PILOT SCALE STRUVITE RECOVERY POTENTIAL FROM CENTRATE AT LULU ISLAND WASTEWATER TREATMENT PLANT

By

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In this study, the workability of a newly designed, scaled up pilot-scale reactor was examined. The work was carried out at Lulu Island Wastewater Treatment Plant (LIWWTP) in Richmond, BC. One of the main objectives of the study was to demonstrate the ability of the reactor to remove at least 70% of the phosphate present in the centrate.

Results showed that the reactor was capable of removing, under controlled conditions, over 90% of phosphate and 4% of ammonia-nitrogen. Phosphate concentration in the effluent could be lowered to 5 mg/L. More than 85% of the phosphate removed was recovered as harvestable struvite pellets. The desired degree of phosphate removal was achieved by controlling the reactor supersaturation ratio. This ratio was in turn, controlled by varying the magnesium input and/or operating pH. Data collected indicated that it was possible to achieve over 90% P-removal at a pH of 7.5; this is contrary to the information found in literature, which recommends that a higher pH (8.2–9.0) is required. Factors that affected phosphate removal were the operating pH, the reactor SSR, the N:P and Mg:P molar ratios.

The determination of struvite solubility product with centrate and distilled water gave different values. The solubility product was dependent on the water tested, the pH of the solution and the temperature. The temperature coefficient and enthalpy at 25°C were 1.21 and 137.5 kJ/mol, respectively.

Analysis of the harvested product showed that the pellets were composed of nearly pure struvite (96% by weight), with small amounts of calcium and traces of aluminum and iron. ICP/MS testing of struvite samples found lower heavy metal content than that present in P-rock. The reactor SSR was determined to be the significant factor for pellet size; Mg:P molar ratio and upflow velocity determining the pellet hardness. Through the process, it was possible to grow pellets larger than 4.75 mm.

Two struvite models, developed by Potts and Britton at UBC, were used to predict the process performance along with the phosphate and ammonia effluent concentrations. The models were validated by comparing the predicted values with the actual operational data.
Comparison of P-removal efficiency, effluent phosphate and ammonia concentrations showed that the former was more accurate in the prediction. A trial was made by using an artificial neural network, Neusciences Neuframe® 4.0 model, to predict the effluent phosphate concentration. This model was found to be inefficient in its prediction, largely due to the small number of data used to train the model.
TABLE OF CONTENTS

ABSTRACT ........................................................................................................ II

TABLE OF CONTENTS .................................................................................. IV

LIST OF TABLES ............................................................................................ X

LIST OF FIGURES ........................................................................................ XI

LIST OF ABBREVIATIONS AND SYMBOLS ................................................... XIII

ACKNOWLEDGEMENTS ............................................................................ XIV

CHAPTER ONE: INTRODUCTION ............................................................... 1

1.1 Previous Research At UBC ................................................................. 1

1.2 Research Objectives ........................................................................... 2

1.3 Lulu Island Wastewater Treatment Plant ....................................... 3

CHAPTER TWO: BACKGROUND AND LITERATURE REVIEW .............. 4

2.1 Introduction ......................................................................................... 4

2.2 Motivation For Phosphorus Recovery ............................................ 4

2.2.1 Global phosphorus supply and demand ........................................ 4

2.2.2 Eutrophication .............................................................................. 5

2.2.3 Problems met in wastewater treatment plants ............................. 5

2.2.4 Economic considerations .............................................................. 5

2.3 Potential Sources For Phosphate Recovery ................................... 6

2.4 Potential Phosphorus Sources in British Columbia ...................... 6

2.5 Struvite Occurrence ........................................................................... 7

2.6 Problems Associated With Wastewater Treatment Plants Having BNR Processes ............................................................. 8

2.6.1 Re-solubilization of phosphorus during anaerobic digestion ....... 8

2.6.2 Struvite formation ........................................................................ 9

2.6.3 Excess sludge production ............................................................. 10

2.7 Phosphorus Removal From Wastewaters ...................................... 11

2.7.1 Biological nutrient removal ........................................................... 11
2.7.2 Chemical phosphorus precipitation .............................................................. 11
2.7.3 Combined biological/chemical treatment ..................................................... 11
2.7.4 Tertiary treatment ....................................................................................... 11

2.8 Methods of Phosphorus Recovery ................................................................. 12
2.8.1 Calcium phosphate precipitation ................................................................. 12
2.8.2 Struvite precipitation (magnesium ammonium phosphate) ....................... 13
2.8.3 Aluminium phosphate and iron phosphate (AlPO₄, FePO₄) ......................... 13
2.8.4 Membrane or ion exchange technologies followed by precipitation .......... 13

2.9 Parameters of P-Recovery .............................................................................. 14
2.9.1 pH Value .................................................................................................. 14
2.9.2 Magnesium addition ................................................................................... 14
2.9.3 Magnesium to phosphorus molar ratio ....................................................... 15
2.9.4 Ammonia to phosphorus molar ratio .......................................................... 15
2.9.5 Turbulence ................................................................................................. 16
2.9.6 Initial reactor seeding .................................................................................. 16
2.9.7 Temperature ............................................................................................... 17

2.10 Chemistry of Struvite .................................................................................. 17
2.10.1 Solubility Product ..................................................................................... 18
2.10.2 Conditional Solubility Product ................................................................. 19

2.11 Factors Affecting Solubility Product ............................................................ 20
2.11.1 pH ........................................................................................................... 20
2.11.2 Temperature ............................................................................................. 20
2.11.3 Impurity ions ........................................................................................... 21

2.12 Pellet Growth ............................................................................................... 22

2.13 Size of Struvite Pellets ................................................................................. 22

2.14 Struvite As A Resource ............................................................................... 22
2.14.1 Fertilizer value ......................................................................................... 22
2.14.2 Slow release fertilizer for rehabilitating oligotrophic streams ................. 23

2.15 Morphology Of Struvite Pellets ................................................................. 24

CHAPTER THREE: MATERIALS AND METHODS ............................................. 27

3.1 Centrate Characteristics ................................................................................. 27
3.2 Process Description ....................................................................................... 30
### TABLE OF CONTENTS

3.3 Materials And Equipment ................................................................. 31

3.3.1 Reactor ........................................................................ 31

3.3.2 Injection port ..................................................................... 33

3.3.3 Harvest zone ...................................................................... 34

3.3.4 Active zone ....................................................................... 34

3.3.5 Fines zone ......................................................................... 34

3.3.6 Clarifier/Seed hopper ....................................................... 35

3.3.7 External clarifier ................................................................. 35

3.4 Chemicals, Storage Tanks and Pumps .......................................... 35

3.4.1 Centrate ........................................................................ 35

3.4.2 Magnesium feed ................................................................ 36

3.4.3 pH control ........................................................................ 36

3.5 Process Monitoring And Maintenance ........................................ 37

3.6 Sample Collection, Storage And Preservation .............................. 38

3.7 Analytical Methods .................................................................. 39

3.7.1 Magnesium ................................................................. 39

3.7.2 Ortho-phosphate ............................................................ 39

3.7.3 Ammonia ......................................................................... 40

3.7.4 Calcium, Aluminum and Iron ............................................ 40

3.7.5 Total Phosphorus .............................................................. 40

3.7.6 Filtration .......................................................................... 40

3.7.7 Conductivity .................................................................... 40

3.8 Pellet Harvest, Drying and Analysis ........................................... 40

3.9 Pellet Quality Determination ...................................................... 41

3.9.1 Composition And Purity ..................................................... 41

3.9.2 Hardness .......................................................................... 41

3.9.3 Density ............................................................................ 42

3.9.4 Morphology of struvite pellets ......................................... 42

3.9.5 Charge of pellets ............................................................. 42

3.10 Terminology ........................................................................... 43

3.10.1 Struvite solubility product (Ksp) ....................................... 43

3.10.2 Struvite conditional solubility product ............................... 44

3.10.3 Supersaturation ratio ....................................................... 45
## TABLE OF CONTENTS

<table>
<thead>
<tr>
<th>Section</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>3.10.4 Inlet supersaturation ratio</td>
<td>45</td>
</tr>
<tr>
<td>3.10.5 Reactor supersaturation ratio</td>
<td>45</td>
</tr>
<tr>
<td>3.10.6 Recycle ratio</td>
<td>46</td>
</tr>
<tr>
<td>3.10.7 Crystal retention time</td>
<td>46</td>
</tr>
<tr>
<td>3.10.8 Mean crystal size</td>
<td>46</td>
</tr>
<tr>
<td>3.10.9 Removal efficiency</td>
<td>47</td>
</tr>
<tr>
<td>3.11 Struvite Solubility Determination</td>
<td>48</td>
</tr>
<tr>
<td>3.11.1 Apparatus</td>
<td>48</td>
</tr>
<tr>
<td>3.11.2 Sampling</td>
<td>49</td>
</tr>
<tr>
<td>3.12 Process Control Models</td>
<td>49</td>
</tr>
<tr>
<td>3.12.1 Potts’ crystallizer model</td>
<td>49</td>
</tr>
<tr>
<td>3.12.2 Britton’s struvite equilibrium model</td>
<td>50</td>
</tr>
<tr>
<td>3.12.3 Neuscienches Neuframe 4.0</td>
<td>50</td>
</tr>
<tr>
<td>3.13 Model Evaluation</td>
<td>52</td>
</tr>
<tr>
<td><strong>CHAPTER FOUR: RESULTS AND DISCUSSION</strong></td>
<td>53</td>
</tr>
<tr>
<td>4.1 Operating Conditions</td>
<td>53</td>
</tr>
<tr>
<td>4.1.1 Phosphate Levels</td>
<td>54</td>
</tr>
<tr>
<td>4.2 Chemistry of Struvite</td>
<td>55</td>
</tr>
<tr>
<td>4.2.1 Struvite Solubility Product</td>
<td>55</td>
</tr>
<tr>
<td>4.2.2 Temperature Coefficient (θ) And Enthalpy (ΔH)</td>
<td>57</td>
</tr>
<tr>
<td>4.2.3 Conditional Solubility Product</td>
<td>57</td>
</tr>
<tr>
<td>4.3 Performance Of The Process</td>
<td>59</td>
</tr>
<tr>
<td>4.3.1 Phosphate removal efficiency</td>
<td>59</td>
</tr>
<tr>
<td>4.3.2 Ammonia removal</td>
<td>60</td>
</tr>
<tr>
<td>4.3.3 Total phosphorus and ammonia</td>
<td>62</td>
</tr>
<tr>
<td>4.3.4 Struvite recovery efficiency</td>
<td>63</td>
</tr>
<tr>
<td>4.4 Factors Affecting P-Removal</td>
<td>64</td>
</tr>
<tr>
<td>4.4.1 Effect of pH</td>
<td>64</td>
</tr>
<tr>
<td>4.4.2 Effect of inlet SSR</td>
<td>65</td>
</tr>
<tr>
<td>4.4.3 Effect of reactor SSR</td>
<td>65</td>
</tr>
<tr>
<td>4.4.4 Effect of magnesium to phosphorus molar ratio</td>
<td>66</td>
</tr>
<tr>
<td>4.4.5 Effect of ammonia to phosphorus molar ratio</td>
<td>68</td>
</tr>
</tbody>
</table>
# TABLE OF CONTENTS

<table>
<thead>
<tr>
<th>Appendix</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>APPENDIX H: CHEMICAL ANALYSIS OF STRUVITE PELLETS</td>
<td>145</td>
</tr>
<tr>
<td>APPENDIX I: TOTAL PHOSPHORUS AND NITROGEN</td>
<td>151</td>
</tr>
<tr>
<td>APPENDIX J: MODEL RESULTS</td>
<td>152</td>
</tr>
</tbody>
</table>
LIST OF TABLES

Table 2.1. Sources of magnesium with their corresponding pH values ........................................ 15
Table 2.2. Side reactions involved in struvite formation ................................................................. 18
Table 3.1 Centrate characteristics – January to April 2004 ............................................................. 27
Table 3.2. Centrate characteristics – June 2004 ........................................................................... 27
Table 3.3. Dimensions of the reactor .............................................................................................. 31
Table 3.4. Upflow velocities and Reynolds number in different sections of the reactor ................. 32
Table 3.5. Equilibrium Constants at various temperatures ............................................................... 44
Table 3.6. Neuframe 5-layer neural network details .................................................................... 51
Table 4.1. Range of operational conditions and results summary ................................................. 53
Table 4.2. Struvite recovery ............................................................................................................ 63
Table 4.3. Summary of pellet composition analysis ..................................................................... 71
Table 4.4. Impurity content of pellet ............................................................................................. 72
Table 4.5. Comparison of heavy metal content in different product ............................................. 72
Table 4.6. Summary of model results ............................................................................................ 84
Table 4.7. Cost of struvite production ............................................................................................ 87
LIST OF FIGURES

Figure 2.1. Struvite accumulation in a centrate liquor pipe .................................................. 10
Figure 2.2. Struvite crystal as seen using a SEM ................................................................. 25
Figure 3.1. Centrate characteristics, (PO₄-P concentration) – January to April 2004 ............. 28
Figure 3.2. Centrate characteristics (NH₄-N concentrations) – January to April 2004 .......... 28
Figure 3.3. Centrate characteristics (PO₄-P concentrations) – June 2004 ............................ 29
Figure 3.4. Centrate characteristics (NH₄-N concentrations) – June 2004 ......................... 29
Figure 3.5. Pilot-scale struvite crystallizer reactor process design ....................................... 30
Figure 3.6. Sketch of the pilot-scale struvite crystallizer injection port assembly .................. 33
Figure 3.7. Pilot-scale struvite crystallizer injection port assembly. (a) centrate and recycle ports, (b) magnesium chloride and caustic ports ......................................................... 34
Figure 3.8. Set-up of the study area ................................................................................. 37
Figure 3.9. Setup of solubility determination ..................................................................... 49
Figure 4.1. Phosphate levels in centrate and effluent (Run1) ................................................. 54
Figure 4.2. Phosphate levels in centrate and effluent (Run2) ............................................... 55
Figure 4.3. Solubility product of struvite in centrate at 20 °C ............................................. 56
Figure 4.4. Solubility product of struvite in centrate and distilled water at 25 °C .......... 56
Figure 4.5. Struvite solubility product at different temperatures .......................................... 57
Figure 4.6. Conditional solubility product of struvite in centrate at 20 °C ......................... 58
Figure 4.7. Conditional solubility product of struvite in centrate and distilled water at 25 °C . 59
Figure 4.8. Percentage phosphate removal (Run1) ......................................................... 60
Figure 4.9. Percentage phosphate removal (Run2) ............................................................ 60
Figure 4.10. Percentage ammonia removal (Run1) .......................................................... 61
Figure 4.11. Percentage ammonia removal (Run2) ............................................................ 62
Figure 4.12. TKN and TP removal efficiency ................................................................. 62
Figure 4.13. Effect of pH on P-removal ................................................................. 64
Figure 4.14. Effect of SSR at the inlet on P-removal .......................................................... 65
Figure 4.15. Effect of reactor SSR on P-removal ............................................................. 66
Figure 4.16. Relation between Mg:P molar ratio and P-removal efficiency (Feb 29-March 12, 2004) .............................................................. 67
Figure 4.17. Relation between Mg:P molar ratio and P-removal efficiency .................. 67
Figure 4.18. Relation between N:P molar ratio and P-removal efficiency ...................... 68
Figure 4.19. Mean pellet size harvested (Run 1) ..................................................... 70
Figure 4.20. Mean pellet size harvested (Run 2) ..................................................... 70
Figure 4.21. Relation between pellet size and reactor SSR ......................................... 71
Figure 4.22. SEM images of struvite surface harvested in June 2004 ......................... 75
Figure 4.23. SEM image of magnified (×800) surface of a 1 mm< D< 2 mm pellet harvested in June, 2004 ..................................................... 76
Figure 4.24. Cut SEM images (×300) of pellets harvested ......................................... 77
Figure 4.25. SEM image of pellet harvested on 28th January, 2004 .......................... 78
Figure 4.26. Struvite loading rate (Run 1) .............................................................. 80
Figure 4.27. Struvite loading rate (Run 2) .............................................................. 81
Figure 4.28. Predicted and actual P-removal efficiency using 2 different models .......... 84
Figure 4.29. Predicted and actual effluent PO$_4$ concentration using 2 different models 85
Figure 4.30. Predicted and actual effluent NH$_4$-N concentration using 2 different models 85
Figure 4.31. Predicted and actual effluent PO$_4$-P concentration using Neusciences Neuframe 4.0 model ........................................................................ 86
### LIST OF ABBREVIATIONS AND SYMBOLS

<table>
<thead>
<tr>
<th>Abbreviation</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>ANN</td>
<td>Artificial Neural Network</td>
</tr>
<tr>
<td>AWWTP</td>
<td>Advanced Wastewater Treatment Plant</td>
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<tr>
<td>BC</td>
<td>British Columbia</td>
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<tr>
<td>BOD</td>
<td>Biochemical Oxygen Demand</td>
</tr>
<tr>
<td>CRT</td>
<td>Crystal Retention Time</td>
</tr>
<tr>
<td>(E)BNR</td>
<td>(Enhanced) Biological Nutrient Removal</td>
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<tr>
<td>EC</td>
<td>Electric Conductivity (mS/cm)</td>
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<tr>
<td>HRT</td>
<td>Hydraulic Retention Time (min)</td>
</tr>
<tr>
<td>ICP</td>
<td>Inductive Coupled Plasma</td>
</tr>
<tr>
<td>K</td>
<td>Kelvin, Unit of Temperature</td>
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<tr>
<td>Ksp</td>
<td>Solubility Product</td>
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<tr>
<td>LIWWTP</td>
<td>Lulu Island Wastewater Treatment Plant</td>
</tr>
<tr>
<td>MAP</td>
<td>Magnesium Ammonium Phosphate Hexahydrate; Struvite</td>
</tr>
<tr>
<td>MS</td>
<td>Mass Spectrophotometer</td>
</tr>
<tr>
<td>Ps</td>
<td>Conditional Solubility Product</td>
</tr>
<tr>
<td>R</td>
<td>Ideal Gas Constant (8.314 J/mol.K)</td>
</tr>
<tr>
<td>Re</td>
<td>Reynolds Number</td>
</tr>
<tr>
<td>RR</td>
<td>Recycle Ratio</td>
</tr>
<tr>
<td>SEM</td>
<td>Scanning Electron Microscope</td>
</tr>
<tr>
<td>SSD</td>
<td>Saturated, Surface Dry</td>
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<tr>
<td>SSR</td>
<td>Supersaturation Ratio</td>
</tr>
<tr>
<td>UBC</td>
<td>University of British Columbia</td>
</tr>
<tr>
<td>WWTP</td>
<td>Wastewater Treatment Plant</td>
</tr>
<tr>
<td>ΔH</td>
<td>Enthalpy Change (J/mol)</td>
</tr>
</tbody>
</table>
I would like to acknowledge the assistance, support and encouragement that I have received throughout my research. Without their cooperation, this study would not be possible.

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1.1 Previous Research At UBC

In 1999, the Department of Civil Engineering at the University of British Columbia started a phosphorus recovery project in collaboration with BC Hydro. One of the driving forces behind this project was the important principle of sustainability (Adnan, 2002). Since 1999, six (including the author) graduate students have been involved with this project. While some of the research work done in this field at UBC has been with bench to pilot scale reactors involving synthetic feed, studies have also been undertaken using real supernatant from treatment plants. Of the six students, two of them worked with synthetic wastewater while three looked into the process of phosphorus recovery from real supernatant. Research carried out by the author involved the use of centrate for phosphorus recovery. The studies were conducted at the UBC Pilot Plant, City of Penticton Advanced Wastewater Treatment Plant (AWWTP) and Lulu Island Wastewater Treatment Plant (WWTP).

Initially, the reactor designed at UBC was used at the bench scale with synthetic wastewater. The equipment was found to be prone to plugging problems, had low recoveries of phosphorus and the recovered struvite crystals were of poor quality (Fred Koch, Environmental Engineering Group, Department of Civil Engineering, UBC, Vancouver, B.C., Canada). After the initial work at the bench scale, some of the problems involving the small reactor were largely rectified with the scale-up of the reactor to approximately 20-25 L in volume. For three years, three students were involved with research using this reactor. One of the graduate students, Ahren Britton, used two reactors at Penticton using real anaerobic digester supernatant having ortho-phosphate concentrations in the range 37 to 71 mg/L. By controlling the operating pH of the reactors and the inlet supersaturation ratio, he was able to achieve 80% removal of the phosphate with up to 91% recovery of the removed phosphorus (Britton, 2002). In 2001, Ali Adnan used a similar reactor, using synthetic feed at the UBC Pilot Plant. He was able to achieve ortho-P removal rates of over 90% for a range (47 mg/L to 220 mg/L) of influent P concentrations. About 80% of the phosphate removed was recovered as struvite, with pellets
having an average mean size of over 2 mm. The reactor supersaturation ratio and the crystal retention time (CRT) were identified as the major factors affecting mean crystal size (Adnan, 2002). In 2002, Hui Huang investigated the recovery of phosphorus from anaerobic digester supernatant from the two treatment plants, as well as synthetic supernatant with high phosphate concentration (100-190 mg/L). Her study showed that the process was capable of removing more than 90% of ortho-phosphate from both the synthetic supernatant and the digester supernatants, with approximately 90% of the removed phosphate recovered as harvestable struvite pellets (Huang, 2003). In 2003, three reactors were built that had a volume of approximately four times that of the previously scaled-up reactor. These reactors, having a volume of about 50 liters, were used at the two treatment plants mentioned above.

1.2 Research Objectives

Although a major component for the recovery of phosphorus in the form of struvite is the chemistry involved, more emphasis on the engineering part of the process will be given in this study. The primary objectives of the research will be

1. To determine the conditions for struvite crystallization and recovery of phosphate from centrate at Lulu Island Wastewater Treatment Plant. At least 70% reduction of phosphate from the centrate and 80% phosphate recovery in the form of struvite was expected at the end of the project.

2. To identify the engineering factors which affect struvite growth, such as loading rates and upflow velocities.

3. Investigating the factors that affect the phosphate removal efficiency.

4. To verify the effectiveness of two struvite crystallization models and an artificial neural network.
1.3  Lulu Island Wastewater Treatment Plant

The Lulu Island Wastewater Treatment Plant (LIWWTP) is a secondary wastewater treatment plant that is operated by the Greater Vancouver Regional District of the Province of British Columbia. Opened in 1973, the plant only had primary treatment until 1988 when the treatment capacity was increased by 50%. The plant was later upgraded to secondary treatment in 1999 and presently serves around 165,000 residents of Richmond, with an average daily flow of 47 million liters (Internet). The plant consists of physical treatment that includes screening, grit removal and primary sedimentation and biological treatment that includes trickling filters, solid contact and secondary clarification. The sludge produced is managed through three processes namely sludge thickening, anaerobic digestion and biosolids dewatering. Solids removed from the primary settling tanks are thickened in a gravity thickener while those from the secondary process are thickened using a dissolved air flotation process. Sludge digestion is accomplished using a mesophilic anaerobic process, which takes place at a temperature of about 35°C. The digestion process reduces the volatile solids in the sludge by about 40 to 60 percent, thereby reducing the amount of sludge to dispose of and making it more stable. The digested sludge, normally having a concentration of about 2%, is mechanically dewatered to decrease the sludge moisture content. The sludge is dewatered in the centrifuge to solids concentration of 25 to 30%. The secondary treated effluent is discharged via a diffuser under the Fraser River south of the plant.

Due to the high concentrations of phosphorus, ammonia and some magnesium in the liquid centrate during the anaerobic digestion process, there is a probability of struvite encrustation in the digester and the centrate lines. Although problems relating to struvite are not common at LIWWTP, there was an occurrence of struvite precipitation in the centrate line to the headworks. The problem was subsequently solved by dilution with effluent water (Brian Hystad, Process Control & Process Systems Engineering Supervisor, Wastewater Treatment Plants Division, Greater Vancouver Regional District, pers. comm.).
CHAPTER TWO
BACKGROUND AND LITERATURE REVIEW

2.1 Introduction

Phosphorus is essential to all life forms because it is a key, irreplaceable element in many physiological and biochemical processes in plants and animals. Yet, of the elements required by plants in the largest amounts, namely nitrogen, phosphorus, potassium, sulphur, calcium and magnesium, phosphorus is the non-renewable resource with the least reserves/resources globally. Phosphorus compounds are present in every domestic wastewater, originating from detergents as well as from metabolism processes, diffuse runoff from agricultural land and inputs from the air. Even at low concentrations of approximately 10μg P/L secondary reactions, also known as eutrophication processes, may occur. Thus, many water bodies in the world are experiencing an increased number of algal blooms. This bloom reduces the amenity value and ecological health of water bodies such as lakes, slow moving rivers and drinking water reservoirs. Apart from an internal release from the sediments (Hart et al., 2002) the main phosphorus source comes from the catchments inputs.

2.2 Motivation For Phosphorus Recovery

2.2.1 Global phosphorus supply and demand

The commercial source of phosphorus is “phosphate rock”, the collective name given to natural calcium phosphates of various forms. Around 38 million tonnes of phosphate (expressed as P₂O₅) are extracted each year (Driver et al., 1999). Although phosphate rocks are present throughout the earth’s crust, most of them are low in phosphate content. Over the last 20 years, the highest-grade deposits have been heavily exploited and are being rapidly depleted. The known reserves of phosphate rock are also limited. The most alarming thing is that the resource base, which could be commercially exploited by methods currently regarded as economic, will last, at best, little more than one hundred years and could be depleted in as little as fifty years (Driver et al. 1999). However, the depletion is not the only concern for the phosphorus resource; the more immediate concern is the decreasing quality of phosphate rocks. The metallic content
of the rocks, such as cadmium, uranium, nickel, chromium, copper and zinc, are increasing steadily. These impurities may make the use of the rocks unacceptable.

The phosphate industry is now seeking a sustainable source of high purity raw material. As a result of agriculture production and consumption, two major sources of phosphorus are now available: human sewage and animal manure and slurries. For the phosphorus to be useful, it must be in a form which is both technically and economically recoverable. Since there is no collection system for animal wastes, it is logical to recover phosphorus from sewage first and develop a technology for further recovery and production.

2.2.2 Eutrophication

Among the nutrients which cause eutrophication and algal growth in freshwaters, phosphorus is, in most cases, the limiting nutrient. Low P concentrations in lakes, reservoirs and rivers have often led to the decrease in its aesthetic values due to biological productivity. In certain conditions, P concentrations as low as 0.01 mg/L can be critical to initiate eutrophication (Lee, 1970). On the other hand, domestic wastewaters can often contain 4 to 15 mg/L of phosphorus (Tchobanoglous et al., 2003). The removal of compounds containing nitrogen and phosphorus is a key element of the European Commission’s Urban Wastewater Treatment Directive (UWWTD) and is intended to reduce and prevent eutrophication of sensitive inland and coastal waters. As a result of human activity, it was estimated that more than 12,000 tonnes of P entered fresh, ground and coastal waters of Canada in 1996. The largest point source was municipal sewage, which added an estimated 5600 tonnes of P (Chambers et al., 2001). In general, the current discharge limit on total phosphorus in North America ranges from 2 to 0.1 mg/L (Tchobanoglous et al., 2003).

2.2.3 Problems met in wastewater treatment plants

This part has been discussed in detail in Section 2.6.

2.2.4 Economic considerations

Two basic considerations may make P-recovery an efficient and sustainable process in terms of economic impetus. The first is the potential for cost savings, in terms of reduced chemical
additions in treatment plants and sludge handling costs. The second is the potential for cost recovery, via the sale of recovered phosphate product.

The economic implication of the sludge volume reduction is dependant on the sludge disposal costs. There is a general consensus among wastewater experts that the cost of sludge disposal will raise significantly in the coming years, because of limitations to agricultural spreading of sludge. Various factors mitigating against agricultural spreading include geographical concentration of sludge production near cities (resulting in long transport distances to farmland), contamination of sludge with chemicals and heavy metals, opposition of residents and specifications in food companies and supermarket quality requirements for the farmers (Woods et al., 1999).

The recovered product may be used either as a source of phosphorus (instead of phosphate rocks) or as a fertilizer (in case of struvite precipitation). Most of the recovered product is of higher purity than the rocks and contain less heavy metals, a factor that may increase the value of the product. Struvite has been found to be a good fertilizer because it can release nutrients slowly. After treatment, the recovered product can also be used as cleaning products, chemicals and fire retardants (Battistoni et al., 2002). In 2001, Unitika Ltd. (Japan) sold their struvite product at about US$ 276 per tonnes. Munch and Barr made a preliminary market research in Australia and determined that it was possible to achieve a price of US$198-380 per tonnes of struvite.

2.3 Potential Sources For Phosphate Recovery

Both human and animal wastes have great potential for phosphorus recycling. Although the nutrient contents of animal manures are higher that in human sewage, most of the recycling to date has been with the latter. One of the reasons for this is the lack of proper collection systems for animal wastes.

2.4 Potential Phosphorus Sources in British Columbia

The bulk of potentially recoverable phosphate in British Columbia (BC) will likely come from agricultural sources in the long run. But until regulations are put into place to limit nutrient
discharges from farms, it is unlikely that the agricultural industry will invest in the necessary infrastructure to support phosphorous recovery. In BC, the total phosphorus in sewage is estimated to be about 3048 tonnes/year (Hall et al., 2001). According to a recent study, 62% of this can be recovered (Yu, 2001). It is currently estimated that about 1900 tonnes of phosphorous are amenable for recovery per year from municipal sewage in British Columbia and about 10,000 tonnes of phosphorous are recoverable from agricultural wastes (Yu, 2001). The total phosphorus generated by all livestock in BC is about 19000 tonnes/year, 54% of which could be recovered (Yu, 2001).

2.5 Struvite Occurrence

Magnesium ammonium phosphate (MAP) hexahydrate (\(\text{MgNH}_4\text{PO}_4\cdot6\text{H}_2\text{O}\)), more commonly known as struvite, is a relatively rare mineral whose important natural source is rotting organic material such as guano deposits and cow manure (Nelson et al., 1991). It has also been observed in sludges derived from the anaerobic digestion of animal farming liquid wastes (Booram et al., 1975) and agricultural wastes. As well as being found in biologically treated wastewater sludge, struvite has been recognized as a common constituent of renal and vesical calculi of both humans and animals (Abbona et al., 1982; Suzuki et al., 1997).

Struvite precipitation was first noticed by wastewater engineers in the Hyperion wastewater treatment plant in Los Angeles in the 1960s. It formed in the digested sludge lines, as well as separating screens, and caused operational problems by clogging pipes or freezing valves (Borgerding 1972). In wastewater treatment, precipitation can lead to capacity loss by compromising process control and negatively affect the performance of sludge and filtrate conveyance system (Benisch et al., 2002). Struvite has also been found in anaerobic sludges from liquid animal production wastes (Booram et al., 1975).

Accumulation of struvite (\(\text{MgNH}_4\text{PO}_4\cdot6\text{H}_2\text{O}\)) on pipe walls and equipment surfaces of anaerobic digestion and post digestion processes is a problem that plagues the wastewater treatment industry. Struvite is well known for plugging pipes and fouling pumps, aerators, screens and other equipment. Remediation is often impractical and, when possible, is costly in terms of labor, materials and system downtime. The pellets anchor to sludge particles in
suspension and to surfaces of equipment, tankage and pipe walls in contact with digestion sludge (Borgerding, 1972). Pellet growth can be rapid and, if left unchecked, struvite accumulation can foul mechanical equipment and clog pipes within months (Westerman et al., 1985; Mohajit et al., 1989).

Although unintentional struvite formation has long been known to be a nuisance in wastewater treatment plants (Munch and Barr, 2001), struvite precipitation can also serve as a process for removing and recovering phosphorus from wastewater streams. Struvite precipitation is attractive for the following reasons.

- Two nutrients (P and N) can be simultaneously removed
- Process is well suited to wastewaters with concentrated nutrients (Dempsey, 1997)
- The process product, MAP, is a premium slow-release fertilizer recycling phosphorus resources and can be marketable (Munch and Barr, 2001)

2.6 Problems Associated With Wastewater Treatment Plants Having BNR Processes

In recent times, more stringent regulations on nutrient discharge to water bodies have brought about the growth of several biological nutrient removal (BNR) plants throughout the world. However, phosphorus removal may prove to be difficult because of its release during either sludge handling or anaerobic digestion. Consequently, three main operational problems arise in treatment plants where nutrient removal is carried out. These are the re-solubilization of biologically removed phosphate during anaerobic digestion, the scaling or encrustation of piping and equipment and the production of excess sludge volumes, when the phosphate is re-precipitated by chemicals.

2.6.1 Re-solubilization of phosphorus during anaerobic digestion

When waste activated sludge from a BNR plant is digested anaerobically, much of the phosphates which were removed in the main treatment train are re-released under the anaerobic conditions. Various studies show that 26% to 90% of the phosphorus entering the head of the treatment plant is due to phosphorus feedback, that is, phosphorus in the return liquors (Mavinic et al., 1998; Niedbala, 1995; Jaffer et al., 2002). Some plants have even reported additional
BACKGROUND AND LITERATURE REVIEW

phosphorus loads of up to 100% (Pitman et al., 1991). Consequently, the phosphorus circulates in a loop within the treatment system, thus increasing the P load. The optimum operation of a BNR process depends heavily on the BOD:P ratio of the wastewater; below a critical BOD:P ratio, a potential system failure can occur (Mavinic et al., 1998). As a result of the recirculation of P to the headworks, BNR process performance decreases.

2.6.2 Struvite formation

A major problem in many wastewater treatment plants is the accumulation of struvite in various sections of the treatment system. A number of treatment plants have reported the occurrence of struvite precipitation in piping and other equipments (e.g. digestion tanks, pumps, valves, etc) (Ohlinger et al., 1998; Webb and Ho, 1992; Booram et al., 1975). Problems related to struvite are especially evident in areas of high turbulence, such as pump impellers and pipe bends (Jaffer et al., 2002; Ohlinger et al., 1999). Struvite precipitation occurs when the combined concentration of Mg\(^{2+}\), NH\(_4^+\) and PO\(_4^{3-}\) exceed the struvite solubility product. The precipitation is also dependent on the pH and the total dissolved concentrations of the three ionic species (Ohlinger et al., 1998). The struvite deposits are hard and often difficult to dislodge, thus needing replacement of the encrusted parts. Struvite accumulation causes damage to pumping systems, reduce the plant flow capacity and have even contributed to major plugging of piping. There have been instances, like at the Slough WWTP in England, where struvite formation has plugged piping systems within the first year of plant operation (Williams, 1999). Although several remedial measures have been suggested (Williams, 1999; Mamais et al., 1994), they are often costly and time consuming. Also, the methods only serve to alleviate the problem without actually eliminating it completely. It was reported that the annual costs for a mid-size treatment plant (25 MGD) related to struvite accumulation exceeded 100,000 US dollars (Doyle and Parsons, 2002). Figure 2.1 shows the severity of struvite accumulation problem where the bore of the pipe reduced to 60 mm from 150 mm over 12 weeks.
2.6.3 Excess sludge production

Phosphorus removal by enhanced BNR (EBNR), or chemical processes increase the sludge production at wastewater treatment plants. Paul et al. (2001) estimated that the average increase in sludge production from biological and chemical phosphorous removal in France was 3 kg of solids per kg of phosphorus removed, or 5 percent of the total sludge production.

There are, however, different opinions on whether P-recovery results in reduction of total sludge production. Jeanmaire (2001) interviewed several specialists and two different opinions were voiced: (1) P-recovery does not reduce sludge production (Day, M., Jaffer, Y., Pearce, P., Heinze, B., Battistoni, P., Nauleau, F and Cretenot) and (2) P-recovery may cause a sludge reduction less than 10% of the total production, but the reduction is unlikely to be visible, given the usual fluctuations that happen almost everyday in sludge production (Klapwijk, B., Roeleveld, P., Churchley, J., Vachon, A., Audic, J.M., Vander, B.H. and Hahn, H.).

In a feasibility study, Woods et al. (1999) estimated that sludge volumes could be reduced by up to 30% (compared to EBNR) and up to 49% (compared to chemical phosphorus precipitation). Another study conducted by Jeanmaire and Evans (2001) concluded that a decrease in sludge mass of 2-8% could be expected, if phosphorous recovery was undertaken at an operating BNR facility with anaerobic sludge digestion.
2.7 Phosphorus Removal From Wastewaters

There are four conventional methods typically applied at municipal and industrial wastewater treatment facilities to remove phosphorus (Sedlak, 1992; WEF, 1998). These are biological nutrient removal, chemical phosphorus precipitation, combined biological/chemical treatment and tertiary treatment.

2.7.1 Biological nutrient removal

In this method, phosphorus is incorporated into the biomass and removed via the waste-activated sludge. In EBNR systems, the provision of an anaerobic zone in the activated sludge bioreactor allows enhanced removal of phosphorus, beyond that needed for biomass synthesis, and compliance with more stringent regulatory permit levels. These EBNR systems also often provide nitrogen removal. In conventional activated sludge plant, bacteria use phosphorus for their metabolic needs. Typical removal rates of 20% to 40% can be achieved through this process (Brett et al., 1997).

2.7.2 Chemical phosphorus precipitation

Precipitating agents (typically ferric chloride, alum and other metal salts) are added at various points in the conventional wastewater treatment train to convert soluble phosphate to a particulate form. The precipitated phosphate is then removed with the waste sludge. Chemical precipitation of phosphorus is a simple and reliable method, and is, therefore, still widely used in North America (Black and Veatch, 1971).

2.7.3 Combined biological/chemical treatment

This combination is often applied to meet more stringent criteria.

2.7.4 Tertiary treatment

Metal salts or lime are added to precipitate phosphorus following conventional treatment. This technology is rarely used, but may be employed to meet very stringent limits (Woods et al., 1999).
Although chemical precipitation is a simple and reliable method, the present trend is more toward the BNR process. Some of the reasons for choosing the BNR process are (Stratful et al., 1999; Paul et al., 2001; Suschka et al., 2001; Edge, 1999):

- The cost of flocculants is increasing and with more stringent discharge regulations, the cost of chemical phosphorus removal could become very high.
- Addition of aluminium and ferric salts as coagulants has, in some cases, resulted in unacceptable concentrations of these cations in the final effluent.
- The additional sludge production from the addition of chemicals is avoided, along with its consequent sludge disposal problems.
- Sludges from biological phosphorus removal would be higher in “plant-available” phosphorus and could make better agricultural fertilizers.

2.8 Methods of Phosphorus Recovery

Most phosphorus recovery technologies are currently in the research and development stage and few have been implemented on a full-scale basis (Woods et al., 1999). Potential technologies for phosphorus recovery from wastewater include: calcium phosphate precipitation, struvite (magnesium ammonium phosphate) precipitation, aluminum and iron phosphates and membrane or ion exchange technologies, followed by precipitation.

2.8.1 Calcium phosphate precipitation

Many other forms (like dicalcic phosphate, apatite hydroxyl etc.) can be obtained depending on the Ca/P ratios and the conditions of the reaction. From the industry’s point of view, phosphorus recovery as calcium phosphate is more promising (Driver et al., 1999). Calcium phosphates of a suitable physical form can be extracted from sewage. The best known example of this method is the Geestmerambacht sewage treatment works in the Netherlands, which is running a fluidized bed reactor (the DHV Crystalactor™) to recover calcium phosphate as a pellet. These pellets are formed around a seed particle of silica sand (Seckler et al., 1996). The calcium phosphate is recovered as small pellets with up to 11% P content, which dry and handle easily (5-10% water). The forms produced are very close to the forms found in
the mined phosphate and can thus be recycled by existing thermal or wet route processes of the P-industry (Driver et al., 1999).

2.8.2 **Struvite precipitation (magnesium ammonium phosphate)**

Struvite cannot be used in the P-industry’s existing processes but can be used as a fertilizer, either directly or in combination with other products (Jeanmaire, 2001). An example of this technology is the Unitika Phosnix Process. In this process, wastewater is fed into the base of the reactor where it is mixed with magnesium chloride to achieve a desired Mg:P molar ratio. There are a number of full-scale and pilot-scale P-recovery operations based on Unitika Phosnix Process (CEEP, 2001).

2.8.3 **Aluminium phosphate and iron phosphate (AIPO₄, FePO₄)**

The AIPO₄ form can only be recycled in the P-industry using the thermal route. In Europe, the only remaining plant using this process is Thermphos International located in Vlissigen, Holland. The FePO₄ form is more common because ferric chloride is most widely used in Europe for the physico-chemical P-removal. Iron phosphates cannot be used in existing P-industry processes and probably have low or zero fertilizer value (Jeanmaire, 2001).

2.8.4 **Membrane or ion exchange technologies followed by precipitation**

An example of this technology is the REM-NUT process using ion exchange, followed by struvite precipitation. This process removes phosphate and ammonia ions from tertiary wastewater. The process consists of three stages-two columns of cationic resin, two columns of ionic resin and the nutrient precipitation process.

In short, we can say that there are basically three main reasons which make the recovery of phosphorus attractive. They are:

- Phosphorus is an essential nutrient for all forms of life and a key element for many physiological and biological processes. It cannot be replaced by another element (Steen, 1998).
- The phosphate rocks reserves are dwindling, so recycling of the phosphorus is a real matter of concern.
• The recovery of phosphorus is very much compatible with biological phosphorus removal, so in order to recover phosphorus, one does not need to go out of the way in terms of installation of a new facility. In fact, it is only an advanced step of biological phosphorus removal. Recovery of phosphorus would not only be useful for phosphorus recycling, but this technique would provide the possibility of producing less sludge in a biological nutrient removal plant (Woods et al., 1999).

2.9 Parameters of P-Recovery

2.9.1 pH Value

Struvite is soluble in acidic conditions and highly insoluble in alkaline solution, with minimum solubility occurring at pH 10.3 (Ohlinger et al., 1998). One of the key parameters in the crystallization process is the saturated condition of the solution. In case of struvite, the solution can be saturated either by increasing the pH or by increasing the struvite constituent ions, but the former method seems to be more preferable for running the process. For struvite crystallization from municipal wastewater, the pH can be increased either by the addition of caustic (Jaffer et al., 2002; Kumashiro et al., 2001; Celen and Turker, 2001) or by CO₂ air stripping (Ohlinger et al., 2000; Munch and Barr, 2001; Battistoni et al., 2001). A pH in the range of 8 to 9 is usually suggested for struvite crystallization process. Some recommended pH values are given in Table 2.1.

2.9.2 Magnesium addition

In most cases, Mg is the limiting factor for struvite crystallization (except for very hard waters). For smooth operation of the process, there is a desired Mg:P molar ratio and thus a magnesium source is usually added. Two main types of magnesium used in the struvite process are magnesium hydroxide and magnesium chloride. The advantage of using the chloride is that it dissociates faster than the hydroxide, therefore resulting in shorter reaction times (Jaffer et al., 2002). A shorter reaction time means that a smaller, full-scale reactor can be constructed as the hydraulic retention time can be reduced. Magnesium hydroxide, however, is generally cheaper and also has the advantage of raising the pH (Munch and Barr, 2001). However, using magnesium hydroxide to serve both the functions of magnesium dose and pH increase means that one cannot be optimized independent of each other (Jaffer et al., 2002; Munch and Barr,
Sea water has also been used successfully as a magnesium source, without affecting the overall performance of the process (Kumashiro et al., 2001). Some sources of magnesium with their corresponding pH values are given in Table 2.1. Munch and Barr recommended that for optimal P-removal, there should be at least 20mg/L soluble magnesium in the effluent.

Table 2.1. Sources of magnesium with their corresponding pH values

<table>
<thead>
<tr>
<th>Base added</th>
<th>Magnesium source</th>
<th>Suggested pH value</th>
<th>Reference</th>
</tr>
</thead>
<tbody>
<tr>
<td>NaOH, Mg(OH)$_2$</td>
<td>MgCl$_2$, Mg(OH)$_2$</td>
<td>pH value ≥ 8.5</td>
<td>Jaffer et al., 2002</td>
</tr>
<tr>
<td>NaOH</td>
<td>MgCl$_2$, MgO</td>
<td>8.5 &lt; pH value &lt; 9</td>
<td>Celen and Turker, 2001</td>
</tr>
<tr>
<td>NaOH</td>
<td>Seawater</td>
<td>pH value ~ 7.7</td>
<td>Kumashiro et al., 2001</td>
</tr>
<tr>
<td>Only CO$_2$ stripping</td>
<td>Not required</td>
<td>8.2 &lt; pH value &lt; 8.8</td>
<td>Battistoni et al., 2001</td>
</tr>
</tbody>
</table>

2.9.3 **Magnesium to phosphorus molar ratio**

Struvite contains magnesium and phosphorus in a 1:1 molar ratio. In most cases with municipal wastewaters, the limiting element in the formation of struvite is magnesium. In order to optimize P-removal in the form of struvite, supplementation of magnesium is usually necessary (Adnan, 2002). Various studies have concluded that the Mg:P molar ratio should be greater than 1:1 for increased P-removal (Jaffer et al., 2002; Munch and Barr, 2001; Stratful et al., 2001). These studies found that optimal P-removal occurred at Mg:P molar ratio of 1.3:1.

2.9.4 **Ammonia to phosphorus molar ratio**

Municipal wastewaters typically contain higher molar concentration of ammonium ions than magnesium and phosphorus. Not much research has been carried out in trying to optimize the N:P molar ratio with regard to phosphorus recovery (Adnan, 2002). Some studies have indicated that P-removal is favored with the increase in ammonia (Suschka et al., 2004; Munch and Barr, 2001). Munch and Barr also determined that, with a higher ammonia concentration, the P-removal ratio was sharper. Suschka et al. (2004) found that the struvite pellet structure was depended on the presence of free ammonia.
2.9.5 Turbulence

Turbulence is a very important parameter in struvite crystallization. Nucleation is controlled by solubility chemistry, while growth rate is believed to be limited by low turbulence or low mixing energy (Ohlinger, 1999). On the other hand, too much turbulence may hinder growth by increasing the collision of individual pellets and thereby breaking the structure which results in reduced pellet size (Durrant et al., 1999). Although it is an important phenomenon, there is no universal quantification for turbulence for fluidized beds. Another important parameter involving turbulence is the minimum fluidization velocity. It marks the transition at which the behavior of an initially packed bed of solids changes into a fluidized bed, and is therefore a crucial parameter in the design of reactors or other contacting devices based on the fluidized bed technology (Asif and Ibrahim, 2002). Reynolds number (Re) has been used previously as a measure of turbulence (Adnan, 2002; Huang, 2002). However, it should be noted that the use of the concept of Reynolds number in fluidized beds should only be used as a guideline, since the number would change with bed density.

2.9.6 Initial reactor seeding

Struvite will not precipitate without a nucleus; however, nucleation is unnecessary if the solution is already “seeded”, i.e. adding materials to serve as embryos for pellet growth. In the case of treated wastewater streams, because of the absence of seeds in liquids, struvite can preferentially grow on the surface of tanks, pipes and other equipment, that act as embryos (Benisch et al., 2002). Even when pH and component ion molar ratios are appropriate, precipitation may cease before true equilibrium is reached because poorly crystallized or amorphous precipitates form initially, resulting in increased solubility (Wang et al., 2003). Reconstitution of such amorphous solids into crystalline solids occurs slowly and requires considerable energy (Sawyer et al., 1994). Wang et al. (2003) evaluated three different seeding materials (quartz sand, granite and struvite pellets) for potential to promote struvite crystal pellet growth and precipitation. They concluded that, with respect to P removal and size distribution of individual pellets, struvite pellets had the best performance among the seeding materials studied. It has also been suggested that seeding is only required at the start-up and the ongoing process eventually becomes self-seeding (Munch and Barr, 2001).
2.9.7 Temperature

There is contrasting information regarding the effect of temperature on struvite crystallization. Borgerding (1972) suggested that the solubility increases from 0°C to a maximum at 20°C; however, above this temperature, the solubility decreases with increasing temperature. Another study found that struvite was more soluble at 38°C than at 25°C (Burn and Finlayson, 1982). In this study, the solubility of struvite harvested at Lulu Island WWTP was tested at two different temperatures. The results are discussed in Chapter Four.

2.10 Chemistry of Struvite

Struvite (MgNH₄PO₄·6H₂O), or commonly called MAP (magnesium ammonium phosphate hexahydrate), is a white crystalline substance consisting of magnesium, ammonium and phosphorus in equi-molar concentrations. Formation of struvite occurs according to the reaction given in Equation 1.

\[
\text{Mg}^{2+} + \text{NH}_4^+ + \text{PO}_4^{3-} + 6\text{H}_2\text{O} \rightarrow \text{MgNH}_4\text{PO}_4\cdot6\text{H}_2\text{O}
\]  

(1)

Although the above equation is a generalized one, several side reactions are involved in the formation of struvite. These equations are given in Table 2.2.

The general struvite formation is given by Equation 1, but another equation has also been proposed. Shimamura et al. (2003) explains that MAP is formed according to the following equation.

\[
\text{Mg}^{2+} + \text{NH}_4^+ + \text{HPO}_4^{2-} + \text{OH}^- + 5\text{H}_2\text{O} \rightarrow \text{MgNH}_4\text{PO}_4\cdot6\text{H}_2\text{O}
\]  

(2)

The difference with the above equation and Equation 1 is that \(\text{HPO}_4^{2-}\) has been used instead of \(\text{PO}_4^{3-}\) and \(\text{OH}^-\) has been included. The reason for the different phosphate ion is that \(\text{HPO}_4^{2-}\) is more dominant than \(\text{PO}_4^{3-}\), in the normal operating pH range.
2.10.1 Solubility Product

The rate at which struvite forms and dissolves in the reactor is an important parameter with regards to phosphorus recovery from wastewater. This rate of reaction can be described in terms of the struvite solubility product. In essence, the solubility product ($K_{sp}$) is defined as the equilibrium constant of a reaction involving a precipitate and its constituent ions (Tchobanoglous and Schroeder, 1985). If a solid solute is placed in a solvent, it will dissolve in it until there is a dynamic equilibrium between the ions leaving the solid to enter the solvent and vice versa. In the case of struvite, if the product of the concentrations of magnesium, phosphate and ammonium in solution exceed the equilibrium solubility of struvite, struvite will form. Otherwise, if the product is lower than the equilibrium solubility, struvite will dissolve.

Many studies have been undertaken to determine the solubility product of struvite (Ohlinger et al., 1998; Stumm and Morgan, 1981; Snoeyink and Jenkins, 1980; Webb and Ho, 1992; Abbona et al., 1982; Taylor et al., 1963). There are two common approaches to determining the solubility product of struvite. These are dissolution and formation of struvite in distilled or other water solutions. The tests are usually carried out under controlled conditions of pH, temperature and mixing energy (Ohlinger et al., 1998; Burns and Finlayson, 1982; Bouropoulos and Koutsoukos, 2000). The formation approach is made by adding chemicals to provide magnesium, ammonium and phosphate ions. The dissolution test allows previously
grown struvite pellets to dissolve in solution. After the solution is assumed to be in equilibrium, the solubility product is determined by analyzing the ionic concentrations left.

Although extensive studies have been carried out to determine this value, there is no value that has been agreed upon universally. Published values of pK$_{sp}$ (-logK$_{sp}$) for struvite range from 12.6 to 13.8 (Dastur, 2001). In their review, Andrade and Schuiling (2001) give a good overview on the chemistry of struvite. They mentioned four reasons which cause the discrepancy in the reported K$_{sp}$ values.

- The solubility products may be derived by using approximate solution equilibria.
- The effects of ionic strengths are often neglected.
- Mass balance and electroneutrality equations are not always used.
- Different chemical species are selected for calculations.

Although a particular value of K$_{sp}$ has not yet been established, there is a general agreement that the value of K$_{sp}$ decreases with increasing pH and that it is only accurate for a specific pH value (Adnan, 2002; Doyle and Parsons, 2002; Ohlinger, 1999). Every wastewater is likely to have a distinct K$_{sp}$ value at a specific pH with regard to its struvite precipitation potential, because wastewater composition will vary from one treatment works to another. Any variation in water chemistry will result in differences in ionic strength and ion activity, changing the struvite precipitation potential of the wastewater (Doyle and Parsons, 2002).

2.10.2 Conditional Solubility Product

The determination of solubility product is complex and time consuming, as it requires a number of calculations and constants. In order to reduce the complexities and uncertainties, Booram et al. (1975) followed the example of Stumm and Morgan (1981) in calculating a conditional solubility product that could then be compared directly with the total concentrations of components in solution. This value represents the product of the measured concentrations of dissolved magnesium, ammonium and phosphate. In the calculation of P$_{s}$, the effect of ionic strength and side reactions are not considered, thus making the determination simpler. However, it should be noted that P$_{s}$ is only accurate for a particular set of conditions and will vary with
pH, ionic strength of solutions and temperature. On the other hand, $K_{sp}$ can be applied at any pH (Doyle and Parsons, 2002).

2.11 Factors Affecting Solubility Product

The solubility of a salt is not governed by the constituent ions but also involves other equilibria occurring in the solution. In the case of struvite, side reactions (Table 2.2) will increase or decrease the solubility. Some of the other factors that may affect the solubility of struvite in solution are pH, temperature and impurity ions.

2.11.1 pH

The general acceptable notion about struvite solubility is that it decreases with increasing pH (Ohlinger et al., 1998; Burns and Finlayson, 1982; Booram et al., 1975). However, as pH continues to rise above a pH of 9, the solubility of struvite begins to increase again, since the ammonium ion concentration will decrease and the phosphate ion concentration will increase (Snoeyink and Jenkins, 1980). This point was also illustrated by results published by Booker et al. (1999). Studies have shown that minimum solubility of struvite occurred at a pH of 10.3 (Ohlinger et al., 1998). Others have reported minimum solubility at pH 9.0 (Buchanan et al., 1994). One of the main reasons for this discrepancy in solubility values is the selection of different $K_{sp}$ values.

2.11.2 Temperature

Temperature affects both the equilibrium position of a precipitation reaction and the reaction rate. In general, solubility increases with increasing temperature, with a few notable exceptions such as CaCO$_3$, Ca$_3$(PO$_4$)$_2$, FePO$_4$. As with the case of pH effect, there are different opinions on the effect of temperature on solubility of struvite. Durrant et al. (1999) reported that the maximum solubility occurred at 20°C. Similar study conducted by Aage et al. (1997) found that the maximum solubility was at 50°C. Doyle and Parsons (2002) found that at high temperature, the structure of struvite pellets changed, which affected its solubility.
The solubility product of struvite is also temperature dependent. Burns and Finlayson (1982) determined the $K_{sp}$ values at different temperatures and the data showed that the $K_{sp}$ value increased with increasing temperature in the range from 25°C to 45°C.

The enthalpy change of a chemical reaction ($\Delta H$) is the amount of heat that is released or taken up during the course of the reaction (Snoeyink and Jenkins, 1980). Burns and Finlayson (1982) reported enthalpy values of 24.23 KJ.mol$^{-1}$ for struvite formation. This shows that the formation of struvite is an endothermic one.

Standard enthalpy change values, $\Delta H$, for reactions are most commonly used in water chemistry to determine the effect of temperature on the position of equilibrium. A useful expression relating $\Delta H$ to $K_{sp}$ is given by Equation 3.

\[
\frac{d \ln K_{sp}}{dT} = \frac{\Delta H}{RT^2}
\]

or,

\[
\ln \frac{K_{sp}(T_2)}{K_{sp}(T_1)} = -\frac{\Delta H}{R} \times \frac{T_1 - T_2}{T_1 \times T_2}
\]

where, $T$ = temperature (in K); $\Delta H$ = enthalpy of reaction (J/mol) and $R$ = ideal gas constant (8.314 J/mol.K).

Struvite crystallization is normally carried out at or near the ambient temperature. Thus, the quantity $\Delta H/RT_1T_2$ in Equation 4 can be assumed to be a constant. Thus, Equation 4 becomes

\[
\ln \frac{K_{sp}(T_2)}{K_{sp}(T_1)} = -\text{Const.}(T_1 - T_2)
\]

or,

\[
\frac{K_{sp}(T_2)}{K_{sp}(T_1)} = \theta^{(T_2 - T_1)}
\]

where, $\theta$ is the temperature coefficient.

### 2.11.3 Impurity ions

The presence of impurity ions has two effects on struvite solubility. The first is the possibility of complex formation by magnesium, ammonium and phosphate ions with each other or with other species present in the solution. This will cause an increase in the solubility of
struvite (Booram et al., 1975). The second effect is the change in ionic strength of the solution. Studies have shown that struvite solubility increases in the presence of calcium, carbonates, acetate and organic acids (Ohlinger et al., 1998; Durrant et al., 1999; Schulze-Rettmer, 1991).

2.12 Pellet Growth

According to the crystal formation theory, the process can be divided into two stages: nucleation and growth. Nucleation is the first stage where constituent ions combine to form crystal embryos. Crystal growth is the second stage as crystals grow continuously until the chemical equilibrium is reached (Ohlinger et al., 1999).

2.13 Size of Struvite Pellets

The desired quality and size of struvite pellets will largely depend on its final application. For struvite pellets to be used for agricultural purposes, it is desirable to have them as hard (or less brittle) so that they can be used with current fertilizer spreading machines and also be easily shipped and handled. On the other hand, use of small struvite for enrichment of nutrient deficient water bodies is desired. The size is also of importance when the release rate is considered as increased size would lead to a slower release rate due to the lower surface area/volume ratio. Overall, harder pellets tend to be denser and thus cheaper to transport in bulk.

2.14 Struvite As A Resource

2.14.1 Fertilizer value

Struvite has been suggested to display excellent fertilizer qualities under specific conditions when compared with standard fertilizers (Ghosh et al., 1996). The fertilizer properties of struvite were demonstrated in the 1960s in Germany and the US (as summarized by Schuiling and Andrade, 1998). Bridger et al. (1961) gave a detailed description of the value of MAP as a slow release fertilizer. MAP has been used commercially for container plants and is also appropriate for use on turf seedlings, ornamentals, vegetables and flower boards. A factor that must be addressed is that struvite may require supplementation with potassium to meet the NPK (nitrogen: phosphorus: potassium) requirements of certain specific crops (Gaterel et al.,
2000). The advantages of MAP as a fertilizer are:

- Because nutrients are released at a slower rate throughout the season compared to soluble fertilizers, plants can take up most of the nutrients without waste by leaching.
- Less frequent application is required.
- Fertilizer burn is not a problem even at high application rates.
- Low heavy metal content compared to phosphate bearing rocks that are mined and supplied to the fertilizer industry.

Unitika Ltd. of Japan has been able to operate full scale MAP reactors at a profit by successfully marketing their MAP product. The product, under the name of “Green MAP II”, has been promoted in Japan as an “environmentally friendly special fertilizer for forest regeneration of artificially created slopes” as well as a “plant friendly, long lasting type gardening fertilizer”. The recovered struvite contained only minute traces of toxic substances and was sold to fertilizer companies for 27,000 yen/tonne. It is used to enhance existing fertilizers, which are widely used on paddy rice, vegetables and flowers (Ueno and Fujii, 2001).

2.14.2 Slow release fertilizer for rehabilitating oligotrophic streams

Human activities during the past century, particularly intensive forest practices, fishing, urbanization, industrialization and impoundment construction, have negatively impacted the health of British Columbia’s numerous wild salmonid stocks (Slaney et al., 1996). Overharvesting and alteration of salmonid habitat reduces the return of spawning adults which naturally fertilize streams for their progeny (Larking and Slaney, 1996). Consequently, there is a lack of marine-derived nutrients to freshwater habitats, resulting in nutrient deficient streams. Oligotrophic stream conditions, both human induced and naturally occurring in granitic coastal systems, can become adequately fertile with low level addition of nutrients.

The use of a slow-release fertilizer is an innovative method for adding inorganic nutrients to nutrient-poor (oligotrophic) streams to increase autotrophic production (Sterling and Ashley, 2003). Liquid fertilizers traditionally have been product of choice for stimulating the base of the food chain, but their use is limited to more accessible streams and rivers, and the application is expensive due to the high cost of maintenance that is required (Ashley and Slaney, 1997). Slow- release fertilizers have the advantage that a once-per-year or per-season
application is much more operationally efficient than continuous drip or monthly application of solid granular fertilizers (Sterling and Ashley, 2003). The first slow-release fertilizers used in British Columbia were introduced to the Keogh River on northern Vancouver Island. Studies indicate that a slow-release fertilizer product can be manufactured to last approximately four months when applied in the spring to stimulate autotrophic production in nutrient deficient streams, thereby increasing forage and salmonid production (Sterling and Ashley, 2003).

2.15 Morphology Of Struvite Pellets

Struvite is a white crystalline substance that has a distinctive orthorhombic structure (Doyle and Parsons, 2002). The structure can be identified via X-ray diffraction (XRD) by matching the intensity and positions of the peaks produced to a database for the crystal structure (Doyle and Parsons, 2002). The internal structure of the pellets consists of regular \( \text{PO}_4^{3-} \) tetrahedral, distorted \( \text{Mg(H}_2\text{O})_6^{2+} \) octahedral and \( \text{NH}_4^+ \) groups which are all held together through hydrogen bonding (Abbona and Boistelle, 1979). Depending on the conditions during growth, struvite pellets can be variable from equant, wedge shaped, short prismatic to thick tubular (Durrant et al., 1999). The crystal habit or morphology is often used loosely to describe the shape and aspect ratio of crystals (Hatakka et al., 2000). Reports on the spontaneous formation of struvite in supersaturated solutions suggested that the pellet habit of struvite depends on the solution pH, the solution supersaturation, the Mg:P molar ratio, impurities in solution and the kinetic factors. (Bourropoulos and Koutsoukos, 2000; Wierzbicki et al., 1997; Abbona and Boistelle, 1979). Figure 2.2 shows a struvite crystal as seen using a scanning electron microscope (SEM).
Struvite solubility is dependant on the solution pH and temperature, and these factors highly influence its formation and morphology (Al-Jibbouri et al., 2002; Booker et al., 1999). Abbona and Boistele found that, at very high levels of supersaturation, formation of bi-dimensional and tri-dimensional twinned pellets were formed. On changing the supersaturation from high to low, the pellet habit changed from tubular to increasing elongation (Abbona and Boistelle, 1979). However, studies made by Bouropoulos and Koutsoukos found that the degree of supersaturation did not have any impact on the morphology of the pellets formed.

Studies on struvite pellets have found that the size of the pellets were influenced by the Mg:P molar ratio and the influent phosphorus concentration. It was reported that, for high influent concentrations, struvite was generated instantaneously near the inlet of the injection port and did not grow larger (Abe, 1995). In a study conducted by Hirasawa et al. (1997), they found that at Mg:P molar ratio of 2, the crystals agglomerated which resulted in large crystals. On the other hand, when the molar ratio was increased to 4, together with fine crystals, needlelike crystals were formed (Hirasawa et al., 1997). Another study showed that aggregation was favored at higher magnesium concentrations (Bourropoulos and Koutsoukos, 2000).

The presence of impurities or additives in a crystallization system can have a radical effect on crystal growth, nucleation, macrostep formation, agglomeration and on the uptake of foreign ions in the crystal structure. Different impurities have different effects on the growth of
crystals; while some may enhance growth, others may exert a highly selective effect, acting only on certain crystallographic faces (Hatakka et al., 2000).

Studies on struvite kinetics have demonstrated that mixing energy was the primary influence in overcoming the transport limitations to crystal growth (Ohlinger et al., 1999, Ohlinger et al., 1998). Another study found that the crystal size decreased with increasing specific power input to the reactor (Franke and Mersmann, 1995).

The pellet habit and polarity of struvite have been discussed in detail elsewhere (Abbona and Boistelle, 1985; Abbona et al., 1984).
3.1 Centrate Characteristics

During the study period, centrate from Lulu Island wastewater treatment plant was used. The centrate was stored in the centrate tanks before being used for the process. During the first part of the study, the tank was filled on alternate days or when the level of the centrate was low. Since new centrate was filled regularly, the centrate characteristics were not constant and it changed with every new fill. A summary of the centrate characteristics during the two study periods are given in Tables 3.1 and 3.2 and shown graphically in Figures 3.1 to 3.4.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>pH</th>
<th>Temp °C</th>
<th>Conduc. mS/cm</th>
<th>PO₄-P mg/L</th>
<th>NH₄-P mg/L</th>
<th>Mg mg/L</th>
<th>Ca mg/L</th>
<th>N:P</th>
<th>Mg:P</th>
</tr>
</thead>
<tbody>
<tr>
<td>Maximum</td>
<td>7.87</td>
<td>24.7</td>
<td>7.38</td>
<td>88.4</td>
<td>907.7</td>
<td>14.4</td>
<td>39.8</td>
<td>37.7</td>
<td>0.3</td>
</tr>
<tr>
<td>Minimum</td>
<td>7.40</td>
<td>15.0</td>
<td>6.14</td>
<td>44.3</td>
<td>410.3</td>
<td>0.02</td>
<td>0.3</td>
<td>21.4</td>
<td>0.0</td>
</tr>
<tr>
<td>Average</td>
<td>7.63</td>
<td>20.2</td>
<td>6.50</td>
<td>59.6</td>
<td>779.4</td>
<td>5.13</td>
<td>32.7</td>
<td>29.4</td>
<td>0.1</td>
</tr>
</tbody>
</table>

Table 3.1 Centrate characteristics – January to April 2004

<table>
<thead>
<tr>
<th>Parameter</th>
<th>pH</th>
<th>Temp °C</th>
<th>Conduc. mS/cm</th>
<th>PO₄-P mg/L</th>
<th>NH₄-P mg/L</th>
<th>Mg mg/L</th>
<th>Ca mg/L</th>
<th>N:P</th>
<th>Mg:P</th>
</tr>
</thead>
<tbody>
<tr>
<td>Maximum</td>
<td>7.7</td>
<td>31.3</td>
<td>6.87</td>
<td>67.9</td>
<td>814.9</td>
<td>18.3</td>
<td>38.8</td>
<td>41.5</td>
<td>0.60</td>
</tr>
<tr>
<td>Minimum</td>
<td>7.5</td>
<td>23.2</td>
<td>5.96</td>
<td>38.8</td>
<td>525.0</td>
<td>3.3</td>
<td>32.66</td>
<td>17.2</td>
<td>0.09</td>
</tr>
<tr>
<td>Average</td>
<td>7.6</td>
<td>27.1</td>
<td>6.38</td>
<td>58.7</td>
<td>719.2</td>
<td>10.6</td>
<td>34.8</td>
<td>27.9</td>
<td>0.24</td>
</tr>
</tbody>
</table>

Table 3.2. Centrate characteristics – June 2004
Figure 3.1. Centrate characteristics, (PO$_4$-P concentration) – January to April 2004.

Figure 3.2. Centrate characteristics (NH$_4$-N concentrations) – January to April 2004.
Figure 3.3. Centrate characteristics (PO₄-P concentrations) – June 2004.

Figure 3.4. Centrate characteristics (NH₄-N concentrations) – June 2004.
3.2 Process Description

The crystallization process used in the study was developed in the Civil Engineering Department of the University of British Columbia. The process consists of the reactor, an external clarifier, storage tanks for centrate, magnesium feed and caustic, pumps for the feed, recycle, magnesium and a pH controller. The basic flow diagram of the crystallization process is shown in Figure 3.5. The centrate is fed into the bottom of the reactor along with the recycle stream. Magnesium chloride and sodium hydroxide is added to the reactor through the injection ports, just above the feed and recycle flows. From known concentrations of ammonia-N and phosphate in the centrate and the total flow rates, it is possible to calculate the amount of standard magnesium chloride that has to be added to provide for a certain supersaturation ratio within the reactor. Similarly, the pH that is needed can be calculated and set. Once set, the pH controller determines when and amount of caustic that has to be added, so that the pH is within ± 0.5 of the set value.

Figure 3.5. Pilot-scale struvite crystallizer reactor process design.
3.3 Materials And Equipment

3.3.1 Reactor

The reactor used in this study was based on a 4× linear scale up of the pilot scale reactor designed by the UBC phosphate recovery team (Fred Koch, Environmental Engineering Group, Department of Civil Engineering, UBC, Vancouver, B.C., pers. comm.). Figure 3.5 shows the basic design of the reactor and associated equipments. The reactor had four distinct zones depending on the diameter of the pipe. The bottom part of the fluidized reactor was called the ‘harvest zone’, above that the ‘active zone’ while the last fluidized section was the ‘fines zone’. There was a settling zone, also called the ‘seed hopper’, at the top.

The inside diameters of the different sections were 7.6 cm, 10.2 cm, 15.2 cm and 38.1 cm for the harvest, active, fines zones and seed hopper, respectively. The total volume of the reactor was 91 liters (39 liters of which was in the fluidized zone) and the total height was approximately 4.55 m. The dimensions of the various sections are given in Table 3.3.

<table>
<thead>
<tr>
<th>Section</th>
<th>Length (cm)</th>
<th>Diameter (cm)</th>
<th>Volume (L)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Harvest zone</td>
<td>74.9</td>
<td>7.6</td>
<td>3.42</td>
</tr>
<tr>
<td>Active zone</td>
<td>154.9</td>
<td>10.2</td>
<td>12.56</td>
</tr>
<tr>
<td>Fines zone</td>
<td>127.0</td>
<td>15.2</td>
<td>23.17</td>
</tr>
<tr>
<td>Seed hopper</td>
<td>45.7</td>
<td>38.1</td>
<td>52.12</td>
</tr>
<tr>
<td>Below harvest zone</td>
<td>52.1</td>
<td>--</td>
<td>--</td>
</tr>
</tbody>
</table>

The variation of reactor diameter, with increasing height was considered in order to develop a certain degree of turbulence above each transition; this ensured that there was sufficient mixing in each zone and also helped to classify the fluidized particles by size. As the pellets grow in size (mass), they are able to overcome higher upflow velocities and thus have a tendency to move down the reactor. High turbulence at the harvest zone enhances crystal growth (Ohlinger, 1999). As a result, only the largest pellets in the reactor were harvested. The removal of the bigger pellets then allows the smaller pellets higher up in the reactor to enter the harvest zone, thus repeating the process. In the seed hopper, the upflow velocity was low so that the
fines at the very top had a tendency to settle down and this avoided their washing out with the recycle flow.

Upflow velocities in the range of 200 - 400 cm/min (hydraulic retention time, HRT, of 10 - 5 mins) was studied in this test. Calculated upflow velocities, based on a flow rate of 14L/min, and corresponding Reynolds numbers are given in Table 3.4. Reynolds numbers have been used to quantify turbulence in this study. However, it should be noted that the Reynolds number determined here is expected to be quite different than that in a reactor full of pellets.

Table 3.4. Upflow velocities and Reynolds number in different sections of the reactor

<table>
<thead>
<tr>
<th>Section</th>
<th>Upflow velocity (cm/min)</th>
<th>Reynolds Number</th>
</tr>
</thead>
<tbody>
<tr>
<td>Harvest zone</td>
<td>307</td>
<td>4367</td>
</tr>
<tr>
<td>Active zone</td>
<td>173</td>
<td>3276</td>
</tr>
<tr>
<td>Fines zone</td>
<td>77</td>
<td>2184</td>
</tr>
<tr>
<td>Seed hopper</td>
<td>12</td>
<td>874</td>
</tr>
</tbody>
</table>

\( ^a \) Calculations provided in Appendix A
\( ^b \) Calculations provided in Appendix B

The crystallizer was constructed of clear PVC piping connected with standard Schedule 40 or Schedule 80 PVC fittings. In the construction of the reactor, the inside joints between piping and fittings were kept as smooth as possible, to minimize dead zones where the fluidized particles could settle and struvite encrustation problems could occur. Clear piping was used in order to monitor the behavior of the struvite crystals in the fluidized bed and to monitor for signs of plugging or encrustation.

The reactor was equipped with a pH probe in the top of the harvest zone and a provision for a conductivity meter was kept opposite to the pH probe. Another pH meter was kept in the external clarifier. The pH probe in the harvest zone was used for feedback control using a proportional flow pH controller. Magnesium was dosed to the reactor in the form of magnesium chloride solution to supply the desired magnesium to phosphorous molar ratio in the reactor.

During the first trial, the reactor was initially seeded with three liters of struvite crystals grown from synthetic supernatant at the UBC pilot plant. During the second run, three liters of struvite generated during the first trial at Lulu Island WWTP was used.
3.3.2 Injection port

The reactor injection port was designed to mix the centrate with the recycle stream from the external clarifier, the magnesium chloride solution from the dosing pump and the sodium hydroxide solution from the pH controller. Figure 3.6 and Figure 3.7 show a simplified cross section and actual assembly of the injection port, respectively.

The injection port assembly was developed so that all inflow points to the reactor could be easily disconnected by means of quick release connectors. This was necessary to clean the port regularly. The magnesium chloride and sodium hydroxide (caustic) injection points are coincident so that a high local supersaturation ratio exists in this zone. This condition was sometimes responsible for some struvite formation in the injection port. Check-valves were installed, with both the caustic and magnesium feeds, to prevent any reactor backflow into the respective pumps in the event of an accidental reactor stoppage.

Figure 3.6. Sketch of the pilot-scale struvite crystallizer injection port assembly.
3.3.3. Harvest zone

The harvest zone was used primarily for harvesting of the struvite pellets and was located immediately above the injection port. It had an internal diameter of 7.6 cm, was 74.9 cm in length and held a volume of 3420 cm$^3$ (3.42 liters). Two ball valves (one at the top and one at the bottom of the zone) were used to isolate the harvest zone during struvite harvesting and injection port cleaning. The harvesting procedure is described in Section 3.8.

3.3.4 Active zone

Immediately above the harvest zone was an expanding section of 10.2 cm inside diameters and 154.9 cm in length. This section had an isolation valve at the top, and together with the top isolation valve of the harvest zone, could be separated completely. The reactor diameter changes were accomplished using standard PVC expansion couplings with rounded transitions.

3.3.5 Fines zone

Above the active zone was another expanded section, having a 15.2 cm internal diameter and a length of 127 cm. During the operation of the reactor, the fluidized bed of struvite crystals expanded to the top of this fines zone and settled in the bottom of clarifier section mounted above.
3.3.6 Clarifier/Seed hopper

Mounted to the top of the reaction zone was a 38.1 cm inside diameter clarifier section, also called the seed hopper, with a height of approximately 45.7 cm. This section was the largest in the reactor and having the lowest upflow velocity. As a result, most of the fine pellets and suspended solids settled by gravity and little escaped the reactor.

Two side outlets were fitted at the top of the hopper, which facilitated overflow from the reactor to the external clarifier. The main overflow was set at approximately 30 cm water depth in the clarifier section, while the backup overflow was set at approximately 35 cm water depth.

3.3.7 External clarifier

The external clarifier was placed to perform two major functions, to store the effluent from the reactor and to trap the washed out fine pellets and suspended solids from the reactor. The storage of the effluent was needed since some of it was recycled back to the reactor through the injection port. The external clarifier was square pyramidal shaped with 61 cm sides and 86.4 cm height. The total volume of clarifier was 154.2 L.

There were two outlets from the clarifier, one used for the recycle flow and the other to carry the final effluent to the drain. The final effluent was obtained in two weirs that were situated 7.6 cm from the top of the clarifier. This set up enabled relatively clear effluent to pass to the drain. Fine pellets and suspended solids deposited at the bottom of the clarifier and, thus, the recycle feed was taken 30.48 cm from the bottom. A three-way valve was used at the entrance to the clarifier, one for the overflow from the reactor, one connecting to the clarifier and the other to measure the flowrate.

3.4 Chemicals, Storage Tanks and Pumps

3.4.1 Centrate

As stated previously centrate from LIWWTP was used for the study. The centrate was stored in a 5600 liters capacity holding tank which was filled on alternate days or when the tank was empty. The centrate was pumped to the reactor through 1.3 cm (½ inch) tubing that was connected to a 1.3 cm (½ inch) ball valve. The valve was placed 20.3 cm (8 inch) from the
bottom of the tank to reduce the amount of suspended solids input into the reactor. Since suspended solids accumulated in the tank, a 5.1 cm (2 inch) valve was also attached to the tank to drain off the sludge. Depending on the suspended solids concentration in the centrate, it was necessary to drain/clean the tank once every 2-3 months. A T was placed at the entrance to the valve to facilitate sampling of the centrate. During the second run, a similar tank was installed. The main purpose of this tank was to allow greater centrate flow into the reactor without having to fill the tank everyday. By doing, so less time was required from the operators at the treatment plant (for filling the tank). From the holding tank the centrate was pumped to the reactor using Monyo Model 500 332 progressive cavity pump with a ½ HP motor and a digital speed controller.

3.4.2 Magnesium feed

The magnesium feed for the study was made from commercial grade magnesium chloride (MgCl₂·6H₂O). The solution was stored in a 1400 L tank and pumped to the injection port using a MasterFlex L/S variable speed peristaltic pump with a standard pump head.

3.4.3 pH control

The struvite crystallization process is highly depended on the pH of the system (struvite being less soluble in alkaline solutions). During the operation of the reactor, it was necessary to set a particular pH for a specific supersaturation ratio. As struvite forms, there is a drop in the pH and so it was necessary to find a way to increase the pH back to the desired value. The required pH can be achieved in several ways. Research performed by Fujimoto et al. (1991) indicated that addition of sodium hydroxide was more effective than addition of lime or magnesium hydroxide. Aeration itself can achieve the desired pH increase, but it requires longer residence times (Battistoni et al., 1998).

The caustic was made from sodium hydroxide pellets and stored in a 120 L tank. A carbon dioxide trap (concentrated caustic in a bottle) was used to strip off carbon dioxide from the air, before the air entered the caustic tank. This setup was made to ensure minimal loss of caustic strength which, in turn, could reduce the amount of caustic needed. The pH of the system was monitored continuously by a pH probe that was placed at the top of the harvest zone. The pH in the external clarifier was monitored using two Oakton continuous pH monitors, equipped with an Oakton gel filled, epoxy body pH probe. The pH meters were regularly
calibrated by the two point method, using standard buffer solutions of pH 7 and pH 10. Figure 3.8 shows the total setup of the study area at Lulu Island Wastewater Treatment Plant.

![Figure 3.8. Set-up of the study area.](image)

### 3.5 Process Monitoring And Maintenance

At the beginning of both runs, the reactor was seeded with 3 L of struvite. Synthetic seed produced at the UBC Pilot plant was used for the first run, while the second run was seeded using struvite produced from the first run. The main purpose of seeding was to reduce the time required for the system to start, that is the nucleation period. A successful attempt was made at the UBC pilot plant to start up a reactor with synthetic feedwater without seeding within a week (Iqbal Hossain Bhuiyan, Ph.D. candidate, Civil Engineering Department, University of British Columbia, per. comm.). Therefore, it is reasonable to assume that the reactor, in this case, could be started without seeding, but due to lack of time this procedure was not verified. Once the crystallization process started, the reactors were self seeded by the fine struvite pellets that were formed above the injection port.

In order to have a better control and understanding of the crystallization process, several parameters were monitored and recorded each day. These include grab samples of centrate and effluent for determining $\text{NH}_4^+$, $\text{PO}_4^{3-}$, $\text{Mg}^{2+}$ and $\text{Ca}^{2+}$ concentrations, the feed and recycle flows,
pH, temperature and conductivity of the centrate, effluent and external clarifier. The methods for the determination of the ions are described in Section 3.7. The feed flow was taken to be the rate of effluent flow from the external clarifier. This included the centrate and magnesium flows. The total flow was measured by opening a three way valve installed at the connection of the overflow pipe from the seed hopper to the external clarifier. The flows were measured using a graduated cylinder and a stop watch. The solubility of struvite in water is temperature dependent and so it was recorded. An alcohol thermometer was used and the data was checked with that given by the conductivity meter. Conductivity is an important parameter in the determination of supersaturation ratio and was thus measured. The conductivity was measured using an Oakton portable meter with built in temperature sensor. The levels in the different tanks were also recorded, in order to calculate the daily usage and simultaneously check with pump settings.

Apart from the collection of data and samples, regular maintenance of the system had to be performed. All the pH probes were calibrated by the two-point calibration method using standard pH 7 and pH 10 buffer solutions to ensure accurate measurements. The pH meter in the reactor was calibrated only when harvesting was carried out. The conductivity meter was calibrated once every month with recommended solutions (Occasionally the injection port became clogged due to the high supersaturation ratio and suspended solids). Thus, the port was cleaned whenever struvite was harvested. Although acid washing is an easy method to remove struvite from the ports (Huang, 2003 and Adnan, 2002) this method was not practiced since acid in the drain could cause problems at the plant. Instead, a screw driver was used to scrap off the struvite. The injection ports for the magnesium and caustic feeds were cleaned with a thin rod.

3.6 Sample Collection, Storage And Preservation

Grab samples of centrate and effluent were collected on each day from the centrate and external clarifier, respectively, while that for magnesium feed was collected from the magnesium holding tank. For the measurement of ions, the samples were filtered through 0.45 microns membrane filter papers (Standard Methods). Since this filter is easily clogged, samples were pre-filtered with 1.25 μm glass fibre filter papers. For NH$_4^+$ and PO$_4^{3-}$, 2 mL samples were taken in small test tubes to which was added one drop of 5% v/v sulphuric acid to lower the pH to below 2. Two drops of concentrated nitric acid were added to 5 mL of samples to preserve the
metal samples. During the second run, the samples were further digested to reduce the organic content of the samples as some clogging of the analyzer tubes were noticed (Susan Harper, Lab Manager, Civil Engineering Department, University of British Columbia, pers. comm.). All the above samples were stored at 4°C, until analysis, which was completed within USEPA recommended times (Berg, 1982).

3.7 Analytical Methods

3.7.1 Magnesium

Magnesium analysis was undertaken in the UBC laboratory and was performed by flame atomic absorption spectrophotometry using a Varian Inc. SpectrAA220 Fast Sequential Atomic Absorption Spectrophotometer. Instrument operational parameter details are given in Appendix C.

An attempt was made to determine the dissolved magnesium concentration in the centrate using EDTA titrations for total and calcium hardness. The assumption is that the difference between the total hardness and the calcium hardness is composed entirely of magnesium hardness. Unfortunately, this method gave results that were orders of magnitude different from the values determined by atomic absorption and so this technique was abandoned.

3.7.2 Ortho-phosphate

Ortho phosphate samples were analyzed at the UBC laboratory using flow injection analysis on a Lachat QuikChem 8000 instrument configured as described in Appendix C. Flow injection analyses were also used for samples from the pellet product analysis and struvite solubility determination experiments described in Sections 3.9 and 3.11.

Initially on-site ortho-phosphate analysis was attempted using a Hach DR2000 spectrophotometer and molybdovanadate method (APHA, AWWA and WPCF, 1995). Molybdovanadate reagent AccuVac Ampuls (Hach) were used to reduce the time needed for analysis. Unfortunately, the color of the centrate interfered with the yellow color developed and measured in this method, and it proved to be unusable.
3.7.3 Ammonia

Ammonia samples were analyzed at the UBC laboratory by flow injection analysis on the same LaChat instrument as the phosphate analysis described above. Instrument operational parameter details are given in Appendix C.

3.7.4 Calcium, Aluminum and Iron

Calcium, aluminum and iron analysis was performed on the samples from the crystal product analysis. Analysis was done by atomic absorption spectrophotometry using the same instrument as for the magnesium analysis described above. Instrument operational parameter details are given in Appendix C.

3.7.5 Total Phosphorus

Total phosphorus analysis for the estimation of full-scale phosphorus loads in the wastewater treatment plant were digested using the sulfuric acid-nitric acid digestion method (APHA, 1995, method 4500-P B.4) and analyzed by flow injection analysis on the same Lachat instrument as the ortho-phosphate samples as described above.

3.7.6 Filtration

All field samples were filtered using Fisher Brand G6 filter papers, with a nominal pore size of 0.45 microns, to remove suspended solids from the samples.

3.7.7 Conductivity

Conductivity was measured using a Hanna Instruments HI9033 multi range conductivity meter for the struvite solubility tests described above.

3.8 Pellet Harvest, Drying and Analysis

The pellets were harvested when there was sufficient quantity in the reactor, or when desired. Sometimes, harvesting had to be done to clean the injection ports when it was felt that the port was getting clogged. Harvesting involved isolating the harvest zone with the help of valves and then opening the harvest pipe. Unlike the previous reactors in which the reactors were shut during harvesting (Huang, 2003, Adnan, 2002, Britton, 2002) this reactor was designed such that the feed and recycle flows were diverted to the active zone through check
valves, even when the injection ports were closed, thereby keeping the reactor operational. The harvested pellets were collected in buckets and allowed to dry on trays. Depending on the atmospheric conditions, the drying usually took couple of days. The dried crystals were then sieved through standard sieves, after which some of them were taken to the laboratory for chemical and physical analysis. W.S. Tyler® sieves of sizes 4.75 mm, 2.83 mm, 2.0 mm, 1.0 mm and 0.5 mm were used to classify the harvested struvite pellets after which each fraction was weighed with an analytical balance. Chemical analysis usually involved the determination of saturated and dry density, concentration of NH$_4$-N, PO$_4$-P, Mg and Ca, Al and Fe.

3.9 Pellet Quality Determination

The quality of the harvested pellets was determined by checking the composition, purity, hardness, density and morphology. Samples of pellets were chosen on random with the assumption that they were representative of the harvested struvite pellets.

3.9.1 Composition And Purity

In order to determine the composition and purity of the pellets grown from centrate at LIWWTP, several random grab samples of the harvested pellets were dissolved in a 0.5% nitric acid solution. For the tests approximately 0.05 g of pellets were dissolved in 50 ml of 0.5% nitric acid solution. In order to accelerate dissolution, the samples were stirred with a magnetic stirrer for 24 hours, after which samples were analyzed for magnesium, ammonia, orthophosphate, calcium, aluminum, iron and potassium. Inductive coupled plasma (ICP)/mass spectrophotometer (MS) analysis of harvested struvite was also carried out for a few samples.

3.9.2 Hardness

Hardness is one measure of the strength of the structure of the mineral relative to the strength of its chemical bonds. Hardness can be tested through scratching with the assumption that a mineral can only be scratched by a harder substance. Therefore, a relative scale can be established, to account for the differences in hardness simply by seeing which mineral scratches another. The Mohs Hardness Scale, starting with talc at 1 and ending with diamond at 10, is universally used around the world as a way of distinguishing minerals. Another preliminary test of hardness is the ability of a material to resist crushing by the fingers. This test is judgmental,
and will largely depend on the person carrying out the test. Hardness can also be reported in terms of its impact and/or compressive resistance.

In this study, hardness was tested by the scratch method and the preliminary hardness test. For the scratch test, two items were chosen as standards: fingernail and industrial potassium nitrate pellets. According to the Mohs Hardness scale, fingernails have a hardness of 2.5. Due to the unavailability of instruments at the scale required, the impact and compressive test was not performed.

3.9.3 Density

In this study, the wet density was determined. The term “saturated, surface dry” (SSD) was used to describe the condition of wet pellets with no water on their outer surface. The SSD condition was approximated by soaking the particles and then drying them by laying them briefly on a paper towel (Potts, 2002). For the tests, random samples were chosen for each size distribution. The variability in the achieved SSD state and the precision of the instruments used were determined to be the chief limitations in the accuracy of the determination. Detailed procedure and calculations for the determination of the density is given in Appendix D.

3.9.4 Morphology of struvite pellets

The morphology of the harvested pellets was examined using a scanning electron microscope (SEM). The examination was performed at the Materials and Metals Engineering Department laboratory at UBC. The machine used was a Hitachi S-3000N SEM that was operated at variable pressure mode.

3.9.5 Charge of pellets

The surface charge of the harvested pellets was investigated by simple static test. This involved charging a plastic container by rubbing it with rubber. The rubbing induces negative charge on the plastic and then checking the interaction with the plastic, the surface charge of the pellet could be determined.
3.10 Terminology

3.10.1 Struvite solubility product (Ksp)

The struvite solubility product or \( K_{sp} \) as defined in this study is the product of the ionic activities of the ionic forms involved in the formation of struvite and is given by the Equation 7. The \{\} brackets indicate ionic concentration in moles per liter, corrected for activity. This involves the speciation of analytically determined concentrations using published acid and base dissociation constants, as well as an adjustment for activity.

\[
K_{sp} = \{Mg^{2+}\}\{NH_4^+\}\{PO_4^{3-}\} 
\]  

(7)

The activity is a function of the concentration of the ion and its activity coefficient, \( \gamma \). The activity is given by the Güntelberg approximation of the Debye-Hückel equation shown in Equation 8 (Sawyer et al., 1994).

\[
\log \gamma = \frac{0.5z^2 \sqrt{\mu}}{1 + \sqrt{\mu}} \tag{8}
\]

Where,

\( \gamma \) = the activity coefficient for the species of interest

\( z \) = the ionic charge of the species of interest

\( \mu \) = ionic strength

The ionic strength of the solution was determined based on conductivity measurements using the conversion factor described in Equation 9 (Tchobanoglous and Schroeder, 1985).

\[
\mu = 1.6 \times 10^{-5} EC 
\]  

(9)

Where, \( EC \) = electric conductivity (\( \mu S/cm \))

In order to determine the solubility product the total soluble ortho-phosphate, ammonia and magnesium have to be determined. The total ion is a combination of species other than
PO$_4^{3-}$, NH$_4^+$ and Mg$^{2+}$, as shown in Equations 10 to 12. The $[ ]$ brackets indicate ion concentration in moles per liter, without correction for activity.

$$T-PO_4 = [H_3PO_4] + [H_2PO_4] + [HPO_4^{2-}] + [PO_4^{3-}]$$ (10)

$$T-NH_3 = [NH_3] + [NH_4^+]$$ (11)

$$T-Mg = [Mg^{2+}] + [MgOH^+]$$ (12)

The different species given by Equations 10 to 12 can be determined by using the respective acid/base dissociation constants given in Table 3.5 (Potts, 2002). Since all samples were filtered prior to analysis, it was assumed that only dissolved species were present.

**Table 3.5. Equilibrium Constants at various temperatures**

<table>
<thead>
<tr>
<th>Description</th>
<th>Temperature</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>10°C</td>
</tr>
<tr>
<td>$[H^+][H_2PO_4]/[H_3PO_4]$</td>
<td>k$_1$</td>
</tr>
<tr>
<td>$[H^+][HPO_4^{2-}]/[H_2PO_4]$</td>
<td>k$_2$</td>
</tr>
<tr>
<td>$[H^+][PO_4^{3-}]/[HPO_4^{2-}]$</td>
<td>k$_3$</td>
</tr>
<tr>
<td>$[Mg^{2+}][OH^]/[MgOH^+]$</td>
<td>k$_{MgOH}$</td>
</tr>
<tr>
<td>$[Mg^{2+}][H_2PO_4]/[MgH_2PO_4^-]$</td>
<td>k$_{Mg1}$</td>
</tr>
<tr>
<td>$[Mg^{2+}][HPO_4^{2-}]/[MgHPO_4]$</td>
<td>k$_{Mg2}$</td>
</tr>
<tr>
<td>$[Mg^{2+}][PO_4^{3-}]/[MgPO_4^-]$</td>
<td>k$_{Mg3}$</td>
</tr>
<tr>
<td>$[NH_4^+][OH^-]/[NH_3]_{Total}$</td>
<td>k$_b$</td>
</tr>
</tbody>
</table>


### 3.10.2 Struvite conditional solubility product

In order to calculate the supersaturation ratio in the reactor, based on the analytical results obtained in the laboratory, the term struvite conditional solubility product ($P_s$) was used. This, as used in this study, is the product of the analytical results for soluble magnesium, ammonia nitrogen and ortho-phosphate (Equation 13). $P_s$ is a product of concentration only and has not been corrected for ionic activity.

$$P_s = [Mg]_{Total} [NH_4-N]_{Total} [PO_4-P]_{Total}$$ (13)
3.10.3 Supersaturation ratio

Supersaturation ratio (SSR) is defined as the ratio of the solubility product in a solution to the equilibrium solubility product for a given set of conditions. This parameter can be used to quantify precipitation potential (Stumm and Morgan, 1981). In this study, conditional solubility product has been used in the determination of supersaturation ratio. The conditional equilibrium solubility product used in this study was determined in the laboratory using LIWWTP centrate. Equation 14 gives the relation used to determine SSR in this study where \( P_s \) and \( P_{s\text{-eq}} \) are the conditional solubility and conditional equilibrium solubility products, respectively.

A solution with a SS ratio greater than 1 is supersaturated with respect to struvite and struvite may form to bring the solution to equilibrium; a solution with a SS ratio of less than 1 is undersaturated and struvite crystals will melt to bring the solution to equilibrium. Very fine crystalline precipitate with a disordered lattice may form in a highly supersaturated condition (Stumm and Morgan, 1981).

\[
SS \text{ Ratio} = \frac{P_s}{P_{s\text{-eq}}} \tag{14}
\]

3.10.4 Inlet supersaturation ratio

The inlet supersaturation ratio was determined by Equation 15 where \( P_{s\text{-inlet}} \) is the conditional solubility product in the reactor. The \( P_{s\text{-inlet}} \) was determined by using the concentrations of magnesium, ammonia and ortho-phosphate in the feed. This parameter represents the saturation conditions expected at the injection ports.

\[
\text{Inlet SS Ratio} = \frac{P_{s\text{-inlet}}}{P_{s\text{-eq}}} \tag{15}
\]

3.10.5 Reactor supersaturation ratio

The supersaturation ratio in the reactor was the primary control parameter used in the study. This factor is important because it determines the actual reaction driving force of the reactor to form pellets, compared to rate of crystal nucleation.

The supersaturation ratio in the reactor was determined by Equation 16 where \( P_{s\text{-reactor}} \) is the conditional solubility product in the reactor. The \( P_{s\text{-reactor}} \) was determined by using the concentrations of magnesium, ammonia and ortho-phosphate in the feed and the recycle flows. This parameter represents the saturation conditions expected just above the injection ports.
Reactor SS Ratio = $\frac{P_{s-reactor}}{P_{s-eq}}$ (16)

3.10.6 Recycle ratio

The recycle ratio is an important parameter in this study and was calculated using Equation 17.

Recycle Ratio = $\frac{Q_r}{Q_t} = \frac{(Q_t - Q_f)}{Q_t}$ (17)

Where: $Q_r$ is the recycle flow

$Q_t$ is the total combined flow through the reactor (feed + recycle).

$Q_f$ is the feed flow (magnesium and centrate flow).

The recycle ratio, therefore, represents the ratio of the flow from the recycle pump to the combined flow from the centrate and chemical pumps. The purpose of the recycle flow was to provide the required upflow velocity and to dilute the centrate for pH set up (thereby help in maintaining desired supersaturation ratio).

3.10.7 Crystal retention time

For pellets to grow to a desired size (2-5 mm), it is necessary for them to stay in the reactor for a certain period of time. The crystal retention time (CRT) is a term analogous to solids retention time (SRT) used in wastewater treatment. In this study the concept of CRT has been used to estimate the number of days a pellet spends in the reactor. The crystal retention time was determined by measuring the volume of collapsed bed of struvite at the time of harvest and the amount of pellets harvested (Equation 18).

$CRT = \frac{\text{volume of collapsed bed of struvite}}{\text{volume of harvested struvite}}$ (18)

For example if the collapsed bed volume was measured to be 15 liters, and 2 liters of pellets were harvested from the reactor every two days, then the CRT would be 15 days. The CRT was calculated after a volume, equal to the initial seeding volume, was harvested.

3.10.8 Mean crystal size

The mean crystal size was used to describe the size of the harvested pellets and was determined from sieve analysis. Pellets in each size fraction were assumed to be of a diameter in
the middle of the size fraction. For example, the pellets that were smaller than 0.5 mm were assumed to be 0.25 mm in diameter and the 0.5-1 mm size fraction a diameter of 0.75 mm. Based on this assumption, the mean diameter of harvested pellets (by mass) was calculated using Equation 19.

$$MCD = \frac{M_1(0.25)+M_2(0.75)+M_3(1.5)+M_4(2.42)+M_5(3.79)}{(M_1+M_2+M_3+M_4+M_5)}$$

Where: $MCD$ = Mean Crystal Diameter (mm)

$M_1$ = mass of crystals of diameter less than 0.5 mm (0.25 mm)

$M_2$ = mass of crystals of diameter from 0.5 to 1 mm (0.75 mm)

$M_3$ = mass of crystals of diameter from 1 to 2 mm (1.5 mm)

$M_4$ = mass of crystals of diameter from than 2 to 2.83 mm (2.42 mm)

$M_5$ = mass of crystals of diameter from than 2.83 to 4.75 mm (3.79 mm)

### 3.10.9 Removal efficiency

The percentage removal efficiencies of phosphate, ammonia and magnesium were calculated using Equations 20 to 22.

$$\%P_{\text{removal}} = \frac{[P_{\text{influent}}] - [P_{\text{effluent}}]}{[P_{\text{influent}}]} \times 100$$

$$\%N_{\text{removal}} = \frac{[N_{\text{influent}}] - [N_{\text{effluent}}]}{[N_{\text{influent}}]} \times 100$$

$$\%Mg_{\text{removal}} = \frac{[Mg_{\text{influent}}] + [Mg_t] - [Mg_{\text{effluent}}]}{[Mg_{\text{influent}}] + Mg_{\text{feed}}} \times 100$$

Where

$[P_{\text{influent}}]$, $[N_{\text{influent}}]$, $[Mg_{\text{influent}}]$ = concentrations of centrate PO$_4$-P, NH$_4$-N and Mg, respectively, at the inlet (mg/L) (i.e. multiplying the respective concentrations with the centrate flow rate and dividing by the total influent flow)
\[ \text{[P}_{\text{effluent}}] = \text{concentration of PO}_4^-\text{-P, NH}_4^-\text{-N and Mg, respectively, in the reactor effluent collected from the external clarifier (mg/L)} \]

\[ \text{[Mg}_{\text{feed}}] = \text{concentration of external Mg at the inlet (mg/L) (determined by multiplying Mg concentration in the magnesium feed tank with the magnesium flow and dividing with the total influent flow)} \]

3.11 Struvite Solubility Determination

The solubility of struvite has been investigated by a number of researchers and there is a wide range of reported values (Dastur, 2001). Solubility product determined by previous researchers at UBC resulted in values that ranged from \(2 \times 10^{-4}\) to \(8.5 \times 10^{-4}\) for different types of waters and temperatures. It was concluded that the solubility product would be different when the tests were performed with different solutions (Britton, 2002). As a result, a new struvite solubility curve was determined over a range of pH values. To generate this curve, struvite pellets grown at Lulu Island WWTP were dissolved in centrate and the solution stirred for 24 hours. As discussed in Section 2.11, there is contradictory information in literature regarding the effect of temperature on the solubility of struvite. Therefore, the solubility product was determined at temperatures 20°C and 25°C.

3.11.1 Apparatus

The setup of the apparatus used for the determination of the solubility product is shown in Figure 3.9. A six-station paddle stirrer (Phipps and Bird) was used with square jars in a temperature controlled room. About 10 grams of struvite was added to 1.5 L of centrate in each jar. The idea was to insert enough struvite in the jars to attain saturation. The paddle stirrers were set to operate at 70 ± 2 RPM. In order to determine the solubility curve over a range of pH values, dilute hydrochloric acid or sodium hydroxide solutions were added to each of the jars. Since previous research at UBC found that the equilibrium was reached within 24 hours after the pH was changed (Ping Liao, Department of Civil Engineering, UBC, pers. comm.), the apparatus was left to equilibrate for at least 24 hours before analysis.
3.11.2 Sampling

When equilibrium was assumed to be established, the pH and conductivity in each jar were measured. Samples were taken from each jar to determine total magnesium, total ammonia-nitrogen, total ortho-phosphate and calcium. The samples were analyzed according to the methods described in Section 3.7.

Figure 3.9. Setup of solubility determination

3.12 Process Control Models

In this study, three available process models were used to determine the effectiveness of each to predict effluent phosphate concentrations. This prediction may be helpful for checking efficiency of the crystallizer with respect to phosphate removal.

3.12.1 Potts’ crystallizer model

Throughout the course of the study, this model (Potts, 2002) was used to determine the operating conditions required for a specific supersaturation ratio. By having the feed concentrations, upflow velocity, recycle ratio, conductivity and desired supersaturation ratio as inputs, the model can be used to predict the operating pH. This process is done by using Microsoft Excel Solver tool to balance the chemical equilibria given in Table 2.2. The model
MATERIALS AND METHODS

also predicts the effluent concentrations of the three main species of interest, the efficiency and the theoretical struvite formation. This model was deemed to be superior to the Britton’s model, in that it takes into account all the side reactions, ionic activity and uses the solubility product. One deficiency of the model is that it uses a specific solubility product value and not one that is generated for the particular supernatant used. The model uses Equation 23 to define the formation of struvite.

\[ \text{Mg}^{2+} + \text{NH}_4^+ + \text{PO}_4^{3-} + 6\text{H}_2\text{O} \rightarrow \text{MgNH}_4\text{PO}_4.6\text{H}_2\text{O} \] (23)

3.12.2 Britton’s struvite equilibrium model

Britton (2002) developed a model to predict the effluent phosphate, ammonia and magnesium concentrations, given the influent feed concentrations and the operating pH of the reactor. The model uses the conditional solubility product (Ps) and assumes that pure struvite is formed, and that the reactor is at equilibrium with respect to struvite (Adnan, 2002).

The general equation used in the model is given in Equation 24.

\[ ([\text{Mg}]_{\text{in}} - \Delta) ([\text{PO}_4]_{\text{in}} - \Delta) ([\text{NH}_4]_{\text{in}} - \Delta) = P_{s-eq} \] (24)

where, \( \Delta = \) molar reductions;

\([\text{Mg}]_{\text{in}}, [\text{PO}_4]_{\text{in}}, [\text{NH}_4]_{\text{in}} = \) influent feed concentrations of magnesium, ortho-phosphate and ammonia respectively

\( P_{s-eq} = \) equilibrium conditional solubility product

By solving for \( \Delta \), prediction of the effluent concentrations can be made. It is important to note that the \( P_{s-eq} \) is dependent on the type of supernatant used. Therefore, it is necessary to generate a \( P_{s-eq} \) curve for the supernatant before using the above equation.

3.12.3 Neusciences Neuframe 4.0

Over the past few years, artificial neural networks (ANNs) have been used successfully in water quality modeling and drinking water treatment process modeling (Lewin et al., 2004). ANN is an artificial intelligence (AI) modeling technique inspired by the structure and operation of a human brain (Rodriguez et al., 1997). Artificial neural networks are capable of self-organizing and learning, and patterns and concepts can be extracted directly from historical data.
(Baxter et al., 1999). Neuframe is a graphical environment for exploring and using advanced intelligence technologies. Data analysis models are easily laid out using drag and drop on a workspace. Once designed, these models can be used to test Neural Nets and other analysis methodologies. This program is based on artificial neural network (ANN).

In the model design stage, an appropriate ANN architecture is chosen to provide optimum performance from the ANN model. This involves choosing the type of network (e.g., back-propagation), learning methods and speed, the number of inputs, outputs, hidden layers and neurons, and the type of activation function used by the neurons (Shariff et al., 2004). The design parameters used are given in Table 3.6.

Table 3.6. Neuframe 5-layer neural network details

<table>
<thead>
<tr>
<th>ANN design and criteria</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>Topology</td>
<td>Back propagation network with three hidden layers</td>
</tr>
<tr>
<td>Input layer 1</td>
<td>Linear function with number of neurons=number of inputs</td>
</tr>
<tr>
<td>Hidden layer 2</td>
<td>Gaussian function</td>
</tr>
<tr>
<td>Hidden layer 3</td>
<td>n/a</td>
</tr>
<tr>
<td>Hidden layer 4</td>
<td>n/a</td>
</tr>
<tr>
<td>Neurons per hidden layer</td>
<td>Number of hidden neurons=(1/2)(Inputs+Outputs)+(Number of patterns)^{1/2}</td>
</tr>
<tr>
<td>Output layer 5</td>
<td>Logistic function with one neuron</td>
</tr>
<tr>
<td>Learning rate factor</td>
<td>0.1</td>
</tr>
<tr>
<td>Momentum factor</td>
<td>0.1</td>
</tr>
<tr>
<td>Initial weights</td>
<td>0.3</td>
</tr>
<tr>
<td>Pattern selection</td>
<td>Random</td>
</tr>
<tr>
<td>Weight update method</td>
<td>Momentum</td>
</tr>
<tr>
<td>Number of patterns for training</td>
<td>65 % of total data set</td>
</tr>
<tr>
<td>Number of patterns for test</td>
<td>15 % of total data set</td>
</tr>
<tr>
<td>Number of patterns for</td>
<td>20 % of total data set</td>
</tr>
</tbody>
</table>
3.13 Model Evaluation

Following completion of model runs, two values were determined as given in Equations 25 and 26.

Average absolute error, $E_{abs} = \text{Abs}(Y_{actual} - Y_{predicted})$  \hspace{1cm} (25)

Relative absolute error (%) = $\text{Abs} \left( \frac{E_{abs} \times 100}{Y_{actual}} \right)$  \hspace{1cm} (26)

Where, $Y_{actual} = \text{laboratory analysis value}$

$Y_{predicted} = \text{model predicted value}$
4.1 Operating Conditions

From January 2004 to June 2004, a pilot-scale struvite crystallizer reactor was installed at the Lulu Island WWTP to investigate the feasibility of P-removal from centrate and subsequent P-recovery in the form of struvite pellets. The study consisted of two runs, Run 1 (January – April 2004) and Run 2 (June 2004).

At the end of the study, it was determined that the reactor was efficient in achieving high phosphorus removal from the centrate. In terms of purity, size and hardness the struvite pellets harvested were always of good quality, with average sizes of over 3.5 mm in diameter. A range of operational conditions and results summary of the study is given in Table 4.1. Detailed operational data is given in Appendix F for runs 1 and 2.

Table 4.1. Range of operational conditions and results summary

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Range</th>
</tr>
</thead>
<tbody>
<tr>
<td>Centrate PO\textsubscript{4}-P (mg/L)</td>
<td>12.0 ~ 88.4</td>
</tr>
<tr>
<td>Effluent PO\textsubscript{4}-P (mg/L)</td>
<td>1.4 ~ 54.2</td>
</tr>
<tr>
<td>Centrate Mg:P molar ratio</td>
<td>0.0 ~ 0.6</td>
</tr>
<tr>
<td>Centrate N:P molar ratio</td>
<td>17 ~ 41</td>
</tr>
<tr>
<td>pH</td>
<td>7.4 ~ 7.9</td>
</tr>
<tr>
<td>Recycle ratio</td>
<td>6 ~ 12</td>
</tr>
<tr>
<td>Reactor SSR</td>
<td>0.7 ~ 5.7</td>
</tr>
<tr>
<td>\textbf{P-removal efficiency} (%)</td>
<td>24 ~ 99.7</td>
</tr>
<tr>
<td>Total flow rate (mL/min)</td>
<td>8310 ~ 23100</td>
</tr>
<tr>
<td>Total upflow velocity (cm/min)</td>
<td>180 ~ 400</td>
</tr>
<tr>
<td>Mean struvite pellet size (mm)</td>
<td>1.4 ~ 3.6</td>
</tr>
</tbody>
</table>
4.1.1 Phosphate Levels

The phosphate concentration of the centrate varied throughout the course of the study. In the first run, the phosphate concentration of the centrate ranged from 42 mg/L to 88 mg/L, while during the second run, the range was 39 mg/L to 68 mg/L. The phosphate concentrations during the two runs are shown graphically in Figures 4.1 and 4.2.

The Figures 4.1 and 4.2 also illustrate the effluent concentrations obtained. It can be seen that the crystallization process was successful in decreasing the phosphate concentration to a low value. Depending on the removal efficiencies, it was possible to keep the phosphate concentration to below 10 mg/L, and sometimes, as low as 2 mg/L.

![Figure 4.1. Phosphate levels in centrate and effluent (Run1).](image_url)
4.2 Chemistry of Struvite

4.2.1 Struvite Solubility Product

The solubility product of struvite, harvested from Lulu Island WWTP, was determined at 20°C and 25°C, in the centrate. For distilled water, the temperatures were 18°C and 25°C. Figures 4.3 and 4.4 show the negative logarithm of $K_{sp}$ values for the Lulu Island WWTP centrate.

At 25°C, the $K_{sp}$ value increased from $2.71 \times 10^{-14}$ to $4.74 \times 10^{-14}$, when the pH was increased from 7.21 to 8.3, with an average value of $3.71 \times 10^{-14}$. At 20°C, the $K_{sp}$ value increased from $6.21 \times 10^{-15}$ (pH 6.61) to $2.62 \times 10^{-14}$ (pH 8.46), with an average value of $1.44 \times 10^{-14}$. As can be seen from the Figure 4.4, the $K_{sp}$ value is higher for the centrate than for the distilled water. This shows that the struvite is more soluble in centrate than in distilled water. One of the reasons for this could be the higher ionic strength of the centrate, since electrostatic interaction of ions in solution will reduce their real activities or effective concentrations (Ohlinger et al., 1998). Other factors that may lead to this condition could be the presence of more impurity ions in the centrate, such as carbonate, which could inhibit the precipitation of struvite and thus increasing the solubility (Huang, 2003).
RESULTS AND DISCUSSION

Thermodynamically, there should be one value of solubility product, given that the activity of each chemical species is known accurately (Adnan, 2002). In this study, it was not possible to determine all the chemical species present in the centrate; this could explain the different solubility products calculated.

![Graph showing solubility product of struvite in centrate at 20 °C.](image1)

\[ y = -0.0947x^2 + 1.1222x + 10.898 \]
\[ R^2 = 0.9631 \]

Figure 4.3. Solubility product of struvite in centrate at 20 °C.

![Graph showing solubility product of struvite in centrate and distilled water at 25 °C.](image2)

\[ y = -0.093x^2 + 1.2562x + 9.3872 \]
\[ R^2 = 0.9508 \]

\[ y = -0.0334x^2 + 0.3045x + 13.106 \]
\[ R^2 = 0.9544 \]

Figure 4.4. Solubility product of struvite in centrate and distilled water at 25 °C.
4.2.2 Temperature Coefficient (θ) And Enthalpy (ΔH)

The temperature coefficient and enthalpy are two important parameters that reflect the effect of temperature on the equilibrium reaction and solubility product. As stated previously, the struvite solubility product for two temperatures was studied, with the data shown in Appendix E.

The solubility of struvite varies with temperature, the solubility increasing with increase in temperature. Thus, the $K_{sp}$ value also increases with increase in temperature. Figure 4.5 shows the change in $K_{sp}$ values with temperature. The average value of $K_{sp}$ increased from $1.44 \times 10^{-14}$ at 20°C to $3.71 \times 10^{-14}$ at 25°C, an increase of 61%. For the Lulu Island WWTP centrate, a temperature coefficient of 1.21 and enthalpy of 137.5 KJ/mol was calculated. Since the enthalpy is positive, struvite formation in solution is an endothermic reaction.

![Figure 4.5. Struvite solubility product at different temperatures.](image)

4.2.3 Conditional Solubility Product

As mentioned earlier, the conditional solubility product ($P_s$) is an easy and less complex method of determining the solubility potential of a material. In this study, $P_s$ has been used to describe struvite solubility potential and also to monitor the struvite crystallization process.
The reactor at Lulu Island WWTP was housed indoors, where the temperature in the reactor varied from 15°C to 29°C from January to June. Since the average temperature, winter and summer, was 20°C and 25°C, the conditional solubility product was determined at these two temperatures. In order to provide a universal understanding of the harvested product solubility, tests were also performed with distilled water. The change in conditional solubility product with pH is shown in Figures 4.6 and 4.7. Equations 27 and 28 describe the polynomial curves for the struvite in centrate at 20°C and 25°C, respectively.

\[ pP_s = -0.1981 \, pH^2 + 4.0555 \, pH - 11.768 \]  \hspace{1cm} (20°C)  \hspace{1cm} (27)

\[ pP_s = 0.222 \, pH^2 - 2.2628 \, pH + 11.447 \]  \hspace{1cm} (25°C)  \hspace{1cm} (28)

Figure 4.6. Conditional solubility product of struvite in centrate at 20 °C.
Figure 4.7. Conditional solubility product of struvite in centrate and distilled water at 25 °C.

4.3 Performance Of The Process

4.3.1 Phosphate removal efficiency

During this study, removal efficiencies above 90 % were possible on a number of days. Figures 4.8 and 4.9 illustrate the percentage phosphate removal during the two runs. From the graphs, it can be seen that there were days when the removal efficiency was low. This may be attributed to operational problems with flows and also the difficulty in maintaining the desired SSR (as mentioned in Section 4.7). Thus, it can be seen that the objective of achieving 70 % P-removal was easily achievable.
4.3.2 Ammonia removal

There are a number of ways to treat nitrogen-containing wastewaters, but these techniques do not offer nitrogen recovery. Through the struvite crystallization process, it is possible to remove, as well as recover ammonia, in the form of struvite. Thus, this process
offers an alternative technique for the partial treatment of high ammonia containing wastewaters (Celen and Turker, 2001; Dempsey, 1997; Shin and Lee, 1997).

Ammonia concentrations in the centrate at Lulu Island WWTP were several times higher than phosphorus. In the formation of struvite, equimolar amounts of ammonia and phosphate are removed. As a result, ammonia reduction by the process is relatively low. However, the molar removal of ammonia is expected to be slightly higher than the molar phosphate removal, since ammonia is volatile, especially at a basic pH.

Figures 4.10 and 4.11 show the percentage ammonia reduction throughout the study period. For both the runs, average ammonia reduction was approximately 4.5%. As can be seen from Figures 4.10 and 4.11, there were days when the ammonia reduction was negative. Two reasons could account for this; operational problems with the reactor, leading to shutdown, and analytical errors.

Figure 4.10. Percentage ammonia removal (Run 1).
RESULTS AND DISCUSSION

4.3.3 Total phosphorus and ammonia

Samples for TKN and TP were tested on six different days to determine the reduction in the total phosphorus and nitrogen through the crystallization process. The test showed that, on an average, about 84% and 79% of total phosphorus and nitrogen were in the dissolved form, respectively. Reduction in the total phosphorus and nitrogen were approximately 86% and 7%, respectively (Figure 4.12). Detailed data are provided in Appendix I.

Figure 4.11. Percentage ammonia removal (Run 2).

Figure 4.12. TKN and TP removal efficiency
RESULTS AND DISCUSSION

4.3.4 Struvite recovery efficiency

One of the primary purposes of this study was to recover phosphate from the centrate in the form of struvite. Along with phosphate removal efficiency, another efficiency that is of importance is the struvite recovery. This efficiency was calculated by comparing the theoretical mass of struvite grown, based on the amount of phosphate removed, against the amount harvested. In order to keep the reactor running at the end of this study, it was not possible to fully empty the reactor, in order to calculate the total mass of struvite left in the reactor. Consequently, the removal efficiency was calculated for a certain period of the study. In reality, the actual mass of struvite harvested was higher than that recovered. Some losses invariably occurred during the recovery process, from harvesting to sieving. Some fine struvite escaped the reactor and accumulated at the bottom of the clarifier; these losses were not taken into account in the calculations.

Table 4.2. Struvite recovery

<table>
<thead>
<tr>
<th></th>
<th>Feb 20-29, 2004</th>
<th>June 21-27, 2004</th>
</tr>
</thead>
<tbody>
<tr>
<td>Total struvite harvested (g)</td>
<td>4922</td>
<td>5661</td>
</tr>
<tr>
<td>Theoretical struvite produced (g)</td>
<td>6114</td>
<td>6576</td>
</tr>
<tr>
<td>Struvite recovery efficiency (%)</td>
<td>81</td>
<td>86</td>
</tr>
</tbody>
</table>

As shown in Table 4.2, struvite recovery efficiency reached 81% and 86% for the two periods calculated. The size of the pellets harvested during the later period was larger than that of the first, and as result there was less loss of fines during recovery process. This could account for the relatively higher efficiency in the later period. Previous trials using Lulu Island WWTP supernatant estimated that it was possible to recover more than 90% of the struvite (Huang, 2003). In full scale installations, with better harvesting and recovery processes, it is expected that the struvite recovery efficiency will increase. Even at the efficiency calculated, it can be concluded that most of the phosphate was being recovered in a form that was easily harvestable.
4.4 Factors Affecting P-Removal

4.4.1 Effect of pH

The solubility of struvite varies with pH conditions, being soluble in acidic conditions and highly insoluble in alkaline conditions ((Ohlinger et al., 1998). Various researchers have investigated the effect of pH on P-removal (Adnan, 2002; Dastur, 2001, Munch and Barr, 2001; Battistoni et al., 2001; Kumashiro et al., 2001; Ohlinger, 1999) and the general agreement is that there is an increase in P-removal with increase in pH.

Figure 4.13 shows the effect of pH on P-removal throughout the study period. It can be seen that there is a range of removal efficiencies at a given pH. This can be attributed to inconsistent operating conditions in the reactor. The struvite solubility is controlled by pH, provided that the other reacting species (Mg$^{2+}$, PO$_4^{3-}$ and NH$_4^+$) and conditions (flows, RR) remains constant. This condition was not achievable in the reactor due to the varying characteristics of the centrate feed and changes in flows.

Several researchers have also recommended high pH values (8.2 ~ 9.0) to ensure high P-removal efficiencies (above 80%) (Jaffer et al, 2002; Munch and Barr, 2001; Battistoni et al., 2001; Ohlinger, 1999). But as can be seen from Figure 4.13, high removal rates (above 80%) can be achieved at relatively low pH of 7.5. Adnan (2002), using synthetic supernatant, also found that it was possible to have above 79% removal rates with a low pH of 7.1.

![Figure 4.13. Effect of pH on P-removal.](image-url)
4.4.2 Effect of inlet SSR

Previous studies with Lulu Island WWTP supernatant found that there was a direct relationship between inlet SSR and P-removal efficiency (Huang, 2003). In this study, no particular relationship could be established (Figure 4.14). At a given recycle ratio, an increase in inlet SSR would increase the reactor SSR. This in turn should increase the P-removal efficiency. But studies have shown that there is very little relationship between the inlet and reactor SSR (Huang, 2003, Adnan, 2002). Huang (2003) reported that there was a somewhat linear relationship between the two SSRs, but her graphical R-squared value was only 0.11. Since P-removal efficiency and pellet growth are more likely to be influenced by the reactor SSR, it is recommended that this SSR be used only as an operating parameter.

![Figure 4.14. Effect of SSR at the inlet on P-removal.](image)

4.4.3 Effect of reactor SSR

In this study, the desired phosphate removal efficiency was achieved by controlling the reactor supersaturation ratio. The supersaturation ratio was maintained by controlling the reactor operating pH. Figure 4.15 illustrates the effect of reactor SSR on P-removal. As can be seen from the figure, increasing the reactor SSR increases P-removal. This is, however, true until a certain SSR is reached. For the Lulu Island centrate, this SSR value was around 2.8. Above this...
value, the P-removal efficiency had a negative trend. As can be seen from the figure, there are some points which do not fall into the general trend. This is attributed to operational problems on these days, which led to stoppage of centrate flow into the reactor or flow at a much reduced rate.

![Graph showing the effect of reactor SSR on P-removal efficiency](image)

**Figure 4.15. Effect of reactor SSR on P-removal.**

### 4.4.4 Effect of magnesium to phosphorus molar ratio

Theoretically, struvite forms when the Mg:N:P molar ratio is 1:1:1. In most cases, since magnesium is the limiting element for struvite formation, external magnesium addition is required. In the first run of this study, the magnesium dose was increased considerably, to determine if high Mg:P molar ratio had any impact on the hardness of pellets harvested. The ratio of Mg:P molar ratio was reduced during the second run. Figure 4.16 shows the relation between Mg:P molar ratio and P-removal efficiency. The data used was taken when the reactor pH was kept constant at 7.6. In terms of P-removal efficiency, there does not seem to be any definite relation in the range tested. It was difficult to keep the Mg:P molar ratio constant because of two main reasons: changing P-concentration of the centrate and difference in set and actual feed flows. The influence of higher Mg:P molar ratio on the hardness and pellet morphology is discussed in Section 4.5. Huang (2003) found that there was a linear relation between Mg:P molar ratio and P-removal efficiency. But it should be noted that the R-squared
value of the graph was only 0.72, indicating that linearity explanation given could not be conclusive.

Figure 4.16. Relation between Mg:P molar ratio and P-removal efficiency (Feb 29-March 12, 2004).

Figure 4.17 shows the effect of Mg:P molar ratio on P-removal efficiency at pH 8.2. It can be seen that, with an increase in Mg:P molar ratio, there was a slight increase in removal efficiency. This increase is, however, not as pronounced as indicated by previous studies carried out at the UBC Pilot Plant (Huang, 2003; Adnan, 2002).

Figure 4.17. Relation between Mg:P molar ratio and P-removal efficiency.
4.4.5 *Effect of ammonia to phosphorus molar ratio*

Susicka *et al.* (2004) in their study emphasized that the effects of phosphate removal depends strongly on the amount of ammonia present. Using clean water and chemical reagents, they found that when the N:P molar ratio was increased from 1 to 5, the P-removal efficiency increased from 70% to 96%, respectively. The average N:P molar ratio of Lulu Island WWTP was 124 in the month of June, with values rising to 200 on certain days. As can be seen from Figure 4.18, there was an increase in the P-removal efficiency with increase in the N:P molar ratio. However, it is felt that it would be premature to conclusively say that there is a linear relationship between the efficiency and molar ratio. Katsuura (1998) investigated the effect of ammonia on the P-removal performance. He found that, as the ammonia concentration increased from 100 to 500 mg/L, so did the P-removal ratio.

![Figure 4.18. Relation between N:P molar ratio and P-removal efficiency.](image)

4.5 *Characteristics Of Harvested Product*

4.5.1 *Pellet size*

Figures 4.19 and 4.20 show the mean diameter of the harvested pellets during the course of the study. For most part of the first run, the pellets’ size were small (average 1.5 mm),
RESULTS AND DISCUSSION

cmpared to those of the second run (average 3.3 mm). This can be attributed to the different operating conditions existing in the reactor. Throughout this study, it has been shown that it is possible to grow pellets as big as 5 mm under suitable conditions. Although there was sufficient variation in the characteristics of the centrate, the results show that it had minimal effect on the size of the pellets. This is true as long as the conditions within the reactor are optimal.

Figure 4.21 shows the effect of reactor SSR on the mean pellet size. From the figure, it is seen that there is a definite relationship between the two parameters. At the beginning, with a low SSR, the pellet size is small. As the SSR was increased, so did the size of the pellets. During the third week of the month, there was an excess of pellets in the reactor and so higher amounts were harvested. During the four continuous days of harvesting, little change was observed in the size of the pellets. Although the reactor SSR dipped to less than 2.5, the conditions were still suitable enough for growth to occur. Another interesting observation indicated that, if the conditions in the reactor were suitable, the pellets did not need a long CRT to grow in size. This phenomenon was also observed by another graduate student working at the Penticton AWWTP (Alexander Forrest, Department of Civil Engineering, UBC, pers. comm.). A study plotted the rate of crystal growth of struvite versus the relative supersaturation and found that there was a parabolic dependence (Bouropoulos and Koutsoukos, 2000). This dependence is indicative of a surface controlled mechanism, according to which the rate determining step is the diffusion of growth units on the surface of the supercritical struvite nuclei, which undergo crystal growth (Sohnel and Garside, 1992). Detailed sieve analysis data for harvested struvite pellets is given in Appendix G.
RESULTS AND DISCUSSION

Figure 4.19. Mean pellet size harvested (Run 1).

Figure 4.20. Mean pellet size harvested (Run 2).
4.5.2 Composition And Purity

The composition of the harvested struvite pellets were tested for purity and composition at the environmental laboratory. Apart from the regular species of struvite, Al, Fe and Ca were tested. Previous results on Lulu struvite found that potassium was present in trace amounts, with most of the analyses being below the detection limit of the method (Huang, 2003).

Samples were chosen randomly and for each of the months studied. For the month of June different size samples were also tested to see if there was any influence of size on the purity of the harvested pellets. Detailed analyses are shown in Appendix H.

Table 4.3. Summary of pellet composition analysis

<table>
<thead>
<tr>
<th>Composition by mass</th>
<th>Theoretical value of</th>
<th>Mean value of</th>
<th>Standard deviation</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mg</td>
<td>9.9</td>
<td>9.5</td>
<td>0.6</td>
</tr>
<tr>
<td>NH$_4$-N</td>
<td>5.7</td>
<td>5.7</td>
<td>0.2</td>
</tr>
<tr>
<td>PO$_4$-P</td>
<td>12.6</td>
<td>12.0</td>
<td>1.0</td>
</tr>
<tr>
<td>Struvite</td>
<td>100</td>
<td>96</td>
<td>3.5</td>
</tr>
</tbody>
</table>
RESULTS AND DISCUSSION

Table 4.3 gives a summary of the analysis. From the results it is seen that the harvested product is of high purity. Previous analysis of struvite pellets grown at the UBC Pilot Plant found that, on an average, the pellets were 94.1% pure (Huang, 2003). Table 4.4 shows the impurity content of the harvested struvite pellet.

Table 4.4. Impurity content of pellet

<table>
<thead>
<tr>
<th>Content by mass (%)</th>
<th>Mean value of analysis (%)</th>
<th>Standard deviation</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al</td>
<td>0.06</td>
<td>0.02</td>
</tr>
<tr>
<td>Ca</td>
<td>0.07</td>
<td>0.06</td>
</tr>
<tr>
<td>Fe</td>
<td>0.12</td>
<td>0.03</td>
</tr>
</tbody>
</table>

Table 4.5. Comparison of heavy metal content in different product

<table>
<thead>
<tr>
<th>Contents (ppm)</th>
<th>Morocco P-rocks</th>
<th>Geestmerambacht Ca₃(PO₄)₂</th>
<th>LIWWTP Struvite</th>
<th>Synthetic struvite (UBC Pilot Plant)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al</td>
<td>200</td>
<td>950</td>
<td>203</td>
<td>101</td>
</tr>
<tr>
<td>Cd</td>
<td>40</td>
<td>&lt;6</td>
<td>0.5</td>
<td>0.82</td>
</tr>
<tr>
<td>Cr</td>
<td>357</td>
<td>8</td>
<td>2.1</td>
<td>3.6</td>
</tr>
<tr>
<td>Fe</td>
<td>1600</td>
<td>1260</td>
<td>2018</td>
<td>389</td>
</tr>
<tr>
<td>Mg</td>
<td>5700</td>
<td>4200</td>
<td>155981</td>
<td>158550</td>
</tr>
<tr>
<td>Na</td>
<td>1700</td>
<td>360</td>
<td>282</td>
<td>83.5</td>
</tr>
<tr>
<td>Ni</td>
<td>67</td>
<td>8</td>
<td>8.7</td>
<td>15.1</td>
</tr>
<tr>
<td>Ti</td>
<td>108</td>
<td>8</td>
<td>5.3</td>
<td>3.0</td>
</tr>
<tr>
<td>Zn</td>
<td>880</td>
<td>310</td>
<td>29</td>
<td>101</td>
</tr>
<tr>
<td>Mn</td>
<td>10</td>
<td>560</td>
<td>145</td>
<td>21</td>
</tr>
<tr>
<td>Cu</td>
<td>23</td>
<td>17</td>
<td>67</td>
<td>265</td>
</tr>
<tr>
<td>As</td>
<td>5</td>
<td>2</td>
<td>1</td>
<td>4</td>
</tr>
</tbody>
</table>

*values given in Jeanmaire (2001).

ICP/MS testing was carried out on five days of harvested struvite to get a full picture of contents in the pellet. Some of the common heavy metals have been compared with values found in literature and with two samples of synthetic pellets grown by the author at the UBC Pilot Plant. The detailed results of the ICP/MS testing are given in Appendix H. As can be seen
from Table 4.5, the struvite pellets harvested from LIWWTP have much lower heavy metal contamination than in the P-rock samples reported.

4.5.3 **Hardness**

In this study, the hardness of the pellets was not determined by any specific method. Instead, the ability of the pellets to resist crushing by fingers and the scratch test were used to qualitatively describe the hardness of the pellets.

In general, it was felt that the quality of the centrate did not have any effect on the hardness of the pellets. Pellets harvested in February and March was found to be very hard, compared to those harvested in June. Some of the pellets harvested in at the earlier part of the study were very hard. There were pellets that could not be crushed by fingers. These pellets were also seen to be more compact and rounded. In comparison, the pellets harvested in June were much softer. Most of the pellets were easily crushed by the fingers. The major difference between the two time period was the Mg:P molar ratio and upflow velocity. The magnesium dosage in the first period was higher than the second period. Also, the upflow velocity in June was approximately 30% higher than the velocity in the first period (January to April, 2004). This increased flow rate may be responsible for some of the breakage (as seen on SEM images), which leads to lower strength. The influence of magnesium dosage on the hardness of pellets was also determined by a previous study (Huang, 2003). Although CRT was thought to be another factor that determined the hardness of harvested pellets, this study did not find any correlation between the two. Scratch test revealed that the struvite harvested in the months of February and March was as hard as the potassium nitrate pellets. Pellets harvested in June was, however, much softer than the potassium nitrate pellets. All harvested pellets were softer than the fingernail.

4.5.4 **Density**

The density of random samples was checked using the SSD conditions. The greatest weakness of the method is in the poorly defined concept of the saturated surface dry (SSD) condition, as it applies to struvite pellets. Another significant source of error is the loss of fines when particles are transferred onto and off of the paper towel. Although care was taken to account for all the weighed pellets, some fines were left on the towel. As the proportion of the fines is small compared to the total mass of pellets, it is expected that this lost will have little
RESULTS AND DISCUSSION

effect on the calculated SSD condition. In case of having an effect, it will probably increase the value slightly.

SSD densities ranging from 1.01 g/mL to 1.30 g/mL were determined. It was noticed that the smallest size fraction had the greatest value. This might be related to the greater loss of fines for the smallest fraction. The average SSD density was determined to be 1.14 g/mL. Potts (2002) determined the SSD density of synthetic struvite and found values in the range of 1.29 – 1.40 g/mL. The presence of impurities in the Lulu struvite might have lowered the value compared to the synthetic one.

4.5.5 Morphology

The pellets harvested from Lulu Island WWTP were of various sizes and shapes. As detailed in Section 2.15, there are operational factors that affect the growth, and thereby the morphology, of the pellets that form. In order to verify these effects, the outside and inside of harvested pellets were observed under a scanning electron microscope (SEM).

Two major categories of product were observed under the SEM. The first batch of pellets was classified according to their sizes. Harvested pellets from the month of June were sieved and then examined under the microscope. This batch was used to see if there was any change in the morphology of pellets with increasing size. During the initial study period, the Mg:P molar ratio was substantially higher than the latter part of the study. This was done to see if increased Mg:P ratio had any influence on the hardness, size and morphology of the pellets. The second batch consisted of pellets that were harvested from each of the five months the study was conducted.

Figure 4.22 shows the outside appearance of the pellets according to the size. Apart from the 0.5mm<D<1mm size fraction, the surface of all the other pellets are predominantly smooth. There are places where the pellets are not smooth and have crevices. In Figure 4.22 (a) the surface is composed of tightly bound needle-like and plate-like structures. In (c) it can be seen that flat plate-like pellets are adhered to a relatively smooth surface. As mentioned in Section 4.7, struvite was cleared off the reactor column by tapping against the sides. The thin plate-like struvite on the pellet in (c) was probably as a result of this clearing. With increase in size, the pellets seem to have more crevices than for the smaller pellets. One reason for this structure may be due to higher upflow velocity in the column, thus breaking open the pellet. The smoothness
of the outside may be a result of the numerous collisions the pellets have among themselves and with the wall of the reactor. This impact grinds off the rough edges from the surface. The smoothness of the pellet harvested in June suggests that higher upflow velocity will cause more collisions, resulting in a higher grinding effect.

Figure 4.22. SEM images of struvite surface harvested in June 2004.
A high resolution SEM image of 1<D<2mm struvite product was taken as illustrated in Figure 4.23. The struvite particle is a combination of numerous fine pellets that are of different sizes. From the image, it can be seen that the individual brick-like and rod-like struvite pellets are tightly packed.

![SEM image of magnified (×800) surface of a 1 mm<D<2 mm pellet harvested in June, 2004.](image)

In order to examine the interior, the pellets were cut in half with a sharp blade. Figure 4.24 shows the interior of pellets harvested for each month of the study period. This set of pellets was chosen to check the influence of Mg:P molar ratio on the compactness of the pellets. The Mg:P molar ratio was the highest (molar ratio of 30) in the month of January and lowest in the month of April (molar ratio of 2). It can be seen that the pellet harvested in March (molar ratio of 15) was more compact from the rest. Previous images on Lulu Island WWTP pellets were found to be less compact than the ones harvested in this study. One of the major differences between the two studies was the Mg:P molar ratio. The average Mg:P molar ratio of the previous study was kept at 3 (Huang, 2003), whereas the present study was conducted with molar ratios varying from 30 to 1.5. The present pellets were also more compact than the synthetic pellets grown at the UBC Pilot Plant under a Mg:P molar ratio of 5 (Adnan, 2002). The different sets of images of harvested pellets under different Mg:P molar ratio suggests that this ratio is an important parameter in determining the compactness of the struvite product.
similar conclusion was also made in another study which found that high magnesium concentration favors struvite crystal aggregation (Bouropoulos and Koutsoukos, 2002).

Figure 4.24. Cut SEM images (×300) of pellets harvested.
The core of the pellet appear to be weaker and less dense than the exterior (Figure 4.25). The interior is composed of numerous small, individual pellets loosely packed. On the other hand, the exterior is subjected to numerous collisions which cause the small pellets to fuse together. This fusing may be responsible for the relatively smooth exterior surface. In Figure 4.25 (a), it can be seen that there is an outer layer of around 0.1 mm which is completely fused. Similar observations were made with other cut pellets. This coating is probably responsible for the hardness of the pellet.

Figure 4.25. SEM image of pellet harvested on 28th January, 2004.
4.5.6 Charge of pellet

The electric charge of the crystalline particles in their suspensions plays an important role in the particle-particle interactions (Bouropoulos and Koutsoukos, 2000). This, in turn, may affect precipitation and pellet growth kinetics. Bouropoulos and Koutsoukos investigated the electric charge of the surface of struvite particles as a function of pH and pMg. They found that struvite particles showed a strong negatively charged surface, the negative charge increasing with pH.

During the first run of this study, the magnesium concentration in the reactor was increased (average 160mg/L) to examine its effect on the hardness of the struvite pellets. During this period, the pellets were checked for surface charge and were found to be positively charged. In the second run, the magnesium concentration was reduced (average 29 mg/L), and the pellets were found to be negatively charged. Thus, it was found that there was a charge reversal with the increase/decrease of magnesium concentration in the reactor. Similar results were also reported (Bouropoulos and Koutsoukos, 2000), where it was found that increasing magnesium concentration in the aqueous phase caused charge reversal. It was found that the isoelectric point was at pMg 1.75. Above pMg 1.75, the electrokinetic charge of the struvite particles is negative, while below the value it is positive.

4.6 Struvite Loading Rate

One of the methods that could be used to scale up future reactors is the struvite loading rate. This is defined as the theoretical mass of struvite grown daily, which is based on the daily mass of phosphate removed (Adnan, 2002). Although the struvite loading rate could be increased by increasing the influent flow rate and the pH, there seems to be a limit to how high it could be set. Increasing the influent and the pH will increase the reactor pH, a condition which causes plugging problems in the reactor (Adnan, 2002). Another problem with increasing the upflow velocity (by increasing the influent flow rate) is that there is a shorter HRT in the reactor. In order for complete reaction of the influent in the reactor, a minimum HRT is required. Before increasing the total flow rate it will be necessary to check the effluent supersaturation ratio to see if complete reaction ($SSR_{effluent} = 1$) takes place in the reactor. Otherwise, it may be necessary to reduce the total flow.
Figures 4.26 and 4.27 show the struvite loading rates for the study period. During the first run, the production was low due to the lower feed flows. During the second run, the struvite loading rate was higher than the first run. This was due to the reactors being subjected to a higher pH value, which in turn allowed for higher consistent P-removal and therefore higher struvite loading rates. There were days when plugging problems reduced the feed flow, and this is shown by the low theoretical production of struvite. Due to lack of time, it was not possible to run the reactor at higher feed rates. From personal observation, it was felt that the present reactor was capable of handling total flows as high as 25 L/min.

![Graph showing struvite loading rates](image)

Figure 4.26. Struvite loading rate (Run 1)
4.7 Operational Problems

Over the course of this study, several operational problems were encountered in running the reactor. This section describes the problems faced and ways they were mitigated or minimized.

4.7.1 Feed flow regulation

Keeping all the flows constant to the set point is vital for smooth operation of the reactor. Any change in the flow of one of the components will have an effect on the reactor supersaturation ratio and thereby on the effectiveness of the reactor. Even though positive displacement pumps were used, the change in pump head between the full level and the empty level in the centrate holding tank and the subsequent increase in the reactor volume caused variations in centrate. This problem was more pronounced during the first run of the study, when the centrate flow was lower (average of 1.1 L/min) than in the second run (average of 2.1 L/min). In order to minimize this problem, it was necessary to measure and readjust the flows by adjusting the pump speed daily. Another solution was to minimize the variation of centrate head in the holding tank. In a full-scale process, it is expected that this problem would be minimal with the use of online flow controllers.
4.7.2 Plugging of tubing

Another reason for the change in flows was plugging of the tubing. Both the centrate and recycle flow tubing were often encrusted with struvite and suspended solids. Occasionally, the encrusted layer broke off from the tubing walls and accumulated at the entrance and exit, thereby plugging the pump and other ports. This problem was solved by tapping with a screwdriver the points of obstruction. This process broke up the encrustation and allowed normal flow to be resumed. At times, when the encrustation was too high, the problem was solved by passing hot water through the tubing. This method required less effort and was very efficient.

4.7.3 Reactor fouling

One of the reasons for using clear piping for the reactor was to be able to observe the condition of the fluidized bed (pellet movement, bed volumes) from outside. But the reactor walls were often coated with an opaque layer of struvite. As was done to clear the tubing, a screwdriver was used to tap off the struvite from the walls. Another way to clean the walls would be to use acid washing, but that would require the reactor to be emptied. It is worth mentioning that care must be taken in removing the amount of encrustation, as they may drop to the bottom of the reactor and plug up the injection ports (personal observation while working at the UBC Pilot Plant Crystallizer).

4.7.4 Injector port fouling

The highest local supersaturation ratio existed at the injection port and so was the place most prone to struvite encrustation. It was necessary to clean this section whenever struvite was harvested. Occasionally, it was necessary to harvest if it was felt that the port was clogging. Although the flow never fully stopped, there was a marked decrease in flows due to the reduction in opening in the port. Cleaning was usually carried out by scraping off the struvite with a screwdriver. The caustic and magnesium entrance was cleaned with a thin wire.

4.7.5 Suspended solids

Although care was taken by the operators at the treatment plant to provide sufficiently clear centrate, there were times when the solids content was high. Since the residence time of the centrate in the holding tank was low (1-2 days), some solids made their way into the reactor. This resulted in solids accumulating in the tubing. Also, depending on the solids content in the
centrate, the holding tank would accumulate solids at the bottom and so it was necessary to flush the tank at least once every 45 days. In a full-scale installation, it would be prudent to install a sedimentation tank between the centrifuge and the crystallizing reactor, to prevent/reduce the entry of solids into the reactor.

4.8 Model Results

4.8.1 Potts’s crystallizer model

Due to the longer time required to verify the effectiveness of the model, the sample size for evaluation was restricted to June data only. The complete analytical results are presented in Appendix J. The average absolute and relative absolute errors are listed in Table 4.6. Besides phosphate, the effluent results for ammonia were also tested along with the P-removal efficiency. Figures 4.28 to 4.30 show the variation of actual and predicted values for the data set verified by Potts’s Crystallizer Model (Potts, 2002).

4.8.2 Britton’s struvite equilibrium model

The sample size for testing this model was the same as that used for the Potts’s Crystallizer Model. The complete analytical results are presented in Appendix J. The average absolute and relative absolute errors are listed in Table 4.6. P-removal efficiency, effluent phosphate and effluent ammonia concentrations were compared by this model. Figures 4.28 to 4.30 show the variation of actual and predicted values for the data set verified by Britton’s Struvite Equilibrium Model (Britton, 2002).

As can be determined from Table 4.6, Potts’s model was more efficient in predicting the P-removal efficiency and PO₄-P effluent results. However, Britton’s model was more efficient in predicting the effluent ammonia concentrations. Despite the shortcomings of both the models, it was felt that both were able to predict concentrations quite efficiently. It was also felt that, few modifications to the models would increase the prediction efficiency significantly.
Table 4.6. Summary of model results

<table>
<thead>
<tr>
<th>Date</th>
<th>Potts P-removal efficiency</th>
<th>Britton P-removal efficiency</th>
<th>Potts PO₄-P concentration</th>
<th>Britton PO₄-P concentration</th>
<th>Potts NH₄-N concentration</th>
<th>Britton NH₄-N concentration</th>
</tr>
</thead>
<tbody>
<tr>
<td>07-Jun</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>12-Jun</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>17-Jun</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>22-Jun</td>
<td></td>
<td></td>
<td></td>
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<td></td>
<td></td>
</tr>
<tr>
<td>27-Jun</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>02-Jul</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Average absolute

<table>
<thead>
<tr>
<th>Average absolute</th>
<th>3.97</th>
<th>10.2</th>
<th>2.2</th>
<th>5.5</th>
<th>26.7</th>
<th>23.4</th>
</tr>
</thead>
</table>

Relative absolute

<table>
<thead>
<tr>
<th>Relative absolute</th>
<th>4.65</th>
<th>11.3</th>
<th>40.6</th>
<th>125.8</th>
<th>4.1</th>
<th>3.6</th>
</tr>
</thead>
</table>

* for P-removal efficiency the unit is in percentage

Figure 4.28. Predicted and actual P-removal efficiency using 2 different models.
Figure 4.29. Predicted and actual effluent $PO_4$ concentration using 2 different models.

Figure 4.30. Predicted and actual effluent $NH_4$-N concentration using 2 different models.
4.8.3 Neusciences Neuframe 4.0 (© Neusciences, 2000)

Effluent PO₄-P concentrations were verified using this model. In comparison to the previous two models, this model was found to be less efficient in predicting the effluent phosphate concentration. Using this model, the average absolute and relative absolute errors were 23.6 mg/L and 370%. The efficiency of this model depends largely on the data set. The more data available for training the model, the better is the efficiency. In most cases, the model over-predicted the phosphate concentrations. Until more data points can be used to train this model, it is not recommended that this method be used. Figure 4.31 illustrates the predicted and actual effluent PO₄-P concentration using the Neusciences Neuframe 4.0 model.

![Figure 4.31. Predicted and actual effluent PO₄-P concentration using Neusciences Neuframe 4.0 model.](image)

**Chemical Costs**

A summary of the cost incurred during the study period, in terms of chemical costs is given in Table 4.7. The prices of caustic and magnesium chloride were taken to be $0.55/kg (Camford Chemicals) and $0.28/kg (from internet), respectively. As can be seen from Table 4.7, the cost of production was lowest in June. During the first run (January to April), the reactor was operated using higher Mg:P molar ratios; this molar ratio was reduced during the second run. By increasing the operating pH, and thereby using higher caustic, the quantity of magnesium...
RESULTS AND DISCUSSION

additive can be reduced substantially. This however, had little effect on the total cost of chemicals. A factor that will determine the amount of caustic used is the initial pH of the centrate used. A high initial pH will require less caustic use than a centrate having low pH. In a full scale operation, it is expected that the cost of caustic will be the more determining factor, with respect to operating chemical costs. Some researchers have reported caustic costs as high as 97% of the total chemical costs (Jaffer et al., 2002). One of the important factors that determine the cost of production is the total centrate flow. In the second run, the centrate flow was increased. This increase resulted in increased struvite production and harvest, thereby bringing down the overall chemical cost.

Table 4.7. Cost of struvite production

<table>
<thead>
<tr>
<th></th>
<th>January</th>
<th>February</th>
<th>March</th>
<th>April</th>
<th>June</th>
</tr>
</thead>
<tbody>
<tr>
<td>Harvest (L)</td>
<td>6.8</td>
<td>23.0</td>
<td>16.9</td>
<td>5.3</td>
<td>15.1</td>
</tr>
<tr>
<td>Reactor (L)</td>
<td>15.0</td>
<td>15.5</td>
<td>15.8</td>
<td>14.0</td>
<td>24.1</td>
</tr>
<tr>
<td>Total growth (L)</td>
<td>23.9</td>
<td>18.4</td>
<td>17.2</td>
<td>3.5</td>
<td>36.2</td>
</tr>
<tr>
<td>Mass harvested (kg)</td>
<td>3.7</td>
<td>10.3</td>
<td>7.5</td>
<td>2.6</td>
<td>29.9</td>
</tr>
<tr>
<td>Caustic used (kg)</td>
<td>1.2</td>
<td>3.6</td>
<td>4.4</td>
<td>1.6</td>
<td>22.6</td>
</tr>
<tr>
<td>MgCl₂ used (kg)</td>
<td>50.0</td>
<td>50.0</td>
<td>57.0</td>
<td>15.4</td>
<td>37.5</td>
</tr>
<tr>
<td>Cost of caustic (CAD)</td>
<td>0.7</td>
<td>2.0</td>
<td>2.4</td>
<td>0.9</td>
<td>12.4</td>
</tr>
<tr>
<td>Cost of MgCl₂ (CAD)</td>
<td>14.0</td>
<td>14.0</td>
<td>16.0</td>
<td>4.3</td>
<td>10.5</td>
</tr>
<tr>
<td>Cost of production (CAD per liter)</td>
<td>0.8</td>
<td>0.9</td>
<td>1.1</td>
<td>1.5</td>
<td>0.6</td>
</tr>
<tr>
<td>Cost of production* (CAD per kg)</td>
<td>0.5</td>
<td>0.5</td>
<td>0.6</td>
<td>0.9</td>
<td>0.4</td>
</tr>
<tr>
<td>Cost of production (CAD per kg of harvest)</td>
<td>4.0</td>
<td>1.6</td>
<td>2.5</td>
<td>2.0</td>
<td>0.8</td>
</tr>
</tbody>
</table>

*assuming struvite density to be 1.7 g/cm³.

Although the cost of producing the struvite is greater than the potential sale of struvite, it must be remembered that there are other factors that must be considered. The cost of production have to be offset by revenue that are lost through increased pumping costs, lost man hours, expensive pipe replacement, possible excavation work if pipes are located underground and WWTP downtime due to blockages. These factors are difficult to quantify financially.
CHAPTER FIVE

CONCLUSIONS

Based on the results obtained from this study on struvite recovery from centrate at Lulu Island Wastewater Treatment Plant, the following conclusions can be drawn.

- The pilot scale recovery crystallizer developed at UBC was effective in removing and recovering phosphate from centrate.
- Under controlled conditions, over 90% of phosphate (range of 39~88 mg/L) and 4% of ammonia-nitrogen (range 410~907 mg/L) was removable through the process. Phosphate concentration in the effluent could be lowered to 5 mg/L.
- A reduction of 86% and 7% in total phosphorus and total nitrogen, respectively, was easily achieved.
- It was possible to achieve over 90% P-removal at a pH of 7.5. Thus it is not always necessary to run the process at a high pH value (8.2~9), as recommended in the literature. This indicates that pH is not the only factor determining the suitability of the struvite pelletization process.
- More than 85% of the phosphate removed was recovered as harvestable struvite pellets. It is expected that, with better harvesting and drying process, the recovery could be increased.
- Factors that affected phosphate removal were the operating pH, the reactor SSR, the N:P and Mg:P molar ratios.
- Solubility tests on centrate and distilled water gave different results for solubility product of struvite. The solubility product was dependant on the water tested, the pH of the solution and the temperature.
- The tested temperature coefficient and enthalpy at 25 °C were 1.21 and 137.5 kJ/mol, respectively. This confirms that struvite formation is an endothermic reaction.
- The reactor supersaturation ratio, magnesium dosage and operating pH were determined to be effective controlling parameters for obtaining the desired phosphate removal efficiency.
- The reactor SSR was determined to be the significant factor with regards to pellet size; Mg:P molar ratio and upflow velocity being the determining factors for pellet hardness.
CRT was not seen as a controlling factor for pellet growth, as long as other conditions in the reactor were suitable. Through the process, it was possible to grow pellets larger than 4.75 mm.

- The harvested struvite pellets were composed of nearly pure struvite (96% by weight) with small amount of calcium and traces of iron and aluminum. ICP/MS testing on four struvite samples found lower heavy metal content than that present in P-rock.
- SSD density of struvite was found to be in the range of 1.01–1.30 mg/L.
- Scanning electron microscope images showed that the pellets were, in fact, aggregates of smaller pellets.
- Under the operating conditions studied, it was possible to have a struvite loading above 1400 g/day. It was also determined that the reactor was capable of handling total flows of 25 L/min.
- Occasional scaling of reactor and tubing were responsible for operational problems, such as reduced flows. However, regular inspection of these parts reduced such problems.
- Potts’ crystallization model (Potts, 2002) was efficient in predicting effluent phosphate and ammonia concentrations. An average absolute prediction error of 2.2 mg/L (40%) and 26.7 mg/L (4%) were determined for phosphate and ammonia, respectively.
- Large deviation from actual values was found when predicting phosphate effluent concentrations by Britton’s struvite equilibrium model (Britton, 2002). The average absolute error of predicting phosphate concentration was 26.7 mg/L (125%). However, this model was as efficient as the Potts’ model (Potts, 2002) in predicting the effluent ammonia concentrations.
- Nuencesciences Neuframe 4.0 model (© Nuencesciences, 2000) was found to be inefficient in predicting effluent phosphate concentrations. This error could be due to the small number of data used to train the model.
Based on the experience gained from this study on phosphorus recovery from centrate, the following recommendations are made.

- Since the crystallization process is dependent on the concentration of ions involved, field methods for their determination should be developed. The possibility and effectiveness of installing online probes should be checked.

- Long term study should be made to determine the usefulness of phosphorus removal, in terms of nutrient removal efficiency in the treatment plant, by struvite formation.

- A curve for the solubility product vs pH should be developed at the beginning of each study. This curve can then serve as a criterion in operating the struvite crystallization process.

- Check valves for the centrate and recycle lines should be installed to prevent backflow in these lines.

- The flow path of chemicals and wastewater to the reactor should be redesigned, so that all flows can be diverted to the active zone during harvesting process.

- Further study on the hydrodynamics of the process should be made. In particular, the effect of upflow velocity on struvite growth should be investigated. Knowledge about the flow patterns and forces to which individual pellets are subjected to could be very useful.

- The use of reactor SSR is recommended as the controlling parameter for the process.

- The possibility of using magnesium to control the reactor SSR should be further explored. It is recommended that a cost comparison be made, using magnesium and pH as controlling parameters for the reactor SSR.
RECOMMENDATIONS

- The effect of N:P and Mg:P molar ratios on the growth and quality of struvite pellets should be studied.

- In order to have a tighter control on the size and hardness of harvested pellets, a better understanding of the aggregation process is required.

- The effect of temperature and conductivity on the process should be further investigated. This could be in the form of more solubility tests in the laboratory.

- Before full scale installation, possibility of automating the process should be examined at pilot scale.

- It is recommended that the upgraded Potts Crystallizer Model (Potts, 2002) be used to control the struvite crystallization process and to predict the process performance.


REFERENCES


REFERENCES


REFERENCES


REFERENCES


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Suschka, J., Kowalski, E. and Poplawski, S. (2004). *Study of the effects of the reactor hydraulics on struvite precipitation at municipal sewage work*. Study carried out for Centre European d'Etudes des Polyphosphates (CEEP) by Polish Academy of Sciences and University of Bielsko-Biala, Poland.


APPENDIX A

CALCULATIONS FOR UPFLOW VELOCITY IN THE REACTOR

Upflow velocity = Flow rate/ cross-sectional area
Flow rate range: 10 - 23 L/min

Table A.1. Upflow velocities in different sections of the reactor

<table>
<thead>
<tr>
<th>Section</th>
<th>Diameter (cm)</th>
<th>Cross-sectional area (cm$^2$)</th>
<th>Upflow Velocity (cm/min)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Harvest</td>
<td>7.6</td>
<td>45.6</td>
<td>219.3 – 504.3</td>
</tr>
<tr>
<td>Active</td>
<td>10.2</td>
<td>81.07</td>
<td>123.3 – 283.7</td>
</tr>
<tr>
<td>Fines</td>
<td>15.2</td>
<td>182.41</td>
<td>54.8 – 126.1</td>
</tr>
<tr>
<td>Seed hopper</td>
<td>38.1</td>
<td>1140.1</td>
<td>8.8 – 20.2</td>
</tr>
</tbody>
</table>
APPENDIX B

CALCULATIONS FOR REYNOLDS NUMBERS

Reynolds number is given by the following equation\(^{(1)}\)

\[ \text{Reynolds Number} = \frac{\rho \times V \times D}{\mu} \]

Where,
\( \rho \) = mass density of the fluid, kg/m\(^3\)
\( V \) = average velocity of the fluid, m/s
\( D \) = diameter, m
\( \mu \) = viscosity of the fluid, N.s/m\(^2\)

At a temperature of 25°C, the values\(^{(1)}\) of \( \rho \) and \( \mu \) are 997 mg/m\(^3\) and \( 8.9 \times 10^{-4} \) N.s./m\(^2\) respectively.

For flow rate range of 10 – 23 L/min, the corresponding Reynolds numbers in the different sections are given in Table B.1.

<table>
<thead>
<tr>
<th>Section</th>
<th>Diameter ( \times 10^3 ) (m)</th>
<th>Velocity (cm/min)</th>
<th>Reynolds number</th>
</tr>
</thead>
<tbody>
<tr>
<td>Harvest</td>
<td>76</td>
<td>219.3 – 504.3</td>
<td>3120 – 7175</td>
</tr>
<tr>
<td>Active</td>
<td>101.6</td>
<td>123.3 – 283.7</td>
<td>2340 – 5380</td>
</tr>
<tr>
<td>Fines</td>
<td>152.4</td>
<td>54.8 – 126.1</td>
<td>1560 – 3585</td>
</tr>
<tr>
<td>Seed hopper</td>
<td>381</td>
<td>8.8 – 20.2</td>
<td>625 – 1435</td>
</tr>
</tbody>
</table>

## APPENDIX C

### INSTRUMENT OPERATIONAL PARAMETERS

**Table C.1.** Instrument operational parameters for flame atomic absorption spectrophotometer

<table>
<thead>
<tr>
<th>Element Analyzed</th>
<th>Magnesium</th>
<th>Calcium</th>
<th>Iron</th>
<th>Aluminum</th>
</tr>
</thead>
<tbody>
<tr>
<td>Concentration Units</td>
<td>mg/L</td>
<td>mg/L</td>
<td>mg/L</td>
<td>mg/L</td>
</tr>
<tr>
<td>Instrument Mode</td>
<td>Absorbance</td>
<td>Absorbance</td>
<td>Absorbance</td>
<td>Absorbance</td>
</tr>
<tr>
<td>Sampling Mode</td>
<td>Autonormal</td>
<td>Autonormal</td>
<td>Autonormal</td>
<td>Autonormal</td>
</tr>
<tr>
<td>Calibration Mode</td>
<td>Concentration</td>
<td>Concentration</td>
<td>Concentration</td>
<td>Concentration</td>
</tr>
<tr>
<td>Measurement Mode</td>
<td>Integrate</td>
<td>Integrate</td>
<td>Integrate</td>
<td>Integrate</td>
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<tr>
<td>Replicates Standard</td>
<td>3</td>
<td>3</td>
<td>3</td>
<td>3</td>
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<tr>
<td>Replicates Sample</td>
<td>3</td>
<td>3</td>
<td>3</td>
<td>3</td>
</tr>
<tr>
<td>Wavelength</td>
<td>202.6 nm</td>
<td>422.7 nm</td>
<td>248.3 nm</td>
<td>309.3 nm</td>
</tr>
<tr>
<td>Range</td>
<td>0-100 mg/L</td>
<td>0-60 mg/L</td>
<td>0.06-15 mg/L</td>
<td>0-20 mg/L</td>
</tr>
<tr>
<td>Flame Type</td>
<td>N₂O/C₂H₂</td>
<td>N₂O/C₂H₂</td>
<td>C₂H₂/Air</td>
<td>N₂O/C₂H₂</td>
</tr>
<tr>
<td>Calibration Algorithm</td>
<td>New rational</td>
<td>New rational</td>
<td>New rational</td>
<td>New rational</td>
</tr>
<tr>
<td>Lamp Current</td>
<td>4.0 mA</td>
<td>10 mA</td>
<td>5 mA</td>
<td>10 mA</td>
</tr>
</tbody>
</table>

**Table C.2.** Instrument operational parameters for flow injection analysis

<table>
<thead>
<tr>
<th>Ion Analyzed</th>
<th>PO₄-P</th>
<th>NH₃-N</th>
</tr>
</thead>
<tbody>
<tr>
<td>Concentration Units</td>
<td>mg/L</td>
<td>mg/L</td>
</tr>
<tr>
<td>Range</td>
<td>0-100 mg/L</td>
<td>0-100 mg/L</td>
</tr>
<tr>
<td>Temperature</td>
<td>63°C</td>
<td>63°C</td>
</tr>
<tr>
<td>Method</td>
<td>Ammonium Molybdate</td>
<td>Phenate</td>
</tr>
<tr>
<td>Reference</td>
<td>1</td>
<td>2</td>
</tr>
</tbody>
</table>

APPENDIX D

STRUVITE PELLET DENSITY ANALYSIS

D.1. Apparatus and Materials

A balance accurate to 0.01g, graduated container of at least 50 mL, 15-25 mL of struvite pellets, paper towel, distilled water (or other solution with known density) and weighing dish.

D.2. Procedure

1. Soak the struvite particles in distilled water or another solution for 5 to 10 minutes to fill all their pores with water. This step may be omitted if particles are already wet.
2. Record the mass of the empty weighing dish ($M_{\text{dish}}$).
3. Record the mass of the graduated container with 50 mL of water (density $\rho_w$) (or other solution) in it ($M_{50}$).
4. Empty the graduated container and put the struvite particles into it. Refill it with distilled water to the 50-mL mark, and record the mass ($M_{50p}$).
5. Carefully decant the distilled water and spread the particles on a paper towel. Place another paper towel on the top and turn both of them around. Do not leave the pellets in the open for more than 3 minutes as more water may evaporate than desired.
6. Transfer the SSD struvite particles to the weighing dish and again record the mass ($M_{\text{SSD+dish}}$).

D.3. Calculations

\[ M_{\text{SSD, in air}} = M_{\text{SSD+dish}} - M_{\text{dish}} \]
\[ M_{\text{SSD, in water}} = M_{50p} - M_{50} \]
\[ V_{\text{particles}} = (M_{\text{SSD, in air}} - M_{\text{SSD, in water}}) / \rho_w \]
\[ \rho_{\text{SSD}} = M_{\text{SSD, in air}} / V_{\text{particles}} \]

where, $\rho_{\text{SSD}}$ is the density at SSD condition.
<table>
<thead>
<tr>
<th>Table D.1. Density determination</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
</tr>
<tr>
<td><strong>$M_{50}$ (g)</strong></td>
</tr>
<tr>
<td>93.80</td>
</tr>
<tr>
<td>90.44</td>
</tr>
<tr>
<td><strong>$M_{50p}$ (g)</strong></td>
</tr>
<tr>
<td>96.22</td>
</tr>
<tr>
<td>95.43</td>
</tr>
<tr>
<td><strong>$M_{dish}$ (g)</strong></td>
</tr>
<tr>
<td>1.2824</td>
</tr>
<tr>
<td>1.2915</td>
</tr>
<tr>
<td><strong>$M_{SSD+dish}$ (g)</strong></td>
</tr>
<tr>
<td>22.6318</td>
</tr>
<tr>
<td><strong>$M_{SSD,in air}$ (g)</strong></td>
</tr>
<tr>
<td>23.538</td>
</tr>
<tr>
<td>21.3403</td>
</tr>
<tr>
<td><strong>$\varepsilon$</strong></td>
</tr>
<tr>
<td><strong>$M_{SSD,in water}$ (g)</strong></td>
</tr>
<tr>
<td>2.42</td>
</tr>
<tr>
<td>4.99</td>
</tr>
<tr>
<td><strong>$V_{particles}$ (ml)</strong></td>
</tr>
<tr>
<td>21.18</td>
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<tr>
<td>16.40</td>
</tr>
<tr>
<td><strong>Temperature (°C)</strong></td>
</tr>
<tr>
<td>24.0</td>
</tr>
<tr>
<td>24.0</td>
</tr>
<tr>
<td><strong>$\rho_{w}$ (g/ml)</strong></td>
</tr>
<tr>
<td>0.99724</td>
</tr>
<tr>
<td>0.99724</td>
</tr>
<tr>
<td><strong>$\rho_{SSD}$ (g/ml)</strong></td>
</tr>
<tr>
<td>1.11</td>
</tr>
<tr>
<td>1.30</td>
</tr>
</tbody>
</table>
## APPENDIX E

### SOLUBILITY DETERMINATION DATA

#### Table E.1. Solubility at 20°C

<table>
<thead>
<tr>
<th>pH</th>
<th>Mg</th>
<th>NH₄-N</th>
<th>PO₄-P</th>
<th>[H⁺]</th>
<th>[Mg]</th>
<th>[NH₄-N]</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>mg/L</td>
<td>mg/L</td>
<td>moles/L</td>
<td></td>
<td>moles/L</td>
<td>moles/L</td>
</tr>
<tr>
<td>Distill</td>
<td>8.07</td>
<td>3.09E+00</td>
<td>5.96E+02</td>
<td>3.79E+01</td>
<td>8.51E-09</td>
<td>1.27E-04</td>
</tr>
<tr>
<td>Water</td>
<td>8.11</td>
<td>5.43E+00</td>
<td>5.93E+02</td>
<td>3.61E+01</td>
<td>7.76E-09</td>
<td>2.24E-04</td>
</tr>
<tr>
<td>at 20C</td>
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Average   | 2.41E-08 | 6.18  | 29.27 | 3591.20 | 147.79 | 7.65| 18.92 |
## APPENDIX F

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## Appendix G

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## APPENDIX H

### CHEMICAL ANALYSIS OF STRUVITE PELLETS

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<td>1.32</td>
<td>1.46</td>
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<td>ug/g (dry)</td>
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## APPENDIX I

### TOTAL PHOSPHORUS AND NITROGEN

Table I.1. Total phosphorus and total nitrogen removal efficiency

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<th>Average</th>
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<td>841</td>
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<td>895</td>
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<td>PO₄-P (mg/L)</td>
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<td>7.7</td>
<td>4.5</td>
<td>6.3</td>
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<tr>
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<td>643</td>
<td>665</td>
<td>837</td>
<td>841</td>
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| Reduction | TP (%) | 84 | 84 | 90 | 85 | 92 | 83 | 86 |
| % Dissolved | TN (%) | 3  | 7  | 15 | 4  | 9  | 8  | 7  |
| PO₄-P (%) | 96     | 93 | 88 | 93 | 89 | 81 | 90 |
| NH₄-N (%) | 2      | 2  | 8  | 7  | 3  | 0  | 4  |
### APPENDIX J

#### MODEL RESULTS

Table J.1. Potts Model

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<th>Error</th>
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<td>PO₄-P (mg/L)</td>
<td>NH₃-N (mg/L)</td>
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<td>670.2</td>
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Table J.3. Neuscience Neuframe Model

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Mean value 23.6 370.9