstitial space will typically have a length scale of the order of millimetres, and the typical length scale of the intra-particle space is of the order of tens of microns.

![Figure 2.6: The solution flow channels through the rock particles of a heap (Petersen and Petrie, 2010).](image)

As described above, the Bond number will be order of 1 for the fluid flow between the particles. However, the existing micro-pores within the particles will have Bond numbers that are many orders of magnitude less than 1, indicating capillary dominated flow (note that a Bond number well below 1 does not mean that gravity does not affect the flow rate, it rather means that the shape of the flow paths is not influenced by gravity). This distinct separation of length scales means that the holdup within the particles will not have the same effect on liquid flow as the holdup between the particles.

The liquid fractions that are inside the rock pores and between the rock particles contribute to the total amount of liquid present in the entire heap (i.e. overall liquid holdup). A part of
the overall liquid content is free flowing (i.e. mobile liquid) but the immobile liquid fractions present in the heap are also reported (eg. Murr, 1979; Murr et al., 1981).

2.7.3. LIQUID HOLDUP IN PACKED BEDS AND HEAPS

Liquid holdup is one of the major factors to be understood to describe the flow behaviour of heap leaching and packed beds (Roman and Bhappu, 1993; de Klerk, 2003; de Andrade Lima, 2006). It determines liquid residence time distribution, mass transfer between the particles and liquid and wetting efficiency of the particles (Yusuf, 1984; Fu and Tan, 1996; Saroha et al., 1998; Al-Dahhan and Highfill, 1999; Lange et al., 2005).

Total liquid holdup, $\theta$, is the ratio of the volume of liquid at any time in the system to the total volume of the system and equals to the multiplication of liquid saturation ($\beta$) and voidage ($\xi$). Further, it is given by the summation of external holdup ($\theta_e$) and internal holdup ($\theta_i$) (Yusuf, 1984; Fu and Tan, 1996; Saroha et al., 1998; Al-Dahhan and Highfill, 1999; de Klerk, 2003; Lange et al., 2005).

The liquid contained in particle pore spaces by capillarity contributes to the internal liquid holdup (for non-porous particles, $\theta_i = 0$, therefore $\theta = \theta_e$) while the liquid volume outside the particles is known as the external liquid holdup (Roman and Bhappu, 1993; Al-Dahhan and Highfill, 1999; de Klerk, 2003). The external liquid volume is the combination of the free flowing liquid volume, which is dynamic holdup, $\theta_{\text{dynamic}}$ (Saroha et al., 1998; Al-Dahhan and Highfill, 1999; de Klerk, 2003) and the liquid volume retained in the system after draining has occurred, which is known as residual holdup (Roman and Bhappu, 1993).

Some authors (eg. de Klerk, 2003; Lange et al., 2005) have considered the residual and the static holdup as the same thing but it is worth noting that this is not necessarily true (i.e. $\theta_{\text{residual}} \geq \theta_{\text{static}}$). The static and residual liquid holdups would be the same thing if the static liquid holdup does not change with the average flow rate. It is not apparent that this has to be the case. Thus, the residual holdup is a more reliable term to define the liquid volume in between the particles in a drained packed bed. It is useful to identify different contributions to the static liquid holdup. This could include the liquid in the pores within the particles, but also liquid held by capillarity between the particles or as thin films around the particles.
Hydrodynamic studies of trickle bed reactors have measured the liquid holdup variation using different methods such as the residence time distribution analysis using tracers, the drainage method and the gravimetric method. Al-Dahhan and Highfill (1999) has presented detailed description of these experimental methods.

2.7.4. LIQUID HOLDUP MODELS

Generally, the researchers who studied the liquid holdup in column leaching (Yusuf, 1984; de Andrade Lima, 2006), trickle bed reactors with porous particles (Schwartz et al., 1976a; Schwartz et al., 1976b; Saroha et al., 1998; Nemec et al., 2001; Lange et al., 2005) and trickle bed reactors with non-porous particles (Schwartz et al., 1976a; Schwartz et al., 1976b; Fu and Tan, 1996; Lange et al., 2005) have plotted the liquid holdup variation against the liquid mass flux or liquid Reynolds number (Reynolds number is the ratio of inertia forces and viscous forces, see Table 2.2 for definitions). Further, various empirical correlations have been formulated to fit the experimental liquid holdup results. Table 2.2 shows some representative correlations to describe liquid holdup in column leaching and trickle bed reactors.

The liquid holdup correlations of Yusuf (1984) and de Andrade Lima (2006) given in Table 2.2 are specific to column leaching studies. Yusuf (1984) reported liquid holdup results with column leaching with three different rock types. Sodium chloride (NaCl) was injected at the top of the column as salt tracer to determine the effluent tracer concentration. The analysis of RTD curved has been employed to calculate the liquid holdup values for different mean size of rock samples. The experimental results were used to formulate the empirical liquid holdup model shown in Table 2.2. The total liquid holdup is expressed as a function of volumetric flow rate \( (Q_v) \), mass of each size fraction of ore particles \( (M_i) \) and particle size of each size fraction \( (d_{pi}) \).

de Andrade Lima (2006) presented liquid holdup results with column leaching tests for 2 mm quartz particles. Liquid holdup values were determined using RTD analysis after injecting a pulse of hydrochloric acid (HCl) at the top of the column. The experimental results indicated an increase of dynamic saturation and a decrease of static saturation with flow rate (Table 2.2). The typical values of dynamic saturation were 5-20% while 2-6% static saturation values were reported. The liquid holdup values were fitted as power law functions against liquid Reynolds numbers.
Table 2.2: Liquid holdup correlations for column leaching and trickle bed reactors.

<table>
<thead>
<tr>
<th>Author</th>
<th>Liquid Holdup Correlation</th>
<th>System of Study</th>
</tr>
</thead>
</table>
| de Andrade Lima (2006) | \( \theta_{\text{dynamic}} = 33.89\xi Re^{0.269} \)  
                      \( \theta_{\text{static}} = 5.86\xi Re^{-0.0472} \) \( [Re = v_s d_p \rho / \mu] \) | Column leaching |
| Fu and Tan (1996)  | \( \theta = 1.505\xi Re^{0.29} Ga^{-0.32} d_0^{-0.22} \) \( [Re = v_s d_p \rho / \mu, \{Ga = \rho^2 d_p^3 g / \mu^2 \}] \) | Trickle bed reactors |
| Specchia and Baldi (1977) | \( \theta_{\text{dynamic}} = 3.86\xi Re^{0.545} Ga^{-0.42} (a_p d_p / \xi)^{0.65} \)  
                      \( [Re = v_s d_p \rho / \mu, \{Ga = \rho^2 d_p^3 g / \mu^2 \}] \) | Trickle bed reactors |
| Saroha et al. (1998) | \( \theta = 0.1 + 10858.54 v_s v_G - 4682.21 v_G^2 + 2987.54 v_s^2 \) | Trickle bed reactors |
| Yusuf (1984)       | \( \theta = b_1 (Q_L) \sum_{i=1}^{n} M_i (d_{pi})^{b_2} + b_3 \)                        | Column leaching |
| Lange et al. (2005) | \( \theta_{\text{dynamic}} = 0.002 (d_R / d_p)^{1.28} (Re)^{0.38} \)  
                      \( \theta = 0.16 (d_R / d_p)^{0.33} (Re)^{0.14}, \{Re = v_s d_p \rho / \xi \mu} \) | Trickle bed reactors |
| Schwartz et al. (1976a) | \( \theta \propto \varphi_L^{0.2} \) and \( \theta_{\text{dynamic}} \propto \varphi_L^{0.26} \) (non-porous)  
                      \( \theta \propto \varphi_L^{0.15} \) (porous) | Trickle bed reactors |
| Urrutia et al. (1996) | \( \theta_{\text{dynamic}} \propto v_s^{1/3} (\mu / \rho)^{1/3} \)                   | Bench scale beds |

\( \xi \) is bed voidage, \( Re \) is Reynolds number, \( Re' \) is modified Reynolds number, \( Ga \) is Galileo number, \( d_0 \) is the diameter based on bed voidage (see Fu and Tan, 1996), \( d_p \) is particle diameter, \( d_R \) is inner reactor diameter, \( a_p \) is packing geometrical area, \( v_s \) is superficial liquid velocity, \( \varphi_L \) is liquid mass flux, \( v_G \) is superficial gas velocity, \( Q_L \) is liquid flow rate, \( \rho \) is density of liquid, \( \mu \) is viscosity of liquid, \( M_i \) is mass of each size fraction of particles and \( b_1, b_2 \) and \( b_3 \) are constants.

The important characteristics of the liquid holdup correlations can also be summarised using the experimental variables such as liquid and gas flow rate, particle size, porosity and liquid viscosity. Table 2.3 shows these parameters according to the liquid holdup results in column leaching and trickle bed reactors (Yusuf, 1984; Fu and Tan, 1996; Saroha et al., 1998; Nemec et al., 2001; Lange et al., 2005).
Table 2.3: Variation of liquid holdup with experimental variables of the system.

<table>
<thead>
<tr>
<th>Experimental Variable</th>
<th>Liquid Holdup Variation</th>
</tr>
</thead>
<tbody>
<tr>
<td>Liquid mass flux or Reynolds number - Increasing</td>
<td>Total liquid holdup and dynamic holdup increases</td>
</tr>
<tr>
<td></td>
<td>Power law relationship</td>
</tr>
<tr>
<td></td>
<td>No significant dependency on the static holdup suggested by some authors while decrease of static liquid holdup with an increase in liquid velocity suggested by others</td>
</tr>
<tr>
<td>Particle size - Decreasing</td>
<td>Total liquid holdup increases</td>
</tr>
<tr>
<td>Liquid viscosity - Increasing</td>
<td>Total liquid holdup increases</td>
</tr>
<tr>
<td>Porosity</td>
<td>Generally, liquid holdup of porous particles are more than twice that of the non-porous particles</td>
</tr>
<tr>
<td>Gas flow rate - Increasing</td>
<td>No significant dependency on the dynamic and static holdup suggested by some authors while decrease of liquid holdup with an increase in gas velocity suggested by others</td>
</tr>
</tbody>
</table>

Non-homogeneous flow characteristics are common in packed beds and heaps along with the previously described flow features through the particles. These affect the uniform liquid content of the system and thus the uniform wetting efficiency of the particles.

2.8. NON-HOMOGENEOUS FLOW FEATURES IN PACKED BEDS AND HEAPS

Non-homogeneous wetting of all the particles is frequently observed in packed beds and heaps. Wall flow is the main uneven liquid distribution behaviour in packed beds as described in following sections. In heaps however, the non-homogeneous flow characteristics are uneven solution distribution, channelling and presence of locally saturated zones.

2.8.1. WALL FLOW

Wall flow is a major source of uneven distribution of fluid in packed beds (Porter, 1968, Porter and Templeman, 1968; Saroha et al., 1998) and it reduces the flow efficiency of the system (Porter, 1968). Several researchers (eg. Fand and Thinakaran, 1990; Eisfeld and Schnitzlein, 2001; Di Felice and Gibilaro, 2004) have investigated this phenomenon in TBRs.
Wall flow is defined as the excess of the fluid flow along the column wall resulting from the larger fractional free space between the particles and reactor wall (i.e. increased voids near the wall) (Saroha et al., 1998). These higher void spaces increase the permeability and decrease the resistance to the liquid flow near the wall (Yusuf, 1984; Saroha et al., 1998). Wall flow also results in localised increase in velocity of the fluid (Cohen and Metzner, 1981; Fand and Thinakaran, 1990). The liquid accumulation on the wall of a randomly packed column depends on the rate at which the liquid moves in the radial direction and on the tendency of the liquid at the wall to return to the packing (Porter and Jones, 1963).

Wall flow depends on the ratio of the column to particle diameter ($d_R/d_p$), liquid and gas flow rates, physicochemical properties of the liquid (density, viscosity, surface tension), wettability, porosity, shape and orientation of the particles (Scott, 1935; Saroha et al., 1998). Several authors have suggested different $d_R/d_p$ values to avoid or minimize the wall effect. Table 2.4 summarises these reported values.

<table>
<thead>
<tr>
<th>Author</th>
<th>$d_R/d_p$ ratio</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cohen and Metzner (1981)</td>
<td>30</td>
</tr>
<tr>
<td>Fand and Thinakaran (1990)</td>
<td>40</td>
</tr>
<tr>
<td>Hoftyzer (1964)</td>
<td>8</td>
</tr>
<tr>
<td>Mehta and Hawly (1969)</td>
<td>50</td>
</tr>
<tr>
<td>Schiesser and Lapidus (1961)</td>
<td>16</td>
</tr>
</tbody>
</table>

The liquid flowing along the wall is assumed to be of negligible thickness (Porter and Jones, 1963). The flow from the packing to the wall is given by the gradient of the flow per unit area per unit time at the interface between the two regions. Cihla and Schmidt (1958) considered the column wall as a perfect reflector but it is an oversimplified assumption due to the presence of the wall flow in packed beds. Porter and Jones (1963) reported that wall should be considered neither as a perfect reflector nor as a perfect sink of liquid.

The results of mathematical modelling and experimental studies of wall flow shows that the flow rate along the wall and in the bed reach equilibrium when the depth of the bed is
sufficiently large. The flow in the packing is then radially independent (Porter and Jones, 1963).

2.8.2. UNEVEN SOLUTION DISTRIBUTION
The percolating liquid should homogeneously wet all the rock particles present in the heap for an optimum metal extraction. This is the ideal fluid flow behaviour but it not usually achieved over the heap volume. However, the liquid addition at the top surface of the heap is not uniform in practice and the liquid trickles as preferential channels through the rock particles from top to bottom (Petersen and Dixon, 2007). Therefore, an inhomogeneous wetting of the ore particles is observed at various depths of the heap. Therefore, the liquid content is highly variable in different regions within the rock mass. This phenomenon is defined as the uneven liquid distribution through the particles. Experimental studies reported in the literature have indicated this behaviour within columns and heaps.

Howard (1968) measured the moisture content variation at several depths in a copper dump leach and found that the moisture content is different at all the depths. Some areas of the dump have zero moisture content while high moisture content was found in other areas. The results indicate that the leached solution did not infiltrate to all the parts of the dumps and fluid flows with different absolute velocities throughout the ore mass.

Armstrong et al. (1971) investigated the liquid flow distribution through a copper dump at Kennecott by injecting Tritiated water as a tracer. The samples of leached liquid were taken periodically at natural surface outflows and from a series of wells drilled in the dump for their Tritium content. The results indicated that the leached solution does not reach all parts within the dump. In some areas the tracer arrived sooner than expected but in other areas a delay was observed.

Murr and co-workers (Murr, 1979; Cathles and Murr, 1980) conducted very large scale column leaching tests to measure leaching behaviour and liquid flow characteristics. Two separate waste columns were flushed with Rhodamine-B dye to investigate the dye distribution within the bed. It was found that the entire bed of rock particles was not in contact with the dye. In one column 40-50% of rock was in contact whereas the dye wetted only 14% of the rock surface area of the second column.
The above experimental studies indicate the presence of uneven liquid flow behaviour in heap leaching. The factors affecting this phenomenon are heap construction procedures, application of leaching reagents and size and distribution of rock particles (Yusuf, 1984). The major uneven flow feature through the ore particles is liquid channelling.

2.8.3. CAUSES OF UNEVEN LIQUID DISTRIBUTION

This section describes causes for the uneven liquid distribution mechanism and these are liquid channelling through the ore particles, presence of locally saturated regions and the effects of compaction and consolidation of the ore.

2.8.3.1. CHANNELLING

All the particles in a heap should be in contact with the leach solution in order to achieve good recovery of metals and generally this will not happen. Preferential flow through channels instead of homogeneous flow is often observed (Yusuf, 1984; Wu et al., 2009). Large channels with very high void volume are responsible for this type of bulk flow (Murr et al., 1981). These high flow channels tend to short circuit the leaching solution (Bartlett, 1992a). This process is called channelling. In packed bed studies, significant scatter in the experimental data was observed due to the channelling phenomenon (Porter and Jones, 1963).

Low grade ores and the mine waste used in dump leaching often have a very broad size distribution ranging from fine particles to boulders. These particles get distributed during the heap construction mainly using haul trucks and Figure 2.7 illustrates the particle segregation.

Figure 2.7: The particle segregation during the heap construction (Yusuf, 1984: p. 12).
The bulk density of the heap was observed to vary across the distribution with distinct high and low bulk density regions (Howard, 1968; Roman, 1977; Yusuf, 1984). The areas of lower bulk density have less resistance to flow or higher permeability and this results in preferential flow through larger openings. The presence of significant quantities of clay results in the localised compaction and increased density with low permeability regions. Often, high permeability channels surround these low permeability areas (Yusuf, 1984).

2.8.3.2. LOCALLY SATURATED REGIONS
The existence of clay layers within the heap generally prevents the solution percolation into the rock particles below this layer. Similarly, gypsum precipitation in some of the leaching operations seals the inter-particle spaces resulting in low flow velocities. In both cases, the locally saturated regions are formed within ore particles above the relatively impermeable material (Yusuf, 1984; Bartlett, 1992a). Sometimes the leaching solution will escape from the sides of the heap rather than percolate downwards through these low permeability regions (Yusuf, 1984).

2.8.3.3. EFFECTS OF COMPACTION AND CONSOLIDATION OF ORE
Compaction of the heap surface usually occurs during the heap construction and it changes the flow characteristics of the leaching solution. The adverse effects from this can be eliminated by trenching the top surface to a depth of few metres below the compacted ore mass (Yusuf, 1984) or by stacking the ore using conveyors rather than trucks and front end loaders.

Rock disintegration takes place with the age of the heap due to weathering of ore particles (eg. acid attack on gangue minerals). It reduces average particle size and permeability of the rock mass. The production of fine particles tends to plug the inter-particle spaces within the heap resulting in uneven liquid flow behaviour through the rock particles (Yusuf, 1984; Bartlett, 1992a). In addition, mathematical modelling of the heap flow behaviour becomes also difficult due to this change in size that occurs over the heap life (Bartlett, 1992a).

2.9. LIQUID DISPERSION AND MASS TRANSPORT IN PACKED BEDS AND HEAPS
The transport of the reagents from the drippers to the surface of the ore particles is an important factor in the overall performance of a heap. This flow behaviour is complex as the
unsaturated fluid flows along a tortuous set of inter-particle channels and connections. Chemical reactions occur between the solution and mineral grains in the ore particles. Transport mechanisms of the dissolved solutes through the heap determine the efficiency of metal extraction. Therefore, liquid dispersion and mass transport processes are very important aspects of the behaviour of heap leaching and intimately coupled to the heap fluid mechanics.

2.9.1. HYDRODYNAMIC DISPERSION

Fluid velocity causes advective solute transport through porous media. If average linear velocity is assumed (i.e. Darcy assumption) and in the absence of other transport mechanisms, the solutes will flow linearly as plug flow. However, hydrodynamic dispersion diminishes the plug flow behaviour. The interactions of the dissolved substances with porous media and micro level variations in each channel and in connections between the channels are responsible for the hydrodynamic dispersion (Decker, 1996).

The coefficient of hydrodynamic dispersion can be defined as the sum of effective molecular diffusion ($D_m$) and mechanical dispersion ($D_h$) (Bear, 1972; Decker, 1996; Matsubayashi et al., 1997; Padilla et al., 1999). Some researchers (e.g. Padilla et al., 1999) have expressed reservations with the addition of effective molecular diffusion and mechanical dispersion to describe hydrodynamic dispersion. Taylor dispersion theory describes how mechanical dispersion along flow lines could be influenced by molecular diffusion. This means that these two mechanisms cannot be considered independently.

$$D = D_m + D_h \quad (2.6)$$

Mechanical dispersion can be expressed as $\lambda v^{n_0}$, where $\lambda$ is dispersivity, $n_0$ is an empirical constant and $v$ is pore water velocity given by $v = \hat{q}/\theta$, where, $\hat{q}$ is Darcy flux and $\theta$ is liquid content (Decker, 1996; Padilla et al., 1999). Further, effective molecular diffusion ($D_m$) is defined as $D_w \tau_i$, where $D_w$ is the diffusion coefficient in bulk water, $\tau_i$ is the tortuosity factor for molecular diffusion (Padilla et al., 1999).

$$D = D_m + \lambda v^{n_0} \quad (2.7)$$

$$D = D_w \tau_i + \lambda v^{n_0} \quad (2.8)$$

Dispersivity ($\lambda$) is generally considered as an intrinsic property of the porous media under fully saturated conditions (Padilla et al., 1999). The constant, $n_0$, has values between 1 and 2
(Bear, 1972). The tortuosity factor accounts for the shape and length of the molecular path (Padilla et al., 1999). For a packed bed of spheres, it is defined as the ratio of the length of the shortest path around a spherical particle to its diameter and has a theoretical value of \( \pi/2 \) or 1.57 (Dixon, 1992).

Mechanical dispersion is likely to dominate around the particles but the molecular diffusion is important in the particles. The effective diffusivity coefficient \( (D_{eff}) \) for the intra-particle reagents diffusion is:

\[
D_{eff} = \frac{D_0 \varepsilon}{\tau_l}
\]  

(2.9)

where \( D_0 \) is the diffusivity, \( \varepsilon \) is the porosity of the particle and \( \tau_l \) is the tortuosity factor for molecular diffusion.

### 2.9.1.1. AXIAL AND RADIAL DISPERSION

The dispersion coefficient along the direction of flow is axial (longitudinal) and the dispersion coefficient in the direction perpendicular to the flow is known as radial (transverse) dispersion coefficient.

### 2.9.1.2. PECLET NUMBER

Peclet number is a dimensionless number, which describes the ratio of the advective to diffusive transport of solutes (Mears, 1971). It can be defined as \( P_e = U l_0 / D \), where \( U \) is the velocity of the system, \( l_0 \) is the characteristic length and \( D \) is the dispersion coefficient. Typically, the particle size \( (d_p) \) can be used as the characteristic length \( (l_0) \). Several authors (eg. Schwartz et al., 1976b; Fu and Tan, 1996; Saroha et al., 1998; de Andrade Lima, 2006) have characterised axial dispersion values using the Peclet number. The Peclet numbers were plotted against liquid Reynolds numbers in these studies (see Table 2.5). Some of the axial dispersion correlations are shown in Table 2.5. The velocity component required to calculate the two dimensionless numbers (Peclet and Reynolds number) was superficial liquid velocity.

Therefore, the calculated axial dispersion values have been typically expressed as a function of liquid superficial velocity. However, the applicability of superficial velocity to describe axial dispersion may not be appropriate (see Chapter 7).
Table 2.5: Axial dispersion correlations for column leaching and trickle bed reactors.

<table>
<thead>
<tr>
<th>Author</th>
<th>Axial Dispersion Correlation</th>
<th>System of Study</th>
</tr>
</thead>
<tbody>
<tr>
<td>de Andrade Lima (2006)</td>
<td>$P_e = 0.0914Re^{0.00532}$</td>
<td>Column leaching</td>
</tr>
<tr>
<td></td>
<td>$P_e = v_s d_p/D$ and $Re = v_s d_p \rho/\mu$</td>
<td></td>
</tr>
<tr>
<td>Fu and Tan (1996)</td>
<td>$P_e = 1.40 \times 10^{-4} \theta d_0^{-0.75} \xi^{-1}$ for $Re &lt; 4$</td>
<td>Trickle bed reactors</td>
</tr>
<tr>
<td></td>
<td>$P_e = v_s d_p/D$ and $Re = v_s d_p \rho/\mu$</td>
<td></td>
</tr>
<tr>
<td>Mears (1971)</td>
<td>$P_e (= v_s Z/D)$ was plotted against $Re (= v_s d_p \rho/\mu)$</td>
<td>Trickle bed reactors</td>
</tr>
<tr>
<td>Saroha et al. (1998)</td>
<td>$P_e (= v_s d_p/D)$ was plotted against $Re (= v_s d_p \rho/\mu)$</td>
<td>Trickle bed reactors</td>
</tr>
<tr>
<td>Schwartz et al. (1976b)</td>
<td>$P_e (= v_s d_p/D)$ was plotted against $Re (= v_s d_p \rho/\mu)$</td>
<td>Trickle bed reactors</td>
</tr>
<tr>
<td>Villanueva et al. (1990)</td>
<td>$D$ values were expressed as $P_e = v_s Z/\theta D$</td>
<td>Column leaching</td>
</tr>
<tr>
<td></td>
<td>$D$ values were expressed as $P_e = v_s Z/\theta D$</td>
<td>Agitation leaching</td>
</tr>
<tr>
<td>Decker (1996)</td>
<td>$D$ values were expressed as $P_e = v_s Z/\theta D$</td>
<td>Column leaching</td>
</tr>
</tbody>
</table>

$Re$ is Reynolds number, $d_0$ is the diameter based on bed voidage (see Fu and Tan, 1996), $d_p$ is particle diameter, $Z$ is column length, $v_s$ is superficial liquid velocity, $\xi$ is bed voidage, $\rho$ is density of liquid, $\mu$ is viscosity of liquid.

2.9.2. RESIDENCE TIME DISTRIBUTION MODELS

Hydrodynamic dispersion has a major impact on solute transport in most packed beds and porous media systems. These dispersion coefficients have been measured in systems with pores ranging in size from tens to hundreds of microns, such as found in hydrology and petroleum studies (eg. Coats and Smith, 1964; De Smedt and Wierenga, 1979; Matsubayashi et al., 1997), to the millimetre or larger scales found in trickle bed and other packed bed reactors (eg. Schwartz et al., 1976b; Fu and Tan, 1996), in agitation leaching (eg. Villanueva et al., 1990; de Andrade Lima and Hodouin, 2005) and column and heap leaching (eg. Murr, 1979; Murr et al., 1981; Decker, 1996; Bouffard and Dixon, 2001; de Andrade Lima, 2006; Bouffard and West-Sells, 2009).
The use of liquid tracers has proved to be simple and effective method to diagnose the underlying flow mechanisms in heap leaching (Bouffard and Dixon, 2001; Bouffard and West-Sells, 2009). The measured effluent tracer concentration of the system, $C(t)$, is normalised such that the area under the curve is unity in pulse injection. The resultant curve is known as the $E(t)$ curve (equation 2.10), where $E(t)$ is the exit age distribution and can be used to calculate the residence time distribution (RTD) curves (Saroha et al., 1998; Marquez et al., 2008). These curves are fitted using RTD models to determine the dispersion coefficient.

$$E(t) = \frac{\int_0^t \frac{C(t)}{C(t)dt}}{\int_0^t}$$  \hspace{1cm} (2.10)

### 2.9.2.1. ADVECTION-DISPERSION EQUATION (ADE)

This is the simplest form of solute transport model in porous media. Several researchers in hydrology (De Smedt and Wierenga, 1979; Matsubayashi et al., 1997; Haga et al., 1999), trickle bed reactors (Fu and Tan, 1996; Saroha et al., 1998) and column and heap leaching (Villanueva et al., 1990; Decker, 1996) have determined hydrodynamic dispersion coefficient using the advection-dispersion model. Table 2.6 summarises these studies including the properties of the respective experimental work.

The advection-dispersion model represents a single Fick’s law type solute transport mechanism superimposed on to plug flow (see Figure 2.8) (Roman and Bhapu, 1993). It assumes steady state liquid flow with uniform water content and velocity profile (Decker, 1996). The advection-dispersion equation can be derived by assuming a saturated porous medium, where Darcy’s law is applicable and considering the conservation of mass of a representative elemental volume. Equation 2.11 describes the equation in one dimension:

$$\frac{\partial C}{\partial t} = D \frac{\partial^2 C}{\partial Z^2} - \nu \frac{\partial C}{\partial Z}$$  \hspace{1cm} (2.11)

where $C$ is solute concentration, $D$ is dispersion coefficient, $\nu$ is pore water velocity and $Z$ is the column length.

The dissolved solutes transfer through two mechanisms. These are an advective process due to bulk flow velocity of liquid and dispersion caused mainly through variability in lengths of flow paths and velocity variations (both spatial and transient) within a flow path. The
hydrodynamic dispersion coefficient in equation 2.11 accounts for both mechanical dispersion and molecular diffusion (Decker, 1996).

**Table 2.6: Experimental studies using advection-dispersion model to calculate the axial dispersion coefficient.**

<table>
<thead>
<tr>
<th>Author</th>
<th>Experimental Properties</th>
<th>System of Study</th>
</tr>
</thead>
<tbody>
<tr>
<td>De Smedt and Wierenga (1979)</td>
<td>Packed beds 100 µm of glass beads</td>
<td>Hydrology</td>
</tr>
<tr>
<td>Haga et al. (1999)</td>
<td>Packed beds 1 mm of glass beads</td>
<td>Hydrology</td>
</tr>
<tr>
<td>Matsubayashi et al. (1997)</td>
<td>-</td>
<td>Hydrology</td>
</tr>
<tr>
<td>Fu and Tan (1996)</td>
<td>Reactors packed with 0.5, 0.9 and 1.9 mm non-porous particles</td>
<td>Trickle bed reactors</td>
</tr>
<tr>
<td>Saroha et al. (1998)</td>
<td>Reactors packed with 1.5 mm porous alumina extrudates</td>
<td>Trickle bed reactors</td>
</tr>
<tr>
<td>Decker (1996)</td>
<td>Gold ore</td>
<td>Column leaching</td>
</tr>
<tr>
<td>Villanueva et al. (1990)</td>
<td>Saltpetre</td>
<td>Column leaching</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Agitation leaching</td>
</tr>
</tbody>
</table>

**Figure 2.8: Schematic representation of the advection-dispersion model.**

The advection-dispersion equation can be used to describe solute transport if solutes can represent all possible velocity variations while travelling through the media and if velocity is
Chapter 2: Literature Review

independent of the initial velocity. Under fully saturated conditions this is often satisfied and is termed the Fickian regime (Padilla et al. 1999). Then, solutes spread linearly with time and dispersive flux is proportional to the concentration gradient (Padilla et al. 1999).

Padilla et al. (1999) described pre-Fickian regime in unsaturated flow, which has greater velocity fluctuations due to small number of flow paths. Solutes in unsaturated conditions must travel longer distances to reach the Fickian conditions than in a saturated media. Therefore, the validity of the advection-dispersion model is questionable for unsaturated systems (Matsubayashi et al., 1997; Padilla et al., 1999). Alternative models have been developed to address solute transport in unsaturated flow such as found in column and heap leaching.

2.9.2.2. MOBILE-IMMOBILE MODEL (MIM)

Large variations of the pore water velocity, including some zero velocities, are observed in unsaturated porous media (Decker, 1996). The approach of the bimodal distribution of velocities (De Smedt and Wierenga, 1979; Nielsen et al., 1986) is to identify two regions of the solute transport system, which are the mobile region and the stagnant region (Coats and Smith, 1964; De Smedt and Wierenga, 1979; Nielsen et al., 1986; Decker, 1996; de Andrade Lima, 2006). This two region model is a better approximation to the variable velocity field compared to the advection-dispersion equation of the uniform velocity (Decker, 1996).

The model consists of plug flow behaviour with axial dispersion in the mobile region (i.e. advection diffusive behaviour) and mass transfer between the bulk and stagnant flow regions. These mechanisms are shown in Figure 2.9 (Yusuf, 1984; Roman and Bhapu, 1993; de Andrade Lima, 2006).

The total liquid content is the sum of the liquid contents in the mobile region ($\theta_d$) and the immobile region ($\theta_s$) (Decker, 1996).

$$\theta = \theta_d + \theta_s$$ (2.12)

The model assumes the dispersion of liquid occurs only in the dynamic or mobile fraction of the liquid within the packed bed and not in the static liquid fraction. Mass transfer of the solute occurs between the mobile and immobile fractions (Coats and Smith, 1964; De Smedt and Wierenga, 1979; Decker, 1996) and is described by the final term in equations 2.13 and
2.14. Hence, the calculated dispersion coefficient represents the hydrodynamic dispersion coefficient in the mobile mass fraction of the liquid (Decker, 1996).

\[
\begin{align*}
\theta_d \frac{\partial C_d}{\partial t} &= \theta_d D \frac{\partial^2 C_d}{\partial Z^2} - \hat{q} \frac{\partial C_d}{\partial Z} - \alpha (C_d - C_s) \\
\theta_s \frac{\partial C_s}{\partial t} &= \alpha (C_d - C_s)
\end{align*}
\]  

(2.13)  

(2.14)

where \(C_d\) is the solute concentration in the mobile liquid, \(C_s\) is the solute concentration in the stagnant region, \(\hat{q}\) is the Darcy flux, \(D\) is the dispersion coefficient, \(\alpha\) is the mass transfer coefficient between the mobile and immobile zones, \(Z\) is the column length and \(t\) is time.

Bouffard and Dixon (2001) modelled solute transfer through an agglomerated and non-agglomerated ore using various two region models. The first model was identical to mobile-immobile model except that it assumed plug flow behaviour in the mobile region (i.e. no axial dispersion). The two other models also assumed plug flow but the stagnant phase has been considered as an array of uniform or variable pore lengths oriented normal to the bulk flow. Then mass transfer takes place at the mobile-immobile interface. Diffusion is the dominant mechanism to describe solute concentration in pores. The rate of mass transfer is proportional to the diffusional flux which depends on the concentration gradient in the
stagnant phase at the interface. Mowla and Sayyad Amin (2008) have also employed the uniform pore length diffusion model to simulate the effluent reagent transport in an oxide copper ore column leaching.

2.9.2.3. MIXING FLOW MODELS
Mixing flow models have been used to simulate RTD in column and heap leaching (Figure 2.10). de Andrade Lima and Hodouin (2005) presented a mixing model for an industrial leaching tank. It was primarily a continuous stirred reactor with the presence of by-pass flow and a stagnant zone.

Similarly, Bouffard and West-Sells (2009) considered the total volume of heap as sum of plug flow (\(V_p\)), well mixed flow (\(V_m\)) and stagnant/dead volume (\(V_d\)) (Figure 2.10). Further, the total flow rate is divided between the active flow (\(V_a\)) and the by-passing flow (\(V_b\)), which has much less residence time. The relative percentage of each volume was determined using salt tracer tests in columns, cribs and heaps of gold ore.

![Figure 2.10: Schematic representation of the mixing flow model during step change of the tracer concentration (Bouffard and West-Sells, 2009).](image)

2.10. LIQUID DISTRIBUTION IN PACKED BEDS AND HEAPS
Liquid introduced into the system flows vertically through particles with simultaneous flow in the radial direction. Several researchers (eg. Yusuf, 1984; Saroha et al., 1998) have
investigated the parameters of liquid distribution theory and its influence on the liquid distribution. The parameters considered were packing and bed characteristics, liquid and gas flow rate, wetting properties of the particles, liquid properties (density, viscosity, and surface tension) and the initial distribution of liquid.

The following section describes the liquid distribution parameters using the results from both heaps and packed bed reactors including trickle bed reactors. The results and conclusions that have been made with packed bed reactors might not be valid for columns and heap leaching heaps due to significant differences between the two systems.

The packing characteristics, such as the particle size, influence the liquid distribution (Lutran et al., 1991) through a parameter called the liquid spreading factor, which is the liquid spread over the cross-sectional area per unit height of the bed (Porter et al., 1968; Brignole et al., 1973). It increases if the particle size increases from 10 mm to 40 mm (Brignole et al., 1973) resulting in lower contacting efficiency of particles and non-uniform liquid circulation (Yusuf, 1984). The shape of the particles may or may not contribute to the liquid distribution (Porter et al., 1968; Lutran et al., 1991). In addition, the size distribution of the particles affects the liquid distribution but porosity does not necessarily influence it (Yusuf, 1984).

Bed height has a strong impact on the radial liquid distribution. The higher the bed height, the more uniform radial spreading results (Yusuf, 1984). The method of packing also influences the liquid distribution (Herskowitz and Smith, 1978; Ng and Chu, 1987; Al-Dahhan and Dudukovic, 1995; Saroha et al., 1998). Lutran et al. (1991) reported that a large particle top layer would improve liquid distribution in the bed significantly. Further, re-packing of the bed will cause changes in the structure which influences the flow patterns through the particles (Porter et al., 1968). Some investigators (eg. Porter et al., 1968; Lutran et al., 1991) reported that the effect of the initial condition of the bed, that is either pre-wetted or dry before the liquid addition affected the liquid distribution. Finally, wall flow contributes to the non-uniform distribution of liquid that affects the liquid spreading mechanisms within the packed bed (Porter, 1968, Porter et al., 1968; Saroha et al., 1998).

Liquid distribution becomes more uniform when liquid flow rate is increased substantially as turbulence in the bed can induce local eddies to minimise stagnant zones (Yusuf, 1984; Lutran et al., 1991; Saroha et al., 1998). Therefore, at high flow rates a significant fraction of the particles in the bed are in contact with the liquid. Higher gas flow rate also influences
liquid spreading because both gas and liquid compete for interstitial pore space (Lutran et al., 1991).

The internal wetting is defined as the liquid volume inside the pore spaces. The external wetting of particles determines the mass transfer between the internal liquid and the bulk flow (Colombo et al., 1976; Burdett et al., 1981). Contacting efficiency is defined as the fraction of particle surface covered by liquid (Herskowitz, 1981; Mills and Dudokovic, 1981). As the percolated liquid trickles from top to bottom of the particle bed, all the particles are not contacted by liquid. Generally, a non-uniform liquid distribution occurs and isolated stagnant liquid regions are possibly present. The contacting efficiency is increased with liquid flow rate due to better liquid distribution that results from lower stagnant volumes present in the packed bed (Colombo et al., 1976).

The liquid properties such as surface tension and density influence the spread of the liquid. The uniformity of the fluid distribution was found to improve with a decrease in surface tension and density of the liquid (Lutran et al., 1991; Saroha et al., 1998).

Several investigators (eg. Yusuf, 1984; Bartlett, 1992a) have studied the effect of the initial liquid distribution in heaps using various liquid distributing devices. A detailed description of some of those devices is given by Yusuf (1984) and Bartlett (1992a). It was reported that a reasonably uniform liquid distribution could be achieved with a minimum number of 60 distributing points per square metre (Ter Veer et al., 1980). However, it is difficult to maintain the uniform initial liquid spreading through the entire height of the bed. Reduced liquid flow results in non-uniform liquid flow and channelling.

2.10.1. MODELS OF SOLUTION DISTRIBUTION
Mathematical models have been developed to describe the distribution of liquid in packed beds. These are based on the random walk and the diffusion theory.

2.10.1.1. RANDOM WALK
The random walk model assumes that liquid follows a random number of steps to left or right with an equal chance of occurrence when a liquid portion strikes a solid particle. The number of shifts that an elemental liquid volume undergoes through the particles from top to bottom of the packed bed is directly proportional to the bed height and inversely proportional to the reactor diameter. The final horizontal displacement of liquid volume is
expressed by the vector summation of the shifts (Tour and Lerman, 1939). The experimental results of Scott (1935) and the studies of Tour and Lerman (1939a, 1939b and 1944) confirm this hypothesis.

The spread of liquid in the packing in the absence of interference from walls, is given by:

\[ Q_x = k_0 e^{-h_0^2 x^2} \]  
\[ h_0^2 = \frac{C_p}{Z} \quad \text{and} \quad k_0 = \frac{h_0}{\sqrt{\pi}} \]  

where \( Q_x \) is the fraction of the liquid that would fall into a trough of unit width located at a distance \( Z \) from the packing and displaced horizontally at a distance \( x \) from the centre line. Both \( h_0 \) and \( k_0 \) are distribution constants (units are per metre) which depend only on the packing height, type and size of the packing materials. \( C_p \) is the packing constant.

The packing constant, \( C_p \), is independent of the flow rate but depends on the type of the packing. Therefore, it is constant for a given type and size of packing. Also, the value of \( C_p \) determines the degree of horizontal distribution of liquid by packing. For a greater value of \( C_p \), the horizontal liquid spreading is lower, as liquid flows in preference vertically through the packing (Tour and Lerman, 1944). Jameson (1966) reported that the random walk model does not calculate the local flow rates and wall flow observed in packed beds.

2.10.1.2. DIFFUSION THEORY

Due to the probabilistic nature of the liquid distribution as proposed by the random walk model, the process can also be described by a diffusion type differential equation (Porter and Jones, 1963; Jameson, 1966). It can be theoretically expressed as in equation 2.17 (Cihla and Schmidt, 1957).

\[ \frac{\partial f_{(rz\phi)}}{\partial z} = D_s \left[ \frac{1}{r^2} \frac{\partial f_{(rz\phi)}}{\partial r} + \frac{\partial^2 f_{(rz\phi)}}{\partial r^2} + \frac{1}{r^2} \frac{\partial^2 f_{(rz\phi)}}{\partial \phi^2} \right] \]  

where \( f_{(rz\phi)} \) is the vertical flow of liquid per unit area in unit time, \( z \) is the vertical co-ordinate, \( r \) is the radial variable in polar coordinates, \( \phi \) is the angular variable and \( D_s \) is the liquid spread factor (dimension metres). The liquid spread factor is a function of liquid flow rate and is assumed to be equal in all horizontal directions but is influenced by the size and shape of the packing (Porter and Jones, 1963).
When the number of steps in the random walk is sufficiently large, predictions made by both theories are identical. However, the mathematics of the diffusion theory is more convenient than that of the random walk (Porter, 1968).

Cihla and Schmidt (1958) obtained a solution for the equation 2.17 in the case of a packed column with different liquid introduction arrangements assuming that the wall behaved as a perfect reflector of liquid. However, some investigators (e.g., Porter and Jones, 1963; Jameson, 1966) observed that the aforementioned assumptions were insufficient.

In the case of circular columns, the liquid is introduced from the top of the column from an axially symmetrical source, liquid distribution can be derived assuming the equilibrium of elemental volume. The derived equation in polar coordinates (equation 2.18) is identical to the equation 2.17 when the derivative of $f_{r(z)}$ with respect to $\phi$ is neglected (Porter and Jones, 1963).

$$\frac{\partial f_{r(z)}}{\partial z} = D_s \frac{1}{r} \frac{\partial}{\partial r} \left[ r \frac{\partial f_{r(z)}}{\partial r} \right]$$

(2.18)

Porter and Jones (1963) solved the equation for $f_{r(z)}$ including the effects of wall flow. Flow rates reach equilibrium with wall flow when the depth of packing is sufficiently large. Equilibrium will be reached with a smaller depth when the liquid spread factor ($D_s$) is large.

Jameson (1966) calculated the mass balance of liquid over the particles in a two-dimensional bed using the same theory. The packed bed flow behaviour was observed for different particle shapes but same particle size of 1.8 mm.

2.10.1.3. RIVULET MODEL

Porter and co-workers (Porter, 1968; Porter et al., 1968; Porter and Templeman, 1968) described the liquid flow behaviour through randomly packed particles as rivulets. These rivulets have stable flow pattern with respect to time and flow through randomly orientated paths. This hypothesis contradicts the theoretical basis of the random walk method as random walk of a single fluid particle assumes that the movement of liquid elements is independent of each other (Porter, 1968).

Liquid introduced on the top of the dry packed bed moves independently and liquid droplets run through the packing according to the random walk theory. However, once this vanguard liquid has traced out wetted paths through the packing, the remaining liquid flows only along these preferred paths in the form of rivulets. The nose of the rivulet continues its
random path until it leaves the bottom of the bed. When the nose of one rivulet runs into a path which has been already wetted by another rivulet, coalescence takes place. Other than that, two or more rivulets could be formed when the nose of one rivulet strikes a solid particle. The rivulets formed through splitting would trace separate paths through the packing (Porter, 1968). The total flow rate at the bottom of the packed bed is given by the product of the total number of rivulets and the flow rate per rivulet (Porter, 1968; Porter et al., 1968).

The rivulet model is a better description of the flow pattern at lower liquid flow rates compared to high flow rates. Additionally, it describes the channelling flow pattern, but it is not applicable for a fully wetted bed (Porter, 1968).

2.11. MEASURING PARTICLE MOTION USING PEPT

Positron emission particle tracking (PEPT) was developed at the University of Birmingham based on the same detector technology associated with Positron Emission Tomography (PET), which was developed for medical imaging (Parker et al., 1993; Parker et al., 2002). Parker and Fan (2008) conducted a comprehensive review of the methodology and many applications of PEPT. The second operational PEPT facility in the world was opened at iThemba Labs in Cape Town with an ECAD “EXACT3D” PET camera (Model: CTI/Siemens 966) (Buffler et al., 2010). A detailed description of the facility is given by Buffler et al. (2010).

This technique involves a labelled tracer particle, a positron camera and a location algorithm to determine the tracer particle location and speed (Parker and Fan, 2008). It can be used to locate the positions of the tracer particle inside an engineering experimental system, measured within the field of view of a positron camera (Figure 2.11). If the tracer particle is neutrally buoyant and hydrophilic, the hydrophilic tracer particle surface ensures that the tracer particle follows the liquid flow rather than becoming attached to one of the many gas liquid interfaces present in these systems. The tracer particle trajectories can be analysed in order to determine parameters such as velocity, acceleration and occupancy of the tracer particle inside the system.

Therefore, PEPT is a potential technique to investigate the tracer particle, and thus fluid, motion in packed beds. The resultant tracer particle locations inside the packed bed system can be used to understand the liquid flow behaviour in column and heap leaching. A
detailed description of the technique and the experimental procedure are given in Chapter 8.

![Figure 2.11: Tracer particle trajectory within the field of view of the positron camera (Cole, 2011).](image)

2.12. SUMMARY
The fluid flow in heap leaching is unsaturated, consisting of both leaching solution and air (Bartlett, 1992). A survey of heap leaching literature has indicated a need to study heap hydrodynamics more extensively to predict underlying flow mechanics more closely (Roman, 1977; Murr et al., 1981; Yusuf, 1984). Fluid flow behaviour has been measured in various systems at different scales, but there is still a debate as to the major factors at work. The hydrodynamic studies in analogous systems are useful to understand general characteristics such as flow textures and dimensionless numbers that can be applied to describe the flow mechanisms in heaps (de Andrade Lima, 2006). However, the direct application of the principles and the models is not entirely possible due to the significant differences between the two systems (Roman and Bhappu, 1993).

Empirical models have been developed at particle scale to simulate the leaching of the individual ore particles. These particle level models have been coupled to formulate heap level models that describe the heap performance. However, the predictions are not
satisfactory in most situations in industrial heap leaching. The reasons could be the application of fundamental equations that are developed for similar systems but with significant differences (eg. Cross et al. (2006) model is based on Richard’s equation, which was originally developed to describe capillary dominated ground water flow problems) and the oversimplified assumptions such as plug flow behaviour (eg. Roman and Olsen, 1974; Roman et al., 1974; de Andrade Lima, 2004) to build these heap scale models. In addition, there have been a number of studies of the fluid flow in heap leaching systems, but these studies have either tended to be purely empirical or produced qualitative descriptions of the fluid flow behaviour (eg. de Andrade Lima, 2006; Yusuf, 1984).

In order to study heap hydrodynamics, the key areas to be investigated are the flow features through the particles in the heap, liquid holdup in the system and solute transfer mechanisms to transfer dissolved minerals from top to bottom of the heap (Roman and Bhappu, 1993).

The flow features and the different flow regimes found in trickle bed reactors are well characterised due to extensive hydrodynamic studies that have been conducted (Jesser and Elgin, 1943; Holub et al., 1993; Saroha et al., 1998; Toye et al., 1998; Maiti et al., 2006, Maiti et al., 2008). At the low superficial velocities employed in industrial heap leaching, droplet and rivulet flow is the dominant flow feature through the crushed ore particles (Roman and Bhappu, 1993). The liquid distribution models such as random walk model and rivulet model have been proposed in order to address liquid flow mechanisms through the different flow features in packed bed systems (Tour and Lerman, 1939a, 1939b and 1944; Porter, 1968; Porter et al., 1968; Porter and Templeman, 1968).

Liquid holdup determines the wetting efficiency of ore particles, which in turn influences the leaching reactions that take place between the reagents and mineral grains. Empirical models of liquid holdup have been developed (eg. Yusuf, 1984; de Andrade Lima, 2006), but inconsistencies between the models indicate the need for work and better theoretical understanding. The problem with all these approaches is that they either treat the liquid holdup as an empirical input parameter, which limits the predictive ability of the method, or they propose a direct relationship between the liquid holdup and the flow permeability of the system. In this work it will be demonstrated that the use of such a direct relationship is not entirely appropriate for two different reasons. Firstly, these systems exhibit hysteresis,
with the steady state liquid holdup depending not only the current flow rate, but also on the flow-rate history. It will also be demonstrated that the presence of porous particles has a marked influence on the liquid hold-up and flow behaviour.

The packed bed that constitutes a typical heap has porous particles that are mainly in the size range of millimetres to a few centimetres. The porosity of the packed particles has two distinct length scales, namely that of the channels between the particles and within the particles. The Bond number will be significantly different in between these two porosity levels. This distinct separation of length scales means that liquid holdup within the particles will not have the same effect on liquid flow as the holdup between the particles and must thus be considered separately. Therefore, this study will address and model the effect of the particle porosity on the overall flow through a heap by comparing the behaviour of a bed consisting of non-porous glass beads with a system consisting of similar sized ore particles.

The existence of hysteresis in trickle bed reactors (i.e. pressure drop and liquid holdup hysteresis) could be expected in column and heap leaching systems, a comprehensive study of this phenomenon in these systems has not been conducted. This is important as hysteresis could be exploited as a strategy to increase the performance of the industrial process. Therefore, a detailed study will be given in this thesis about the hysteresis behaviour in packed bed and column leaching systems and it could be very important for industrial heap operation in future.

In addition, the transport of dissolved products and reagents under advective and dispersive processes has to be investigated. The two region models which were used to describe the solute transport mechanisms in packed beds systems are more appropriate than the simple advection-dispersion models, which assumed uniform velocity in the porous media. Thus, this study will investigate the mass transfer and dispersion behaviour more closely by using both salt tracers tests and PEPT technique.
CHAPTER 3

EXPERIMENTAL METHOD WITH PACKED BEDS

3.1. INTRODUCTION

This chapter describes the design and commissioning of the 1-D column leaching rig that was used to perform the liquid holdup and the hydrodynamic dispersion experiments. The main features of the rig are the bottom plate to support the Perspex column and the liquid distributor to introduce the liquid into the packed bed. These Perspex columns can be used to measure the internal flow behaviour through the particles.

As the experimental work is based on the determination of liquid holdup in the system gravimetrically, reliable load cell measurements are required. The gravimetrically determined liquid holdup values were compared with an independent drainage method for obtaining the liquid holdup in order to verify the accuracy of the load cell measurements.

The empty column was randomly packed with different mono-sized and poly-dispersed glass beads to make a model system. The determination of steady state and residual liquid holdup values with the model system was performed. Copper ore particles of several narrow size fractions were also used as the packing media. The methods used for both the model glass bead and ore systems will be described in this chapter.

This 1-D packed bed was used to quantify the axial dispersion coefficients using salt tracer experiments. The methodology and the data acquisition system of these experiments will be described, followed by a summary of this chapter.
3.2. 1-D EXPERIMENTAL RIG

Figure 3.1: 1-D column leaching rig used in this study.
To investigate heap hydrodynamics, the liquid flow experiments need to be carried out at a range of flow rates and different particle sizes and types. These included mono-dispersed and poly-dispersed glass beads, and copper ore particles in narrow size ranges. Figure 3.1 shows the total system for the 1-D column leaching rig and the individual components are labelled. The different components of the leaching experimental setup are described in the following sections.

3.2.1. EMPTY COLUMN

Flanged Perspex cylinders were fitted onto a Perspex and steel base plate to form the empty column used in this work (Figure 3.1).

3.2.1.1. FLANGED PERSPEX CYLINDERS

A number of cylindrical circular Perspex columns of 243 mm internal diameter and 6 mm wall thickness and of different lengths were fabricated. At each end of the column there were flanges of 325 mm outside diameter and 15 mm thickness, with 8 holes of 10.5 mm diameter equally spaced on a 290 mm pitch circle diameter (PCD) to mount the cylinders on the load cell and attach the bottom plate. Two lengths of Perspex column of 300 mm and 500 mm were used individually (Figure 3.2) and in combination in order to perform experiments in the height range of 300-800 mm.

![Figure 3.2: 300 mm (left) and 500 mm (right) cylindrical Perspex sections.](image)
When connecting the two pieces to each other, a neoprene ring of 3 mm thickness was included to provide a water-tight seal between them.

### 3.2.1.2. BOTTOM PLATE FOR THE PACKED COLUMNS

The bottom plate supports the weight of the Perspex column, packed particles and water held within the column and altogether is capable of withstanding more than 100 kg. Further, it allows water to flow freely out of the bottom of the bed.

The bottom plate has a circular Perspex section of 325 mm outside diameter and 40 mm thickness (Figure 3.3a) and was machined to have a slight conical depression to ease the passage of water from the column. A groove was machined to place the O-ring (5 mm thickness) (Figure 3.3a) which stops any leakage of water from the contact surface area of the packed bed and the bottom plate.

Three grooved Perspex rings were fitted into three inner recesses machined into the bottom plate (Figure 3.3b). These Perspex rings acted as drainage pathways and supported the heavy weight of the packed bed from above.

![Figure 3.3: Design and the main components of the bottom plate including the drainage pathways and the O-ring.](image)

A perforated sheet of 250 mm diameter with 1.7 mm round holes was placed on top in an additional machined recess positioned between the outermost circular Perspex ring and the O-ring groove (see Figure 3.3 and 3.4). The final bottom plate assembly is shown in Figure 3.4. A 316 grade stainless steel supporting plate (330 mm outside diameter, 12.5 mm
thickness) was added to the bottom of the Perspex plate to support the weight of the packed bed.

**Figure 3.4:** Perspex and steel bottom plates are shown separately.

The bottom plates were fixed on the cylinder flange through 6 of the 8 holes with 10 mm stainless steel nuts and bolts. Stainless steel studding of 10 mm diameter was used to suspend the entire vessel on the metal frame, through the final pair of holes in the plate and flange (Figure 3.5). The metal frame was constructed of stainless steel and enclosed the experimental system and supported the weight of the packed bed (Figure 3.1). The total height was 1.72 m.

**Figure 3.5:** The bottom plate attached to the 300 mm Perspex column with the stud sections to mount the column to the metal frame.
3.2.2. LOAD CELL AND DATA LOGGER
At the top of the supporting frame the load cell was mounted between two parallel square metal sections as shown in Figure 3.6.

![Safety nuts to prevent the failure of the system](image)

Figure 3.6: The top section of the experimental rig. The safety nuts and the load cell are shown.

Liquid flow experiments were mainly conducted by measuring the liquid holdup in the packed bed. The gravimetric method measures the liquid holdup by subtracting the dry weight of the column from the weight of the packed bed at steady state liquid flow. The S-type high sensitivity stainless steel load cell (Model: LCM 101-100 cable type) of 0-100 kg range was selected to measure the liquid holdup. It has ± 0.03% full scale linearity and the output is 3 mV/V (± 0.0075 mV/V) when load cell is excited by 10 V (DC) (i.e. input) (see Table 3.1). The load cell has 150 kg of safe overload capacity and it has been pre-calibrated in tension (Note: this calibration was verified in-situ).

The load cell was coupled to a 24 bit high resolution data logger (Model: DP41-B) and data acquisition module (National Instruments), which connects to a computer in order to continuously read and record the weights measured from the load cell. The data logger also provided inbuilt excitation (i.e. 10 V (DC) at 30 mA) to the load cell and the precision of the data logger was ± 0.005% reading. Table 3.1 shows resolution and accuracy of the system for the liquid holdup measurement.
Table 3.1: Resolution and accuracy of the connected system.

<table>
<thead>
<tr>
<th>Description</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Range of measurement</td>
<td>0-100 kg</td>
</tr>
<tr>
<td>Excitation</td>
<td>10 V (inbuilt)</td>
</tr>
<tr>
<td>Output signal</td>
<td>3 mV/V</td>
</tr>
<tr>
<td>Resulting output signal at full range</td>
<td>3*10 mV=30 mV</td>
</tr>
<tr>
<td>Number of divisions in the data logger</td>
<td>16777216</td>
</tr>
<tr>
<td>Unit step of measurement</td>
<td>0.00178 µV</td>
</tr>
<tr>
<td>Resolution of the system</td>
<td>(100<em>1000 g</em>0.00178 µV)/30000 µV = 5.93 mg</td>
</tr>
<tr>
<td>Full scale accuracy of load cell</td>
<td>0.03*100 kg/100 = 30 g</td>
</tr>
</tbody>
</table>

The operational diagram of the load cell and data logger system is shown in Figure 3.7.

Despite the factory calibration of the load cell, a separate manual calibration was carried out to obtain a calibration curve. The load cell was mounted on the column leaching rig and weights of known mass were attached in the range of 0-80 kg. The load cell calibration was linear (106 data points) and is shown in Figure 3.8.
The relationship between the data logger reading and the weight suspended by the load cell (Equation 3.1) can be used to calculate the weight given by the load cell. As all of the measurements in this work require differences in weight, the most important aspect of this calibration is the slope of this line.

\[
weight \ (kg) = \frac{Panel \ meter \ reading - 0.1793}{1.105}
\]  
(3.1)

3.2.3. LIQUID DISTRIBUTOR

Even liquid distribution at the top surface of the particles and the mounting method of the distributor as a separate unit from the packed bed are crucial when the experimental method requires, measurement of the total liquid holdup in the system gravimetrically.

3.2.3.1. EVEN DISTRIBUTION OF WATER AT THE TOP OF THE PACKED BED

Several designs were tested to produce a liquid distributor capable of consistently even addition but these initial designs were unsuccessful. This section only describes the successful liquid distributor. It was designed to ensure even liquid distribution at low flow rates (i.e. 1 L/h or even less in this study) in the relatively high column diameter of 243 mm used in this study. Silicon tubes (1mm bore) and PVC tubes (1.5 mm bore) were cut to be the same length to the nearest millimetre, then joined with T-connectors to make a 16 drip point tube network by repeatedly branching the tubes as shown in Figure 3.9 (Cole, 2010c).
This design maintains the same pressure at every final drip point, if the tubes at each level are of the same length. Two 16 drip point networks were coupled to each other using PVC tubing to make a single 32 drip point tube network (Figure 3.10a).

Figure 3.9: The tube network used in the liquid distributor.

Figure 3.10: Vertically mounted drip points (left) and the total liquid distributor of 32 drip points mounted over the column (right).
Evenly spaced holes were drilled into a 253 mm diameter nylon disc to form 32 drip points. Two identical nylon discs were used, sandwiched by a 60 mm height Perspex cylinder. Therefore the 32 drip points were formed from vertical tubes, which minimised the occurrence of air bubbles that tended to form in curly bends in the tubes (Figure 3.10a). These bubbles would have led to an uneven liquid distribution at the top surface of the packed bed.

Two peristaltic pumps were used to pump water through the liquid distributor (Figure 3.1). Pump A (range of flow rate, 0-22 L/h) was used at first to remove air bubbles from the tubes by operating it at a higher flow rate because air bubbles adversely affected the even liquid distribution. After removing all the air bubbles (i.e. flushing) the liquid distribution was uniform. Then pump B (range of flow rate, 0-5 L/h) was switched on at the desired flow rate depending on the experimental conditions. Finally, the speed of pump A was gradually reduced to zero to achieve the desired low flow rate with pump B, while preventing the formation of air bubbles. The outlet flow rate of the liquid distributor was measured manually before each of the experiments.

**3.2.3.2. NUMBER OF DRIP POINTS**

Types of liquid distributors were discussed by Yusuf (1984) and the novel liquid distributor developed in this study is a uniform type liquid distributor to uniformly distribute liquid over the entire surface area of the pack. Ter Veer *et al.* (1980) have investigated initial liquid distribution and quantified the liquid maldistribution by introducing a parameter called liquid maldistribution index which goes to minimum when the liquid distributor has at least 60 distributing points per square metre. In order to have more uniform liquid distribution, the liquid distributor must be designed with more than 60 drip points per square metre. This criteria has been checked in Table 3.2 for the liquid distributor used in this work.

**Table 3.2: Comparison of the number of drip points per square metre for this study and Ter Veer *et al.* (1980).**

<table>
<thead>
<tr>
<th>Study</th>
<th>Number of Drip Points</th>
<th>Drip Points per m$^2$</th>
</tr>
</thead>
<tbody>
<tr>
<td>This Study</td>
<td>32</td>
<td>690</td>
</tr>
<tr>
<td>Ter Veer <em>et al.</em> (1980)</td>
<td>1,5,9,12, 21,37</td>
<td>6.5,33,59,79,138,243</td>
</tr>
</tbody>
</table>
The uniform type 32 drip points liquid distributor used in this study have more than ten times the drip points per square metre than the criteria suggested by Ter Veer et al. (1980) and indeed the liquid distributor wets the top of the bed uniformly.

3.2.3.3. MOUNTING OF THE LIQUID DISTRIBUTOR
The liquid distributor was mounted separately to the rest of the vessel so that the weight of the tubing and the liquid in the distributor did not affect the weight of the column measured by the load cell. To achieve this, the liquid distributor was fixed to a metal bar which was attached to a vertical stand (Figure 3.10b). The liquid distributor was levelled and could move forward and backward with the fixed stand. In every experiment, the liquid distributor was moved forward along the table to keep it exactly above the packed bed. Then, it was placed 10-15 mm above the packed bed to ensure that there was a gap between the liquid distributor and the top of the packed bed (Figure 3.10b).

3.2.4. PACKING MEDIA
The liquid flow experiments were conducted through packed beds consisting of mono-dispersed and poly-dispersed glass beads and relatively narrow size ranges of copper ore particles.

3.2.4.1. GLASS BEADS
Initially hydrodynamic tests were performed with mono-dispersed packed beds. This model system is used as the particle size can be well defined and the surface properties of the glass beads are well characterised. Four different sizes of glass spheres were used as the packing material: 2, 10, 14 and 18 mm (Figure 3.11). The typical particle size range employed in industrial heap leaching operations is 10–20 mm. Thus, 10 mm, 14 mm and 18 mm particles were selected in relevance to the industrial application, which is reflected in the choice of larger fractions in the laboratory scale vessel. However, the smallest size (2 mm) was selected to represent flow through regions of fines. It is in a different Bond number regime (see chapter 4).

In terms of surface properties, the glass beads were smooth and non-porous. Sphericity was almost one according the manufacturer (i.e. high sphericity particles). The contact angle was determined with the 18 mm beads experimentally and found to be within the range of 15-20 degrees.
Poly-dispersed packing material was created by thoroughly mixing equal weight proportions of two (i.e. binary mixture) or three individual particle sizes (i.e. ternary mixture). Three binary mixtures were employed and those were 10 mm and 14 mm, 10 mm and 18 mm and 14 mm and 18 mm. However, the mixture of 10, 14 and 18 mm beads was the only ternary blend.

![Figure 3.11: Glass beads of different sizes that used in this experimental study to make the model system.](image)

**3.2.4.2. COPPER ORE PARTICLES**

The same experiments performed with the non-porous glass beads were repeated with slightly porous copper ore particles in order to check the applicability of the liquid holdup model developed in this study. A sample of copper ore of around 285 kg was collected from Kennecott Utah Bingham Canyon mine.

Kennecott Utah Bingham Canyon mine is a low grade ore deposit that contains finely disseminated sulphide minerals, primarily copper and iron sulphides within a predominantly quartz monzonite host rock (Lufkin, 2010; Rio Tinto, 2009). This porphyry copper ore body has zones of both primary, containing mainly Chalcopryite (CuFeS₂) and Bornite (Cu₅FeS₄), and secondary, containing Chalcocite (Cu₂S) and other species, sulphide mineralisation. The current grade of this deposit is less than about 0.75% copper (Lufkin, 2010). After drilling
and blasting, the rock is crushed to less than 250 mm (10 inches) in diameter in a gyratory crusher (Rio Tinto, 2012). The ore sample used in this project was obtained from the product of this primary crusher and it composed of particles from dust size to around 250 mm. The original ore sample thus has a size distribution that is wider and coarser than that typically encountered in heap leaching and is more typical of a dump leaching size distribution. The original size distribution is not relevant to the results obtained as narrow size intervals were obtained from the sample for use in the experiments presented in this work.

Sieve analysis was performed using the sieve sizes of 2, 4, 8, 11.2, 13.2, 16, 20, 26.5, 31.5, 37.5 and 45 mm. The resultant particle size distribution curve is shown in Figure 3.12.

Narrow size fractions of copper ore particles in the size ranges of 4-8 mm, 8-11.2 mm, 11.2-13.2 mm, 13.2-16 mm, 16-20 mm, 20-26.5 mm, 26.5-31.5 mm, 31.5-37.5 mm and 37.5-45 mm were used in the liquid holdup experiments in this work (Figure 3.13).

![Particle size distribution of the original copper ore sample.](image)

Geometric mean size (square root of the product of the smallest size and largest size of the sample) was used in this work to represent average particle size of each of the above copper ore fractions. The calculated geometric mean sizes were 5.7, 9.5, 12.2, 14.5, 17.9, 23.0, 28.9,
34.4, 41.1 mm respectively. All the copper ore experiments were conducted in the 300 mm column, as there were insufficient particles in each size range to fill a 500 mm column.

<table>
<thead>
<tr>
<th>Size Range</th>
</tr>
</thead>
<tbody>
<tr>
<td>4-8 mm</td>
</tr>
<tr>
<td>8-11.2 mm</td>
</tr>
<tr>
<td>11.2-13.2 mm</td>
</tr>
<tr>
<td>13.2-16 mm</td>
</tr>
<tr>
<td>16-20 mm</td>
</tr>
<tr>
<td>20-26.5 mm</td>
</tr>
<tr>
<td>26.5-31.5 mm</td>
</tr>
<tr>
<td>31.5-37.5 mm</td>
</tr>
<tr>
<td>37.5-45 mm</td>
</tr>
</tbody>
</table>

Figure 3.13: Copper ore particles of selected narrow size ranges used in this study.

The copper ore particles in each size range were re-used during subsequent liquid flow experiments. During the experiments, the loss of some smaller particles (i.e. smaller than 1.7 mm, which is the size of the apertures in the bottom plate) was observed. These small particles probably remained attached to the larger particles during the dry sieving and only became detached upon liquid addition. The weight loss of these small particles is negligible compared to the overall liquid holdup within the packed bed. During the pre-soaking of the particles and their packing into the columns, some damage is inflicted and fines are produced. To ensure that the particles do not become finer with a size class over a series of experiments, they are re-screened after each experiment and assigned to their correct size interval.

3.2.4.3. VOIDAGE AND POROSITY

The inter-particle space in the packed bed is referred to as voidage whereas porosity implies the intra-particle pore spaces of individual particles. The water accessible porosity was obtained by subtracting the weight of ore particles which were soaked for 3 days from the dry weight of the particles. The soaked particles were screened and patted down with a cloth to remove any external water. This can cause some inaccuracies and therefore
porosity values are only estimates. Also this will be less than the actual porosity as some of the pore volume might not be connected to the particle surface. In addition, for a randomly packed column of the glass beads or the copper ore, the average voidage was measured experimentally. The porosity and voidage values are shown in Table 3.3. The resultant voidage values agree with typical voidage percentages found in packed bed studies (Sato and Hirose, 1973; Bartlett, 1992a).

Table 3.3: Experimentally determined voidage and porosity values of the packed beds.

<table>
<thead>
<tr>
<th>Packing media</th>
<th>Voidage (%)</th>
<th>Estimated Porosity (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Glass bead system</td>
<td></td>
<td></td>
</tr>
<tr>
<td>2 mm</td>
<td>38.9</td>
<td>0</td>
</tr>
<tr>
<td>10 mm</td>
<td>39.9</td>
<td>0</td>
</tr>
<tr>
<td>14 mm</td>
<td>40.5</td>
<td>0</td>
</tr>
<tr>
<td>18 mm</td>
<td>40.8</td>
<td>0</td>
</tr>
<tr>
<td>Mixture of 10 and 14 mm (50% - 50%)</td>
<td>40.1</td>
<td>0</td>
</tr>
<tr>
<td>Mixture of 10 and 18 mm (50% - 50%)</td>
<td>40.2</td>
<td>0</td>
</tr>
<tr>
<td>Mixture of 14 and 18 mm (50% - 50%)</td>
<td>40.7</td>
<td>0</td>
</tr>
<tr>
<td>Mixture of 10, 14 and 18 mm (33.3% - 33.3% - 33.3%)</td>
<td>40.0</td>
<td>0</td>
</tr>
<tr>
<td>Copper ore system</td>
<td></td>
<td></td>
</tr>
<tr>
<td>4-8 mm (5.7 mm)</td>
<td>34.7</td>
<td>7.1</td>
</tr>
<tr>
<td>8-11.2 mm (9.5 mm)</td>
<td>35.3</td>
<td>4.8</td>
</tr>
<tr>
<td>11.2-13.2 mm (12.2 mm)</td>
<td>35.4</td>
<td>2.7</td>
</tr>
<tr>
<td>13.2-16 mm (14.5 mm)</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>16-20 mm (17.9 mm)</td>
<td>36.2</td>
<td>3.0</td>
</tr>
<tr>
<td>20-26.5 mm (23.0 mm)</td>
<td>36.3</td>
<td>2.2</td>
</tr>
<tr>
<td>26.5-31.5 mm (28.9 mm)</td>
<td>37.1</td>
<td>2.0</td>
</tr>
<tr>
<td>31.5-37.5 mm (34.4 mm)</td>
<td>38.5</td>
<td>3.8</td>
</tr>
<tr>
<td>37.5-45 mm (41.1 mm)</td>
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<td>2.2</td>
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<td>Mixture of 8-11.2 mm and 16-20 mm (50% - 50%)</td>
<td>35.2</td>
<td>4.6</td>
</tr>
<tr>
<td>Mixture of 11.2-13.2 mm and 20-26.5 mm (50% - 50%)</td>
<td>33.7</td>
<td>3.5</td>
</tr>
<tr>
<td>Mixture of 13.2-16 mm and 26.5-31.5 mm (50% - 50%)</td>
<td>33.7</td>
<td>3.0</td>
</tr>
<tr>
<td>Mixture of 16-20 mm and 31.5-37.5 mm (50% - 50%)</td>
<td>35.2</td>
<td>3.4</td>
</tr>
</tbody>
</table>

3.2.4.4. LIMITATIONS OF THIS STUDY
This study employs laboratory scale packed bed experiments using non-porous glass beads and slightly porous ore particles and hence there are significant limitations in terms of fully representing an industrial heap leaching system. Narrowly sized ore particles were used during these liquid flow experiments, whereas the actual size range of the heap material is
wider than those used in this study. In addition, the particle packing in the columns is quite regular compared to the often inhomogeneous packings produced during heap construction. The techniques used in industrial heap construction lead to particle segregation and compaction during the process. Furthermore, the height of the packed bed was less than a metre, which is much less than typical industrial heap heights of a few meters. The pressure exerted by the overlying heap can cause compaction and reduced voidage and permeability.

The main aim of the study is to investigate the underlying flow mechanisms, many of which will be the same irrespective of the details of the specific heap or packed bed and thus using a well defined system helps to isolate the mechanisms and dependencies. In order to make quantitative predictions of the behaviour of real heaps, experiments using more realistic particle size distributions and packings will need to be conducted.

### 3.3. EXPERIMENTAL CONDITIONS

A summary of the experimental conditions for the 1-D column tests are listed in Table 3.4. The particle sizes and flow rates were chosen to represent the typical values found in packed beds and column leaching.

Before each experiment, the empty column was vertically suspended from the load cell and levelled. The weight of the empty column was recorded. Then, the packing media was gradually and randomly loaded into the column to make the packed bed. The height of the bed was equal to the height of the Perspex column. The weight of the dry packed bed was then recorded. The liquid distributor was placed 10-15 mm above the top of the packed bed and initiated at the desired flow rate. The next steps were followed depending on whether dry or wet bed operation was required. For dry bed operation the liquid distributor was used to introduce water to the dry bed at the desired flow rate. However, for wet bed operation the suspended bed was initially irrigated at a higher flow rate with the outlet sealed. This flooded the bed and when the water level reached the top of the bed, the desired flow rate for liquid introduction was set. Then the outlet of the bed was opened to drain the excess water from the bed.
3.4. LIQUID HOLDUP MEASUREMENTS WITH NON-POROUS PARTICLES

This section details steady state and residual liquid holdup measurements with non-porous particles in the 1-D packed bed. Glass beads were used as the non-porous packing media. This is referred to as the model system in later sections.

3.4.1. STEADY STATE LIQUID HOLDUP

The main measurement technique used was the gravimetric method. An additional method, the drainage method, was used to validate the accuracy of the gravimetric method. These two independent measurement techniques are described below.

Table 3.4: Summary of the experimental conditions in this study.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Flow rate (L/h)</td>
<td>1.26, 2.52, 5.04, 10.08, 20.16</td>
</tr>
<tr>
<td>Superficial flow rate (mm/s)</td>
<td>0.0075, 0.015, 0.03, 0.06, 0.12</td>
</tr>
<tr>
<td>Liquid</td>
<td>Deionised water</td>
</tr>
<tr>
<td>Operating temperature</td>
<td>Room temperature</td>
</tr>
<tr>
<td>Operating pressure</td>
<td>Atmospheric pressure</td>
</tr>
<tr>
<td>Column height (mm)</td>
<td>300, 500, 800</td>
</tr>
<tr>
<td>Non-porous system</td>
<td></td>
</tr>
<tr>
<td>Particle size of glass beads (mm)</td>
<td>2, 10, 14, 18</td>
</tr>
<tr>
<td>Packing</td>
<td>Mono-dispersed random and poly-dispersed random</td>
</tr>
<tr>
<td>Start-up condition</td>
<td>Dry or wet bed</td>
</tr>
<tr>
<td>Investigation of hysteresis behaviour</td>
<td>Continuous operation for three cycles of flow rate increasing and subsequent flow rate decreasing</td>
</tr>
<tr>
<td>Porous system</td>
<td></td>
</tr>
<tr>
<td>Particle size ranges of copper ore (mm)</td>
<td>4-8, 8-11.2, 11.2-13.2, 13.2-16, 16-20, 20-26.5, 26.5-31.5, 31.5-37.5, 37.5-45</td>
</tr>
<tr>
<td>Packing</td>
<td>Random packing of narrow size ranged particles and poly-dispersed ore particles</td>
</tr>
<tr>
<td>Start-up condition</td>
<td>Dry bed and Externally dried pore saturated ore particles</td>
</tr>
</tbody>
</table>
3.4.1.1. GRAVIMETRIC METHOD
The gravimetric method quantifies the total liquid holdup value by subtracting the initial weight of the system from the weight at steady state of the system. The method is more direct than other methods to measure the liquid holdup as long as the measurement system can accurately record weights. The data logger reading was recorded every 30 seconds after the introduction of the water to the packed bed. This continued until the steady state of the packed bed was reached which corresponds to a constant reading of the data logger.

The time taken to reach the steady state of the bed depends on the particle size, flow rate, column height and the start-up condition of the bed. This usually took 60-90 minutes. For practical purposes a constant reading over five minutes was considered to be steady state and that constant reading was recorded as the steady state liquid holdup.

3.4.1.2. DRAINAGE METHOD
The drainage method was carried out to confirm the accuracy of the gravimetric method. A measuring beaker was kept below the column outlet to collect the liquid after the introduction of water to the packed bed as shown in Figure 3.14.

![Figure 3.14: Experimental set-up to measure the drained liquid from the column using an electronic balance.](image)

It was more practical to weigh the drained liquid with an electronic balance, rather than measure its volume, over extended periods of time. The drained weight of water was recorded every 30 seconds until the bed reached its steady state. After calculating drained
liquid volume from the mass of liquid, the volume was integrated to calculate the total liquid holdup, \( \theta \), (Equation 3.2).

\[
\theta = \frac{\int (V_{in} - V_{out}) dt}{V_c}
\]  \hspace{1cm} (3.2)

where \( V_{in} \) is water volume in, \( V_{out} \) is water volume out and \( V_c \) is volume of the empty column.

As the total volume out was measured and assuming \( V_{in} \) is constant, equation 3.2 becomes equation 3.3 for the calculation of total liquid holdup.

\[
\theta = \frac{V_{int} - M_{out}}{V_c} \rho
\]  \hspace{1cm} (3.3)

where \( M_{out} \) is the total mass of liquid drained out of the packed bed, \( \rho \) is density of water and \( t \) is time.

### 3.4.1.3. ACCURACY OF THE MEASUREMENT TECHNIQUES

Liquid holdup values from both techniques are compared to check their agreement to each other (Figure 3.15). It provides a possible way to investigate the accuracy of load cell measurements because gravimetric method is the main liquid holdup measurement procedure in this work.

The total liquid mass given by the load cell measurements and the integrated liquid content by collecting the drained liquid of the same bed is compared for all the particle sizes. Figure 3.15 shows the weight of liquid retained in a 300 mm column packed with different size glass beads and with a superficial velocity of 0.0075 mm/s (the lowest used in this work and thus the one requiring the highest accuracy from the load cell).

These two completely independent methods measure the amount of liquid held up in the column and show excellent agreement with one another. This implies that the results obtained from the gravimetric measurements are reliable under these experimental conditions and the measurements given by the load cell are thus accurate enough to be used in the subsequent experiments.
Chapter 3: Experimental Method with Packed Beds

Figure 3.15: Holdup variation of drainage and gravimetric methods for all the particle sizes of the model system. The packed bed was initially dry and superficial velocity was 0.0075 mm/s. a) 2 mm b) 10 mm c) 14 mm d) 18 mm.

3.4.2. RESIDUAL LIQUID HOLDUP

After the column packed with glass beads reached steady state, the liquid flow to the top of the bed was suddenly stopped (Figure 3.16). The column was then allowed to drain for a sufficient time, usually 15-20 minutes for all the sizes of glass beds used in this study. When there was no more drainage, the data logger reading was constant and recorded as the weight of the drained bed (Figure 3.16). The weight difference between the drained bed and the dry bed gives the residual holdup for that particular flow rate.
Most investigators (e.g. de Klerk, 2003; Lange et al., 2005) have defined this liquid holdup value as the static liquid holdup. In this work, the term residual liquid holdup is used as it is not obvious that the static liquid in the operating bed must be the same as the residual liquid holdup.

The same procedure was carried out by changing the experimental variables such as liquid flow rate, particle size, packed bed height and start up condition to cover the entire range of experimental conditions listed in Table 3.4.

3.5. LIQUID HOLDUP MEASUREMENTS WITH POROUS ORE PARTICLES

Liquid holdup experiments in the 1-D model system were conducted using the ore particles in narrow size ranges to validate the liquid holdup model developed for the model glass beads system.

The 300 mm column was filled with a selected narrowly sized fraction of ore particles as described in section 3.2.4.2. Generally about 18 kg of ore particles were required to fill the column. Initially the particles were dry and the column was randomly packed. The same
methodology described in section 3.4.2. was followed to obtain a set of steady state and residual liquid holdup values for each flow rate studied.

3.6. HYDRODYNAMIC DISPERSION MEASUREMENTS

A pulse, step increase or decrease tracer injection into the packed bed makes it possible to investigate the residence time distribution (RTD) of a packed bed. The RTD can be used to determine the hydrodynamic dispersion coefficient. During this study, the pulse tracer injection was carried out for mono-dispersed packed beds of glass beds of four different sizes. The experiments were performed in the 300 mm column for both dry bed and wet bed start up conditions and the flow rates mentioned in Table 3.4.

3.6.1. TRACER TYPE AND INJECTION

The tracer used in RTD experiments should be conservative and non-absorbing, such as bromide and chloride (Decker, 1996). For the experiments using packed beds of mono-dispersed glass beads, calcium chloride (CaCl₂) was used. The concentration of the input CaCl₂ solution was 2 mol/dm³ in all the experiments. A small amount of tracer was injected into the flow path of one drip point in the liquid distributor at the top of the packed bed. During the initial experiments, the tracer volume was varied to check for any variations in the normalised effluent tracer concentration curve due to the changes in tracer volume.

Figure 3.17 shows the scaled effluent conductivity curves for different tracer volumes in the range of 0.05-1 ml against time, obtained from the packed bed of 18 mm particles at a superficial liquid addition rate of 0.06 mm/s. It is important to mention that each tracer volume used gives a similar effluent concentration but would change the volume of the system due to the addition of higher tracer volume. Thus the tracer volume of 0.05 ml was injected in all the experiments performed during this study.
Figure 3.17: The scaled effluent tracer conductivity curves for different tracer volumes injected at the inlet of the column. The 300 mm column packed with 18 mm particles was used and the liquid superficial velocity was 0.06 mm/s.

3.6.2. MEASUREMENT OF THE EFFLUENT TRACER CONCENTRATION

At the outlet of the column, a small reservoir was installed to hold a pre-calibrated conductivity meter which was configured to ensure that the conductivity cell was fully submerged when the reservoir was completely filled. Therefore, the small reservoir continuously spilled when measuring the current conductivity value at a particular time. This method was similar to the experimental method employed by de Andrade Lima (2006) despite the fact that the small reservoir had a hole at the bottom to continuously remove the collected solution. Initially three reservoirs of 25, 30 and 35 ml volume were made and their effect on the measured tracer concentration was checked by comparing the RTD curves at the same flow rate.

Figure 3.18 shows the RTD curves for a packed bed of 18 mm particles at a superficial liquid flow rate of 0.03 mm/s. There is a small effect on the RTD curve when changing the reservoir volume. However, 25 ml and 30 ml reservoirs were difficult to use to measure the effluent conductivity because their water level did not always entirely cover the conductivity cell, likely introducing errors in the conductivity measurement. Therefore, a 35 ml reservoir
Experimental Method with Packed Beds

was used in all the experiments during this study to measure the effluent tracer conductivity.

Figure 3.18: Residence time distribution curves using different small reservoirs at the outlet of the column. The 300 mm column packed with 18 mm particles was used and the liquid superficial velocity was 0.03 mm/s.

Despite the shown suitability of such a small reservoir to measure the effluent tracer concentration, it is known that it induces an error due to the residence time of the reservoir, especially at the low liquid flow rates used in column leaching. This error can be corrected mathematically by assuming that the small reservoir behaves as a well-mixed system when liquid from the column falls into it.

Figure 3.19 illustrates the small reservoir, which has a volume $V_r$.

The liquid flow rate into the reservoir ($Q_{res}$) is equal to the liquid flow rate at top of the column at steady state ($Q_L$). $C_{in}(t)$ is the actual tracer concentration in the liquid flowing into the small reservoir at time, $t$ and $C_m(t)$ is the measured tracer concentration using the conductivity cell in the small reservoir at time, $t$.

The tracer mass in the small reservoir ($m_r$) is given by:

$$m_r = m_{initial} + m_{in} - m_{out}$$  \hspace{1cm} (3.4)
where, \( m_{\text{initial}} \) is the initial mass of the tracer in the small reservoir, \( m_{\text{in}} \) is the tracer mass addition and \( m_{\text{out}} \) is tracer mass removal from the small reservoir.

![Schematic representation of the small reservoir with parameters to perform conductivity corrections.](image)

Figure 3.19: Schematic representation of the small reservoir with parameters to perform conductivity corrections.

\[
C_m(t)V_r = \int_0^t C_{in}(t)Qdt - \int_0^t C_m(t)Qdt \tag{3.5}
\]

The residence time of the small reservoir \((t_r)\) is:

\[
\frac{\nu_r}{Q_{res}} = \frac{V_r}{Q_L} \tag{3.6}
\]

Now equation 3.5 can be rewritten as:

\[
C_m(T)t_r = \int_0^T (C_{in} - C_m)dt \tag{3.7}
\]

\[
\int_0^T C_{in}dt = C_m(T)t_r + \int_0^T C_m dt \tag{3.8}
\]

Equation 3.8 can be approximated as follows:

\[
\sum_{i=0}^n C_{in,i} \Delta t \approx C_{in,n}t_r + \sum_{i=0}^n C_{m,i} \Delta t \tag{3.9}
\]

Now, the corrected tracer concentration in the small reservoir:

\[
C_{in,n} \approx \frac{C_{m,n}t_r}{\Delta t} + \sum_{i=0}^n C_{m,i} - \sum_{i=0}^{n-1} C_{in,i} \tag{3.10}
\]

The measured effluent tracer concentration at a particular time can be corrected using equation 3.10 to determine the corrected tracer concentration at that time without the influence of the residence time of the small reservoir. This correction is based on the
assumption that the liquid volume in the small reservoir is well mixed. Ideally, the liquid in the small reservoir needs to be stirred using a magnetic stirrer which would be difficult with the presence of the conductivity probe inside the small reservoir.

Figure 3.20 shows the RTD plots of the measured and the corrected tracer concentrations at two liquid superficial velocities for a packed bed of 18 mm particles. Figure 3.20 on the left illustrates the RTD curves for a superficial velocity of 0.03 mm/s whereas right shows the RTD curves at a superficial velocity of 0.12 mm/s.

Figure 3.20: Comparison of the corrected and measured RTD curves for 300 mm packed bed of 18 mm particles at superficial velocities of 0.03 mm/s (left) and 0.12 mm/s (right).

Figure 3.20 clearly shows the effect of the liquid flow rate on the resultant RTD curve measured using a small reservoir. At higher flow rates, the lower residence time of the small reservoir, results in a RTD curve with a small error. The difference between the mean residence time of the corrected and the measured curves (i.e. $t_{R_{corrected}} - t_{R_{measured}}$) is 6 seconds. However, a significant error is observed at low flow rates due to the much higher residence time of the small reservoir and the larger difference between the residence times of the two curves, which is 26 seconds. Therefore, the correction of the measured conductivities following equation 3.10 is important, especially at low liquid superficial velocities in order to remove the error associated with the residence time of the small reservoir.
3.6.3. DATA ACQUISITION SYSTEM

The conductivity meter has an analog output which was configured using the high frequency data acquisition hardware module (National Instruments) and LabVIEW software to record the effluent tracer concentration data every 500 ms to a computer (Figure 3.21). This data acquisition procedure is continuous and highly accurate compared with the discrete parameter measurements using selected samples within relatively large time intervals employed by some researchers during similar experiments (e.g., De Smedt and Wierenga, 1979; Haga et al., 1999).

![Figure 3.21: The components of the data acquisition system used to record the effluent tracer conductivity.](image)

3.6.4. METHOD OF SALT TRACER TESTS

A Perspex column suspended via the load cell was randomly packed with mono-dispersed glass beads before liquid flow to the column was started at the lowest flow rate. The packed bed typically reached steady state after an one hour of operation which was indicated by a constant reading of the data logger coupled to the load cell. This reading was also recorded to ascertain the steady state liquid holdup value. At this stage, the effluent conductivity measuring system was installed at bottom of the packed bed by placing the conductivity meter inside the small reservoir of 35 ml (Figure 3.22). Care was taken to ensure the probe of the conductivity cell was fully submerged in the 35 ml volume of deionised water. Furthermore, the analog output of the conductivity meter was connected directly to the data acquisition module which was coupled to a computer, as described in the Section 3.6.3.
While the tracer tests are non-steady state measurements, the liquid flow through the packed bed is at steady state. At the top of the packed bed, a 0.05 ml volume pulse of calcium chloride (CaCl₂) solution was introduced simultaneously with the recording of conductivity in LabVIEW to mark the start of the tracer addition (time, \( t = 0 \)). Then, the effluent conductivities were recorded continuously at 500 ms intervals using the data acquisition system. This was continued out until the conductivity value of the effluent liquid returned to the starting value at \( t = 0 \). The same procedure was followed to cover the experimental conditions listed in Table 3.4 for all the glass beads. The conductivity data was normalised to obtain the RTD curves.

### 3.7. SUMMARY

This work developed a 1-D column leaching rig to investigate flow behaviour between non-porous particles of the model glass bead system and the ore particles. The specially designed and commissioned system had a bottom plate to support the weight of the entire packed bed while also allowing the effluent liquid to exit at the centre of the bottom plate. A novel liquid distributor was designed to introduce the liquid into the packed bed and to
maintain an even liquid distribution at liquid flow rates typically found in packed bed studies and column leaching.

The empty column was loaded randomly with either non-porous glass beads or the slightly porous ore particles. The gravimetric method was selected in this work to measure the average liquid holdup of the system, requiring a load cell coupled to a high resolution data logger and a computer. The accuracy of the load cell measurements were tested independently by comparing them to the liquid holdup results obtained using the independent drainage method.

A methodology was adapted to measure steady state liquid holdup of the model system. The procedure was repeated to cover all the experimental conditions during this work. A method for obtaining both steady state and corresponding residual liquid holdups was described.

Similar steady state and residual liquid content measurement experiments were performed for the ore system, which was packed with narrow size range ore particles and mixtures of two different size fractions.

Finally, a method for using salt tracer tests to determine the residence time distribution of the 1-D packed bed was described. The effect of the added tracer volume and the volume of the small reservoir to measure the effluent tracer conductivity on the resultant residence time distribution curve were investigated during the preliminary experiments. A very accurate data acquisition system was utilised to record the effluent tracer conductivity values during the test work.
CHAPTER 4

HYSTERESIS AND INTER-PARTICLE FLOW MODEL WITH THE MODEL SYSTEM

4.1. INTRODUCTION
This chapter describes all the liquid holdup results for the liquid flow experiments performed with the model glass bead system. The liquid holdup for the model system packed with the different sized mono-dispersed glass beads was investigated and the liquid holdup hysteresis behaviour in packed beds was studied using the results from both the dry and wet bed start-up conditions.

While the liquid holdup hysteresis data and modelling presented in this work should be applicable to a wide range of processes, the specific application envisaged for this study is in understanding the fluid flow in column and heap leaching. One of the main objectives of this study is to explain the existence of hysteresis behaviour in packed bed systems and column leaching. In heaps due to the unsaturated flow, the hysteresis manifests itself as different steady state liquid contents for the same liquid addition rates. Previous studies of fluid flow modelling in heaps did not incorporate this behaviour, thus it is significant to study this experimentally before applying to heap level simulations.

To explain the liquid holdup hysteresis in the model system, several hypotheses were presented and a theoretical model was developed. The effect of the particle size on the flow behaviour was studied and the volume of the residual liquid connection was estimated. In addition, the effect of the contact angle and rivulet orientation was explained in order to understand the flow behaviour within the model system. Finally, the general applicability of the inter-particle flow model was investigated using poly-dispersed beds of glass beads.
4.2. STEADY STATE LIQUID HOLDUP
The total liquid holdup values for all of the particle sizes were obtained from the steady state experiments. Figure 4.1 shows the liquid holdup variations for 2 mm, 10 mm, 14 mm and 18 mm glass beads when the packed bed was started dry. In addition, Figure 4.2 shows the same variation for the wet bed start-up condition. The wet bed start-up is when the bed is initially flooded (see section 3.3).

![Figure 4.1](image_url)

*Figure 4.1: Total liquid holdup for all the particle sizes in 300 mm column using the dry bed start-up condition.*

Figure 4.3 shows the comparison between the wet bed and dry bed start-up conditions. The liquid contents in the column which was started wet are markedly higher than those in the column started dry for the same superficial liquid flow rate. This indicates that the system experiences hysteresis. Hysteresis means that the liquid holdup depends not only on the current liquid addition rates but also on the history of the system as well.

The random packing of the particles in both dry and wet bed start-up conditions influences the reproducibility of the measured liquid holdup. For a 300 mm packed bed, the liquid holdup values with 95% confidence interval are calculated for 2, 10, 14 and 18 mm mono-dispersed particles. Figures B1-B4 in Appendix B show the results of this analysis and these imply relatively higher liquid holdup variability in the dry bed compared to the wet bed.
Chapter 4: Hysteresis and Inter-Particle Flow Model with the Model System

Figure 4.2: Total liquid holdup for all the particle sizes in 300 mm column using the wet bed start-up condition.

Figure 4.3: Comparison of dry bed and wet bed liquid holdups in the model system for all the particle sizes in 300 mm column.
4.3. LIQUID HOLDUP HYSTERESIS

For a particular particle size, the steady state liquid holdup value was measured with the gravimetric method (as described in section 3.4.1.1.) for the lowest flow rate of 0.0075 mm/s. After that the flow rate was increased to the next flow rate (i.e. 0.015 mm/s) until it reached steady state when the liquid holdup was again determined. This was repeated for each flow rate up to the highest liquid flow rate (i.e. 0.03, 0.06 and 0.12 mm/s). The trend of the steady state liquid holdup measurement with increasing flow rate is referred to as the increasing arm (see Figure 4.4). The flow rates were then reduced in the same sequence to the lowest flow rate, while measuring the steady state liquid content for each flow rate. This sequence is referred to as the decreasing arm (see Figure 4.4).

Completion of both the increasing and decreasing arms of liquid holdup measurement is considered as the first cycle of operation (see Figure 4.4). This method was used to quantify the hysteresis in the liquid holdup in the packed bed, which is indicated by differences in the liquid holdup at the same flow rate between the increasing and decreasing arms.

![Graph showing liquid holdup vs. superficial flow](image)

**Figure 4.4:** Increasing and decreasing arms of flow rate and first cycle of liquid holdup measurements using 18 mm particles in 300 mm column.

Figures 4.5 shows a significant difference in the liquid holdup obtained for the same flow rate between the increasing and decreasing arms for the dry bed experiments. This is especially true for the larger particle sizes. That there is a big difference in the liquid holdup for the same liquid addition rate indicates that the system experiences hysteresis.
Figure 4.5: Dry bed liquid holdup hysteresis for all the particle sizes in 300 mm column. a) 2 mm b) 10 mm c) 14 mm d) 18 mm.

Figure 4.6 shows the wet bed liquid holdup values for the same packed bed following the same sequence of liquid addition rates. Despite the significant hysteresis behaviour observed in the dry bed start-up condition (Figure 4.5), the wet bed start-up condition gives similar liquid holdup values for both increasing and decreasing arms of the superficial velocities of this study (Figure 4.6). This means no or very little of liquid holdup hysteresis can be seen after the bed is started wet.
Chapter 4: Hysteresis and Inter-Particle Flow Model with the Model System

Figure 4.6: Wet bed liquid holdup hysteresis for all the particle sizes in 300 mm column. a) 2 mm b) 10 mm c) 14 mm d) 18 mm.

The same liquid content determination procedure was continued for the subsequent second and third cycles for both the dry and wet bed start-up conditions to investigate the liquid holdup hysteresis after the first cycle of operation. Figure 4.7 illustrates dry bed liquid holdup as a function of superficial velocity for the first three cycles with 14 mm and 18 mm particles and it indicates no or very little hysteresis after the first cycle. This implies that the subsequent cycles result in a small additional increase in liquid content before settling down to a hysteresis free cycle. Similar results were observed for other particle sizes.
Figure 4.7: Dry bed liquid holdup variation in the first three cycles of operation in 300 mm bed (C1, C2 and C3 indicate first, second and third cycles). a) 14 mm particles b) 18 mm particles.

Figure 4.8 shows the wet bed liquid contents for the first three cycles of operation with 2 mm and 18 mm glass beads. It is shown that for all the three cycles of operation with the wet bed start-up condition, no or very little liquid holdup hysteresis is measured. Therefore,
the packed bed settles down to a hysteresis free status when it was flooded before the initial liquid addition.

![Graph](image)

Figure 4.8: Wet bed liquid holdup variation in first three cycles of operation in 300 mm bed (C1, C2 and C3 indicate first, second and third cycles). a) 2 mm particles b) 18 mm particles.

In order to investigate the main factors that influence the current liquid holdup, the liquid flow rate into the packed bed was changed as listed in Table 4.1. In the first set of flow rates, liquid was introduced at a superficial velocity of 0.0075 mm/s to achieve steady state.
After recording the steady state liquid holdup gravimetrically, flow rate was changed to 0.015 mm/s. At the steady state, the corresponding liquid content was recorded and again the flow rate was reduced to 0.0075 mm/s. Finally, the resultant steady state liquid holdup value was measured in a similar way to other two flow rates. This procedure was continued for second, third and fourth set of flow rates respectively, which are listed in Table 4.1.

**Table 4.1: Set of flow rates and the sequence of flow rate during the experimental run.**

<table>
<thead>
<tr>
<th>Set of flow rates</th>
<th>Sequence of superficial flow rates in mm/s</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.0075 – 0.015 – 0.0075</td>
</tr>
<tr>
<td>2</td>
<td>0.0075 – 0.015 – 0.03 – 0.015 – 0.0075</td>
</tr>
<tr>
<td>3</td>
<td>0.0075 – 0.015 – 0.03 – 0.06 – 0.03 – 0.015 – 0.0075</td>
</tr>
<tr>
<td>4</td>
<td>0.0075 – 0.015 – 0.03 – 0.06 – 0.12 – 0.06 – 0.03 – 0.015 – 0.0075</td>
</tr>
</tbody>
</table>

This procedure gives a variation in liquid holdup for all four sets of flow rates as shown in Figure 4.9. It illustrates the liquid holdup variation of the 300 mm packed bed containing 18 mm glass beads, which was started dry. Similar results were observed for the other particle sizes.

![Figure 4.9: The effect of the maximum flow rate on liquid holdup hysteresis behaviour. The 300 mm dry packed bed with dry 18 mm beads was wetted as listed in Table 4.1.](image-url)
Figure 4.9 implies that the maximum superficial velocity of a particular set of flow rates affects the liquid content corresponding to the subsequent set of flow rates. Therefore, the current flow rate and the maximum flow rate experienced by the packed bed are the main factors that influence the current liquid content. This is why the packed bed under wet bed start-up condition results in no or little liquid holdup hysteresis compared with the dry bed start-up condition, because the packed bed has already experienced the equivalent of the maximum possible flow rate due to flooding before the normal liquid addition.

4.4. UNDERSTANDING OF LIQUID HOLDUP HYSTERESIS

In order to model the liquid flow behaviour, it is important to explain why liquid holdup hysteresis exists in the packed beds. A number of hypotheses to explain the occurrence of the hysteresis behaviour have been proposed (e.g. Maiti et al., 2006; Maiti et al., 2008) including:

1. The difference between the advancing and receding three phase contact angles over the surface of the particles (i.e. changes in the shape of the liquid rivulets in the bed).
2. Spatial variations in the wettability of the surfaces (contact lines advancing over regions of high wettability, but not being able to retreat over these regions).
3. The change in tortuosity of the flow paths.
4. A variation in the number of flow paths between the increasing flow and decreasing flow (i.e. changes in the number of rivulets in the bed).

While these different explanations for the flow hysteresis in packed beds have been offered, there has been no proof as to which of these effects is dominant. In this work a new experimental and analysis procedure has been developed in order to distinguish between the mechanisms (see sections 4.5.2. and 4.5.3.)

The way in which these mechanisms are distinguishable from one another is that mechanisms 1, 2 and 3 have changes in the shape of the rivulet (in cross-section for 1 and 2, and both cross-section, through a change in the average gravitational force, and length for mechanism 3), while for mechanism 4, it is the number of rivulets that change. This means that in mechanisms 1-3, the relationship between the size of the rivulet and the flow down
the rivulet is assumed to change, while it does not need to change for mechanism 4 to explain the hysteresis.

4.5. THEORETICAL MODELLING OF FLOW PATHS
The observed rivulet flow behaviour at low liquid superficial velocities is modelled theoretically to obtain a relationship between the average flow per rivulet and the average liquid holdup of that rivulet. In addition, it will also be used to explain the existence of liquid holdup hysteresis in packed bed and heaps.

4.5.1. OBSERVATIONS OF RIVULET FLOW
At very low liquid flow rates, the unsaturated liquid percolation through the packed bed is discrete and at the particle level the different liquid flow features can be identified as liquid films, rivulets over the particles, pendulum structures, liquid filled channels and liquid filled pockets (Liu et al., 2002; Maiti et al., 2008) (see section 2.7.1). Some of these flow features were observed visually during the laboratory experiments of this study. Examples are shown in Figures 4.10, 4.11 and 4.12.

Initially the paths of the rivulets are more or less vertical through the packed bed and the inter-connections in between them are relatively small. Such rivulets were clearly observed during the liquid flow experiments in this study. The average area of the rivulets depends on the amount of liquid available for flowing. As the liquid flow increases, the rivulets can grow bigger and bigger and the number of rivulets gradually increases (see Figure 4.11).

The experimental observations of unsaturated gravity dominated flow can be used to explain the occurrence of droplet and rivulet flow through particles. Initially at lower flow rates, the percolated liquid trickles as a few individual droplets and liquid pockets in between the interconnected particles (Figure 4.12). The movement of these liquid features can be stopped by capillary forces becoming liquid filled connections between the particles or pendular drops (see Figure 4.11 and 4.12). As the packed bed gets more and more liquid, the discrete liquid features can combine with each other to make a flow path. Hence, liquid flows as rivulets through the particles of the packed bed.
Figure 4.10: Initial liquid introduction into the top surface of the packed bed.

Figure 4.11: Random flow behaviour of rivulets through liquid filled connections between the 18 mm particles.
Figure 4.12: Rivulet flow behaviour through the 18 mm particles of the packed bed.

4.5.2. RESIDUAL LIQUID HOLDUP OF THE MODEL SYSTEM

Figure 4.13 shows the complete flow cycle used to measure the steady state and associated residual liquid holdup. For each flow rate, two values of steady state liquid holdup and corresponding residual liquid holdup were obtained: one for increasing flow rate and the second one for decreasing flow rate (see section 3.4.2 for explanation of the methodology). These values were used to theoretically model the flow paths and also to explain the existence of the hysteresis in the model system of glass beads.

The residual holdup within the particle bed mainly consists of liquid held between neighbouring particles by capillarity. The amount of liquid that will be held in an individual connection will depend on the gravitational force as well as the capillarity, the ratio of which is the Bond number \( B_o = \frac{\rho g d_p^2}{\gamma} \), where \( \gamma \) is the surface tension, \( \rho \) is the density of the
Chapter 4: Hysteresis and Inter-Particle Flow Model with the Model System

liquid, \( d_p \) is the particle size and \( g \) is gravitational acceleration). The contact angle will also influence the amount of liquid held in an individual connection. This means that the residual holdup is proportional to the number of liquid filled connections in the bed, with a proportionality that depends on the Bond number and contact angle only.

Figure 4.13: Steady and residual liquid holdup during the increasing and decreasing flow rates in 500 mm column packed with 2 mm particles. The superficial velocities in mm/s are shown.

Figure 4.14 shows the residual holdup for different liquid addition rates for the increasing and decreasing legs of the experiment. As the liquid addition rate is increased, the residual holdup also increases. This implies that the number of connections within the bed increases as the liquid flow rate is initially increased. It also implies that the new connections are relatively hard to form, since they require a change in the liquid addition rate to be formed, rather than the liquid spontaneously filling all of the available connections.

On the decreasing leg the residual holdup either stays relatively constant or even increases slightly as the liquid addition rate is decreased. This implies that once formed, these connections are hard to destroy. The small increase in the residual liquid content as the flow
is decreased is possibly due to disturbance as the flow is reintroduced after the measurement of the residual holdup.

Figure 4.14: Residual holdup variation of all the particle sizes in 500 mm dry packed bed for both increasing and decreasing arms of flow. a) 2 mm b) 10 mm c) 14 mm d) 18 mm.

It was found a higher liquid content difference for 18 mm particles than 2 mm particles between the increasing and decreasing arms of the flow (see Figure 4.5 and 4.14). The reason for this could be that the higher Bond number makes the creation of new flow paths easier, since gravity is more able to influence the shape of the rivulets.
Since these residual connections are the remnants of rivulets through the particles, this change implies that the number of rivulets in the heap changes between the increasing and decreasing arms of the experiment. This is consistent with the explanation that a change in the number of flow paths is a factor in the hysteresis, but does not prove that this is the dominant mechanism. To test this explanation it is desirable to check if the relationship between the flow per rivulet and the size of the rivulet is different on the increasing and decreasing arms of the experiments.

4.5.3. EXPLANATION OF LIQUID HOLDUP HYSTERESIS
The number of liquid filled connections is proportional to the residual liquid holdup. Assuming that their average size depends on Bond number and contact angle only, it can be assumed that the residual holdup is proportional to the number of rivulets per cross-sectional area \( N_{rivulets} \).

\[
\theta_{residual} \propto N_{rivulets} \quad (4.1)
\]

\[
\theta_{residual} = N_{rivulets}A_r \quad (4.2)
\]

The proportionality constant \( (A_r) \) is the average cross-sectional area of the residual liquid in a rivulet. In this work, the size of the rivulets are characterised by means of their average cross-sectional area, which can equivalently be thought of as the volume of the rivulet per height of the column. If \( V \) is the volume of liquid per connection, \( r_p \) is the particle radius and \( c \) is a dimensionless geometric constant that represents the vertical spacing of connections along a rivulet relative to the particle size.

\[
\theta_{residual} = N_{rivulets} \left( \frac{V}{c r_p} \right) \quad (4.3)
\]

The average flow per rivulet can be calculated as follows:

\[
\text{Average flow per rivulet} = \frac{\text{Flow rate of the column}}{N_{rivulets}} \quad (4.4)
\]

\[
\text{Average flow per rivulet} = \frac{v_s A_r}{\theta_{residual}} \quad (4.5)
\]

where \( v_s \) is the superficial liquid velocity (flow rate per area) into the column.

A relative velocity, \( v_s^* \), that is proportional to the flow per rivulet can thus be defined:

\[
v_s^* = \frac{v_s}{\theta_{residual}} \quad (4.6)
\]
Similarly, the holdup per rivulet can also be calculated:

\[
\text{Holdup per rivulet} = \frac{\theta}{N_{\text{rivulets}}} \quad (4.7)
\]

\[
\text{Holdup per rivulet} = \frac{\theta A_r}{\theta_{\text{residual}}} \quad (4.8)
\]

A relative holdup, \( \theta^* \), that is proportional to the holdup per rivulet can thus also be defined:

\[
\theta^* = \frac{\theta}{\theta_{\text{residual}}} \quad (4.9)
\]

These ratios can be used to investigate the relationship between the average flow per flow path (proportional to \( \nu^*_s \)) and holdup per flow path (proportional to \( \theta^* \)). The \( \nu^*_s \) versus \( \theta^* \) plots for all the particles sizes are shown in Figure 4.15. It can be seen that for each of the particle sizes the relationship between \( \nu^*_s \) and \( \theta^* \) follows a single curve for both the increasing and decreasing flow rates. In other words the relationship between \( \nu^*_s \) and \( \theta^* \) does not exhibit hysteresis.

Earlier, in section 4.3, the occurrence of hysteresis was illustrated for different particle sizes (Figure 4.5), with significant differences in holdup at equivalent liquid addition rates when increasing and decreasing the flow. It was not obvious what caused this hysteresis and a number of hypotheses are mentioned (see section 4.4). It is shown that the relationship between \( \nu^*_s \) and \( \theta^* \) follows a single curve irrespective of the underlying hysteresis. That \( \nu^*_s \) versus \( \theta^* \) follows a single curve for both increasing and decreasing flow indicates that the hysteresis is not caused by a change in the relationship between the size of the rivulet and the flow down it (which would occur if hypotheses 1-3 were the dominant mechanisms). Instead, this shows that the biggest contribution to the hysteresis is the increase in the number of rivulets as the flow is increased.
Figure 4.15: The variation of $\nu_2^*$ against $\theta^*$ in the increasing and decreasing arms of flow for all the particle sizes in 500 mm packed bed. a) 2 mm b) 10 mm c) 14 mm d) 18 mm.

4.5.4. INTER-PARTICLE FLOW MODEL
Consider a rivulet (Figure 4.16) at any instant. Some liquid is held in reservoirs between the particles by capillarity. These separate reservoirs are connected by flowing drops and rivulets. Note that the liquid in the reservoirs is continuously being replaced and thus this liquid is slow moving rather than truly stationary.

The area of rivulet that is actively flowing ($A_f$) is the difference between the average area of the rivulet ($A_t$) and the area of the rivulet held relatively stationary in the reservoirs between the particles ($A_s$):

$$A_f = A_t - A_s$$  \hspace{1cm} (4.10)
Assuming that the shape of the flowing portion of the rivulet remains roughly constant, the average velocity of the liquid in the rivulet ($v_{rivulet}$) is given by equation 4.11, where $C_{drag}$ is a dimensionless drag coefficient (this is the Darcy-Weisbach law). The derivation of this equation is given in Appendix C.

$$v_{rivulet} = \frac{\rho g}{\mu C_{drag}} A_f$$ (4.11)

where $\rho$ is the density of the liquid, $g$ is gravitational acceleration and $\mu$ is the viscosity of the liquid.

The assumption that the cross-sectional shape of the rivulet is reasonably constant is reasonable, if the rivulet is small compared to the size of the particles. Under these conditions the shape of the rivulet will mainly be determined by the contact angle, though gravity will also distort the rivulets at higher Bond numbers.

Then, the flow down one rivulet ($Q_r$) is,
Chapter 4: Hysteresis and Inter-Particle Flow Model with the Model System

\[ Q_r = \frac{\rho g}{\mu C_{\text{drag}}} A_r^2 \]  \hspace{1cm} (4.12)

However, \( v_s = Q_r N_{\text{rivulets}} \); where \( v_s \) is the total flow rate per area of the column (i.e. superficial liquid velocity).

\[ \frac{v_s}{N_{\text{rivulets}}} = \frac{\rho g}{\mu C_{\text{drag}}} A_r^2 \]  \hspace{1cm} (4.13)

It will be assumed that capillarity ensures that connections are not broken as the column drains and that the stationary reservoirs do not change size with flow rate. The reason why this assumption can be made is because there is a flow independent (but Bond number dependent) size above which a connection must release liquid. During flow and during the subsequent drainage, these connections will thus remain roughly this size:

\[ A_s = A_r \]  \hspace{1cm} (4.14)

At the low Reynolds numbers encountered in this work, this is likely to be a good assumption, though at higher Reynolds numbers, inertial effects may well cause changes in the size and shape of these reservoirs. This assumption is also only likely to be valid at low saturations, since at high saturation the concept of distinct rivulets flowing between the particles will be less valid.

By definition the following two equations hold:

\[ \theta = N_{\text{rivulets}} A_t \]  \hspace{1cm} (4.15)

\[ \theta_{\text{residual}} = N_{\text{rivulets}} A_r \]  \hspace{1cm} (4.16)

Thus, by using equation 4.10, together with the definitions 4.15 and 4.16, equation 4.13 can be rewritten as:

\[ A_s \frac{v_s}{\theta_{\text{residual}}} = \frac{\rho g}{\mu C_{\text{drag}}} A_r^2 \left[ \frac{\theta - \theta_{\text{residual}}}{\theta_{\text{residual}}} \right]^2 \]  \hspace{1cm} (4.17)

By using the definitions 4.6 and 4.9 the following final form is obtained:

\[ v_s^* = K[\theta^* - 1]^2 \]  \hspace{1cm} (4.18)

where \( K \) is the pre-factor given by, \( K = \rho g A_r / \mu C_{\text{drag}} \) (assuming that \( A_s = A_r \) according to equation 4.14)

For the reasons mentioned above, \( A_r \) is likely to be a function of the particle size and the Bond number and not a function of the flow rate. \( v_s^* \) is plotted against \( \theta^* - 1 \) on log-log
axes for all the particle sizes for the dry bed start-up condition to test whether the power law relationship proposed in equation 4.18 holds (Figure 4.17).

It is evident from Figure 4.17 that, the experimental data for all the particle sizes in the dry bed operation is consistent with the squared relationship between $v_s^*$ and $\theta^* - 1$ proposed by the model. In addition, Figure 4.17 shows a very similar relationship for a particular particle size in different length columns.

Figure 4.18 illustrates the relationship between $v_s^*$ and $\theta^* - 1$ for all the particle sizes of this study with the wet bed start-up condition. A similar squared correlation can also be seen between the two parameters of the model despite the change in the start-up procedure for the packed bed.

![Figure 4.17: The relationship between the relative flow rate ($v_s^*$) and the additional liquid content of that rivulet ($\theta^* - 1$) for dry bed start-up condition of the model system.](image)

There is some slight scatter in the data at low liquid holdup in both graphs (see Figure 4.17 and Figure 4.18), but this will be due to the calculated value of $\theta^* - 1$ being very sensitive to measurement errors in both the steady and residual liquid holdups, especially when the two values are quite similar.
Chapter 4: Hysteresis and Inter-Particle Flow Model with the Model System

Figure 4.18: The relationship between the relative flow rate ($v_s^*$) and the additional liquid content of that rivulet ($\theta^* - 1$) for wet bed start-up condition of the model system.

To test the fact that for a particular particle size the same relationship between the relative flow rate and the relative holdup applies irrespective of the start-up condition (i.e. either wet bed or dry bed), it is desirable to show all the results plotted above in a single graph. This is shown in Figure 4.19 and indicates that the same correlation holds for a particular particle size in both dry and wet bed start-up conditions.

Earlier in this results section, it was demonstrated that the different hysteresis behaviour occurs for dry bed and wet bed start-up conditions. But now it is shown that there is the same correlation between the average flow rate per rivulet and flowing holdup of that rivulet. This can be explained as follows.

In the dry bed start-up condition, the number of flow paths in the flow rate decreasing arm is greater than that in the flow rate increasing arm and this results in liquid holdup hysteresis. In the wet bed start-up condition all the possible connections are pre-wetted and therefore the flowing liquid does not need to create flow paths. Since there are no additional flow paths to create, virtually no hysteresis occurs.
Figure 4.19: The relationship between the relative flow rate \( v_s^* \) and the additional liquid content of that rivulet \( \theta^* - 1 \) for all the particle sizes of the model system in both dry and wet bed start-up conditions.

The theoretical value of the exponent between \( v_s^* \) and \( \theta^* - 1 \) is 2 for the rivulet flow according to the derived relationship expressed in equation 4.18. This can be investigated using the experimental results plotted in Figure 4.19 for all the particle sizes and different column lengths in both dry and wet bed start-up conditions. The actual slope of each line in Figure 4.19 for a particular experimental condition was determined and these are given in Table 4.2 with the corresponding goodness of fit values. Table 4.2 shows that the actual slopes for all the particle sizes and different column lengths are very close to the theoretical value of 2 and the slight deviations appear to be random, with no apparent trends with particle size or flow rate. This would suggest that the variation in this exponent is mainly due to the experimental variability. This analysis implies that the experimental data for all the particle sizes in both start-up conditions in the model system is consistent with the squared relationship between \( v_s^* \) and \( \theta^* - 1 \) proposed by the inter-particle model.
Table 4.2: Actual slopes of the lines between \( v_s^* \) and \( \theta^* - 1 \) in Figure 4.19 for all the particle sizes and different column lengths of the model system in both dry and wet bed start-up conditions.

<table>
<thead>
<tr>
<th>Column height and start-up condition</th>
<th>Particle size (mm)</th>
<th>Slope of the line between ( v_s^* ) and ( \theta^* - 1 )</th>
<th>Goodness of fit</th>
</tr>
</thead>
<tbody>
<tr>
<td>300 mm column – Dry bed start-up</td>
<td>2</td>
<td>1.96</td>
<td>0.99</td>
</tr>
<tr>
<td></td>
<td>10</td>
<td>1.66</td>
<td>0.97</td>
</tr>
<tr>
<td></td>
<td>14</td>
<td>2.06</td>
<td>0.83</td>
</tr>
<tr>
<td></td>
<td>18</td>
<td>2.24</td>
<td>0.98</td>
</tr>
<tr>
<td>300 mm column – Wet bed start-up</td>
<td>2</td>
<td>2.19</td>
<td>0.97</td>
</tr>
<tr>
<td></td>
<td>10</td>
<td>2.37</td>
<td>0.92</td>
</tr>
<tr>
<td></td>
<td>14</td>
<td>1.97</td>
<td>0.97</td>
</tr>
<tr>
<td></td>
<td>18</td>
<td>2.28</td>
<td>0.91</td>
</tr>
<tr>
<td>500 mm column – Dry bed start-up</td>
<td>2</td>
<td>2.39</td>
<td>0.98</td>
</tr>
<tr>
<td></td>
<td>10</td>
<td>2.16</td>
<td>0.97</td>
</tr>
<tr>
<td></td>
<td>14</td>
<td>2.28</td>
<td>0.97</td>
</tr>
<tr>
<td></td>
<td>18</td>
<td>1.99</td>
<td>0.93</td>
</tr>
</tbody>
</table>

4.5.4.1. EFFECT OF PARTICLE SIZE

Figure 4.17 and Figure 4.18 show different pre-factor values for the relationship between \( v_s^* \) and \( \theta^* - 1 \) when particle size changes. This pre-factor (\( K \)) in equation 4.18 depends on the Bond number, which, together with the contact angle, influences the shape of the liquid rivulet, its tortuosity (which will depend on the particle packing) and as well as the rheological properties of the liquid. As the inter-particle flow model is semi-empirical and only has one fitting parameter (i.e. \( K \)), the effect of the particle size on this main model parameter needs to be determined.

Figure 4.20 shows the variation of the pre-factor as a function of the particle size with the dry bed start-up condition. It can be seen that \( K \) is quite a strong function of particle size in the case of spherical glass beads.
4.5.4.2. ESTIMATING THE EFFECT OF THE CONTACT ANGLE

The effect of particle size, and thus Bond number, was studied experimentally. However, the contact angle was not varied in the liquid holdup experiments and therefore a model will be used to estimate the impact of changes in contact angle on this value of $K$.

The flow rate of a rivulet with a circular cross-section when the flow is vertical and laminar (see Equation C.17 in Appendix C):

$$Q_r = \frac{\rho g}{8 \mu} \pi R^4 \quad (4.19)$$

where $\rho$ is the density of the liquid, $g$ is gravitational acceleration and $\mu$ is the viscosity of the liquid and $R$ is the radius of the circular rivulet.

In flow through more complex geometries, the above form can typically not be derived analytically. A good approximation is to use the hydraulic radius in the above equation.

The hydraulic radius $(d_h)$ is the characteristic dimension which is most useful in flow through open channels. It is defined as the ratio of the cross-sectional flow area of the channel (i.e. $A_f$) and the wetted perimeter of the surface of the channel ($W$) (Streeter and Wylie, 1983, 1985).

$$d_h = \frac{A_f}{W} \quad (4.20)$$
The hydraulic radius for a circular cross-section is \( d/4 \), where \( d \) is the diameter and the hydraulic diameter \( (D_H) \) is four times the hydraulic radius (Streeter and Wylie, 1983, 1985).

Now for a circular cross-section:

\[
D_H = d = 2R
\]  

(4.21)

Therefore, equation 4.19 becomes:

\[
Q_r = \frac{\rho g \pi}{\mu} \frac{1}{128} D_H^4
\]  

(4.22)

Figure 4.21 shows a schematic of the actual flow path of a rivulet between two liquid filled connections, \( L \), relative to the straight line distance, \( l \), and the vertical displacement, \( h \).

Figure 4.21: Schematic representation of rivulet flow behaviour with tortuous flow paths between the two liquid filled connections.

Equation 4.22 can be rewritten as:

\[
Q_r = \frac{\Delta P}{L \mu} \frac{\pi}{128} D_H^4
\]  

(4.23)

where \( \Delta P \) is pressure difference between the two points.

The macroscopic length covered by the rivulets between the liquid filled connections is \( l \). However, the liquid elements cannot travel linearly from one contact point to the next due to impediments provided by the particles of the packed bed. As the packed bed is unsaturated and the liquid content is relatively small compared with a saturated system, the
small rivulets should closely follow the surface of the particles. This distance is the actual flow path, $L$, which is associated with the tortuosity of the flow paths. Therefore, $L \geq l$.

The relationship between $l$ and $L$ is:

$$L = k_T l$$  \hspace{1cm} (4.24)

where $k_T$ is the tortuosity factor for the flowing rivulets.

Using equation 4.24, equation 4.23 becomes:

$$Q_r = \frac{\Delta \rho}{k_T \mu} \frac{\pi}{4} D_H^4$$  \hspace{1cm} (4.25)

The pressure change due to gravity is $\rho gh$ and therefore equation 4.25 can be rewritten as:

$$Q_r = \frac{\rho g h}{\mu} \frac{1}{k_T} \frac{\pi}{128} D_H^4$$  \hspace{1cm} (4.26)

If $g^*$ is the gravitational component in the direction along the rivulet:

$$g^* = \frac{h}{l} g$$  \hspace{1cm} (4.27)

Equation 4.25 becomes:

$$Q_r = \frac{\rho g^*}{k_T \mu} \frac{\pi}{128} D_H^4$$  \hspace{1cm} (4.28)

Figure 4.22 illustrates a cross-section of the flowing portion of the rivulet over the surface of a glass bead. In this figure, $W$ is the wetted perimeter, $r_c$ is the radius of curvature of the rivulet interface and $\Theta$ is the contact angle. In this simple model it is assumed that the cross-sectional curvature of the rivulet is much smaller than that of the particle.

For contact angles less than 90 degrees, the following relationships can be defined:

$$\frac{W}{2} = r_c \sin \Theta$$  \hspace{1cm} (4.29)

$$A_f = \pi r_c^2 \left( \frac{2 \Theta}{2 \pi} \right) - \frac{W}{2} r_c \cos \Theta$$  \hspace{1cm} (4.30)

where $A_f$ is the cross-sectional area of the channel.
Chapter 4: Hysteresis and Inter-Particle Flow Model with the Model System

Figure 4.22: Schematic representation of the cross-section of the flowing rivulet over the surface of the glass bead.

Using equation 4.29, equation 4.30 becomes:

\[ A_f = \varphi r_c^2 - r_c^2 \sin \varphi \cos \varphi \]  \hspace{1cm} (4.31)

\[ A_f = r_c^2 (\varphi - \sin \varphi \cos \varphi) \]  \hspace{1cm} (4.32)

Hydraulic diameter can be applied to an open channel flow and it can be defined using equation 4.33 (Streeter and Wylie, 1983, 1985). In this case the rivulet flow over the surface of particles in the packed bed is the corresponding open channel flow behaviour, which was illustrated in Figure 4.22.

\[ D_H = \frac{4A_f}{W} = \frac{4A_f}{2r_c \sin \varphi} \]  \hspace{1cm} (4.33)

Substituting from equation 4.32, equation 4.33 gives:

\[ D_H = \frac{2 \sqrt{A_f \sin \varphi \cos \varphi}}{\sin \varphi} \]  \hspace{1cm} (4.34)

Now, the general equation for a rivulet that flows over the surface of the particles of the packed bed can be defined by substituting equation 4.34 into equation 4.28.
Chapter 4: Hysteresis and Inter-Particle Flow Model with the Model System

\[ Q_r = \frac{\rho g' \pi (\phi - \sin \phi \cos \phi)^2}{k_T \mu \sin^4 \phi} A_f^2 \]  
\[ Q_r = \frac{\rho g' \pi (\phi - \sin \phi \cos \phi)^2}{\mu k_T \sin^4 \phi} A_f^2 \]  

Equation 4.36 resembles equation 4.12, which also defines the flow rate of the rivulet using the dimensionless drag coefficient \( C_{\text{drag}} \). Thus, \( C_{\text{drag}} \) can be defined using the terms of equation 4.36:

\[ C_{\text{drag}} = \frac{\mu k_T \sin^4 \phi}{\pi (\phi - \sin \phi \cos \phi)^2} \]  

Figure 4.23 shows the variation of the dimensionless drag coefficient with the contact angle.

![Figure 4.23: The dimensionless drag coefficient as a function of the contact angle of the rivulets \( k_T = 1 \) and \( g' = g \).](image)

\( C_{\text{drag}} \) will be infinity for a contact angle of zero and it steadily decreases when the contact angle increases. This model will not be applicable at very low contact angles. At these contact angles the rivulets will spread out to the extent that they will become thin films rather than true rivulets. The scaling behaviour of thin films is different to that of rivulets (see Appendix D).

**4.5.4.3. EFFECT OF THE RIVULET ORIENTATION**

The range of different orientations of the rivulets influences the effective gravitational acceleration. Using equation 4.27, the acceleration of gravity \( g \) is related to the effective
acceleration of gravity ($g^*$) in the direction of rivulet flow in order to account the orientation of the flow paths. Equation 4.27 can be rewritten as:

$$g^* = \frac{g}{m}$$

(4.38)

where $m$ is a constant.

If the Bond number is very low, then the orientation of the rivulets will be essentially random as capillarity means that gravity has little impact on the orientation. If the rivulets are randomly oriented, then the effective gravitational force will be approximately one third of the vertical value (i.e. $m \approx 3$).

As Bond numbers increase, the orientation of the rivulets will become influenced by gravity and will align more vertically. The average orientation will never become vertical because of the presence of the particles. Instead the rivulets will tend toward the steepest possible descent.

### 4.5.4.4. SIZE AND VOLUME OF THE RESIDUAL LIQUID CONNECTIONS

The two unknowns in $K$ are the average cross-sectional area of a residual rivulet, $A_r$, and the dimensionless drag coefficient, $C_{drag}$. The ratio of these two parameters, $A_r / C_{drag}$, is plotted against the particle size in Figure 4.24 for all the particles in the 300 mm and 500 mm columns. The parameter $A_r / C_{drag}$ follows approximately a power law relationship with the particle size with an exponent of between 0.38 and 0.42 for the two column heights.

Since the shape of the rivulets is unlikely to vary much with particle size (contact angle and the geometry of the packing is virtually constant), the drag coefficient is unlikely to be a strong function of particle size. The biggest variation is likely to be due to the size of the residual liquid channels. The data shows that, while these residual channels get bigger with particle size, they do not increase nearly as quickly as would be expected if they stayed the same relative size (this would imply a squared relationship). This means that as the particle size increases, the relative size of the residual liquid connections between particle decreases. This is consistent with the qualitative visual observations of the experiments. This is most likely because at higher Bond numbers the liquid connections between particles are more distorted by gravity and thus stop shedding liquid when they are smaller relative to the size of the particle.
In addition, the ratio, $A_r/C_{drag}$, is $0.004 \text{ mm}^2$ for the smallest particle size (i.e. 2 mm) and $0.01 \text{ mm}^2$ for the largest particle size (i.e. 18 mm) (see Figure 4.24). It is very reasonable given that the drag coefficient is likely to be of order 10 or 100 depending on the contact angle as estimated above (see Figure 4.23). Therefore the $A_r$ values are of order 0.04-0.4 mm$^2$ for 2 mm particles and order 0.1-1 mm$^2$ for 18 mm particles.

Using equations 4.2 and 4.3, the volume of liquid per connection ($V$) is given by:

$$V = A_r c r_p$$  \hspace{1cm} (4.39)

where $r_p$ is the particle radius.

The dimensionless geometric constant ($c$) that represents the vertical spacing of connections along a rivulet relative to the particle size is of order 1.

Thus the typical volumes of residual connections are of order 0.04-0.4 mm$^3$ for 2 mm particles and order 0.9-9 mm$^3$ for 18 mm particles.

**4.6. FLOW BEHAVIOUR IN POLY-DISPERSED MODEL SYSTEM**

There were three binary mixtures and one ternary mixture studied in this work in order to investigate whether the liquid holdup model is robust over the poly-dispersed particles of the packed beds (see Table 3.3). Similar to the mono-dispersed packed beds, the relative
velocities ($v^*_s$) and the relative liquid holdups ($\theta^*$) were calculated for all poly-dispersed beds to plot the $v^*_s$ values as a function of $\theta^* - 1$.

The results are shown in Figure 4.25 for all the four mixtures of glass beads. In each plot, the correlation between the two parameters is plotted together with the results from the individual mono-dispersed particles that make up the mixture in order to ascertain the characteristic length scale most applicable to mixtures.

Figure 4.25 implies that each poly-dispersed packed bed follows a squared relationship between the relative flow rate and the additional liquid content of that rivulet. It proves that the applicability of the inter-particle flow model for the poly-dispersed particles. In addition, the data for the poly-dispersed bed lies within the range of the data for the constituent particles, but towards the smaller particles.

Table 4.3 compares the fitted values of pre-factor, $K$, for the mono-dispersed and all the poly-dispersed packed beds shown in Figure 4.25. It can be seen that $K$ values of the mixtures are similar or slightly lower than the same of the smaller particle size. This might be expected as particle mixtures have smaller inter-particle spaces compared to the mono-dispersed bed of larger particles. In addition, the smaller particles can pack in between the larger particles by creating even smaller inter-particle spaces for liquid flow compared to the mono-dispersed beds of the smaller particle size.

Finally these smaller channels slow down the average velocity of rivulets indicated by a lower value of the pre-factor. This is crucial during the simulation of the flow behaviour in packed beds and heaps as characteristic particle size needs to be defined from the size range of particles in the packed bed or the ore mixture.
Figure 4.25: The relationship between $v_s^*$ and $\theta^* - 1$ for dry bed start-up condition using the poly-dispersed beds of non-porous glass beads. All the mixtures were prepared by mixing equal weights from each particle size.

Table 4.3: Pre-factor of the mono-dispersed and poly-dispersed model systems.

<table>
<thead>
<tr>
<th>Packed bed (mono- or poly-dispersed)</th>
<th>Pre-factor, $K$ (m/s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>10 mm</td>
<td>0.064</td>
</tr>
<tr>
<td>14 mm</td>
<td>0.085</td>
</tr>
<tr>
<td>18 mm</td>
<td>0.093</td>
</tr>
<tr>
<td>10 mm+14 mm (50% - 50% by weight)</td>
<td>0.064</td>
</tr>
<tr>
<td>10 mm+18 mm (50% - 50% by weight)</td>
<td>0.058</td>
</tr>
<tr>
<td>14 mm+18 mm (50% - 50% by weight)</td>
<td>0.078</td>
</tr>
<tr>
<td>10 mm+14 mm+18 mm (33.3% - 33.3% - 33.3% by weight)</td>
<td>0.06</td>
</tr>
</tbody>
</table>
4.7. SUMMARY

This chapter presented liquid holdup results for the model system of glass beads containing both the mono-dispersed particles of different diameter and the poly-dispersed particles. It was found that the liquid holdup in the model system depends not only on the current input conditions, but also on the system history and this implies that the packed bed experiences hysteresis. It was demonstrated that the liquid holdup hysteresis is significant during the first cycle of operation when the bed was started dry, but was virtually absent when the bed was started wet. After considering a few theories to explain this phenomenon, the theoretical modelling of flow paths proved that the dominant cause for the liquid holdup hysteresis behaviour is the change in the number of flow paths rather than a change in the shape of the rivulet between the increasing and decreasing arms of the flow.

As the rivulet flow behaviour is the major flow feature through the particles of the packed bed, the theoretical modelling of flow paths was extended to obtain a relationship between the average flow rate of the rivulet and the liquid holdup of the packed bed. It was found that for the model system, the relative flow rate of the rivulet is proportional to the square of the additional liquid content of that rivulet. The inter-particle flow model is consistent with the experimental results of all the mono-dispersed particle sizes in both wet and dry bed start-up condition. The pre-factor \( K \) of the model is a function of particle size and Bond number and was thus a strong function of the particle size.

A relationship for the dimensionless drag coefficient was proposed and it was found that it is order 10 or 100 for the model system depending on the tortuosity of the rivulets and the solid-liquid contact angle. With that the average volume of the residual connections was theoretically estimated.

Finally, the validity of the inter-particle flow model was investigated for the poly-dispersed packed beds and it was found that the model is robust over all the poly-dispersed mixtures. In addition, the pre-factor \( K' \) of the mixture is similar or less than the same given by a mono-dispersed bed of the smallest particle size of the mixture.
CHAPTER 5

LIQUID HOLDUP RESULTS FOR THE ORE SYSTEM

5.1. INTRODUCTION

One of the objectives of this work is to investigate the validity of the inter-particle flow model for an ore system considering the model was shown to describe the flow behaviour in the model system very well.

The liquid holdup hysteresis behaviour of the ore system was studied when the bed was started dry. The inter-particle flow model accounts for the liquid volume held in between the particles; therefore, a method was developed with the ore bed in order to determine the steady state and residual liquid contents. The resulting liquid holdup results were investigated with the flow model using different narrow size fractions of ore. The inter-particle flow model was also tested for mixtures made from the narrow size fractions of the ore.

Finally, the liquid holdup results of the ore system were compared with the model system of glass beads. The main parameter of the flow model was analysed for both systems and the differentiation between the flow mechanisms that are involved in the two systems was presented. In addition, the residual liquid holdup variation was studied for both narrow size ore particles and their mixtures, followed by a summary of the liquid holdup results in the ore system.
5.2. LIQUID HOLDUP OF THE ORE BED

In a set of preliminary experiments, the liquid content of the ore was determined with a bed which was started dry (see Figure 5.1).

Figure 5.1: The initial liquid flow behaviour through dry ore particles in 300 mm column. The particle size range of the ore particles was 8-11.2 mm.
Figure 5.2 shows the resultant liquid holdup values for the narrow size fraction of 8-11.2 mm particles. The total liquid content of the dry ore mixture increases with the liquid addition rate.

The geometric mean size (square root of the product of the smallest and largest particle sizes) of each size fraction of ore particles was considered as the average particle size. For the size fraction of 8-11.2 mm, the geometric mean size is 9.5 mm. To compare the liquid contents between the ore and model systems, the liquid holdup values of the 10 mm glass beads are also shown in Figure 5.2. The mean size of the ore bed is similar to the particle size of the model system therefore it is possible to compare the liquid content between the two systems.

The liquid holdup of the ore particles is approximately an order of magnitude higher than the liquid contents of the glass beads for the same particle size. The ore particles are slightly porous (estimated porosity of 5%) therefore the pore volumes contain substantial amount of liquid. This liquid fraction contributes to the internal liquid holdup ($\theta_i$) whereas it is zero for the non-porous glass beads.

Figure 5.2: Comparison of liquid contents between the model and ore systems in 300 mm column which were started dry.
Figure 5.3 illustrates total and external liquid contents for the 8-11.2 mm ore particles. The external liquid content is approximately half the total holdup, though this fraction increases with flow rate. The total liquid holdup was measured gravimetrically with a packed bed that contains the dry ore particles. During the external liquid holdup measurement, the ore particles were soaked in water for 3-4 days and then the additional liquid was removed by screening and patting them with a cloth before filling the 300 mm empty column. It was assumed that the water accessible pores within the ore particles are almost saturated when the external liquid holdup is measured.

![Graph showing liquid holdup vs. superficial velocity]

**Figure 5.3: Contribution of internal pore volume on the total liquid holdup in the ore system.**

In addition, the external liquid holdup in the ore system is approximately five times higher than the corresponding values for the glass beads. This can be explained using the difference in the shape of particles of the two systems. The higher tortuosity of flow paths in the ore system contributes to higher liquid contents as liquid remains within the packed bed due to longer residence time compared to the less tortuous flow paths in the model system.

In addition, the low solid-liquid contact angle of the ore particles compared to the glass beads may give higher liquid contents in the ore system.
5.2.1. LIQUID HOLDUP HYSTERESIS
Liquid holdup hysteresis was observed in the model system (see section 4.3) and it was demonstrated that changes in the number of flow paths between the flow increasing and decreasing arms was the major cause for this behaviour. Significant liquid holdup hysteresis in the ore system was observed in the preliminary experiments performed with dry ore particles though the magnitude is slightly smaller than in the model system. It is shown in Figure 5.4 for the ore packed bed containing the ore particles in the size range of 8-11.2 mm. The behaviour of this system will be explored in a similar manner to that used for the model glass bead system.

![Figure 5.4: Liquid holdup hysteresis of the initially dry ore particles in 300 mm column.](image)

5.3. STEADY AND RESIDUAL LIQUID CONTENTS
When starting with a dry bed, some significant differences in behaviour between the ore system and the glass bead system were observed. These include:

1) Longer time required to achieve the steady state

The breakthrough time for the first drop to come out of the bottom of the packed bed at the lowest flow rate was dramatically longer than with the model system (about one hour compared to five minutes in the model system when using a 300 mm column). The reason for this difference is that the ore particles are slightly porous and, having very fine channels and a relatively low contact angle, there is strong capillary suction into the ore particles. This
means that as soon as a water droplet touches an unsaturated ore particle the liquid gets sucked into the ore particle by capillarity. This water is also distributed to neighbouring dry particles by capillary wicking.

Only once virtually all the particles had been wet by the capillary wicking was there much evidence of the formation of extended liquid flow features such as liquid filled connections, rivulets and droplets. This is because the external liquid is only able to resist the capillary suction into the particles when they are near saturated. This meant that it took over 3 hours for the 300 mm column to reach steady state, compared to one hour for the non-porous glass beads.

2) Difficulty of separating internal and external liquid holdup

With a dry bed start-up condition, it is very hard to tell the proportion of the liquid held in the particles (i.e. internal liquid holdup) and between the particles (i.e. external liquid holdup). The liquid holdup model developed for the model system only considering the external liquid holdup as glass beads are non-porous. The external liquid holdup with a dry bed start-up condition cannot be determined with the porous ore particles as the load cell measures the total liquid holdup of the packed bed. Hence, an experimental method is required to measure the liquid in between the particles by separating its contribution from that of the liquid within the particles.

3) Inability to measure the residual liquid holdup accurately

The steady state packed bed should be allowed to drain until there is no more drainage to measure the residual holdup. The ore column continued to drip very slowly for more than 24 hours rather than stopping dripping within half an hour, as was the case with the glass beads. In preliminary experiments the ore column was allowed to drain overnight until it was switched back on to the next flow rate on the following morning. This meant that a single set of these preliminary results took 2 weeks to collect. However, the evaporation from the top section of the ore bed was significant, resulting in erroneously small liquid holdup values. This residual also included both the residual within the particles and around the particles.
A new method was thus developed to measure the steady state liquid volume in between the ore particles (external liquid holdup) and the residual liquid holdup between the particles.

### 5.3.1. STEADY STATE EXTERNAL LIQUID HOLDUP

To distinguish the two fluid classes in the ore system, the experiments were performed with saturated particles (note that it is the ore particles that are initially saturated and not the bed). This was achieved by placing the particles in water for 3-4 days and then screening them and patting them down with a cloth to remove any external water. The particles were then placed in the column and liquid flow to the column was started at the lowest flow rate (i.e. 0.0075 mm/s) straight away.

Starting with saturated ore particles, the breakthrough time was more comparable to that of the non-porous glass bead system, unlike the ore system which was started dry. Table 5.1 compares these breakthrough times for the ore system and the model system which contains the particles of the same average size (i.e. 10 mm because the geometric mean size of the 8-11.2 mm ore particles is 9.5 mm).

**Table 5.1: Comparison of the breakthrough times for the same particle size in the model and ore systems.**

<table>
<thead>
<tr>
<th>Particle size and condition in 300 mm column</th>
<th>Breakthrough time (s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>8-11.2 mm ore particles (Initially dry)</td>
<td>2855</td>
</tr>
<tr>
<td>8-11.2 mm ore particles (Pores saturated)</td>
<td>878</td>
</tr>
<tr>
<td>10 mm glass beads (Initially dry)</td>
<td>318</td>
</tr>
</tbody>
</table>

In addition, Table 5.2 compares the breakthrough times for all the particle sizes in both the model glass bead and the ore systems.

As both dry mass of the particles and their wetted weight is known, it is possible to calculate the amount of liquid initially held within the particles and thus the porosity of the ore can be estimated (see Table 3.3 in Chapter 3). This is the water accessible porosity rather than the actual porosity.

Assuming that liquid is being continuously added to the system, capillarity will ensure that the particles remain saturated or close to saturated. Then any extra liquid within the system will be held between the particles, allowing the steady state liquid holdup value (external
liquid holdup) to be calculated for a particular liquid flow rate. Typically, one hour was required to reach steady state with a 300 mm column. The steady state liquid contents were measured for every superficial velocity in both the increasing and decreasing arms of the flow.

Table 5.2: Comparison of the breakthrough times for all the particle sizes in the model and ore systems.

<table>
<thead>
<tr>
<th>Particle Size (mm)</th>
<th>Breakthrough Time (s) at 0.0075 mm/s</th>
<th>Particle Size Range (mm)</th>
<th>Breakthrough Time (s) at 0.0075 mm/s</th>
</tr>
</thead>
<tbody>
<tr>
<td>2</td>
<td>396</td>
<td>4-8 (pore saturated)</td>
<td>1709</td>
</tr>
<tr>
<td>10</td>
<td>318</td>
<td>8-11.2 (pore saturated)</td>
<td>878</td>
</tr>
<tr>
<td>14</td>
<td>250</td>
<td>11.2-13.2 (pore saturated)</td>
<td>852</td>
</tr>
<tr>
<td>18</td>
<td>239</td>
<td>13.2-16 (pore saturated)</td>
<td>827</td>
</tr>
<tr>
<td></td>
<td></td>
<td>16-20 (pore saturated)</td>
<td>562</td>
</tr>
<tr>
<td></td>
<td></td>
<td>20-26.5 (pore saturated)</td>
<td>530</td>
</tr>
<tr>
<td></td>
<td></td>
<td>26.5-31.5 (pore saturated)</td>
<td>518</td>
</tr>
<tr>
<td></td>
<td></td>
<td>31.5-37.5 (pore saturated)</td>
<td>503</td>
</tr>
<tr>
<td></td>
<td></td>
<td>37.5-45 (pore saturated)</td>
<td>340</td>
</tr>
</tbody>
</table>

5.3.2. RESIDUAL LIQUID HOLDUP

By attempting to ascertain the residual holdup in the ore column by turning off the flow, a large amount of liquid drains out in the first 10 minutes but then continues to drain very slowly for over a day. In comparison, the non-porous glass bead system, stopped dripping after about 20 minutes, with most of the liquid lost in the first few minutes (again in a 300 mm tall column). This results in quite different liquid content profiles as a function of time during the drainage portions of these experiments.

This is shown in Figure 5.5 where the liquid holdup of the glass bead system asymptotically reaches to a constant value very rapidly, while the ore system has a liquid content that initially decreases rapidly, but then continues to slowly decrease and does so for many hours. Note that the liquid holdup in this figure (i.e. Figure 5.5 right) is based on the difference in weight between the operating bed weight and the weight of the SATURATED
particles. The model system’s curve is a bit less smooth, but this is mainly due to the lower liquid contents involved and thus the finite precision of the measurements. An automatic data logger was also used for the ore experiments, resulting in more frequent data points.

![Graph showing liquid holdup over time for different particle sizes.](image)

**Figure 5.5:** Variation of the liquid holdup during the drainage in the 300 mm column at superficial velocity of 0.12 mm/s containing 10 mm glass beads (left) and 8-11.2 mm ore particles (right).

While the liquid holdup profiles look quite different, the profiles for the drainage rate out of the columns are very similar (Figure 5.6). That these curves are so similar, especially at short times, while the liquid content curves are so different, is surprising since one is simply the integral of the other. Looking at the flow curves there is a subtle, but important difference: For the model system the flow out of the column rapidly drops to zero, while for the ore system the flow out drops off very rapidly not to zero, but to a very small and near constant value (Figure 5.6).

In Figure 5.7 the drainage velocity is plotted on a log axis and it can be seen that this flow is two orders of magnitude lower than the initial flow (which is the same as the flow through the bed at steady state) and that it remains constant. This small flow out was observed in all the ore experiments and was virtually identical in a particular experiment (see Figure 5.9). This small flow rate remains virtually constant for hours before tapering off.
Chapter 5: Liquid Holdup Results for the Ore System

Figure 5.6: Comparison between the drainage velocity from glass beads and ore particles in 300 mm column. Initial liquid addition rate was 0.12 mm/s.

Figure 5.7: Drainage velocity for the 300 mm column containing 8-11.2 mm ore particles at superficial velocity of 0.12 mm/s (log-normal axes).

Visually this flow is accompanied by the slow drying out of the ore particles from the top of the column downwards, with the flow tapering off as the drier ore region approaches the bottom of the column. It is thus a good assumption that this small flow is caused by liquid...
motion within the ore particles. At steady state this flow is not a major contribution to the overall fluid motion, being over an order of magnitude smaller than the total liquid flux, but it does have a significant effect on the final drainage of the column due to the length of time over which it acts.

Therefore, it is possible to use this to estimate the residual liquid held between the ore particles. If the flow within the particles, rather than around them, is assumed to be constant at short times and have the same flow rate as it asymptotes to longer times, then the residual liquid content between the particles can be estimated by adding the liquid flowing out of the particles back onto the liquid content as given by equation 5.1.

\[ \theta_{ex} = \theta + \frac{v_{end}t}{Z} \]  

(5.1)

where \( \theta_{ex} \) is the estimated external residual liquid between the particles, \( v_{end} \) is the roughly constant flow out of the column at longer times and \( Z \) is the length of the column. Now \( \theta \) is the liquid content obtained by subtracting the weight of the bed from the initial weight of the bed containing saturated ore particles.

Unlike the actual liquid holdup of initially saturated particles, this estimated external liquid content between the ore particles, \( \theta_{ex} \), rapidly asymptotes towards a constant value. Figure 5.8 shows 8-11.2 mm ore particles in 300 mm column after the bed reached steady state at a superficial velocity of 0.12 mm/s. The liquid content after 40 minutes drainage was taken as the residual liquid holdup of the ore system for any particular flow rate.
After 25-30 minutes, it was found that the drainage rate was relatively constant for every flow rate for a particular size range of particles (see Figure 5.9). This constant end drainage velocity up to 40 minutes ($v_{end}$) was used to estimate the residual liquid holdup using equation 5.1.

Figure 5.9 shows the end drainage velocities for each superficial liquid velocity with the different size range of ore particles. Relatively similar values of drainage velocities can be seen for all the five superficial velocities applied for a particular size range of particles despite the slight variability of the experimental data. This is mainly caused by the variations of the load cell measurements during the drainage. In addition, Figure 5.9 implies that higher drainage velocities were obtained for the smaller size ore particles compared to the larger particle sizes. This is probably due to the larger water accessible porosities of the smaller particles (see Table 3.3). There is more than one possible reason for the larger water accessible porosities for the smaller particles. The most likely reason is that the average distance to the surface is smaller in the smaller particles and therefore there are less likely to be unconnected pore spaces in the smaller particles. Another possible contribution is that
more porous regions of the ore might fracture more easily and thus be over-represented in the smaller size fractions.

Figure 5.9: End drainage velocity of different ore fractions at different superficial velocities in the 300 mm packed bed.

The steady state and residual liquid holdup values were calculated as described above to cover the experimental conditions listed in Table 3.4 (in Chapter 3). This method provides a set of steady state and residual liquid holdup values for the increasing and decreasing arms of the flow. An experimental run of 72 hours was required to obtain these set of data using a method similar to that illustrated in Figure 4.13. Importantly, the ore column was not allowed to drain overnight to prevent evaporation, so liquid was introduced into the system overnight. In the morning it was drained for 40 minutes before being switched back on to the next available flow rate.

5.4. MODELLING OF FLOW PATHS

Figure 5.10 shows the liquid contents (i.e. external liquid holdup of the ore bed) for two narrow size range ore particles in the 300 mm column. The liquid holdup values decrease
with particle size. The liquid holdup hysteresis behaviour can also be seen especially at low superficial liquid velocities.

Figure 5.10: Liquid holdup hysteresis of pore saturated externally dried ore particles in 300 mm packed bed.

It can be assumed that flow paths through the ore particles are small rivulets, even though due to the opaque nature of the ore, it was hard to visually observe them, unlike in the beds of spherical glass beads. To explain the existence of hysteresis behaviour in these ore beds, the two parameters to be calculated are the relative flow rate ($v_s^*$) and the relative liquid holdup of the rivulets ($\theta^*$), similar to the model system.

The inter-particle steady state external liquid holdup and residual liquid holdup were used to calculate the relative flow rate ($v_s^*$) and the relative liquid holdup ($\theta^*$) for each superficial velocity of the ore system using equations 5.2 and 5.3.

$$v_s^* = \frac{v_s}{\theta_{ex(t=40 \text{ minutes})}}$$  \hspace{1cm} (5.2)

$$\theta^* = \frac{\theta_{ex(t=0)}}{\theta_{ex(t=40 \text{ minutes})}}$$  \hspace{1cm} (5.3)

The relationship between $v_s^*$ and $\theta^*$ is obtained for all the size fractions of ore particles and is shown in Figure 5.11 for only four size ranges.

It can be seen that the relationship between the two parameters is a single curve for the increasing and decreasing arms of the flow rates despite the presence of liquid holdup.
hysteresis. Similar behaviour was observed in the model system as well. The results given by the ore beds imply that the hysteresis behaviour is caused by the change of number of flow paths between the increasing and decreasing arms of flow rather than due to a change in the shape of the rivulets. This is consistent with the conclusion obtained with the model system.

![Graphs showing variation of \( v_s^* \) against \( \theta^* \)](image)

**Figure 5.11:** Variation of \( v_s^* \) against \( \theta^* \) for the increasing and decreasing arms of the flow for different size ranges of ore particles in 300 mm column. a) 4-8 mm b) 13.2-16 mm c) 20-26.5 mm d) 37.5-45 mm.

**5.4.1. VALIDITY OF THE INTER-PARTICLE FLOW MODEL**

The inter-particle flow model developed with the model system predicts a squared relationship between the two parameters in the model, which are \( v_s^* \) and \( \theta^* - 1 \) (equation 4.18). The same model can be tested for the ore system. Figure 5.12 shows the \( v_s^* \) values for
all size fractions are plotted against the corresponding $\theta^* - 1$ values for the ore particles. In Figure 5.12, the data follows the same power law relationship for all the particle sizes. It implies that the flow model developed in this study can be applied to the typical size range of the crushed ore particles employed in column leaching and industrial leaching operations.

Figure 5.12: The relationship between the relative flow rate ($v_s^*$) and the additional liquid content of that rivulet ($\theta^* - 1$) for all the size fractions of ore particles.

It can be seen that in Figure 5.12, the data deviates slightly from a power law relationship of two. During the theoretical modelling of the flow paths in Chapter 4, it was assumed that the cross-sectional shape of the rivulets is constant. The shape of the rivulet will be mainly dictated by the contact angle and the Bond number. The relationships given in equations 4.11 and 4.12 will only hold if the shape and aspect ratio of the flow channel remains constant. Then, the flow rate of a rivulet ($Q_r$) is proportional to the square of the flowing area of the rivulet ($A_r$). For this a square relationship between the relative flow rate of the rivulet ($v_s^*$) and the additional liquid content of that rivulet ($\theta^* - 1$) was predicted. A very good agreement with this model was found for the glass bead system (see Figure 4.19 and Table 4.2). But equations 4.11 and 4.12 do not apply to flow down a thin film as the film changes aspect ratio as the flow rate changes.
For a thin film falling over the surface of the particle, a relationship can be derived for the flow rate in the liquid flow \( Q_{film} \), which has a cubed relationship with the cross-sectional area of the film \( A_{film} \) (see Appendix D). This correlation thus predicts a cubic relationship between the relative flow rate in a film and the additional liquid content of that film.

Similar to the model glass bead system, the slope of the line between \( \nu_s^* \) and \( \theta^* - 1 \) was calculated to determine the actual slope of the experimental data in the ore system. The actual slopes and their corresponding goodness of fit values are given in Table 5.3 for all the ore size ranges. It shows that the data in Figure 5.12 is slightly steeper than quadratic; this deviation could be due to some of the liquid flow occurring in films rather than rivulets. The extra film flow compared to the glass bead system could be due to lower contact angles and more angular particles.

**Table 5.3: Actual slopes of the lines between \( \nu_s^* \) and \( \theta^* - 1 \) in Figure 5.12 for all the particle sizes in the ore system in 300 mm column. The ore particles were initially saturated and externally dried before placed in the 300 mm column.**

<table>
<thead>
<tr>
<th>Particle size range (mm)</th>
<th>Slope of the line between ( \nu_s^* ) and ( \theta^* - 1 )</th>
<th>Goodness of fit</th>
</tr>
</thead>
<tbody>
<tr>
<td>4-8</td>
<td>2.16</td>
<td>0.98</td>
</tr>
<tr>
<td>8-11.2</td>
<td>2.54</td>
<td>0.96</td>
</tr>
<tr>
<td>11.2-13.2</td>
<td>2.58</td>
<td>0.94</td>
</tr>
<tr>
<td>13.2-16</td>
<td>2.48</td>
<td>0.94</td>
</tr>
<tr>
<td>16-20</td>
<td>2.63</td>
<td>0.87</td>
</tr>
<tr>
<td>20-26.5</td>
<td>2.44</td>
<td>0.89</td>
</tr>
<tr>
<td>26.5-31.5</td>
<td>2.85</td>
<td>0.99</td>
</tr>
<tr>
<td>31.5-37.5</td>
<td>2.49</td>
<td>0.97</td>
</tr>
<tr>
<td>37.5-45</td>
<td>2.66</td>
<td>0.91</td>
</tr>
</tbody>
</table>

**5.5. RESULTS OF THE POLY-DISPERSED ORE BEDS**

Typically industrial heap leaching heaps have a wide particle size range. To understand the hydrodynamics in columns and heaps it is crucial to study the heap performance using these wider particle size distributions. This can be started with a flow behaviour experimental study using a poly-dispersed ore mixture, which was prepared by mixing two or more narrow size fractions of the ore.
All the liquid flow experiments presented in this chapter were conducted using beds consisting of comparatively narrow size range of particles to determine the direct relationship between the flow and the particle size. In addition, a set of experiments were conducted using well defined mixtures of different narrow size particles in order to determine the validity of the inter-particle model. It provides the effect of the particle size distribution on the flow behaviour and in particular, tries to ascertain the characteristic particle size to describe the flow mechanics.

In this section, the results of four binary mixtures prepared using equal weight proportions of the narrow size range ore particles will be described. The experimental methods used and the conditions studied were similar to those used for the narrow size range ore beds.

For the poly-dispersed mixtures of ore particles, the relative flow rate ($v_\theta^*$) and the relative liquid holdup ($\theta^*$) were again determined. The relationship between the two parameters followed a single curve for all the poly-dispersed mixtures. In addition, the relationship between $v_\theta^*$ and $\theta^* - 1$ is shown in the following figures (Figure 5.13, 5.14, 5.15 and 5.16).

All the poly-dispersed mixtures follow a similar relationship to the individual narrow size fractions, despite the slight experimental variability of the data. It demonstrates the applicability of the inter-particle flow model for the ore mixtures as well.

![Figure 5.13: The relationship between $v_\theta^*$ and $\theta^* - 1$ for the binary mixture of 8-11.2 mm and 16-20 mm. The same relationship for the individual size fractions is also shown.](image-url)
Chapter 5: Liquid Holdup Results for the Ore System

Figure 5.14: The relationship between $\nu_s^*$ and $\theta^* - 1$ for the binary mixture of 11.2-13.2 mm and 20-26.5 mm. The same relationship for the individual size fractions is also shown.

Figure 5.15: The relationship between $\nu_s^*$ and $\theta^* - 1$ for the binary mixture of 13.2-16 mm and 26.5-31.5 mm. The same relationship for the individual size fractions is also shown.
Figure 5.16: The relationship between $\nu_s^*$ and $\theta^* - 1$ for the binary mixture of 16-20 mm and 31.5-37.5 mm. The same relationship for the individual size fractions is also shown.

5.6. COMPARISON OF THE MODEL AND THE ORE SYSTEMS

The effect of the particle porosity on the overall flow through a heap was investigated by comparing the behaviour of a bed consisting of non-porous glass beads with a system consisting of similar sized ore particles. The main reason is both inter-particle porosity and intra-particle porosity have significantly different effects on heap flow behaviour due to their different length scales. Thus the Bond numbers are also different in the two classes of the porosities. Table 5.4 shows the Bond numbers for the flow between the particles and these are of order 1 or larger for both the model glass bead and ore systems.

The theoretical inter-particle flow model was developed to describe the flow between the particles in a way that accounts for hysteresis in both systems. If the inter-particle liquid content is separated from the liquid held in the pore spaces of the ore particles, it is shown that both model and ore systems follow a similar power law relationship for all the particle sizes studied. This can be clearly shown by plotting $\nu_s^*$ as function of $\theta^* - 1$ for both of the packed bed systems. Figure 5.17 shows this relationship.
Table 5.4: Bond numbers of the model and the ore systems using particle size as the characteristic length scale.

<table>
<thead>
<tr>
<th>Model System</th>
<th>Bond Number</th>
<th>Ore System</th>
<th>Particle Size Range (mm)</th>
<th>Geometric Mean Size (mm)</th>
<th>Bond Number</th>
</tr>
</thead>
<tbody>
<tr>
<td>Particle Size (mm)</td>
<td>Bond Number</td>
<td>Particle Size Range (mm)</td>
<td>Geometric Mean Size (mm)</td>
<td>Bond Number</td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>0.5</td>
<td>4-8</td>
<td>5.6</td>
<td>4.0</td>
<td></td>
</tr>
<tr>
<td>10</td>
<td>12.4</td>
<td>8-11.2</td>
<td>9.5</td>
<td>11.1</td>
<td></td>
</tr>
<tr>
<td>14</td>
<td>24.3</td>
<td>11.2-13.2</td>
<td>12.1</td>
<td>18.4</td>
<td></td>
</tr>
<tr>
<td>18</td>
<td>40.2</td>
<td>13.2-16</td>
<td>14.5</td>
<td>26.2</td>
<td></td>
</tr>
</tbody>
</table>

From Figure 5.17 (and Figure 5.18), it can see that the pre-factor in the relationship between \( \nu_0^* \) and \( \theta^* - 1 \) is over 5 times larger for the glass beads compared to the copper ore system. There are two main reasons for this. Firstly, the tortuosity of the rivulets will be higher in the ore system. This has the effect of increasing the apparent drag coefficient (see equation 4.37) and thus decreasing \( K \). The other reason is that the particle contacts in the ore system are more angular than those in the glass beads. This means that they will hold less liquid, which will result in a lower average residual area of the rivulet and thus a lower value of \( K \).

A difference in contact angle could also account for some of the difference as it will influence both the shape of the flowing rivulet (and thus the drag coefficient) and the size of the residual connections (see section 4.5.4.2).
5.6.1. PRE-FACTOR

In Figure 5.18, the pre-factor in the relationship between the relative flow rate and the relative holdup is plotted against the particle size for both the glass beads and the ore particles. The geometric mean size of each size fraction was used as the average particle size in the narrow size range ore particles. It can be seen that K is quite a strong function of the particle size in the case of the spherical glass beads, more than doubling over the particle size range measured. In the ore system, though, there is far less variation in the pre-factor, with an average value of 0.0063 \pm 0.0008 m/s, with very little systematic change in this value with particle size.

The reason for this is probably due to the angularity of the ore particles. In angular particles the local shape of the particles as seen by a rivulet flowing over it and, particularly, the local shape of the particle in the vicinity of particle to particle contacts is not a strong function of
the particle size. For instance, the rivulet and connection shape will be influenced by the angles between facets and the number of facets coming together at points, neither of which necessarily depends on the macroscopic size and shape of the ore particles. On the other hand, the local shape of the glass particles experienced by the rivulets in the model system is directly related to the size of the particles.

That the pre-factor in the model is virtually independent of particle size does not mean that the total liquid holdup is independent of particle size. This only says that the relationship between the relative flow rate and the relative holdup is virtually independent of the particle size in the angular particle ore system.

![Graph](image)

Figure 5.18: Comparison of the pre-factor ($K$) in the model and the ore systems.

5.6.2. RESIDUAL LIQUID HOLDUP

Figure 5.19 illustrates the maximum residual liquid holdups in the ore system at a superficial velocity of 0.12 mm/s as a function of the geometric mean size (hysteresis means that the residual is not constant). The residual liquid content, though, is quite a strong function of the particle size, as can be seen from Figure 5.19, which means that the actual (rather than relative) liquid content will depend quite strongly on the particle size. The reason for this is that the number of potential rivulet paths through the bed increases as the particle size
decreases and therefore the number of residual connections within the drained bed also increases.

**Figure 5.19:** Variation of the residual liquid holdup as a function of geometric mean size of the different ore size fractions in 300 mm column.

The geometric mean size of the four binary mixtures of the ore particles was calculated and the residual liquid holdup, which correspond to a superficial velocity of 0.12 mm/s, were plotted on the same graph (see Figure 5.20).

**Figure 5.20:** Variation of the residual liquid holdup as a function of geometric mean size of the ore size fractions and binary mixtures in 300 mm column.
Figure 5.20 shows this comparison for both the narrow size range fractions and the binary mixtures of ore particles. When represented by their geometric mean size, the residual holdup of the mixtures quite closely follows that of the narrow size fractions. This mainly implies that the smaller particles have a larger impact on the residual than the larger particles. This is consistent with the results for the mixtures of different sized glass beads.

5.7. SUMMARY

The results and analysis of the liquid holdup experiments conducted with the narrow size range ore particles and their binary mixtures were described in this chapter, followed by a comparison of the results with the model system. During the initial experiments with dry ore particles it was found that the total liquid holdup is approximately an order of magnitude higher than the similar sized glass beads. The pore volumes in the ore particles contribute nearly half of the overall liquid holdup.

In the preliminary experiments, the ore system behaved quite differently to the model glass bead system. The results surmised that this was due to the porosity of the ore particles compared to the non-porous glass beads. Thus it was necessary to separate the effect of the externally and internally held liquid.

A new methodology was developed to measure the external liquid holdup and the external residual liquid holdup accurately using the ore systems. Initially, the ore particles were saturated and the additional liquid over the surface of the particle was removed in order to prepare a packed bed with pore saturated but externally dry ore particles. The steady state external liquid content was determined accurately and the external residual liquid content was estimated to determine the model parameters. These liquid holdup values fit the inter-particle flow model with a near squared correlation between the two parameters of the model, which are the relative flow rate of the rivulet and the additional liquid content of that rivulet. The slight deviation of the experimental data from a quadratic relationship can be explained as the presence of some film flow features through the particles of ore bed. However, the flow behaviour is not completely film flow dominated as it would give a cubic relationship between the two model parameters.

Similar to the narrow size range ore particles, the model was tested with binary mixtures in order to investigate the validity of the flow model for wider size distributions. The results
presented show that the binary mixtures follow the same inter-particle flow model as the narrow size ranges.

The pre-factor in the flow model for the model system is over five times higher than in the ore system. Unlike the model system, the main model parameter, \( K \), is not a strong function of the average particle size, which was calculated as the geometric mean size. However, the steady state and residual liquid holdups are strong functions of the average particle size.

The main conclusion of this chapter is that the average liquid content in a heap is not a good basis on which to model its flow. Instead, the externally and internally held liquid needs to be considered separately as they have very different effects on the flow behaviour.
CHAPTER 6

TRANSIENT BEHAVIOUR IN THE MODEL GLASS BEAD AND ORE SYSTEMS

6.1. INTRODUCTION

Studying the transient or unsteady state behaviour of a system can contribute significantly to the understanding of the detailed fluid flow behaviour in that system. This chapter investigates the transient changes in the liquid holdup in the model glass bead system and the ore system.

A discussion about the transient changes of the liquid holdup will be given with examples from the literature before going on to describe the results of this chapter. This section highlights the importance of the unsteady flow investigation and it also implies that the gravimetric method can be used to measure transient liquid holdup changes.

The movement of the initial wetting front of the model glass bead system was investigated until the packed bed reaches steady state. The results of the drained weight of the mono-dispersed particles from different column lengths will be presented in order to analyse the flow behaviour.

The drainage behaviour of the ore system was simulated using the inter-particle flow equations developed in this study coupled with a continuity equation. The simulated liquid content variation of the ore bed will be presented and these results can also be used to prove the applicability of the residual holdup estimation technique used in Chapter 5, followed by a summary of this chapter.
6.2. TRANSIENT CHANGES OF LIQUID HOLDUP

A system is defined as being unsteady state or transient when its appearance changes with time (Wilkes, 2006). In heaps, intermittent leaching is seldom practiced and it has shorter solution application periods followed by much longer rest periods (Bartlett, 1992a). However, this solution application technique can be employed to increase the heap performance significantly. Intermittent leaching facilitates slow mineral oxidation in order to provide higher recoveries during the next solution application cycle. Typically, the flow behaviour in these heaps during the rest periods is under the transient state. A similar operation can be seen in trickle bed reactors (TBRs) which are analogous systems to heap leaching. The unsteady state operation of TBRs by periodically modulating the liquid or gas supply also leads to transient fluid hydrodynamics (Khadilkar et al., 1999). Tukac et al. (2007) reported an increase in the reactor performance compared with the steady state flow behaviour. These examples highlight the importance of the transient flow behaviour in packed beds and heap leaching heaps.

Figure 6.1 shows the three stages of the liquid holdup variation in a system and the first stage is the initial phase after the start of the liquid addition. The breakthrough of liquid at the bottom of the system occurs and the liquid holdup increases gradually until it reaches the steady state. The second stage is the steady state with an asymptotic liquid content. After stopping the liquid addition, the drainage continues until the subsequent liquid addition commences again and it is shown in the third stage. The liquid holdup variation in both the first and the third stages follows transient changes as the liquid holdup changes with time.

Despite the extensive studies in order to investigate the liquid holdup in the systems of different scales, Standish (1968) reported that the significance of the measurement of transient changes of liquid holdup. The gravimetric method determines the weight of the packed bed continuously without any practical experimental difficulties to monitor all the transient changes in total liquid holdup under the actual operating conditions. In addition, the technique is more practical and more accurate than the other liquid holdup measurement techniques (Euzen et al., 1993). Hence, Standish (1968) performed the liquid flow experiments (without any gas flow) in a glass column packed with 6.3 mm ceramic and
glass Raschig rings using the gravimetric technique to measure the transient changes of the liquid holdup.

![Diagram of liquid holdup variation](image)

**Figure 6.1: Schematic illustration of liquid holdup variation in the periodic operation (Liu et al., 2009).**

The objective of this chapter is to investigate the transient liquid holdup variations in both the model glass bead system and the ore system. The liquid holdup changes until steady state is reached, and this was investigated using the model system whereas the drainage behaviour was investigated in the ore system only.

### 6.3. MOVEMENT OF THE WETTING FRONT FOR THE MODEL GLASS BEAD SYSTEM

Liquid is introduced into the model glass bead system when it was dry and a measuring cylinder was kept on top of the electronic balance in order to measure the drained liquid weight continuously. The drained weight of water was recorded every second by coupling the high resolution balance into a computer. This methodology was similar to the drainage method described in section 3.4.1.2., but the objective of the current measurement method is to investigate the movement of the wetting front in the column until it reaches the steady state. In order to ascertain the wetting front movement, the experiments were performed in three different column lengths: 300, 500 and 800 mm and with different particle sizes. All the experiments were performed with mono-dispersed packed beds and the liquid superficial velocity was 0.0075 mm/s.
The movement of the liquid within the packed bed is potentially influenced by both gravitational and capillary/dispersive effects. When the flow is completely gravity dominated a sharp wetting front is expected, whereas capillary dominated flow acts to even out the liquid content of the packed bed because of the greater dispersive effects. The effects of both capillarity and gravity are expected when the flow is in the transition region between the capillary and gravity dominated flow. If this is the case, both capillarity and gravity forces are acting where gravity acts to sharpen the wetting front, but capillarity tries to spread it out (this is illustrated in Figure 6.2). The reason why gravity sharpens up the front is that the liquid velocity increases with liquid content. This means that liquid that moves ahead of the front will slow down thus be caught by the front.

When the two effects are balanced the movement of the wetting front forms a standing wave, where the shape of the front does not change as it moves.

![Figure 6.2: Schematic illustration of the mechanisms within a standing wave.](image)

The standing waves are parallel to each other for a particular particle size when packed bed height increases and thus the drainage experiments using the same particle size and with different column lengths can be used to investigate this behaviour. Figure 6.3 illustrates these parallel standing waves. What would characterise a standing wave would be an initial
time period in which liquid does not drain out of the column followed by a rapid transition towards a uniform drainage rate. If there is a standing wave, then in different length columns the initial time without any drainage would change, but the shape of the transition region would be the same.

![Diagram of liquid holdup and distance from top of the column](image)

**Figure 6.3:** Schematic illustration of standing waves for different column lengths.

Figure 6.4a shows the variation of the drained weight as a function of time since the start of the liquid addition for 10 mm particles. The packed bed requires around one hour to reach steady state and the data logging was carried out over that period. However, Figure 6.4a shows the packed bed behaviour for the first half an hour because the movement of wetting front should be analysed especially during the initial period of the liquid addition. The curves for three different column lengths are shown and the breakthrough time increases with the column length.

In Figures 6.4 and 6.5, which are for the superficial liquid velocity of 0.0075 mm/s, it can be seen that when the curves are shifted onto one another for different column lengths, the transitions are very similar and all very rapid, which is characteristic of a standing wave. Differences in the slope of the curves at longer times are due to experimental variability in the input liquid flow rate.
Figure 6.4: Drained weight of water for 10 mm particles in different column lengths for a superficial velocity of 0.0075 mm/s. a) as a function of time b) as a function of time shifted relative to 800 mm column.
Figure 6.5: Drained weight of water for 14 mm particles in different column lengths for a superficial velocity of 0.0075 mm/s. a) as a function of time b) as a function of time shifted relative to 800 mm column.
6.4. SIMULATION OF LIQUID DRAINAGE FOR THE ORE SYSTEM

Since a relationship for the liquid flux as a function of the liquid content has been developed (i.e. equation 4.18), it can be used to predict the evolution of the liquid content in the column as it drains. This will take the form of a partial differential equation (PDE) as the liquid content in the column will vary both spatially and temporally as it drains.

The liquid content of the ore bed can be expressed using the volume continuity equation:

$$\frac{\partial \theta_e}{\partial t} = -\nabla F$$  \hspace{1cm} (6.1)

where $\theta_e$ is the external liquid holdup, $F$ is liquid flux within the bed.

If residual liquid holdup ($\theta_{\text{residual}}$) is constant with respect to time ($t$) and distance ($Z$) and the only liquid content variations are in the vertical direction, equation 6.1 can be written as:

$$\frac{\partial}{\partial t} \left( \frac{\theta_e}{\theta_{\text{residual}}} \right) = -\frac{\partial}{\partial Z} \left( \frac{F}{\theta_{\text{residual}}} \right)$$  \hspace{1cm} (6.2)

Assuming that the effect of capillarity on the external liquid drainage is small, the quantity of the right hand side in equation 6.2 can be approximated using equation 4.18 (in Chapter 4):

$$\frac{F}{\theta_{\text{residual}}} = K \left( \frac{\theta_e}{\theta_{\text{residual}}} - 1 \right)^2$$  \hspace{1cm} (6.3)

The assumption that capillarity has only a small impact in the vertical direction can be made since the inter-particle spaces are quite large. The capillary pressures ($P_c$) will be of order (see Appendix E):

$$P_c \approx O \left( \frac{\gamma}{r_{ps} \sqrt{\theta}} \right)$$  \hspace{1cm} (6.4)

where $\gamma$ is the surface tension of the liquid, $r_{ps}$ is the particle spacing and $\theta$ is the liquid holdup of the system.

Since the surface tension of water is approximately 0.07 N/m, the particle spacing is of the order of millimetres and the external liquid holdups are a few percentage, the capillary pressure will thus be of order tens to hundreds of Pascals. Since the column is tens of centimetres tall, the pressure gradient associated with capillarity will thus be of order 100-
1000 Pa/m. This is compared to the effect of gravity, which is of order 10,000 Pa/m (i.e. ρg).

This implies that gravity will be the dominant factor in the flow, with capillarity as a secondary, though not necessarily totally insignificant factor.

The Bond number can be contributed in two ways during the liquid drainage. The first one is to determine the shape of the connections between the particles and the second one is on the liquid flow when both gravity and capillary forces are acting. The Bond number for the shape of flow paths is given by \( \rho gd_p^2 / \gamma \), where \( d_p \) is the particle size.

The driving force due to gravity during the drainage is given by \( \rho g \) whereas the driving force exerted by capillarity can be expressed as \( \gamma / r_c d_p \), where \( r_c \) is the radius of curvature of the rivulets. Now, the corresponding Bond number on the flow can be expressed as the ratio of the gravity and capillary driving forces and it is given by \( \rho g r_c d_p / \gamma \), which is not the same Bond number acting on the shape of the connections.

The ratio of the steady state external and the residual liquid holdup \( (\theta_e / \theta_{residual}) \) is defined as \( \theta^* \) in this work.

Substituting equation 6.3, into equation 6.2:

\[
\frac{\partial \theta^*}{\partial t} = -\frac{\partial}{\partial z} [K(\theta^* - 1)^2] \tag{6.5}
\]

Equation 6.5 was solved numerically using the first order upwind method. The discretised equation that describe the liquid holdup in the packed bed of length, \( Z \), and at any time can be written as follows.

\[
\frac{\theta^*_i^{j+1} - \theta^*_i^j}{\Delta t} = -K \left[ \frac{(\theta^*_i^{j-1})^2 - (\theta^*_i^{j-1-1})^2}{\Delta z} \right] \tag{6.6}
\]

where \( i \) is index of the space, \( j \) is the index of the time.

The following initial condition was used:

\[
\theta^* (0, Z) = \frac{\theta_e}{\theta_{residual}} \tag{6.7}
\]

The top boundary condition is:

\[
Q_L = 0 \tag{6.8}
\]

Therefore, as no capillarity: \( \theta_e = \theta_{residual} \)
There was no boundary condition at the bottom of the bed as the order of the PDE is one.

For each set of experimental drainage curves, the simulated drainage velocities were formulated using the solution of equation 6.6. In all of the simulations presented below the flux due to flow through the particles, \( v_{end} \), was added onto that obtained from the above equations, which are only for the flow around the particles. Therefore, assuming the estimated residual liquid values according to the equation 5.1 (in Chapter 5) are reliable, the liquid drainage of the ore bed can be simulated.

The following figures (Figure 6.6a and 6.6b) compare the agreement between the experimental and simulated data for two superficial velocities. Since the steady state and residual liquid holdups are known experimentally, the value of \( K \) can be obtained using these values together with the known liquid flux through the system. This means that these predictions of the drainage rates out of the bed are made with **NO** fitting parameters.

Figure 6.6a shows the comparison of the two sets of data for a 300 mm column packed with 8-11.2 mm ore particles for a superficial velocity of 0.12 mm/s. Figure 6.6b shows the same for a superficial velocity of 0.03 mm/s. A good fit to the experimental data can be observed, with very high \( R^2 \) values obtained despite the lack of adjustable fitting parameters. It also implies that the estimated residual liquid holdup values using equation 5.1 (in Chapter 5) are reliable and the methodology which was developed in this work to estimate the external residual liquid holdup between the ore particles is reasonably accurate.

In addition, for each set of experimental drainage curves, the simulated average liquid holdups of the ore bed were also obtained using the solution of equation 6.6. This is thus the integral of the data from Figure 6.6 starting from the initial liquid holdup, though the simulation gave out both results without the need to integrate from one to the other. Figures 6.7a and 6.7b compare the agreement between the experimental and the simulated liquid contents for the same two superficial velocities (i.e. 0.12 mm/s and 0.03 mm/s) employed in Figure 6.6a and 6.6b.
Figure 6.6: Simulated and experimental drainage curves for different superficial velocities with 8-11.2 mm ore particles in 300 mm column (solid line is simulated, diamonds are the experimental results). a) 0.12 mm/s b) 0.03 mm/s.
Figure 6.7: Simulated and experimental liquid contents during the drainage for different superficial velocities with 8-11.2 mm ore particles in 300 mm column. a) 0.12 mm/s b) 0.03 mm/s.
Chapter 6: Transient Behaviour in the Model Glass Bead and Ore Systems

It can be seen that in Figures 6.7a and 6.7b, the experimental liquid holdup values at both liquid superficial velocities agree with the simulated liquid contents at both shorter and higher drainage times. At shorter time intervals (i.e. just after the drainage) the actual and simulated liquid contents are similar and it is possible that this is due to gravity dominating over capillary forces for higher liquid contents. At moderate time intervals the simulated liquid holdups are less than the experimental values obtained during the drainage. This implies that this simple model, which was used to simulate the transient drainage behaviour, slightly underestimates the liquid content of the bed at moderate drainage times. Since the main factor that is not included in this simple transient model is capillarity, this is the likely reason for this discrepancy. This is supported by the fact that the model overestimates the drainage velocity (underestimates the liquid content), since capillarity will act down liquid content gradients and will thus act to retard the drainage. Figures 6.8 and 6.9 show the simulated liquid content profiles with height at different times after liquid addition is stopped. The liquid flow out of the column is determined by the conditions at the bottom of the column (height zero). Since these simulations only consider gravity, this means that the flow out of the column is simply a function of liquid content.

At very short times the average liquid content of the column drops, but the liquid content at the bottom remains constant and thus the liquid flow out also remains constant. This is most pronounced at lower liquid addition rates (Figure 6.9). This can be seen in both the simulated and experimental data in Figure 6.10 when flow out is constant for a hundred seconds or so. Once the liquid content at the bottom of the column starts to drop, this will have a significant effect on the flow out of the column, which can be seen in the very rapid drop in outflow after the initial constant flow. While not included in the model, capillarity will have an effect on out flowing liquid when there is a gradient in the liquid content at the bottom of the column. This means that at short times, when the liquid content near the bottom is constant (Figures 6.8 and 6.9) capillarity will have no effect. As soon as the drying nears the bottom, the liquid content gradient reaches a maximum and thus the effect of capillarity will be at its largest. Subsequently, both the liquid content and its gradient decrease with time and it is in this intermediate time period that the discrepancy in the predicted liquid contents is the greatest (Figure 6.7). This implies that the simulations can be improved by including capillarity.
Figure 6.8: External liquid content of 8-11.2 mm ore particles in 300 mm column as a function of column height for the superficial velocities of: a) 0.12 mm/s b) 0.03 mm/s.
Figure 6.9: External liquid content of 8-11.2 mm ore particles in 300 mm column as a function of column height for the superficial velocities of: a) 0.015 mm/s b) 0.0075 mm/s.
Figure 6.10: Simulated and experimental drainage curves with 8-11.2 mm ore particles in 300 mm column for the superficial velocities of: a) 0.015 mm/s b) 0.0075 mm/s.
6.5. SUMMARY

The transient changes of the liquid holdup in the model glass bead system and the ore system were investigated as these changes are crucial for understanding the hydrodynamics in packed bed and heap leaching systems. The experiments on different column heights show that the initial wetting front moving through the packed bed follows a standing wave.

The drainage behaviour of the ore system was simulated using the inter-particle flow model equations developed in this study. The effect of the capillary forces was assumed to be negligible compared with the gravitational forces, which was shown to be a reasonable, but not a perfect assumption. Thus a good agreement between the experimental and the simulated drainage curves was observed. Therefore, this simple drainage model can be used to simulate the liquid drainage in the gravity dominated flow. The simulation also proves that the residual liquid holdup estimation methodology performed in Chapter 5 is reasonably accurate.

Despite the fact that the capillary forces are assumed to be negligible, the results showed that these forces play an important role during the intermediate times of drainage in the bed as the experimental liquid contents are higher than the simulated liquid contents. However, these liquid contents are in very good agreement for both the initial and late time intervals during the drainage of the ore bed.

The variation of the external liquid content with the column height of the bed at very low superficial velocities was simulated and it can be used to understand the drainage behaviour in different column lengths. Further, this analysis shows that the intermediate times are when capillary effects are likely to be largest. The results of this analysis are also consistent with the corresponding temporal variations of the external liquid content for both the lowest and highest superficial liquid flow rates studied.
CHAPTER 7

HYDRODYNAMIC DISPERSION ANALYSIS

7.1. INTRODUCTION
The investigation of hydrodynamic dispersion in porous media such as packed beds and hydrocarbon reservoirs is found in petroleum engineering and research related to groundwater flow, trickle bed reactors, column leaching and heap and dump leaching. Due to its engineering relevance, it is important to understand the flow behaviour in these systems. As the porosity ranges from a few microns to millimetre or greater in systems such as leaching, it results in the simultaneous existence of different flow and transport mechanisms in the same system.

Many of the dispersion studies in packed bed, column leaching and heap leaching systems have been conducted by using salt tracers and dyes to determine the dispersion coefficients. However, the resultant dispersion values vary significantly among the reported experimental studies. The objective of this study is to obtain similar axial dispersion values through residence time distribution (RTD) analysis after injecting the salt tracers into the packed bed system. In addition, this work investigates the characterisation of the calculated dispersion values with the appropriate characteristic velocity.

The RTD analysis, including the normalised tracer response curves, will be described in this chapter using experimental data. The accuracy of the RTD curves and the liquid holdup values was investigated by comparing mean residence time values independently. The numerical method used to fit the experimental RTD curves in order to obtain the axial dispersion values will be detailed. The resultant axial dispersion coefficients for different
superficial liquid velocities and particle sizes will be presented with different characteristic
velocities.

Finally, the liquid holdup in the mobile region and the immobile region and the mass
transfer coefficient between the mobile and immobile regions will be presented, followed
by a summary of this chapter.

### 7.2. RESIDENCE TIME DISTRIBUTION ANALYSIS

Following the pulse injection, the measured tracer response curve, \( C(t) \), is the variation of
effluent conductivity (\( \mu S \)) with time. Figure 7.1 shows the effluent tracer curves for a 300
mm column packed with 18 mm glass beads at each superficial liquid velocity.

![Figure 7.1: Effluent tracer conductivity variation with time in 300 mm column packed with 18 mm glass beads.](image)

The effluent tracer curves were normalised for pulse injection using equation 2.10 (Chapter
2). The resultant exit age distributions, \( E(t) \), are shown in Figure 7.2. All curves are
asymmetric around the mean residence time. A long tail in the distribution can be observed
and is most significant at low liquid superficial velocities. This is consistent with other
residence time distribution studies conducted for column leaching, heap and dump leaching and packed beds (e.g. Murr, 1979; Decker, 1996; de Andrade Lima, 2006). The stagnant zones present in packed beds are indicated by a long tail. For porous particles, it can be argued that the fluid volume in the pores accounts for the immobile fluid. However, as glass beads are non-porous, any stagnant liquid must be held between the particles. The stationary liquid in this system will be that held by capillarity at particle contacts and possibly as thin films around the particles. Mass transfer between the stationary and mobile fractions will occur by mixing and molecular diffusion in the capillary connections. The fact that a salt tracer and a particulate tracer (with essentially no molecular diffusivity) have essentially the same dispersion behaviour (see Chapter 8) would suggest that hydrodynamic effects are dominant. The reason why stagnant regions result in a long tail is that they release tracer back into the flow long after the main pulse has passed.

![Figure 7.2: Normalised tracer response curves for 300 mm column packed with 18 mm glass beads.](image)
The cumulative distribution function, $F(t)$, was obtained using $E(t)$ curves. The relationship between $F(t)$ and $E(t)$ is:

$$F(t) = \int_0^\infty E(t)dt$$  \hspace{1cm} (7.1)

For each $E(t)$ curve in Figure 7.2, the resultant $F(t)$ curve was computed and these are shown in Figure 7.3. Residence time distribution (RTD) graphs are bounded between 0 and 1 and represent the fraction of the salt tracer that has a residence time less than a particular value.

The first moment of the $E(t)$ curve is the mean residence time ($t_R$) of the tracer and $t_R$ is given by:

$$t_R = \int_0^\infty tE(t)dt$$  \hspace{1cm} (7.2)

![Figure 7.3: Cumulative RTD curves for 300 mm column packed with 18 mm glass beads.](image)

As the total liquid holdup at each liquid superficial velocity was also measured, the mean residence time can be determined using equation 7.3.
where $V_e$ is volume of the empty column and $Q_L$ is volumetric flow rate into the column. $t_R$ values are calculated from both equations (i.e. equation 7.2 and 7.3) and these calculations are independent of each other. Hence, the two values of $t_R$ at each superficial velocity can be used to validate the RTD curves shown in Figure 7.3. Table 7.1 compares these mean residence times.

**Table 7.1: Calculated $t_R$ values from both liquid holdup measurements and RTD curves.**

<table>
<thead>
<tr>
<th>Superficial Velocity (mm/s)</th>
<th>$t_R$ based on liquid holdup (s)</th>
<th>$t_R$ based on RTD (s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.0075</td>
<td>424.04</td>
<td>423.02</td>
</tr>
<tr>
<td>0.015</td>
<td>249.52</td>
<td>250.61</td>
</tr>
<tr>
<td>0.03</td>
<td>126.69</td>
<td>126.10</td>
</tr>
<tr>
<td>0.06</td>
<td>70.14</td>
<td>70.36</td>
</tr>
<tr>
<td>0.12</td>
<td>43.31</td>
<td>44.00</td>
</tr>
</tbody>
</table>

It is shown same mean residence times at each liquid superficial velocity and therefore RTD curves shown in Figure 7.3 are accurately measured. In addition, the liquid holdup measurements from this experimental work are in agreement with the measurements carried out using the gravimetric method. The accuracy is of utmost significance as RTD curves will be fitted using the experimentally determined liquid holdup values to calculate the axial dispersion coefficients. The details are provided in the next section.

Further RTD curves were obtained for increasing and decreasing arms of both dry bed and wet bed start-up conditions to cover each superficial liquid velocity. The particle size of the packed bed was also changed. In this work, a mobile-immobile model was used to fit the RTD curves in order to calculate the axial dispersion coefficients. A detailed description of the mobile-immobile model is given in section 2.9.2.2 (Chapter 2).

**7.3. NUMERICAL SOLUTION OF MOBILE-IMMOBILE MODEL**

Assuming the liquid dispersion occurs only in the mobile fraction of the liquid within the packed bed and mass transfer of the solutes occurs between the mobile and immobile
fractions, the equations which describe the mobile-immobile model can be written as follows:

\[
\theta_d \frac{\partial C_d}{\partial t} = \theta_d D_a \frac{\partial^2 C_d}{\partial z^2} - u \theta_d \frac{\partial C_d}{\partial z} - \alpha (C_d - C_s) \tag{7.4}
\]

\[
\theta_s \frac{\partial C_s}{\partial t} = \alpha (C_d - C_s) \tag{7.5}
\]

where \(C_d\) is the tracer concentration in the mobile liquid, \(C_s\) is the tracer concentration in the stagnant region, \(\theta_d\) is the mobile liquid holdup, \(\theta_s\) is the static liquid holdup, \(u\) is the liquid velocity in the mobile region (\(u = v_s/\theta_d\), where \(v_s\) is the superficial liquid velocity down the column), \(D_a\) is the axial dispersion coefficient and \(\alpha\) is the mass transfer coefficient between the dynamic and static zones.

In the present study, the above equations (i.e. equation 7.4 and 7.5) were solved numerically using the finite difference method. The central differences of classical forward-explicit scheme for space and time derivatives are expressed as follows, where \(i\) is index of the space, \(j\) is the index of the time and \(C\) is the concentration:

\[
\frac{\partial C}{\partial z} = \frac{C_{i+1}^j - C_{i-1}^j}{2\Delta z} \tag{7.6}
\]

\[
\frac{\partial^2 C}{\partial z^2} = \frac{C_{i+1}^j - 2C_i^j + C_{i-1}^j}{\Delta z^2} \tag{7.7}
\]

\[
\frac{\partial C}{\partial t} = \frac{C_{i}^{j+1} - C_i^j}{\Delta t} \tag{7.8}
\]

The discretised equations that describe the tracer concentration in the mobile zone and the immobile zone in the packed bed of length, \(Z\), and any time \(j\) can be written as follows.

\[
C_{d_i}^{j+1} = \left[-\frac{2D_a\Delta t}{\Delta z^2} - \frac{\alpha \Delta t}{\theta_d} + 1\right] C_{d_i}^j + \left[\frac{\theta_d u \Delta t}{\Delta z^2} - \frac{\alpha \Delta t}{\theta_d}\right] C_{d_{i+1}}^j + \left[\frac{\theta_d u \Delta t}{\Delta z^2} + \frac{\alpha \Delta t}{\theta_d}\right] C_{d_{i-1}}^j + \frac{\alpha \Delta t}{\theta_d} C_{s_i}^j \tag{7.9}
\]

\[
C_{s_i}^{j+1} = \left[1 - \frac{\Delta t \alpha}{\theta_s}\right] C_{s_i}^j + \frac{\Delta t \alpha}{\theta_s} C_{d_i}^j \tag{7.10}
\]

\[
t^{j+1} = t^j + \Delta t \tag{7.11}
\]

The following initial, top and bottom boundary conditions were used. Initially at all positions in the packed bed, the tracer concentration in the mobile liquid is zero:

\[
C_d(0,Z) = 0 \tag{7.12}
\]
Similarly, at all positions in the packed bed before the tracer is introduced, the tracer concentration in the immobile liquid is zero:

\[ C_i(0, z) = 0 \]  

(7.13)

A mass balance can be written over an elemental region at the top of the packed bed considering advective and diffusive terms into and from that elemental volume. It gives a top boundary condition known as the Danckwerts condition (Danckwerts, 1953):

\[ uC_d(t, 0^+) = uC_d(t, 0^-) - D_a \left( \frac{\partial C_d}{\partial z} \right)|_{z=\text{top}} \]  

(7.14)

At the bottom of the packed bed, the concentration gradient in mobile liquid is assumed to be zero (a free flow condition):

\[ \left( \frac{\partial C_d}{\partial z} \right)|_{z=\text{bottom}} = 0 \]  

(7.15)

The tracer pulse was introduced as a Dirac delta.

\[ C_d(t, 0) = \delta(t) \]  

(7.16)

For each set of experimental RTD curves, the axial dispersion coefficient \( D_a \), the mass transfer coefficient \( \alpha \) and the mobile liquid fraction \( \theta_d \) in the model was adjusted using a minimisation algorithm in order to achieve the best least squares fit between the model and the experimental data. The total liquid holdup, \( \theta = \theta_d + \theta_i \), and the liquid superficial velocity through the column, \( v_s \), were set using the experimental values.

RTD curves for each superficial velocity were fitted against the mobile-immobile model to determine the axial dispersion coefficient of the packed bed, as shown in Figure 7.4. It shows the experimental data for a 300 mm column packed with 18 mm glass beads at a superficial velocity of 0.015 mm/s. Strong agreement was obtained between the modelled and experimental residence time distribution when using the mobile-immobile model, with a \( R^2 \) value of greater than 0.999 for all flow rates. The dispersion coefficient is also shown in Figure 7.4 with other parameters of the specified experimental conditions.

Similarly, Figure 7.5 shows both experimental and fitted data from the mobile-immobile model at a superficial velocity of 0.03 mm/s for the same packed bed with 18 mm particles.
Chapter 7: Hydrodynamic Dispersion Analysis

Figure 7.4: The mobile-immobile model fitted to the experimental data of 18 mm particles at a superficial velocity of 0.015 mm/s in dry bed operation using a 300 mm column.

![Graph showing RTD](image1)

- $D_a = 3.14E-05 \text{ m}^2/\text{s}$
- $\alpha = 4.29E-06 \text{ s}^{-1}$
- $\theta_d = 0.0111$
- $\theta (\text{experimental}) = 0.0125$
- $v_s = 0.015 \text{ mm/s}$

Figure 7.5: The mobile-immobile model fitted to the experimental data of 18 mm particles at a superficial velocity of 0.03 mm/s in dry bed operation using a 300 mm column.

![Graph showing RTD](image2)

- $D_a = 5.85E-05 \text{ m}^2/\text{s}$
- $\alpha = 5.71E-06 \text{ s}^{-1}$
- $\theta_d = 0.0114$
- $\theta (\text{experimental}) = 0.0129$
- $v_s = 0.03 \text{ mm/s}$
The axial dispersion coefficient, mass transfer coefficient and the mobile mass fraction increase with the superficial liquid velocity of the packed bed. A detailed analysis of this will be given in the subsequent sections of this chapter. Axial dispersion coefficients were calculated similarly for each superficial velocity in increasing and decreasing arms of dry and wet bed start-up conditions and for each particle size of glass beads studied in this work.

7.4. ANALYSIS OF AXIAL DISPERSION COEFFICIENT

Generally, axial dispersion coefficient values resulting from tracer studies have been analysed as a function of superficial liquid velocity. Peclet numbers based on superficial liquid velocity have been plotted against the Reynolds numbers based on same velocity component in these studies (eg. Schwartz et al., 1976b; Fu and Tan, 1996; Saroha et al., 1998; de Andrade Lima, 2006). However, the objective of this study is to investigate the applicability of the superficial liquid velocity as the appropriate characteristic velocity for describing the axial dispersion.

During the discussion on liquid holdup results in Chapter 4, liquid holdup hysteresis was observed when liquid contents plotted against the superficial liquid velocity for the increasing and decreasing arms of the flow. Figure 7.6 shows dry bed and wet bed liquid holdup results corresponding to the salt tracer dispersion tests conducted in a 300 mm column packed with 18 mm glass beads.

It can be observed that the curves indicate substantial hysteresis in the dry bed operation compared to very little in the wet bed. In addition, it gives multiple liquid contents for a particular superficial flux. For example, if the packed bed operates at a superficial liquid velocity of 0.0075 mm/s (i.e. lowest superficial velocity in Figure 7.6), it has four distinct liquid holdup values. These were 0.01, 0.015, 0.025 and 0.026.

Liquid content determines the actual velocity of the liquid when it interacts with particles, whereas the superficial velocity is constant irrespective of the liquid holdup. As it is the velocity in the fluid in which dispersion is occurring, the velocity in the mobile liquid fraction will be tested as a characteristic velocity:

$$ u = \frac{v_s}{\theta_d} $$  \hspace{1cm} (7.17)
where $v_s$ is the superficial liquid velocity down the column and $\theta_d$ is the mobile liquid holdup.

Equation 7.17 can be used to calculate the liquid velocities in the mobile region. The mobile mass fractions are different in dry and wet bed start-up conditions as well as in the increasing and decreasing arms of the hysteresis curves. If a packed bed gives multiple liquid contents for each superficial flow velocity (Figure 7.6), it must have different liquid velocities in the mobile region for every liquid holdup value.

![Graph showing liquid holdup values measured gravimetrically during salt tracer tests](image)

**Figure 7.6:** Liquid holdup values measured gravimetrically during the salt tracer tests for the first cycle of operation in dry and wet bed start-up conditions using 18 mm particles in 300 mm column.

Figure 7.7 shows the mobile liquid velocity variation against the superficial liquid velocity based on the liquid holdup data in Figure 7.6. The existence of multiple actual velocities corresponding to each superficial velocity, depending on the operating condition of the system can be observed from this analysis. Every actual velocity of liquid has its own axial dispersion coefficient. The variation of that against liquid superficial velocity does not provide a tight relationship although it has been used by several researchers (eg. Schwartz et al., 1976b; Fu and Tan, 1996; Saroha et al., 1998). This can be observed from Figure 7.8a,
which shows axial dispersion coefficient for the 300 mm column packed with 18 mm glass beads. Figure 7.8a shows all the dispersion values for both dry and wet bed operation conditions including the increasing and decreasing arms of the flow. A greater variability of the axial dispersion values at each superficial velocity is observed and it is caused by multiple liquid velocities that correspond to each liquid flux.

The applicability of mobile liquid velocity can be investigated by plotting the same axial dispersion coefficients against their respective mobile velocities. This variation is shown in Figure 7.8b for the same sets of data shown in Figure 7.8a. The plot of axial dispersion values as a function of mobile liquid velocity gives a much tighter relationship, collapsing all the data onto the same curve. The small variations in Figure 7.8b can be assumed to be mainly due to the experimental variability of the system. Similarly, the variation of axial dispersion coefficients in a 300 mm column packed with 10 mm particles are plotted against both the superficial liquid velocity and the mobile region liquid velocity respectively. Figure 7.9a shows the dispersion coefficient against the superficial liquid velocity whereas the mobile region liquid velocity is the independent variable in Figure 7.9b.

**Figure 7.7:** Mobile liquid velocities at each superficial liquid velocity based on the liquid contents in Figure 7.6.

The applicability of mobile liquid velocity can be investigated by plotting the same axial dispersion coefficients against their respective mobile velocities. This variation is shown in Figure 7.8b for the same sets of data shown in Figure 7.8a. The plot of axial dispersion values as a function of mobile liquid velocity gives a much tighter relationship, collapsing all the data onto the same curve. The small variations in Figure 7.8b can be assumed to be mainly due to the experimental variability of the system. Similarly, the variation of axial dispersion coefficients in a 300 mm column packed with 10 mm particles are plotted against both the superficial liquid velocity and the mobile region liquid velocity respectively. Figure 7.9a shows the dispersion coefficient against the superficial liquid velocity whereas the mobile region liquid velocity is the independent variable in Figure 7.9b.
Chapter 7: Hydrodynamic Dispersion Analysis

Figure 7.8: Variation of axial dispersion coefficient in 300 mm column packed with 18 mm glass beads as a function of: a) superficial liquid velocity b) mobile region liquid velocity.
Figure 7.9: Variation of axial dispersion coefficient in 300 mm column packed with 10 mm glass beads as a function of: a) superficial liquid velocity b) mobile region liquid velocity.
This analysis clearly proves that the mobile region liquid velocity is the suitable characteristic velocity to characterise the axial dispersion coefficient in the packed bed, rather than the superficial liquid velocity.

### 7.5. MOBILE AND STATIC LIQUID FRACTION

The mobile liquid content is one of the fitting parameters in the mobile-immobile model and it is assumed that this mobile liquid content is where both solute convective and dispersive motion occur. The variation of this fitting parameter is shown in Figure 7.10a. It shows the mobile liquid holdup values as a function of superficial liquid velocity for both dry and wet bed start-up conditions.

In both start-up conditions the mobile liquid holdup increases with the superficial liquid flow velocity and the trend is consistent with the experimental results presented by de Andrade Lima (2006) for a leaching column with 2 mm particles. The results shown in Figure 7.10a indicate that the mobile liquid contents are higher in the decreasing arms of the flow. The higher overall liquid contents in the decreasing arm compared to the increasing arm can make additional flow paths through the particles and therefore the mobile liquid contents can also be higher.

In addition, Figure 7.10a implies that the mobile liquid holdup values for the wet bed operation are higher than those for the dry bed start-up conditions. This is mainly due to the higher total liquid holdup of the flooded bed. If the mobile liquid contents relative to the total liquid holdup are calculated (i.e. ratio of the mobile liquid holdup and the total liquid holdup), those are substantially higher in the dry bed start-up condition compared to the same in the wet bed start-up condition (see Figure 7.10b). The mobile liquid fractions for all the particle sizes in the dry bed start-up condition are in the range of 70-98% whereas the same is in the range of 40-80% for the packed bed which was started wet. Flooding the packed bed before the liquid addition introduced a significant number of relatively stationary liquid pockets in between the particles (see Figure 7.10b), which are less likely to contribute to the mass transport of the solutes.
Figure 7.10: Mobile liquid holdup (a) and mobile liquid fraction as a percentage (b), in 300 mm column packed with 14 mm glass beads.
Figure 7.10b also indicates that the mobile liquid fractions in the decreasing arm are slightly higher than the increasing arm. This is consistent with the explanation of the higher mobile liquid holdup values in the decreasing arm of the flow compared with the increasing arm of the flow (see Figure 7.10a).

The experimentally determined total liquid holdup value ($\theta$) and the fitted mobile liquid content ($\theta_d$) can be used to calculate the corresponding static liquid holdup ($\theta_s$):

$$\theta_s = \theta - \theta_d \quad (7.18)$$

The calculated static liquid holdup values using equation 7.18 are presented in Figure 7.11 for both the dry bed and wet bed start-up conditions. These imply that the superficial liquid velocity was a small influence on the static or immobile liquid content. A similar trend was observed in the relationship between the static saturation and the Reynolds number based on the superficial liquid velocity presented by de Andrade Lima (2006). The method of startup does have an impact on the static liquid fraction.

![Figure 7.11: Static liquid holdup variation for both dry and wet bed start-up conditions in 300 mm column packed with 14 mm glass beads.](image-url)
7.5.1. STATIC VERSUS RESIDUAL LIQUID HOLDUP

Many researchers have made the assumption that the immobile and the residual liquid content are the same thing, but this is not necessarily the case. The fact that a residual liquid pocket remains once the liquid has drained does not mean that it was not involved in the flow.

Even the idea that there are two distinct flow classes is a big simplification, since the reality is that there will be a range of velocities, the slower of which are assumed to be stationary and the faster of which are given a single representative velocity.

This means that the velocities that can be considered stationary will depend upon the effect that velocity has on the system. For instance the velocities, and thus region of the fluid, where dispersion is strong may not be exactly the same as the regions in which viscous losses are large. Figure 7.12 suggests that this is indeed the case, since using the residual as the stationary fraction was good approximation for the fluid flow, but for dispersion the stationary fraction is lower than the residual indicating that more of the fluid is involved in dispersion than in viscous losses.

Figure 7.12: Relationship between the residual and static liquid contents of 18 mm particles in 300 mm column for both dry and wet bed start-up conditions.
7.6. MASS TRANSFER COEFFICIENT

The mass transfer coefficient ($\alpha$ in equations 7.4 and 7.5) is another fitting parameter in the mobile-immobile model and it describes the rate of mass exchange between the mobile and immobile regions. Figure 7.13 shows the variation for the mass transfer coefficient for both dry and wet bed start-up conditions against the superficial liquid velocity. The values increase with the superficial liquid velocity. de Andrade Lima (2006) presented the fitted mass transfer coefficients using the same model (however the model equations are slightly different with a small error in their formulation) and found a similar trend with the superficial liquid velocity.

In addition, it can be seen that the mass transfer coefficients are higher when the packed bed was started wet, compared with those in the dry bed start-up condition (see Figure 7.13a and 7.13b). The mobile-immobile model (i.e. equations 7.4 and 7.5) accounts for the static liquid fraction in order to determine the total mass transfer and it was found that the static liquid pockets, which are not involved in the mass transfer, are substantially higher in the flooded bed. To balance this static liquid volume in the model, it should give higher mass transfer coefficients and this might be a reason for the higher mass transfer coefficients in the wet bed.
Figure 7.13: Mass transfer coefficient between the mobile and immobile regions as a function of superficial liquid velocity a) 14 mm b) 10 mm glass beads in 300 mm column.
7.7. SUMMARY
Residence time distribution analysis is used extensively in the experimental studies of porous media in order to determine the dispersion coefficients. Despite the number of studies that have been conducted on the dispersion coefficients, there is still debate as to the mechanisms at work. Much of this debate is due to the fact that the details of the flow behaviour usually need to be inferred from dispersion measurements obtained by measuring the spread of a soluble tracer such as a salt or a dye. The main reason for using this method is that the technique is both simple to carry out and the results are easy to analyse (Bouffard and Dixon, 2001; Bouffard and West-Sells, 2009).

The objective of some of the RTD studies based on salt tracers was to calculate the liquid holdup in the packed bed system. Using this technique, however, there is no way to investigate the accuracy of the effluent tracer detection methodology. As this study measures the liquid holdup in the system accurately using the gravimetric method, the resultant liquid holdup values could be used to determine the mean residence time of the system. In addition, the experimental RTD curves provide the mean residence times independently. It was proved that both methods give very similar mean residence times and this implies that the effluent salt tracer detection system used in this study is accurate.

During the analysis of the experimental RTD curves, a mobile-immobile model was used to fit the curves in order to calculate the dispersion values. This model implies a bi-dispersed liquid flow velocity compared to the uniform liquid flow velocity assumed by the advection-dispersion model. A good fit of the experimental data is observed and, unlike similar experimental studies that have used the mobile-immobile model, the liquid holdup is not a fitting parameter (eg. Decker, 1996; de Andrade Lima, 2006). As the liquid contents were experimental rather than fitted by the model, it provides better accuracy on the calculated axial dispersion coefficients.

When the axial dispersion values are plotted against the superficial liquid velocity, variability in the axial dispersion coefficient can be observed for the same packed bed with the same particle size during the increasing and decreasing arms of dry and wet bed start-up conditions. This is explained as the existence of multiple average liquid flow velocities resulted from multiple liquid contents of the packed bed due the presence of liquid holdup hysteresis even though the superficial liquid velocity is identical. The mobile region liquid
velocities are calculated using the mobile-immobile model and are used as the characteristic liquid flow velocities in the system. All the axial dispersion coefficients can be collapsed onto a single curve using the mobile region liquid velocity. This implies that the superficial liquid velocity is not the appropriate velocity to characterise the axial dispersion values despite most previous investigators have used it (eg. Mears, 1971; Schwartz et al., 1976b; Villanueva et al., 1990; Decker, 1996; Fu and Tan, 1996; Saroha et al., 1998; de Andrade Lima, 2006). This study concludes that the average velocity in the mobile liquid should be used to characterise the axial dispersion coefficients as there is liquid holdup hysteresis in these systems and thus there is no simple relationship between different velocities.

The mobile liquid content and the mass transfer coefficients were the other fitting parameters in the mobile-immobile model as the total liquid holdup was determined experimentally. It was found that the mobile liquid content increases with the superficial liquid velocity whereas the immobile liquid content has little dependency on the liquid velocity. Further, the mobile liquid fraction in the bed is higher for the dry bed start-up condition compared with the bed which was flooded initially. This was mainly due to the substantially higher immobile liquid pockets in the wet bed condition (Figure 7.10b). Due to the same reason the mass transfer coefficients in the wet bed are higher compared with the dry bed start-up condition.
CHAPTER 8

POSITRON EMISSION PARTICLE TRACKING (PEPT) IN PACKED BEDS

8.1. INTRODUCTION
Positron emission particle tracking (PEPT) is a technique to measure particle motion in opaque and multiphase systems. A neutrally buoyant hydrophilic solid tracer particle with sufficient radioactivity can be used to map out the rivulet flow behaviour in column leaching. The tracer particle should be small enough to travel through the inter-particle spaces in the packed bed.

The residence time distribution analysis described in Chapter 7 used salt tracers to determine the axial dispersion coefficient in the packed bed. By using a bed composed of solid particles in the size range employed in industrial heap leaching, PEPT can also be used to calculate the axial dispersion coefficient. The objective of this study is to compare dispersion values measured with salt tracers and with PEPT using the same packed bed and at the same time observe the details of the flow path.

This chapter will describe the experimental method used to measure the tracer particle motion in the rivulets within the packed bed. The tracer coordinates were continuously tracked using a triangulation algorithm in order to obtain their trajectories. Furthermore, a time weighting function was used to remove high frequency noise associated with the original PEPT data. In the results section, the axial dispersion values were determined using smoothed PEPT data and compared with the axial dispersion coefficients found using the salt tracer tests in Chapter 7. The radial dispersion results will also be presented as the PEPT
technique provided the horizontal displacements of the tracer particle required to calculate this.

8.2. POSITRON EMISSION PARTICLE TRACKING (PEPT)

The following sections describe the basics of the PEPT technique and its applications in various experimental systems.

8.2.1. BASICS OF PEPT

Tracer particles must be labelled with a positron emitting radionuclide such as $^{18}$F (half-life, $t_{1/2} = 109$ minutes), $^{22}$Na ($t_{1/2} = 2.6$ years), $^{61}$Cu ($t_{1/2} = 204$ minutes), $^{64}$Cu ($t_{1/2} = 162$ minutes), $^{66}$Ga ($t_{1/2} = 9.45$ hours) and are often composed of ion exchange resins impregnated with an appropriate radionuclide (Fan et al., 2006a, 2006b). A detailed description of the tracer fabrication and labelling techniques is given by Fan et al. (2006a, 2006b). Tracers can be made neutrally buoyant by adjusting the density of the impregnated solution, to use the particles to represent fluid or bulk flow (Fangary et al., 2000; Pianko-Oprych et al., 2009). Recently, Cole et al. (2012) labelled chelating resins with the radioisotope $^{68}$Ga ($t_{1/2} = 68$ minutes) to make tracers as small as 50 µm.

The radioisotope emits positrons and they annihilate with local electrons. The annihilation event forms pairs of 511 keV gamma rays released almost back-to-back. If both gamma rays are detected, their path forms a line of response (LoR) and the basis of the PEPT technique is the simultaneous detection of the pair of gamma rays using the positron camera. The location of the tracer particle must be situated somewhere along the LoR and, theoretically, two intersecting LoRs are enough to locate the particle.

Typically, the location of the tracer particle is determined by the intersection of multiple lines of response in order to reduce or eliminate the effect of corrupted events. The corrupted events record when the camera detects the gamma rays after they have undergone Compton scattering or due to the coincident detection of two gamma rays not belonging to the same annihilation event. Figure 8.1 illustrates the detected LoRs with time during the tracer particle trajectory and it can be seen that corrupted events are also recorded at each position when it moves.

After removing the corrupted events the tracer triangulation algorithm generates the time sequential particle trajectory through a medium (Parker et al., 1993; Parker et al., 2002).
The iterative triangulation algorithm has two main parameters to determine the location of the tracer particle. These are the slice size \((N)\) and the final fraction of detected events \(f\). The slice size is the number of LoRs used in determining each location and the final number of LoRs in the location after iteration is termed as the final fraction of detected events or \(f\). With these two parameters, the triangulation algorithm locates the position of the particle which minimises the location error.

**Figure 8.1:** Detected line of responses (LoRs) of the tracer particle and the corrupted events with PEPT (Cole, 2011).

Figure 8.2 illustrates the location of the tracer particle using a given slice size and final fraction of detected events and it can be seen that the corrupted events have been removed in that particular slice of the detected LoRs. A detailed description of the PEPT location algorithm can be found in Parker et al. (1993).

Generally, the PEPT technique is only capable of detecting a single tracer at a time (Yang et al., 2007). However, multiple particles of distinct radioactivity can be tracked with a technique called Multiple-PEPT which was developed by Yang et al. (2006). Yang et al. (2007) gave a detailed study about Multiple-PEPT including its application in a gas-solid fluidized bed. Bickell et al. (2012) have just published a paper on a new way to locate the
tracer particle, using the spatial density of LoRs in coincidence. It can track lots of particles, unless they collide.

![Figure 8.2: Final position of the tracer particle using the triangulation algorithm (Cole, 2011).](image)

The position of the tracer particle can be located accurately up to 1000 times per second for tracer velocities up to 0.5 m/s with PEPT, but the accuracy of the technique decreases as the measured velocities increases (Bakalis et al., 2004). The new positron camera at Birmingham, the ADAC Forte, can be used to locate a slow moving tracer to within 100 μm, 50 times/s and a fast moving tracer (at 1 m/s) to within 0.5 mm, 250 times/s (Parker et al., 2002). Parker et al. (2002) determined the root mean square (r.m.s) location error of a PEPT measurement of a tracer particle rotating on a turntable at a speed of 1.4 m/s. By comparing PEPT trajectory to visual observation of the tracer rotation, the r.m.s of the deviation of the PEPT location data from the true path of the tracer was calculated as 0.6 mm. Recently, Volkwyn et al. (2010) reported the statistical uncertainties associated with different kinematic PEPT measurements at the PEPT centre in Cape Town.

8.2.2. APPLICATIONS OF PEPT

In a solid-liquid suspension, some other flow field velocity measurement techniques are laser doppler anemometry (LDA), particle imaging velocimetry (PIV) and magnetic resonance imaging (MRI). Both LDA and PIV techniques require optical access to the system while an experimental system containing certain types of metal such as steel, cannot to be used with MRI. Moreover, PEPT can provide detailed information on the fluid motion in opaque systems to determine realistic velocity distributions (Bakalis et al., 2004). There are numerous applications of PEPT to obtain flow fields in liquids, organic polymers and
granular materials such as metallic and mineral particles (Parker and Fan, 2008). The current developments can track 1 to 3 tracer particles simultaneously to provide the time averaged flow pattern, but not the instantaneous dynamic flow behaviour (Parker and Fan, 2008).


Chan et al. (2009) applied PEPT technique to study the real time solid particle motion in standpipes used in circulating fluidized beds (CFB). Guida et al. (2009) investigated the particle and fluid trajectories and the two phase flow field with PEPT in a mechanically agitated solid-liquid suspension.

Hoff et al. (1996) used liquid tracers for mapping unsaturated water flow within porous clay bricks. The experimental results were used to obtain the absorption profiles for advance of a wetting front into a clay brick. Richard’s equation in a horizontal direction was used to describe liquid flow using PET data and then to determine the hydraulic diffusivity of the material.

Waters et al. (2008a, 2008b) applied the technique in froth flotation by studying dynamic behaviour in a flotation vessel. Then, Cole et al. (2010a) and Cole et al. (2010b) used both positron emission particle tracking and digital image analysis to describe particle motion in froths. Buffler et al. (2010) presented the trajectories associated with the rotating rock charge in a tumbling mill. The occupancy plot of the tracer particle was used to characterise the charge motion inside the tumbling mill.

**8.3. PEPT TRAJECTORIES IN PACKED BEDS OF PARTICLES**

Although PEPT can be used to trace particle motion in opaque and multiphase systems, the application of the technique in minerals processing has been limited to froth flotation and comminution. The present study investigates the applicability of PEPT in column leaching. This study is the first PEPT based study on column leaching according to the best of knowledge of the author.

PEPT tracer location data in a packed bed can be used to calculate both the axial and radial dispersion coefficients. The objective of this work is to compare axial dispersion values
measured with PEPT and salt tracers using the same packed bed. The main difference between the dispersion coefficients based on salt tracers and those from PEPT is that the particle tracers used in PEPT do not experience molecular diffusion. Therefore this comparison separates the effects of inter rivulet mixing (i.e. hydrodynamic effects) on dispersion from the influence of molecular diffusion. Radial dispersion coefficients given by PEPT cannot be compared, as residence time distribution experiments using a salt tracer only provide axial dispersion values.

A tracer of average size of 400 µm was tracked with PEPT through the 18 mm glass beads of the packed bed and a time weighting function was applied to the location data given by PEPT to interpolate and smooth the data. Finally, the smoothed location data was used to determine the dispersion coefficients of the packed bed.

8.4. EXPERIMENTAL METHOD FOR PEPT

PEPT experiments were performed at the University of Cape Town PEPT facility located at iThemba Labs, South Africa (see Figure 8.3).

![PEPT Gantry](image)

**Figure 8.3**: PEPT camera located in iThemba Labs, South Africa. The 300 mm column packed with 18 mm particles is shown inside the positron camera.
A cylindrical Perspex column was used to perform PEPT measurements. The column was the same column which was used to measure liquid holdups and for carrying out the salt tracer tests in this research. The internal diameter and height of the column were 243 mm and 300 mm respectively.

The bottom plate described in the section 3.2.1.2 was not used due to limited clearance (580 mm) inside the PEPT gantry (see Figure 8.3). A 325 mm diameter metal sieve with 9.7 mm aperture size was used as an alternative bottom plate as shown in Figure 8.4. Due to the limited space it was also not possible to suspend the column from a load cell. This meant that simultaneous PEPT and liquid holdup measurement were not possible. It assumed that the liquid flow behaviour was the same as in earlier experiments (i.e. in Chapter 4).

**Figure 8.4:** Empty 300 mm Perspex column with bottom plate inside the PEPT gantry.

Figure 8.4 shows the empty Perspex column with the bottom plate attached, mounted inside the active area of the positron camera. The empty column was fixed approximately
centrally inside the active area to maintain the same position of the packed bed in every experiment. Figure 8.5 shows a schematic representation of the coordinate system relative to the Perspex column. This coordinate system was maintained throughout this Chapter when illustrating and analysing the tracer particle trajectories.

Figure 8.5: Schematic representation of the PEPT coordinate system used in this work relative to the empty Perspex column inside the PEPT gantry.
At the bottom of the column a plastic collection tray was placed in order to collect the liquid from the packed column and also to retain the tracer particle which leaves the packed bed after completing its trajectory (see Figure 8.4).

The interior of the column was mapped with a sealed radioactive source, to provide a frame of reference for the tracer trajectories through the packed bed. The sealed source contained $^{22}\text{Na}$ ($t_{1/2} = 2.6$ years) was in a vial of 10 mm diameter and 8 mm long. For the mapping, the sealed source was kept in five locations inside the column and 60 seconds of list mode data was recorded with PEPT in each location. The locations were bottom left, bottom right, top left, top right and bottom centre of the empty packed bed.

These measured points were used to generate a 3-D image of the empty column. Figure 8.6 shows the five locations and trajectories of the sealed source of $^{22}\text{Na}$ between these locations. Especially at the bottom of the column, the sealed source of $^{22}\text{Na}$ does not indicate the true inner edge of the empty column as the sealed source was not a point source. The dimensions of the vial create a small gap between the location of the tracer particle and the column (see Figure 8.6).

18 mm glass beads were used as the packing material to prepare a randomly packed mono-dispersed bed and then the liquid distributor was placed on the top of the bed (Figure 8.7). A space of 10-15 mm (see Figure 8.7) was maintained between the liquid distributor and the packed bed so that the tracer particle could be introduced.

The novel liquid distributor of 32 drip points was used to distribute liquid onto the packed bed. A detailed description of the design of the liquid distributor is given in the section 3.2.3 (Chapter 3). The liquid was deionised water for all the PEPT experiments and the same flow rates used in the liquid holdup experiments and the salt tracer tests were also used. Thus, the investigated flow rates were 1.26, 2.52, 5.04, 10.08 and 20.16 L/h and they correspond to the superficial flow within the range of 0.0075-0.12 mm/s.
Figure 8.6: Empty column (300 mm) mapping inside the active area of the positron camera.

Figure 8.7: The 300 mm packed bed with 18 mm particles and mounted liquid distributor inside the PEPT gantry.
Ion-exchange techniques described in Cole et al. (2012) were used to label resin particles (Purolite NRW-100) which were hydrophilic and composed of a polystyrene divinylbenzene matrix with a specific gravity of around 1.1. The radionuclide $^{68}$Ga ($t_{1/2} = 68$ minutes) was adsorbed from solution onto the resin surface and then sealed with two layers of superglue to prevent leaching. The initial activities of the tracer particles were in the range of 50 to 100 µCi. Different tracer sizes were used, ranging from 350 – 450 µm with an average size of about 400 µm. Figure 8.8 shows the images of the tracer particle viewed with a macro lens system.

Figure 8.8: Images of the tracer particle. The top image shows the scale of the tracer particle.
Chapter 8: Positron Emission Particle Tracking (PEPT) in Packed Beds

Liquid was introduced into the packed bed at a particular flow rate for 1 hour until it reached steady state. Once the packed bed achieved steady state, the tracer particle was introduced into the column carefully using a paint brush at the bottom of a randomly selected drip point.

The location data of the tracer particle was determined with time until it left the packed bed at the bottom of the column. However, the main limitation of this technique is that the tracer particle tended to get stuck between the glass beads of the bed especially at very low superficial velocities ranging from 0.0075-0.03 mm/s, which correspond to low liquid contents and thus narrow rivulets.

8.5. PEPT DATA ANALYSIS

The list mode data was triangulated with the location algorithm developed by Parker et al. (1993) with slice size, $N$, of 250 and the final fraction of detected events, $f$, of 0.15. This creates the tracer trajectory inside the packed bed by removing corrupt events such as from Compton scattering (Parker et al., 1993). With low $N$ and $f$ values, the location algorithm has a lot of locations, but the location error of the tracer particle is high. The location error can be reduced by increasing the number of line of responses (LoRs) in each location calculation by increasing the $N$ and $f$ values at the expense of the location frequency. Hence, the selection of $N$ and $f$ should be a compromise to minimize the location error of the tracer particle while maximising the location frequency. The optimisation of the tracer location by minimising the location error is a continuing research area at PEPT Cape town.

The triangulated PEPT data contains a sequence of $x$, $y$ and $z$ Cartesian coordinates of positions in millimetres as a function of time in milliseconds. The artificial contribution of the measurement of noise is expected with the triangulated PEPT data (Parker et al., 1993). This noise will be due to a combination of errors in the triangulation and the finite volume in which the gamma rays are being created. The effect of the high frequency noise can be investigated by analysing the triangulated coordinates of the tracer particle when it was relatively stationary. For instance, when the tracer particle in the bottom collection tray after leaving the packed bed. In this case, the average position of the particle should be constant as the particle was virtually stationary. Figure 8.9 shows the deviations of $y$ and $z$ coordinates of the tracer particle from its average $y$ and $z$ coordinates for 10 seconds respectively.
Figure 8.9: High frequency noise measurements when the tracer particle was stationary in the collection tray. The standard deviations of the y and z data are 850 µm and 450 µm respectively.

This clearly shows that the presence of high frequency noise in the original triangulated PEPT data. The frequency of PEPT data in Figure 8.9 is relatively higher compared with the
tracer particle within the packed bed. The tracer particle is much closer to the detector elements of the positron camera and thus it emits gamma rays more frequently and also there was no surrounding media (i.e. glass beads) around the tracer particle.

Therefore, smoothing of the triangulated PEPT data was performed and the details of the interpolation and smoothing of the tracer trajectory will be described in the following section.

### 8.5.1. TIME WEIGHTING FUNCTION

Noise and effects associated with axes of lower sensitivity in the positron camera can be removed with a weighting function to interpolate and smooth the recorded PEPT data as suggested by Cole et al. (2010a). A cubic spline weighting function is used as it is continuous and smooth, has a near Gaussian shape, but goes to zero at a finite distance (i.e. has compact support). This makes the computation efficient as the weighting function is only non-zero in the vicinity of the time being considered. The weighting function was developed with a varying kernel width and it is shown in Figure 8.10.

![Figure 8.10: The kernel for a weighting function composed of cubic splines for interpolating and smoothing the original PEPT data (Cole, 2010c: p. 119).](image)

The piecewise polynomial of the weighting function can be mathematically represented as follows.
\[ q_i = \frac{|t-t_i|}{h'} \]  

where \( t \) is the time, \( t_i \) is the time of a PEPT coordinate and \( h' \) is half the kernel width.

If \( (q_i < 0.5) \), \[ W_i = 1 - 6q_i^2 + 6q_i^3 \]  

If \( (0.5 < q_i < 1) \), \[ W_i = 2(1-q_i)^3 \]

else \( W_i = 0 \)

For each original PEPT coordinate of \( A_i(x_i, y_i, z_i) \) at time \( t_i \) within the kernel width, the final PEPT coordinate, \( A(x,y,z) \) is calculated using equation 8.5.

\[ A(t) = \frac{\sum_{i=0}^{\infty} A_i(t_i)W_i}{\sum_{i=0}^{\infty} W_i} \]  

It indicates that the time weighting function calculates the final PEPT coordinate at time \( t \) considering not only the influence of the original PEPT coordinate at time \( t \) but also considering the influence of neighbouring coordinates within the kernel width. PEPT coordinates beyond the kernel width have no importance. Hence, the calculation of each final PEPT coordinate is based on the data points within a particular kernel width and not based on the data points of the entire time interval of the original PEPT data (Cole, 2010c).

The time weighting function both interpolates and smoothes the original PEPT data and the kernel width must be long enough to have a number of data points within it to be averaged (Cole, 2010c). Further, the selected kernel width should minimise the high frequency noise without losing the actual location data. Cole (2010c) investigated the optimum kernel width for both vertical and horizontal directions. The measured location data of the system with PEPT was verified by image analysis of the tracer particle trajectory. The optimum kernel width was found to be 200 milliseconds for both vertical and horizontal directions at a 95% confidence interval, to locate the tracer particle accurately by using the two independent measurement techniques.

However, in this study the tracer location could not be measured with other techniques, such as image analysis, as the introduced tracer particle was surrounded by the glass beads of the packed bed. As both studies are based on the movement of similar tracer particles measured with PEPT, the kernel width was assumed to be 200 milliseconds to remove the high frequency noise in this study as well. The selection of the kernel width should be a...
compromise between noise removal and accuracy of the location data, especially where the velocity is changing rapidly.

The time weighting function with 200 milliseconds kernel width was applied on the same data plotted in Figure 8.9 to remove high frequency noise measurements. Figure 8.11 shows the deviations of the smoothed tracer particle locations in $y$ and $z$ directions from the average tracer particle position for $y$ and $z$ directions respectively together with the original location data without smoothing. It can be seen that the time weighting function has removed a lot of the high frequency noise. The standard deviation of the smoothed data is approximately 200-350 µm compared to about 850 µm for the unsmoothed data. As the tracer particle is about 400 µm, the error is thus comparable to the size of the tracer.

Figure 8.12 shows the original and smoothed PEPT data for a period of 4.5 seconds when the tracer particle was moving in the packed bed. The graphs on the left hand side of Figure 8.12 shows the original PEPT data for the $y$, $x$ and $z$ directions respectively whereas the smoothed data using a kernel width of 200 milliseconds can be seen in the graphs on the right hand side. The smoothed PEPT data indicates the removal of small amplitude, high frequency noise associated with the original locations.
Figure 8.11: High frequency noise measurements and the smoothed coordinates when the tracer particle was stationary in the collection tray (black line illustrates the deviations from the average tracer particle position for the smoothed PEPT data). The standard deviations of the smoothed $y$ and $z$ data are 360 µm and 200 µm respectively.
Figure 8.12: Graphs of the $y$, $x$, $z$ positions of the tracer particle measured with PEPT as a function of time during a period of 4.5 seconds (only a part of a trajectory to show noise removal). The top graph is the vertical ($y$) direction and the middle and the bottom graphs are horizontal $x$ and $z$ directions respectively. The original data is shown on left and the smoothed data with a time weighting kernel width of 200 milliseconds is on the right.
8.5.2. SMOOTHED PEPT TRAJECTORY
Smoothed PEPT location data will be used in the subsequent analysis of tracer trajectory and dispersion coefficients. Figure 8.13 shows the total tracer trajectory determined by PEPT, where the tracer particle descended from the top to the bottom of the column.

Figure 8.13: The PEPT tracer trajectories of the tracer particles through a 300 mm column packed with 18 mm glass beads (blue-450 μm, red-420 μm, magenta-380 μm, black-370 μm).
The motion consists of periods of relatively rapid descent interspersed with periods where there is little downward movement.

**8.6. DISPERSION COEFFICIENTS FROM THE SMOOTHED PEPT DATA**

For each superficial velocity, tracer locations from the smoothed tracer trajectory in the \( y \)-direction (downwards through the axis of the packed bed) were used to calculate the axial dispersion coefficient \((D_a)\). The tracer displacements \((\Delta y)\) were calculated across different sets of time intervals. The axial dispersion coefficient in the \( y \)-direction, which corresponds to bulk flow direction, is the ratio of the variance of the displacement and the time interval \((\Delta t)\) of the displacement in the \( y \)-direction downwards:

\[
D_a = \frac{\sum (\Delta y - \overline{\Delta y})^2}{(n' - 1) \cdot \Delta t}
\]

where \(\overline{\Delta y}\) is the mean tracer displacement in the \( y \)-direction and \(n'\) is the sample size.

The effect of the selected time base on the calculated dispersion coefficient was investigated and Figure 8.14 shows the results of this analysis.

![Figure 8.14: The effect of the time base on the calculated axial dispersion coefficient.](image-url)
The selection of the time base of 500 milliseconds can be justified as over the time interval of 200-500 milliseconds, the axial dispersion coefficient is uniform. When measurement error is not a factor and assuming that dispersion is the result of a random walk, the time base chosen should not influence the calculated dispersion coefficient as long as there is sufficient data. The calculated axial dispersion coefficient increases slightly at the lower time bases, but this is due to the influence of high frequency noise in the measured particle locations rather than real dispersion behaviour.

Unlike the salt tracer tests, which can only give the axial dispersion coefficients in these column experiments, the PEPT data can also be used to estimate the radial dispersion coefficient. The tracer displacements in the horizontal direction were measured over the same 500 milliseconds time intervals as used to calculate the axial dispersion coefficient. The variances of both horizontal displacements were combined to calculate the radial dispersion coefficient.

At the low end of the superficial liquid flow rates that covered in this experimental work (i.e. 0.0075-0.03 mm/s) the tracer particle tended to get stuck in between the particles. However at moderate superficial liquid velocities of 0.06 and 0.12 mm/s, the tracer particles passed completely through the column providing full 300 mm trajectories from top to the bottom of the packed bed.

The locations of the tracer particle using the triangulated and smoothed PEPT data are shown in Figure 8.15 and it shows y position as a function of time. Figure 8.15 also shows the coordinates corresponding to the tracer introduction, the tracer leaving the packed bed and the stationary tracer particle in the bottom liquid tray. The tracer particle became stuck within the bed during the time intervals from A to B and from C to D.

The exact mechanisms responsible for the tracer particle gets stuck within the packed bed are not known as there were not any visualisation techniques available at the Cape Town PEPT centre (eg. Live imaging system to track the tracer particle). The tracer particle can gets stick with the glass beads of the packed bed and also it can be trapped into stagnant zones while it descents. The future studies with liquid tracers rather than solid particle tracers will be employed to determine the fractions of dynamic and static liquid and thus, these can be used address the possible tracer particle mechanisms more closely.
When the tracer got stuck between particles in the packed bed (for instance over the intervals from A to B and from C to D in Figure 8.15, after the smoothing and interpolation), the stationary location data of the tracer particle were excluded prior to calculating the dispersion coefficient (see Figure 8.16).

The resultant axial and radial dispersion values are plotted as a function of superficial liquid velocity and are shown in Figure 8.17 for the moderate flow rates covered in this work. These experiments show that the radial dispersion coefficient is approximately a factor of four times smaller than the axial dispersion coefficient.

![Graph showing the y coordinates of the tracer descent within the packed bed. Superficial velocity of the liquid is 0.12 mm/s.](image)

**Figure 8.15:** Graph showing the $y$ coordinates of the tracer descent within the packed bed. **Superficial velocity of the liquid is 0.12 mm/s.**
Chapter 8: Positron Emission Particle Tracking (PEPT) in Packed Beds

Figure 8.16: Smoothed mobile tracer trajectories in y-direction downwards through the 18 mm glass beads in 300 mm column after removing the stationary location data.

Figure 8.17: Variation of the axial and radial dispersion coefficients for 18 mm glass beads in 300 mm column based on the PEPT location data of the tracer particle.
8.6.1. COMPARISON OF AXIAL DISPERSION WITH SALT TRACER METHOD

As stationary tracer locations were removed from the smoothed PEPT trajectory at each superficial velocity, the resultant axial dispersion coefficient was calculated from the mobile tracer descent. The determination of the axial dispersion coefficient using salt tracer tests for the same packed bed was explained and presented in Chapter 7. Both the measurement techniques are independent of each other and the resultant axial dispersion coefficients can be compared in order to investigate the agreement of the methods.

Figure 8.18 compares PEPT based and salt tracer based axial dispersion coefficient values for a 300 mm packed bed containing 18 mm glass beads. At moderate flow rates, the two independent techniques are in good agreement. At lower flow rates, it was not possible to obtain dispersion coefficients from PEPT as the 400 µm tracer particles tended to get stuck in the bed. This is probably caused by the rivulets becoming comparable in size to the tracer particles. As the PEPT tracer particle technology is continuously improving, future experiments will be conducted using smaller tracer particles or liquid tracers.

![Figure 8.18: Comparison of the axial dispersion coefficients for 18 mm glass beads in 300 mm column calculated using the RTD and PEPT methods. Figure also shows radial dispersion coefficients with PEPT.](image-url)
Figure 8.1 also shows the radial dispersion coefficients calculated from the same PEPT data used to calculate the axial dispersion coefficients. It is not possible to compare the radial dispersion values as the salt tracer tests in a packed bed only provide axial dispersion coefficients.

8.7. SUMMARY

PEPT is a technique for measuring internal flow behaviour in multiphase systems. It is of particular importance for mixing and separation processes, where particle and fluid behaviour cannot be observed optically. PEPT was used to measure the trajectories of neutrally buoyant tracer particles as they moved through an unsaturated packed bed. From the motion of these tracers, dispersion coefficients were obtained. In this work with PEPT, an experimental methodology was developed to locate the tracer particles as they moved through the glass beads in an unsaturated packed bed. The packed bed used consisted of 18 mm beads representing the relevant size range of particles found in column and heap leaching heaps.

The resin particles with an average size of 400 µm were prepared by adsorption of radionuclide $^{68}$Ga. The half-life of the radionuclide, which is 68 minutes, was long enough to carry out a few tracer trajectory experiments and also short enough to permanently dispose after the experimental run. The list mode data from the Cape Town positron camera were triangulated according to the triangulation algorithm developed by Parker et al. (1993). However, the resultant PEPT tracer trajectories with any positron camera are influenced by measurement noise (Parker et al., 1993). Therefore, a weighting function was applied on the original triangulated PEPT location data to perform interpolation and smoothing in order to remove the high frequency noise, as was originally proposed by Cole et al. (2010a).

It was proved that the time weighting function of a kernel width of 200 milliseconds removes most of the high frequency noise by comparing the original and smoothed data. The main objective of these PEPT experiments was to determine the dispersion coefficients for a packed bed with unsaturated liquid flow behaviour. Therefore, the ratio of the variance of the displacement and the time interval of the displacement in the relevant direction (i.e. in parallel or perpendicular direction with the axis of the packed bed) was used to determine both the axial and radial dispersion coefficients using the smoothed PEPT location data. The effect of the time base on the calculated dispersion coefficient was
investigated and found to be insignificant over the time interval of 200-500 milliseconds. However, the influence of noise rather than actual dispersion behaviour is greater at lower time bases; therefore, a 500 milliseconds time base was selected to calculate the dispersion coefficients.

RTD of a salt tracer test was used to provide an independent measure of the dispersion coefficients. It was shown that the dispersion coefficient obtained from this PEPT location data was in excellent agreement with that obtained from salt tracer experiments, which were described in Chapter 7. The PEPT experiments have the added advantage of allowing the radial dispersion coefficients to be simultaneously measured; however, it is not possible to compare them with salt tracer tests as these only provide the axial dispersion coefficients. The main current limitation with the technique is that the 400 µm tracer particles used in this work tend to get stuck within the bed at lower flow rates. In future experiments, the use of actual ore particles, smaller tracer particles and PET (liquid tracer) experiments will reveal more about the underlying flow mechanics in column and heap leaching systems.
Chapter 9: Conclusions and Future Work

CHAPTER 9

CONCLUSIONS AND FUTURE WORK

9.1. CONCLUSIONS
This work developed a novel experimental methodology to derive an inter-particle flow model that describes the fluid flow behaviour in packed beds, column leaching and heap leaching heaps. The developed model incorporates the liquid holdup hysteresis behaviour that can be observed in the packed bed and column leaching systems. In addition, the 1-D column leaching rig can be used to measure hydrodynamic dispersion by using salt tracer residence time distribution analysis and by tracking tracer particle motion with PEPT.

9.1.1. LIQUID HOLDUP HYSTERESIS
If was found that the liquid holdup of the model system depends not only on the current input conditions but also on the history of the system. This means that for the same superficial liquid velocity and particle size, multiple liquid contents are possible especially in the dry bed start-up condition. Liquid holdup hysteresis behaviour of the model system increases with the particle size of the packed bed.

Heap performance depends upon liquid content as it influences many things such as liquid residence time, air flow and particle wetting. This means that there is some scope to vary liquid holdup or saturation independently of liquid addition rate and thus optimise performance. The possible mechanisms for the existence of the liquid holdup hysteresis are hypothesised and investigated. In addition, the theoretical modelling work developed using the steady state and residual liquid holdup values illustrates a single curve between the relative flow rate \( (\nu^*_t) \) and relative holdup \( (\theta^*) \) for the increasing and decreasing arms of flow. It proves that the dominant mechanism for the liquid holdup hysteresis is the change in the number of flow paths in the increasing and decreasing arms of the flow rather than a
change in the shape of the rivulet. Similar liquid holdup hysteresis was found in the ore system and the same explanation is valid for the ore system as well.

**9.1.2. INTER-PARTICLE FLOW MODEL**

The liquid holdup model developed in this work incorporates liquid holdup hysteresis as well. The model describes inter-particle flow behaviour as it was derived based on the liquid content results of the non-porous glass beads system. The relative flow rate \( \nu_s^* \) of the rivulet is proportional to the square of the additional liquid content of that rivulet \( \theta^* - 1 \). For both dry and wet bed start-up conditions all the particle sizes of the model system fit the model accurately giving a square relationship between the two parameters. The main model parameter of the inter-particle model is the pre-factor of the relationship \( K' \) and it demonstrates a strong relationship with the particle size.

In the ore system, the particles are slightly porous with 2-7% of porosity. Starting the liquid addition into a dry bed and the subsequent steady state and residual liquid holdup results did not fit the liquid holdup model. The main reason for this is that the liquid holdup within the pore spaces does not have the same effect on the overall flow behaviour as with the liquid content between the particles. It proves that it is not appropriate to treat the liquid holdup as an empirical input parameter or propose a direct relationship between the liquid holdup and the flow permeability of the system, as was the case in some of the heap leaching models.

**9.1.3. EFFECT OF PARTICLE POROSITY**

The characteristic length scales of the pores within the particles and in between the particles are significantly different. Therefore, the effect of particle porosity needs to be investigated and this is possible by comparing the flow behaviour of the non-porous model system and the porous ore system. The ore particles, which were initially saturated, and then externally dried, in a packed column can be used to measure the external liquid content of the system. The results of these modified liquid flow experiments with the ore system fit the same inter-particle flow model accurately, which was developed with the model system. However, there is no strong relationship between the particle size of the ore particles and the main model parameter, \( K' \).

202
The results prove that after separating the effect of porosity, the same model can be applied to describe the inter-particle liquid flow behaviour in both systems. That means this model is appropriate to use in heap leaching systems and especially, this can be coupled with the future computational models of heap leaching.

The main difference in the inter-particle flow behaviour between the two systems is the pre-factor in the model. The pre-factor in the relationship for the model system is approximately five times greater than that for the ore system. The higher tortuosity of the rivulets in the ore system and the small liquid content held between the angular ore particles compared to the spherical glass beads are possible reasons for the smaller pre-factor for the ore system.

The less dependency of the main model parameter on the particle size does not necessarily say that the steady state and residual liquid holdup values are independent of the particle size of the rock particles. Both the steady state and residual liquid holdup values decrease with the particle size.

9.1.4. TRANSIENT FLOW BEHAVIOUR

In order to understand the fluid flow mechanisms, not only the steady state, but also the transient flow behaviour in both the model and ore systems needs to be studied. During the initial liquid addition into the model system with dry glass beads, the movement of the wetting front follows a standing wave, where the shape of the front does not change as it moves. This standing wave is characterised by a period of no liquid outflow from the bed followed by a rapid transition to the final steady state flow rate. The shape of this transition also did not change much with the length of the column.

Simulations of the transient liquid holdup within the ore bed as it drains were performed by combining the inter-particle flow model developed in this study with a continuity equation and then solving the equations numerically. The simulated liquid holdups and drainage velocities agree very well with the experimental values. The simple model used in this work only accounts for gravitational and viscous forces and assumes that capillary forces are negligible in the vertical direction. Some of the slight discrepancy between the simulated and experimental results at moderate drainage times is probably due to these capillary forces.
9.1.5. HYDRODYNAMIC DISPERSION MEASUREMENTS
The 1-D column leaching system accurately provides axial dispersion values for different particle sizes of the model system using salt tracer tests. The mobile-immobile model fits the experimental data very well. In this study, liquid content was not a fitting parameter, which improves the accuracy of the calculated dispersion values.

As the model system exhibits liquid holdup hysteresis, it has multiple liquid contents and that means multiple average velocities for the same superficial liquid velocity. These multiple average velocities result in distinct axial dispersion values. Thus the variability of the dispersion values with the superficial velocity is much higher whereas the dispersion values provide much tighter relationship with the average velocity of the system. It implies that the axial dispersion should be characterised with the average velocity in the packed bed, not with the superficial velocity.

A tracer particle (400 µm) was tracked with PEPT to obtain particle location data through the glass beads of the model system. The location data given by PEPT can be used to determine both axial and radial dispersion coefficients in the packed bed. The resultant axial dispersion values with PEPT agree well with the same given by salt tracer tests. It concludes that the role of molecular diffusion of the salt tracer on the hydrodynamic dispersion is negligible, whereas the mixing of solutes in between the connections of rivulets is the dominant mechanism.

The radial dispersion coefficients are approximately four times smaller than the axial dispersion coefficients. Unfortunately, over the range of very low superficial velocities of this work (0.0075-0.03 mm/s) the 400 µm tracer particle tended to get stuck in between the 18 mm glass beads. Therefore, it is not possible to track the motion of the 400 µm tracer particle at very low superficial velocities in order to obtain both the axial and the radial dispersion coefficients with PEPT.

9.2. FUTURE WORK
This thesis presented results from the experiments which were performed within the course of PhD study over the last three years. All the flow aspects of heap leaching heaps could not be covered due to the time constraints of this three year course, however a number of interesting phenomena have been observed and described and useful models and
methodologies developed. The results and conclusions developed in this thesis can be further improved and then applied in heap scale computational models and industrial heap leaching systems by minimising the limitations of the current study.

9.2.1. IMPROVED MODELLING AND SIMULATION OF INDUSTRIAL HEAP LEACHING
This study clearly highlights the need to differentiate between the flow in-between the particles and the flow within the particles. The continuum fluid flow models used in existing heap scale simulators do not account for this effect and use a single relationship and, by implication, length scale to describe the fluid flow. By implementing separate models for these two fluid classes more accurate heap scale simulators could be developed. These descriptions for the fluid flow would need to be coupled to models for mass transfer, heat transfer and chemical reaction in order to produce a full heap scale simulator. This simulator could be used to develop improved heap design and operating strategies, as well as forming the basis for model based control.

9.2.2. MORE REALISTIC PARTICLES AND PACKINGS
The inter-particle flow behaviour has been described using the laboratory scale 1-D systems of both glass beads and ore particles. In the model system both mono-dispersed and also a few poly-dispersed mixtures of two and three particles sizes were employed. In addition, the particle size ranges of the ore mixtures were relatively narrow except the few experiments that have been performed with ore mixtures of two narrow size fractions. Therefore, this inter-particle flow model needs to be validated by using wider size fractions, especially for the ore particles, that more closely represent those found in industrial heap leaching. This would be used to both validate the form of the model, as well as determining the parameters to be used to model industrial scale heaps.

In real heaps the packing is typically inhomogeneous. The effect of inhomogeneity could be investigated by deliberately introducing inhomogeneity of a known and controlled degree into the system. This would probably require carrying out the experiments in much larger columns and possibly even at crib or pilot scale.
9.2.3. RESIDUAL AND INTRA-PARTICLE LIQUID HOLDUP
This study pointed out that the residual liquid holdup is history dependent and also a strong function of the particle size. The liquid holdup model requires the residual holdup for each liquid addition rate in order to calculate the two model parameters. This implies that a residual liquid holdup model is required to enhance the inter-particle flow model. Using the residual liquid holdup values for various particle sizes and analysing the residual liquid holdup features of the packed bed, it is possible to formulate an appropriate residual liquid holdup model. This could be coupled with simulations using software called “Surface Evolver” to model these residual liquid connections in order to improve the current flow model.

Furthermore, the flow model only describes the flow between the particles (i.e. inter-particle flow) and this study does not investigate the flow within the ore particles, which would also be very important in heap leaching. It limits the applicability of current model in large scale flow simulations and therefore, it is a potential subject area for future investigators. Micro-CT scanning would be a suitable experimental technique to investigate the flow within the pore spaces and to form the basis for a model of the intra-particle flow mechanisms.

9.2.4. 2-D LIQUID FLOW BEHAVIOUR
The liquid flow experiments were limited to a 1-D column leaching rig in this study, but similar experiments can be performed using a 2-D column, which is narrow compared to its length and height. It can be used to investigate horizontal liquid transport, which is not included in the inter-particle models developed in this research. The spread of liquid horizontally is important in heap leaching as it dictates, for instance, the spacing of drippers on the heap surface. The relative importance of capillarity to other dispersion mechanisms needs to be investigated.

9.2.5. PEPT TECHNIQUE TO MAP THE FLOW BEHAVIOUR IN PACKED BEDS
The PEPT experiments carried out in this study were only initial trial ones that were only used to calculate the dispersion coefficient within a packed bed. However, PEPT is a promising experimental technique for investigating the underlying flow mechanisms when the system is opaque such as column leaching systems. It was, however, found that the 400 µm tracer particles tended to get stuck in between the glass beads, a problem that is likely
to be even more acute in an ore system. As the PEPT tracer particle technology is continuously improving, future studies can be performed using smaller tracer particles, which should help to alleviate this problem. Recently Cole et al. (2012) labelled resin particles as small as 50 µm at the Cape Town PEPT centre. Furthermore, radioactive liquid tracers can also be used to map the flow behaviour. Future researchers can investigate the applicability of this technique more closely based on the fundamentals described in this study.


References


Cihla, Z., Schmidt, O., 1958. Studies of the behaviour of liquid when freely trickling over the packing of a cylindrical tower II. Collection of Czechoslovak Chemical Communications 23, 569-578.


References


References


Wu, A., Yin, S., Qin, W., Liu, J., Qiu, G., 2009. The effect of preferential flow on extraction and surface morphology of copper sulphides during heap leaching, Hydrometallurgy 95, 76-81.


NOMENCLATURE

\( A (t) \) Final PEPT co-ordinate at time, \( t \), after time weighting function within the kernel width (m)

\( A (x, y, z) \) Final PEPT co-ordinates of \( x, y, z \) at time, \( t \), after time weighting function within the kernel width (m)

\( A_i (t_i) \) Original PEPT co-ordinates at time, \( t_i \), within the kernel width (m)

\( A_i (x_i, y_i, z_i) \) Original PEPT co-ordinates of \( x_i, y_i, z_i \) at time, \( t_i \), within the kernel width (m)

\( A_f \) Area of rivulet that is actively flowing or cross-sectional area of the flow channel (m²)

\( A_{film} \) Average cross sectional area of the liquid film (m²)

\( A_r \) Average cross sectional area of the residual liquid in a rivulet (m²)

\( A_s \) Area of the rivulet held relatively stationary in the reservoirs between the particles (m²)

\( A_t \) Average area of the rivulet (m²)

\( A' \) Area of the channel between the particles occupied by liquid (m²)

\( a_p \) Packing geometrical area (1/m)

\( B \) Capillary capacity of the medium (1/m)

\( Bo \) Bond number (\( = \rho g d_p^2 / \gamma \)) (dimensionless)

\( b_1, b_2 \) and \( b_3 \) Constants in Yusuf’s (1984) liquid holdup model

\( C \) Concentration of diffusing species in solution (kg/m³)

\( C(t) \) Measured effluent tracer concentration of the system (kg/m³)

\( C_d \) Tracer concentration in the mobile liquid in mobile-immobile model (kg/m³)
<table>
<thead>
<tr>
<th>Symbol</th>
<th>Definition</th>
</tr>
</thead>
<tbody>
<tr>
<td>$C_{drag}$</td>
<td>A drag coefficient (dimensionless)</td>
</tr>
<tr>
<td>$C_{in}(t)$</td>
<td>Actual tracer concentration into the small reservoir at time, $t$ (kg/m$^3$)</td>
</tr>
<tr>
<td>$C_{m}(t)$</td>
<td>Measured tracer concentration using the conductivity cell in the small reservoir at time, $t$ (kg/m$^3$)</td>
</tr>
<tr>
<td>$C_{out}(t)$</td>
<td>Tracer concentration of the liquid which leaves the small reservoir at time, $t$ in Figure 3.19 (kg/m$^3$)</td>
</tr>
<tr>
<td>$C_p$</td>
<td>Packing constant (1/m)</td>
</tr>
<tr>
<td>$C_s$</td>
<td>Tracer concentration in the stagnant region in mobile-immobile model (kg/m$^3$)</td>
</tr>
<tr>
<td>$c$</td>
<td>A geometric constant in Equation 4.3 (dimensionless)</td>
</tr>
<tr>
<td>$D$</td>
<td>Hydrodynamic dispersion coefficient (m$^2$/s)</td>
</tr>
<tr>
<td>$D_a$</td>
<td>Axial dispersion coefficient (m$^2$/s)</td>
</tr>
<tr>
<td>$D_{eff}$</td>
<td>Effective diffusivity in porous ore particles corrected for porosity and tortuosity (= $D_o \varepsilon / \tau_i$) (m$^2$/s)</td>
</tr>
<tr>
<td>$D_H$</td>
<td>Hydraulic diameter (m)</td>
</tr>
<tr>
<td>$D_h$</td>
<td>Mechanical dispersion (m$^2$/s)</td>
</tr>
<tr>
<td>$D_m$</td>
<td>Effective molecular diffusion (m$^2$/s)</td>
</tr>
<tr>
<td>$D_o$</td>
<td>Diffusivity (m$^2$/s)</td>
</tr>
<tr>
<td>$D_s$</td>
<td>Liquid spread factor (m)</td>
</tr>
<tr>
<td>$D_w$</td>
<td>Diffusion coefficient in bulk water (m$^2$/s)</td>
</tr>
<tr>
<td>$d$</td>
<td>Diameter of a circular cross-section (m)</td>
</tr>
<tr>
<td>$d_h$</td>
<td>Hydraulic radius (m)</td>
</tr>
<tr>
<td>$d_0$</td>
<td>Diameter based on bed voidage (Fu and Tan, 1996) (m)</td>
</tr>
<tr>
<td>$d_p$</td>
<td>Particle diameter (m)</td>
</tr>
<tr>
<td>$d_{pl}$</td>
<td>Particle diameter of each size fraction (m)</td>
</tr>
<tr>
<td>$d_R$</td>
<td>Inner reactor or column diameter (m)</td>
</tr>
<tr>
<td>Symbol</td>
<td>Definition</td>
</tr>
<tr>
<td>--------</td>
<td>------------</td>
</tr>
<tr>
<td>$d_R/d_p$</td>
<td>Ratio of column or reactor to particle diameter (dimensionless)</td>
</tr>
<tr>
<td>$E(t)$</td>
<td>Exit age distribution (1/s)</td>
</tr>
<tr>
<td>$F$</td>
<td>Liquid flux within the bed (m/s)</td>
</tr>
<tr>
<td>$F(t)$</td>
<td>Cumulative distribution function (dimensionless)</td>
</tr>
<tr>
<td>$F_{t,r_p}$</td>
<td>Fractional extraction in time $t$ for a spherical particle of radius $r_p$ (dimensionless)</td>
</tr>
<tr>
<td>$f$</td>
<td>Final fraction of detected events in the PEPT location algorithm (dimensionless)</td>
</tr>
<tr>
<td>$f_{(r,z,\phi)}$</td>
<td>Vertical flow of liquid per unit area in unit time (m/s)</td>
</tr>
<tr>
<td>$Ga$</td>
<td>Galileo number (dimensionless)</td>
</tr>
<tr>
<td>$g$</td>
<td>Gravitational acceleration (m/s$^2$)</td>
</tr>
<tr>
<td>$g^*$</td>
<td>Gravitational component in the direction along the rivulet (m/s$^2$)</td>
</tr>
<tr>
<td>$H$</td>
<td>Pressure head (m)</td>
</tr>
<tr>
<td>$h$</td>
<td>Vertical distance between the liquid filled connections (m)</td>
</tr>
<tr>
<td>$h'$</td>
<td>Half the kernel width of time weighting function (s)</td>
</tr>
<tr>
<td>$h_0$ and $k_0$</td>
<td>Distribution constants (1/m)</td>
</tr>
<tr>
<td>$K$</td>
<td>Pre-factor of the inter-particle flow model in Equation 4.18 (m/s) ($= p g A_r/\mu C_{drag}$)</td>
</tr>
<tr>
<td>$K_c$</td>
<td>Unsaturated hydraulic conductivity (m/s)</td>
</tr>
<tr>
<td>$k_T$</td>
<td>Tortuosity factor for the flowing rivulets (dimensionless)</td>
</tr>
<tr>
<td>$L$</td>
<td>Actual distance between two liquid filled connections (m)</td>
</tr>
<tr>
<td>$l_0$</td>
<td>Characteristic length (m)</td>
</tr>
<tr>
<td>$l$</td>
<td>Shortest distance between two liquid filled connections (m)</td>
</tr>
<tr>
<td>$l'$</td>
<td>Thickness of the liquid film (m)</td>
</tr>
<tr>
<td>$M_i$</td>
<td>Mass of each size fraction of particles (kg)</td>
</tr>
</tbody>
</table>
Nomenclature

\( M_{out} \) Total mass of liquid drained out of the packed bed (kg)

\( m \) A constant in Equation 4.38 (dimensionless)

\( m_{in} \) Tracer mass addition into the small reservoir (kg)

\( m_{initial} \) Initial mass of the tracer in the small reservoir (kg)

\( m_{out} \) Tracer mass removal from the small reservoir (kg)

\( m_r \) Tracer mass left in the small reservoir (kg)

\( N \) Slice size in PEPT location algorithm (dimensionless)

\( N_{rivulets} \) Number of rivulets per cross-sectional area (1/m\(^2\))

\( n \) Index of the infinite series in equation 2.2 (dimensionless)

\( n_0 \) An empirical constant between 1 and 2 (dimensionless)

\( n' \) Sample size (dimensionless)

\( P_c \) Capillary pressure (Pa)

\( P_e \) Peclet number (= \( UI/D \)) (dimensionless)

\( \Delta P \) Pressure difference between the two liquid filled connections (Pa)

\( Q \) Liquid flow rate (m\(^3\)/s)

\( Q_{film} \) Liquid flow rate for a thin film flow falling over the surface of the particle (m\(^3\)/s)

\( Q_L \) Liquid flow rate into the column (m\(^3\)/s)

\( Q_r \) Liquid flow rate down one rivulet (m\(^3\)/s)

\( Q_{res} \) Liquid flow rate into the small reservoir in axial dispersion measurements (m\(^3\)/s)

\( Q_x \) Fraction of the liquid that would fall into a trough of unit width located at a distance \( Z \) from the packing and displaced horizontally at a distance \( x \) from the centre line

\( \hat{q} \) Darcy flux (m/s)

\( q_t \) Independent variable of the weighting function (dimensionless)
<table>
<thead>
<tr>
<th>Symbol</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>$R$</td>
<td>Radius of the circular rivulet (m)</td>
</tr>
<tr>
<td>$Re$</td>
<td>Reynolds number (dimensionless)</td>
</tr>
<tr>
<td>$Re'$</td>
<td>Modified Reynolds number (dimensionless)</td>
</tr>
<tr>
<td>$r$</td>
<td>Radial variable in polar co-ordinates</td>
</tr>
<tr>
<td>$r_c$</td>
<td>Radius of curvature of the rivulet (m)</td>
</tr>
<tr>
<td>$r_p$</td>
<td>Radius of the particle (m)</td>
</tr>
<tr>
<td>$r_{ps}$</td>
<td>Particle spacing (m)</td>
</tr>
<tr>
<td>$t$</td>
<td>Time (s)</td>
</tr>
<tr>
<td>$t_i$</td>
<td>Time of a PEPT co-ordinate (s)</td>
</tr>
<tr>
<td>$t_R$</td>
<td>Mean residence time (s)</td>
</tr>
<tr>
<td>$t_{R,corrected}$</td>
<td>Mean residence time of the corrected RTD curve (s)</td>
</tr>
<tr>
<td>$t_{R,measured}$</td>
<td>Mean residence time of the measured RTD curve (s)</td>
</tr>
<tr>
<td>$t_r$</td>
<td>Residence time of the small reservoir (s)</td>
</tr>
<tr>
<td>$t_{1/2}$</td>
<td>Half-life of the radionuclide (minutes, hours, years)</td>
</tr>
<tr>
<td>$\Delta t$</td>
<td>Time interval (s)</td>
</tr>
<tr>
<td>$U$</td>
<td>Velocity of the system (m/s)</td>
</tr>
<tr>
<td>$u$</td>
<td>Liquid velocity in the mobile region in mobile-immobile model ($= v_s/\theta_d$) (m/s)</td>
</tr>
<tr>
<td>$V$</td>
<td>Volume of liquid per connection (m$^3$)</td>
</tr>
<tr>
<td>$V_a$</td>
<td>Active flow rate (m$^3$/s)</td>
</tr>
<tr>
<td>$V_b$</td>
<td>By-passing flow rate (m$^3$/s)</td>
</tr>
<tr>
<td>$V_c$</td>
<td>Volume of the empty column (m$^3$)</td>
</tr>
<tr>
<td>$V_d$</td>
<td>Volume of the stagnant/dead volume (m$^3$)</td>
</tr>
<tr>
<td>$V_{in}$</td>
<td>Water volume into the packed bed (m$^3$)</td>
</tr>
<tr>
<td>$V_m$</td>
<td>Volume of the well mixed flow (m$^3$)</td>
</tr>
</tbody>
</table>
Nomenclature

\( V_{\text{out}} \) Water volume out from the packed bed (m³)

\( V_p \) Volume of the plug flow (m³)

\( V_r \) Volume of the small reservoir in Figure 3.19 (m³)

\( v \) Pore water velocity (= \( \bar{q}/\theta \)) (m/s)

\( v_{\text{end}} \) Roughly constant flow out of the column at longer times (m/s)

\( v_G \) Superficial gas velocity (m/s)

\( v_{\text{rivulet}} \) Average velocity of the liquid in the rivulet (m/s)

\( v_s \) Superficial liquid velocity (m/s)

\( v_s^* \) Relative velocity (= \( v_s/\theta_{\text{residual}} \)) (m/s)

\( W \) Wetted perimeter of the surface of the channel (m)

\( W_i \) Weighting or dependant variable of the time weighting function (dimensionless)

\( W_0 \) Width of the liquid film (m)

\( x \) Horizontal co-ordinate or distance (m)

\( \Delta y \) Tracer displacements in y direction in PEPT location data (m)

\( \bar{\Delta y} \) Mean tracer displacement in the y direction in PEPT location data (m)

\( Z \) Column length or height (m)

\( z \) Vertical co-ordinate or distance (m)

Greek Symbols

\( \alpha \) Mass transfer coefficient between the dynamic and static zones in mobile-immobile model (1/s)

\( \beta \) Liquid saturation (= \( \theta/\xi \)) (dimensionless)

\( \gamma \) Surface tension of liquid (N/m)

\( \varepsilon \) Porosity of the particle (dimensionless)

\( \theta \) Total liquid holdup or the moisture content (dimensionless)
<table>
<thead>
<tr>
<th>Symbol</th>
<th>Definition</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\theta_d$</td>
<td>Liquid content in the mobile region or mobile liquid holdup in mobile-immobile model (dimensionless)</td>
</tr>
<tr>
<td>$\theta_{dynamic}$</td>
<td>Dynamic liquid holdup (dimensionless)</td>
</tr>
<tr>
<td>$\theta_e$</td>
<td>External liquid holdup (dimensionless)</td>
</tr>
<tr>
<td>$\theta_{ex}$</td>
<td>Estimated external residual liquid between the particles (dimensionless)</td>
</tr>
<tr>
<td>$\theta_l$</td>
<td>Internal liquid holdup (dimensionless)</td>
</tr>
<tr>
<td>$\theta_{residual}$</td>
<td>Residual holdup (dimensionless)</td>
</tr>
<tr>
<td>$\theta_s$</td>
<td>Liquid content in the immobile region or static liquid holdup in mobile-immobile model (dimensionless)</td>
</tr>
<tr>
<td>$\theta_{static}$</td>
<td>Static liquid holdup (dimensionless)</td>
</tr>
<tr>
<td>$\theta^*$</td>
<td>Relative holdup ($= \theta / \theta_{residual}$) (dimensionless)</td>
</tr>
<tr>
<td>$\lambda$</td>
<td>Dispersivity (m)</td>
</tr>
<tr>
<td>$\mu$</td>
<td>Viscosity of liquid (Pa.s)</td>
</tr>
<tr>
<td>$\xi$</td>
<td>Bed voidage (dimensionless)</td>
</tr>
<tr>
<td>$\rho$</td>
<td>Density of liquid (kg/m$^3$)</td>
</tr>
<tr>
<td>$\tau$</td>
<td>Shear stress (kg/m$^2$s)</td>
</tr>
<tr>
<td>$\tau_l$</td>
<td>Tortuosity factor for molecular diffusion or average tortuosity of open pore paths in the ore particle (dimensionless)</td>
</tr>
<tr>
<td>$\phi$</td>
<td>Contact angle (degrees)</td>
</tr>
<tr>
<td>$\varphi_L$</td>
<td>Liquid mass flux (kg/m$^2$s)</td>
</tr>
<tr>
<td>$\phi$</td>
<td>An angular variable</td>
</tr>
<tr>
<td>Abbreviation</td>
<td>Description</td>
</tr>
<tr>
<td>--------------</td>
<td>-------------</td>
</tr>
<tr>
<td>1-D</td>
<td>1-Dimensional</td>
</tr>
<tr>
<td>2-D</td>
<td>2-Dimensional</td>
</tr>
<tr>
<td>3-D</td>
<td>3-Dimensional</td>
</tr>
<tr>
<td>AC</td>
<td>Alternating Current</td>
</tr>
<tr>
<td>ADE</td>
<td>Advection-Dispersion Equation</td>
</tr>
<tr>
<td>CFB</td>
<td>Circulating Fluidized Beds</td>
</tr>
<tr>
<td>DC</td>
<td>Direct Current</td>
</tr>
<tr>
<td>LDA</td>
<td>Laser Doppler Anemometry</td>
</tr>
<tr>
<td>LoRs</td>
<td>Line of Responses</td>
</tr>
<tr>
<td>Micro-CT</td>
<td>Micro-Computer Tomography</td>
</tr>
<tr>
<td>MIM</td>
<td>Mobile-Immobile Model</td>
</tr>
<tr>
<td>MRI</td>
<td>Magnetic Resonance Imaging</td>
</tr>
<tr>
<td>PCD</td>
<td>Pitch Circle Diameter</td>
</tr>
<tr>
<td>PDE</td>
<td>Partial Differential Equation</td>
</tr>
<tr>
<td>PEPT</td>
<td>Positron Emission Particle Tracking</td>
</tr>
<tr>
<td>PET</td>
<td>Positron Emission Tomography</td>
</tr>
<tr>
<td>PIV</td>
<td>Particle Imaging Velocimetry</td>
</tr>
<tr>
<td>PVC</td>
<td>Polyvinyl Chloride</td>
</tr>
<tr>
<td>ROM</td>
<td>Run-of-Mine</td>
</tr>
<tr>
<td>RTD</td>
<td>Residence Time Distribution</td>
</tr>
<tr>
<td>r.m.s.</td>
<td>root mean square</td>
</tr>
<tr>
<td>TBRs</td>
<td>Trickle Bed Reactors</td>
</tr>
</tbody>
</table>
APPENDIX A: AGITATED TANK LEACHING AND VAT LEACHING

**Agitated Leaching:** An ore is grounded by using operations such as crushing and grinding to a liberation size, which is typically less than 1 mm and the size that mineral grains expose to the leaching reagent. The ore particles are placed in leaching tanks with a leaching reagent to leach from the ore into the fluid phase. Once leaching is completed, the solution enriched with metals is separated from the leaching tank for subsequent metal extraction techniques such as solvent extraction followed by electro-winning (Altman et al., 2002).

The main operating parameters that directly affect the performance of the agitated leaching technique are grind size, slurry density, number of leaching tanks and leaching reagents (Altman et al., 2002). The capital and operating costs for heap leaching is generally less than the capital costs associated with agitated leaching but the metal recoveries are typically higher in agitated leaching than recoveries in heap leaching. Furthermore, less than 72 hours are needed to leach the ore using agitated leaching compared to greater time required for heap leaching (eg. for gold heap leaching 90-180 days, for copper heap leaching 180-360 days) (Altman et al., 2002).

**Vat Leaching:** Agitated tank leaching is limited to high grade finely ground ores due to high capital and operation costs. Therefore, the crushed medium grade ores to sizes that are too fine for heap leaching and too coarse for agitated leaching, can be leached in large tanks called vats (INNOVAT). The vat leaching technique is similar to agitated tank leaching; however the particle size of the crushed ore is greater than the same in ground ore used in tank leaching (see Figure 2.1). There are two types of vat leaching techniques, which are conventional vat leaching and continuous vat leaching. The conventional vat leaching is occurred in separate batches but this is expensive due to loading and unloading the batches of ore. Therefore, continuous vat leaching is practiced and it has the same kinetics as agitated tank leaching. Furthermore, the leaching time measures in hours, not days, weeks, months or years as is common in heap leaching (INNOVAT).
### APPENDIX B: EXPERIMENTAL DATA

#### Table B1: Liquid contents for glass beads in 300 mm column with dry bed start-up.

<table>
<thead>
<tr>
<th></th>
<th>2 mm</th>
<th>10 mm</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Increasing arm</td>
<td>Decreasing arm</td>
</tr>
<tr>
<td>Superficial flow (mm/s)</td>
<td>Liquid holdup (%)</td>
<td>Liquid holdup (%)</td>
</tr>
<tr>
<td>0.0075</td>
<td>0.943</td>
<td>1.119</td>
</tr>
<tr>
<td>0.015</td>
<td>1.047</td>
<td>1.177</td>
</tr>
<tr>
<td>0.03</td>
<td>1.145</td>
<td>1.268</td>
</tr>
<tr>
<td>0.06</td>
<td>1.288</td>
<td>1.359</td>
</tr>
<tr>
<td>0.12</td>
<td>1.483</td>
<td>1.483</td>
</tr>
<tr>
<td>14 mm</td>
<td>Increasing arm</td>
<td>Decreasing arm</td>
</tr>
<tr>
<td>Superficial flow (mm/s)</td>
<td>Liquid holdup (%)</td>
<td>Liquid holdup (%)</td>
</tr>
<tr>
<td>0.0075</td>
<td>0.761</td>
<td>1.093</td>
</tr>
<tr>
<td>0.015</td>
<td>0.820</td>
<td>1.151</td>
</tr>
<tr>
<td>0.03</td>
<td>0.950</td>
<td>1.177</td>
</tr>
<tr>
<td>0.06</td>
<td>1.093</td>
<td>1.268</td>
</tr>
<tr>
<td>0.12</td>
<td>1.392</td>
<td>1.392</td>
</tr>
</tbody>
</table>

#### Table B2: Steady and residual liquid contents for 2 mm particles in 300 mm column with dry bed start-up condition.

<table>
<thead>
<tr>
<th>Superficial velocity (mm/s)</th>
<th>Water (g)</th>
<th>Steady liquid holdup (%)</th>
<th>Superficial velocity (mm/s)</th>
<th>Water (g)</th>
<th>Residual liquid holdup (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.0075</td>
<td>140.27</td>
<td>1.008</td>
<td>0.0075</td>
<td>122.17</td>
<td>0.878</td>
</tr>
<tr>
<td>0.015</td>
<td>161.99</td>
<td>1.164</td>
<td>0.015</td>
<td>135.75</td>
<td>0.976</td>
</tr>
<tr>
<td>0.03</td>
<td>191.86</td>
<td>1.379</td>
<td>0.03</td>
<td>150.23</td>
<td>1.080</td>
</tr>
<tr>
<td>0.06</td>
<td>215.38</td>
<td>1.548</td>
<td>0.06</td>
<td>159.28</td>
<td>1.145</td>
</tr>
<tr>
<td>0.12</td>
<td>247.06</td>
<td>1.776</td>
<td>0.12</td>
<td>167.42</td>
<td>1.203</td>
</tr>
<tr>
<td>0.06</td>
<td>226.24</td>
<td>1.626</td>
<td>0.06</td>
<td>168.33</td>
<td>1.210</td>
</tr>
<tr>
<td>0.03</td>
<td>214.48</td>
<td>1.542</td>
<td>0.03</td>
<td>171.04</td>
<td>1.229</td>
</tr>
<tr>
<td>0.015</td>
<td>198.19</td>
<td>1.424</td>
<td>0.015</td>
<td>169.23</td>
<td>1.216</td>
</tr>
<tr>
<td>0.0075</td>
<td>189.14</td>
<td>1.359</td>
<td>0.0075</td>
<td>170.14</td>
<td>1.223</td>
</tr>
</tbody>
</table>
Table B3: Steady and residual liquid contents for 10 mm particles in 300 mm column with dry bed start-up condition.

<table>
<thead>
<tr>
<th>Superficial velocity (mm/s)</th>
<th>Water (g)</th>
<th>Steady liquid holdup (%)</th>
<th>Superficial velocity (mm/s)</th>
<th>Water (g)</th>
<th>Residual liquid holdup (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.0075</td>
<td>110.41</td>
<td>0.794</td>
<td>0.0075</td>
<td>100.45</td>
<td>0.722</td>
</tr>
<tr>
<td>0.015</td>
<td>128.51</td>
<td>0.924</td>
<td>0.015</td>
<td>110.41</td>
<td>0.794</td>
</tr>
<tr>
<td>0.03</td>
<td>148.42</td>
<td>1.067</td>
<td>0.03</td>
<td>121.27</td>
<td>0.872</td>
</tr>
<tr>
<td>0.06</td>
<td>166.52</td>
<td>1.197</td>
<td>0.06</td>
<td>123.98</td>
<td>0.891</td>
</tr>
<tr>
<td>0.12</td>
<td>194.57</td>
<td>1.398</td>
<td>0.12</td>
<td>132.13</td>
<td>0.950</td>
</tr>
<tr>
<td>0.06</td>
<td>181.00</td>
<td>1.301</td>
<td>0.06</td>
<td>135.75</td>
<td>0.976</td>
</tr>
<tr>
<td>0.03</td>
<td>172.85</td>
<td>1.242</td>
<td>0.03</td>
<td>140.27</td>
<td>1.008</td>
</tr>
<tr>
<td>0.015</td>
<td>165.61</td>
<td>1.190</td>
<td>0.015</td>
<td>142.99</td>
<td>1.028</td>
</tr>
<tr>
<td>0.0075</td>
<td>170.14</td>
<td>1.223</td>
<td>0.0075</td>
<td>142.08</td>
<td>1.021</td>
</tr>
</tbody>
</table>

Table B4: Steady and residual liquid contents for 14 mm particles in 300 mm column with dry bed start-up condition.

<table>
<thead>
<tr>
<th>Superficial velocity (mm/s)</th>
<th>Water (g)</th>
<th>Steady liquid holdup (%)</th>
<th>Superficial velocity (mm/s)</th>
<th>Water (g)</th>
<th>Residual liquid holdup (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.0075</td>
<td>99.55</td>
<td>0.715</td>
<td>0.0075</td>
<td>87.78</td>
<td>0.631</td>
</tr>
<tr>
<td>0.015</td>
<td>115.84</td>
<td>0.833</td>
<td>0.015</td>
<td>94.12</td>
<td>0.676</td>
</tr>
<tr>
<td>0.03</td>
<td>139.37</td>
<td>1.002</td>
<td>0.03</td>
<td>110.41</td>
<td>0.794</td>
</tr>
<tr>
<td>0.06</td>
<td>163.80</td>
<td>1.177</td>
<td>0.06</td>
<td>128.51</td>
<td>0.924</td>
</tr>
<tr>
<td>0.12</td>
<td>197.29</td>
<td>1.418</td>
<td>0.12</td>
<td>147.51</td>
<td>1.060</td>
</tr>
<tr>
<td>0.06</td>
<td>190.05</td>
<td>1.366</td>
<td>0.06</td>
<td>153.85</td>
<td>1.106</td>
</tr>
<tr>
<td>0.03</td>
<td>182.81</td>
<td>1.314</td>
<td>0.03</td>
<td>157.47</td>
<td>1.132</td>
</tr>
<tr>
<td>0.015</td>
<td>174.66</td>
<td>1.255</td>
<td>0.015</td>
<td>153.85</td>
<td>1.106</td>
</tr>
<tr>
<td>0.0075</td>
<td>172.85</td>
<td>1.242</td>
<td>0.0075</td>
<td>152.04</td>
<td>1.093</td>
</tr>
</tbody>
</table>

Table B5: Steady and residual liquid contents for 18 mm particles in 300 mm column with dry bed start-up condition.

<table>
<thead>
<tr>
<th>Superficial velocity (mm/s)</th>
<th>Water (g)</th>
<th>Steady liquid holdup (%)</th>
<th>Superficial velocity (mm/s)</th>
<th>Water (g)</th>
<th>Residual liquid holdup (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.0075</td>
<td>114.93</td>
<td>0.826</td>
<td>0.0075</td>
<td>103.17</td>
<td>0.742</td>
</tr>
<tr>
<td>0.015</td>
<td>130.32</td>
<td>0.937</td>
<td>0.015</td>
<td>111.31</td>
<td>0.800</td>
</tr>
<tr>
<td>0.03</td>
<td>158.37</td>
<td>1.138</td>
<td>0.03</td>
<td>132.13</td>
<td>0.950</td>
</tr>
<tr>
<td>0.06</td>
<td>191.86</td>
<td>1.379</td>
<td>0.06</td>
<td>154.75</td>
<td>1.112</td>
</tr>
<tr>
<td>0.12</td>
<td>224.43</td>
<td>1.613</td>
<td>0.12</td>
<td>171.95</td>
<td>1.236</td>
</tr>
<tr>
<td>0.06</td>
<td>219.91</td>
<td>1.581</td>
<td>0.06</td>
<td>180.09</td>
<td>1.294</td>
</tr>
<tr>
<td>0.03</td>
<td>215.38</td>
<td>1.548</td>
<td>0.03</td>
<td>185.52</td>
<td>1.333</td>
</tr>
<tr>
<td>0.015</td>
<td>211.76</td>
<td>1.522</td>
<td>0.015</td>
<td>190.05</td>
<td>1.366</td>
</tr>
<tr>
<td>0.0075</td>
<td>209.95</td>
<td>1.509</td>
<td>0.0075</td>
<td>189.14</td>
<td>1.359</td>
</tr>
</tbody>
</table>
Table B6: Steady and residual liquid contents for 2 mm particles in 300 mm column with wet bed start-up condition.

<table>
<thead>
<tr>
<th>Superficial velocity (mm/s)</th>
<th>Water (g)</th>
<th>Steady liquid holdup (%)</th>
<th>Superficial velocity (mm/s)</th>
<th>Water (g)</th>
<th>Residual liquid holdup (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.0075</td>
<td>352.04</td>
<td>2.530</td>
<td>0.0075</td>
<td>320.36</td>
<td>2.303</td>
</tr>
<tr>
<td>0.015</td>
<td>365.61</td>
<td>2.628</td>
<td>0.015</td>
<td>323.98</td>
<td>2.329</td>
</tr>
<tr>
<td>0.03</td>
<td>380.09</td>
<td>2.732</td>
<td>0.03</td>
<td>323.08</td>
<td>2.322</td>
</tr>
<tr>
<td>0.06</td>
<td>393.67</td>
<td>2.829</td>
<td>0.06</td>
<td>323.08</td>
<td>2.322</td>
</tr>
<tr>
<td>0.12</td>
<td>407.24</td>
<td>2.927</td>
<td>0.12</td>
<td>319.46</td>
<td>2.296</td>
</tr>
<tr>
<td>0.06</td>
<td>390.95</td>
<td>2.810</td>
<td>0.06</td>
<td>323.08</td>
<td>2.322</td>
</tr>
<tr>
<td>0.03</td>
<td>377.38</td>
<td>2.712</td>
<td>0.03</td>
<td>318.55</td>
<td>2.290</td>
</tr>
<tr>
<td>0.015</td>
<td>363.80</td>
<td>2.615</td>
<td>0.015</td>
<td>318.55</td>
<td>2.290</td>
</tr>
<tr>
<td>0.0075</td>
<td>350.23</td>
<td>2.517</td>
<td>0.0075</td>
<td>319.46</td>
<td>2.296</td>
</tr>
</tbody>
</table>

Table B7: Steady and residual liquid contents for 10 mm particles in 300 mm column with wet bed start-up condition.

<table>
<thead>
<tr>
<th>Superficial velocity (mm/s)</th>
<th>Water (g)</th>
<th>Steady liquid holdup (%)</th>
<th>Superficial velocity (mm/s)</th>
<th>Water (g)</th>
<th>Residual liquid holdup (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.0075</td>
<td>170.14</td>
<td>1.223</td>
<td>0.0075</td>
<td>152.94</td>
<td>1.099</td>
</tr>
<tr>
<td>0.015</td>
<td>185.52</td>
<td>1.333</td>
<td>0.015</td>
<td>164.71</td>
<td>1.184</td>
</tr>
<tr>
<td>0.03</td>
<td>194.57</td>
<td>1.398</td>
<td>0.03</td>
<td>166.52</td>
<td>1.197</td>
</tr>
<tr>
<td>0.06</td>
<td>211.76</td>
<td>1.522</td>
<td>0.06</td>
<td>168.33</td>
<td>1.210</td>
</tr>
<tr>
<td>0.12</td>
<td>237.10</td>
<td>1.704</td>
<td>0.12</td>
<td>171.04</td>
<td>1.229</td>
</tr>
<tr>
<td>0.06</td>
<td>215.38</td>
<td>1.548</td>
<td>0.06</td>
<td>171.04</td>
<td>1.229</td>
</tr>
<tr>
<td>0.03</td>
<td>208.14</td>
<td>1.496</td>
<td>0.03</td>
<td>173.76</td>
<td>1.249</td>
</tr>
<tr>
<td>0.015</td>
<td>199.10</td>
<td>1.431</td>
<td>0.015</td>
<td>173.76</td>
<td>1.249</td>
</tr>
<tr>
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<td>194.57</td>
<td>1.398</td>
<td>0.0075</td>
<td>175.57</td>
<td>1.262</td>
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</tbody>
</table>

Table B8: Steady and residual liquid contents for 14 mm particles in 300 mm column with wet bed start-up condition.

<table>
<thead>
<tr>
<th>Superficial velocity (mm/s)</th>
<th>Water (g)</th>
<th>Steady liquid holdup (%)</th>
<th>Superficial velocity (mm/s)</th>
<th>Water (g)</th>
<th>Residual liquid holdup (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.0075</td>
<td>208.14</td>
<td>1.496</td>
<td>0.0075</td>
<td>181.90</td>
<td>1.307</td>
</tr>
<tr>
<td>0.015</td>
<td>226.24</td>
<td>1.626</td>
<td>0.015</td>
<td>188.24</td>
<td>1.353</td>
</tr>
<tr>
<td>0.03</td>
<td>235.29</td>
<td>1.691</td>
<td>0.03</td>
<td>185.52</td>
<td>1.333</td>
</tr>
<tr>
<td>0.06</td>
<td>259.73</td>
<td>1.867</td>
<td>0.06</td>
<td>193.67</td>
<td>1.392</td>
</tr>
<tr>
<td>0.12</td>
<td>296.83</td>
<td>2.133</td>
<td>0.12</td>
<td>211.76</td>
<td>1.522</td>
</tr>
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<td>0.06</td>
<td>215.38</td>
<td>1.548</td>
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<td>0.015</td>
<td>217.19</td>
<td>1.561</td>
</tr>
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<td>0.0075</td>
<td>249.77</td>
<td>1.795</td>
<td>0.0075</td>
<td>211.76</td>
<td>1.522</td>
</tr>
</tbody>
</table>
Table B9: Steady and residual liquid contents for 18 mm particles in 300 mm column with wet bed start-up condition.

<table>
<thead>
<tr>
<th>Superficial velocity (mm/s)</th>
<th>Water (g)</th>
<th>Steady liquid holdup (%)</th>
<th>Superficial velocity (mm/s)</th>
<th>Water (g)</th>
<th>Residual liquid holdup (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.0075</td>
<td>251.58</td>
<td>1.808</td>
<td>0.0075</td>
<td>203.62</td>
<td>1.464</td>
</tr>
<tr>
<td>0.015</td>
<td>250.68</td>
<td>1.802</td>
<td>0.015</td>
<td>219.00</td>
<td>1.574</td>
</tr>
<tr>
<td>0.03</td>
<td>276.92</td>
<td>1.990</td>
<td>0.03</td>
<td>224.43</td>
<td>1.613</td>
</tr>
<tr>
<td>0.06</td>
<td>295.02</td>
<td>2.120</td>
<td>0.06</td>
<td>225.34</td>
<td>1.620</td>
</tr>
<tr>
<td>0.12</td>
<td>315.84</td>
<td>2.270</td>
<td>0.12</td>
<td>236.20</td>
<td>1.698</td>
</tr>
<tr>
<td>0.06</td>
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<td>2.264</td>
<td>0.06</td>
<td>244.34</td>
<td>1.756</td>
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<tr>
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<td>0.03</td>
<td>246.15</td>
<td>1.769</td>
</tr>
<tr>
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<td>292.31</td>
<td>2.101</td>
<td>0.015</td>
<td>250.68</td>
<td>1.802</td>
</tr>
<tr>
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<td>0.0075</td>
<td>249.77</td>
<td>1.795</td>
</tr>
</tbody>
</table>

Table B10: Steady and residual liquid holdup during the increasing and decreasing flow rates in 500 mm column packed with 2 mm particles.

<table>
<thead>
<tr>
<th>Increasing arm</th>
<th>Superficial velocity (mm/s)</th>
<th>Time (minutes)</th>
<th>Total holdup (%)</th>
<th>Superficial velocity (mm/s)</th>
<th>Time (minutes)</th>
<th>Total holdup (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.0075</td>
<td>0</td>
<td>0</td>
<td>14.5</td>
<td>1.0147</td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.0075</td>
<td>0.5</td>
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Figure B1: Liquid holdup for the 2 mm particles in 300 mm column with 95% confidence. a) dry bed (0.07-0.18%) b) wet bed (0.09-0.17%).
Figure B2: Liquid holdup for the 10 mm particles in 300 mm column with 95% confidence. 
a) dry bed (0.09-0.17%) b) wet bed (0.06-0.14%).
Figure B3: Liquid holdup for the 14 mm particles in 300 mm column with 95% confidence. 

a) dry bed (0.1-0.18%) b) wet bed (0.06-0.11%).
Figure B4: Liquid holdup for the 18 mm particles in 300 mm column with 95% confidence.
   a) dry bed (0.09-0.16%) b) wet bed (0.1-0.14%).
APPENDIX C: DERIVATION OF THE AVERAGE LIQUID VELOCITY IN A RIVULET

The Darcy-Weisbach relationship is as follows, with a constant proportionality for a particular cross sectional shape:

\[ Q \propto \frac{\Delta P}{Z \mu} R^4 \quad (C.1) \]

where \( Q \) is the flow rate, \( \Delta P \) is the pressure difference, \( Z \) is the column length, \( \mu \) is the viscosity of the liquid and \( R \) is the radius of the flow channel.

For gravity driven flow:

\[ Q \propto \frac{\rho g}{\mu} R^4 \quad (C.2) \]

The hydraulic diameter \( (D_H) \) is the characteristic dimension used in flow through both open and closed channels (Streeter and Wylie, 1983 and 1985).

\[ Q \propto \frac{\rho g}{\mu} D_H^4 \quad (C.3) \]

If the shape remains the same, then \( A_f \propto D_H^2 \) and equation C.2 gives:

\[ Q \propto \frac{\rho g}{\mu} A_f^2 \quad (C.4) \]

\[ Q = \frac{\rho g}{\mu C_{drag}} A_f^2 \quad (C.5) \]

where \( A_f \) is the cross-sectional area of the flow channel and \( C_{drag} \) is a dimensionless drag coefficient.

The small rivulets that flow over the surface of the particles in the packed bed are not necessarily circular and therefore, the derivation of the dimensionless drag coefficient applicable to those rivulets is given in Section 4.5.4.2.

The relationship in equation C.5 comes from dimensionless analysis, but it can be demonstrated quite easily for other simple geometries.

For instance in a rivulet of circular cross section, the momentum balance over an increment in radius is as follows:

\[ 2\pi r \tau_r - 2\pi (r + \Delta r) \tau_{r+\Delta r} = 2\pi r \Delta \rho g \quad (C.6) \]
where $r$ is the radial variable that defines the radius of the circular rivulet and $\tau_r$ is shear stress.

Taking the limit as $\Delta r$ tends to zero:

$$\frac{1}{r} \frac{d(r\tau)}{dr} = \rho g$$  \hfill (C.7)

$$r\tau = \frac{1}{2} r^2 \rho g + C_1$$  \hfill (C.8)

where $C_1$ is a constant.

At $r = 0$, symmetry implies that $\tau = 0$. Therefore, $C_1 = 0$

$$\tau = \frac{1}{2} r \rho g$$  \hfill (C.9)

$$\mu \frac{dU}{dr} = -\tau$$  \hfill (C.10)

where $U$ is liquid velocity.

Equation C.10 becomes:

$$\mu \frac{dU}{dr} = - \frac{1}{2} r \rho g$$  \hfill (C.11)

$$U = - \frac{1}{4} r^2 \frac{\rho g}{\mu} + C_2$$  \hfill (C.12)

where $C_2$ is a constant.

At $r = R$, $U$ becomes $U = 0$. Therefore, $C_2 = \frac{1}{4} R^2 \frac{\rho g}{\mu}$

where $R$ is the radius of the circular rivulet.

$$U = \frac{1}{4} \frac{\rho g}{\mu} (R^2 - r^2)$$  \hfill (C.13)

The flow rate of a rivulet ($Q_r$) is given by:

$$Q_r = 2\pi \int_0^R U r dr$$  \hfill (C.14)

Integration of this equation from 0 to $R$, results:

$$Q_r = \frac{2\pi \rho g}{4 \mu} \int_0^R (R^2 - r^2) r dr$$  \hfill (C.15)

$$Q_r = \frac{2\pi \rho g}{4 \mu} \left[ \frac{R^4}{2} - \frac{R^4}{4} \right]$$  \hfill (C.16)

$$Q_r = \frac{\pi \rho g}{8\mu} R^4$$  \hfill (C.17)
However, for a relationship for the velocity of the rivulet, the flow rate of a rivulet \( Q_r \) is substituted by the multiplication of the area of the flowing fraction of the rivulet \( A_f \) and the velocity of the rivulet \( v_{rivulet} \). Further, \( R^4 \) can be replaced by \( A_f^2/\pi^2 \), if the shape of the rivulet is circular and the flow is laminar and vertical.

\[
A_f v_{rivulet} = \frac{\rho g}{8\pi\mu} A_f^2
\]  

(C.18)

Now equation C.18 becomes:

\[
v_{rivulet} = \frac{\rho g}{\mu C_{drag}} A_f
\]  

(C.19)

\[
Q_r = \frac{\rho g}{\mu C_{drag}} A_f^2
\]  

(C.20)

where \( C_{drag} \) is a dimensionless drag coefficient and equals to \( 8\pi \).

Equation C.20 is a special case of the Darcy-Weisbach law for circular cross sections and it is known as Poiseuille law.
APPENDIX D: DERIVATION OF THE AVERAGE LIQUID VELOCITY IN THE FILM FLOW

Assuming that the flow is down a thin film rather than rivulet, the dependency of flow on liquid content can be derived. Assuming that the film is thin compared to the radius of curvature of the particles, it can be treated as a flat layer:

\[ \frac{d\tau}{dx} = \rho g \]  \hspace{1cm} (D.1)
\[ \tau = \rho gx + C_3 \]  \hspace{1cm} (D.2)

At \( x = l' \), there is no shear stress and thus, \( \tau = 0 \). Therefore, \( C_3 = -\rho gl' \)

\[ \tau = \rho g(x - l') \]  \hspace{1cm} (D.3)
\[ \mu \frac{dU}{dx} = -\tau \]  \hspace{1cm} (D.4)

where \( \tau \) is shear stress, \( l' \) is the thickness of the film and \( U \) is the velocity of the liquid.

Now equation D.4 becomes:

\[ \mu \frac{dU}{dx} = -\rho g(x - l') \]  \hspace{1cm} (D.5)

\[ U = -\frac{\rho g}{\mu} \left( \frac{x^2}{2} - l'x \right) + C_4 \]  \hspace{1cm} (D.6)

At \( x = 0 \), there is no slip and thus, \( U = 0 \). Therefore, \( C_4 = 0 \)

\[ U = -\frac{\rho g}{\mu} \left( \frac{x^2}{2} - l'x \right) \]  \hspace{1cm} (D.7)

The flow rate down the film is given by equation:

\[ Q_{fim} = \int U dx W_0 \]  \hspace{1cm} (D.8)

where \( W_0 \) is the width of the liquid film.

Integration of this equation from 0 to \( l' \), results:

\[ Q_{fim} = -\frac{\rho g}{\mu} W_0 \int_0^{l'} \left( \frac{x^2}{2} - l'x \right) dx \]  \hspace{1cm} (D.9)

\[ Q_{fim} = -\frac{\rho g}{\mu} W_0 \left[ -\frac{l'^3}{3} \right] \]  \hspace{1cm} (D.10)

\[ Q_{fim} = \frac{\rho g}{3\mu} W_0 l'^3 \]  \hspace{1cm} (D.11)

\[ A_{fim} = W_0 l' \]  \hspace{1cm} (D.12)
where $A_{film}$ is the average cross-sectional area of the liquid film.

$$Q_{film} = \frac{\rho g A_{film}^3}{3\mu W_0^2} \quad (D.13)$$

where $W_0 \propto r_p$ (i.e. radius of the particle), assuming the film covers the particle. This means that the flow rate of the liquid film can be expressed as:

$$Q_{film} = \frac{\rho g A_{film}^3}{\mu c_{drag} W_0^2} \quad (D.14)$$

Therefore, the flow rate of the film has a cubic relationship with the average area of the liquid film.

$$Q_{film} \propto A_{film}^3 \quad (D.15)$$

Thus the film flow relationship is different from that of the rivulet flow through the particles of the packed bed.
APPENDIX E: DERIVATION OF A RELATIONSHIP FOR THE CAPILLARY PRESSURE

The dependency of the capillary pressure on the channel size and liquid content can be estimated as follows.

The capillary pressure is given by:

\[ P_c = -\frac{\gamma}{r_c} \]  \hspace{1cm} (E.1)

where \( \gamma \) is the surface tension of the liquid and \( r_c \) is the radius of the curvature. Note that as the water is generally a wetting phase, the capillary pressure is negative as interface will curve into the liquid phase (i.e. water lower pressure than air).

\[ r_c \propto \sqrt{A'} \]  \hspace{1cm} (E.2)

where \( A' \) is the area of the channel between the particles occupied by liquid.

Since the total area of the channel will be proportional to the square of the particle spacing, \( r_{ps} \), and the liquid holdup (\( \theta \)) is proportional to the ratio of the area occupied by liquid to the total area.

\[ \theta \propto \frac{A'}{r_{ps}^2} \]  \hspace{1cm} (E.3)

Using equations E.2 and E.3:

\[ \theta \propto \frac{r_c^2}{r_{ps}^2} \]  \hspace{1cm} (E.4)

Now the relationship for \( r_c \) becomes:

\[ r_c \propto r_{ps}\sqrt{\theta} \]  \hspace{1cm} (E.5)

Using equations E.1 and E.5, the relationship for the capillary pressure is:

\[ P_c \propto \frac{\gamma}{r_{ps}\sqrt{\theta}} \]  \hspace{1cm} (E.6)
APPENDIX F: JOURNAL ARTICLES


