DEVELOPMENT OF A NOVEL SYSTEM FOR PROGRESSIVE FREEZE CONCENTRATION PROCESS

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CONCENTRATION PROCESS

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A thesis submitted in fulfilment of the
requirements for the award the degree of
Doctor of Philosophy (Chemical Engineering)

Faculty of Chemical and Natural Resources Engineering
Universiti Teknologi Malaysia

AUGUST 2010

DEDICATION

This thesis is especially dedicated to my ever loving wonderful husband, Zaki Yamani Zakaria and my beautiful, beloved children, Ikhwan Firdaus, Marsya Syazweena and Alya Humaira, as well as my beloved parents. You are all the sources of my strength. May Allah keep us all together forever.

ACKNOWLEDGEMENTS

Praise be to Allah. I wish to express my profound gratitude to my supervisors, Prof. Dato' Dr. Rosli Mohd Yunus and Associate Professor Dr. Mohd. Ariffin Abu Hassan, for their constant comments, suggestions, guidance, encouragement and support throughout this work. May all your contributions invested on me will be paid off not only by my success indirectly, but also by blessings from the merciful Allah Azzawajalla.

A very special thanks to my ever loving and helpful husband, Mr. Zaki Yamani Zakaria, for his wisdom in offering me advices either academically or otherwise. He has always been the person I turn to in times of troubles and rough endeavour, whenever I need comfort. Your insights, knowledge, act of wisdom and love, I will cherish forever.

Grateful appreciation to En. Mohd. Rafiza Othman, Puan Noraidah Zhwal and other laboratory staffs for the assistance they offered throughout my experimental work. The hardships I encountered were certainly more or less lightened by the aid offered.

Also to all my dear fellow academicians in FKKKSA who have touched even a tiny part of my research, which would be a long list if I were to mention, millions of thanks for all the inputs you have ever given me, for there are times that I was in a dire need for those helpful ideas and sometimes motivation.

I would also like to acknowledge Universiti Teknologi Malaysia, not only for their financial support but for all other assistances in enlightening my route to this academic success.

Last but certainly not the least, a mountain of love and appreciation towards my father and mother for their encouragement and moral support and doa. May Allah reward all of you here and in the hereafter.

ABSTRACT

Freeze concentration (FC) was investigated focusing in terms of assessment towards benefiting industrial wastewater treatment. A new more productive crystalliser was aimed to be designed as the main component in a progressive freeze concentration (PFC) process, a more economical version of FC which forms ice crystals as a layer or a block on cooled surface. Subsequent analysis on its performance, a process optimisation and heat transfer study were intended following completion of design. As a result, a helical structured copper crystalliser was successfully developed and fabricated named coil crystalliser, designed in such a way to provide high surface area per coolant volume, easy scale up and practical. In the subsequent performance analysis carried out using glucose solution as simulated industrial wastewater treatment, it was found that the effective partition constant (K) was satisfactorily low at high circulation flowrates, low initial concentration and intermediate coolant temperature. Low coolant temperature of -10 °C was observed to cause high ice front growth rate which subsequently promote high solute entrapment in the ice layer formed, affecting its purity. High circulation flowrates of 1000 ml/min on the other hand resulted in low solute inclusion in solid phase thus giving out high ice purity. In terms of volume reduction, the highest achieved was 76.65 % and coolant temperature seems to influence the most where low temperatures resulted in high solution volume reduction as a consequence of high ice growth rate. A process optimisation employing Response Surface Methodology (RSM) in Statistica software to yield the optimum conditions to produce the best K, ice purity (IP) and solution volume reduction (ΔV) generated three regression models, which have been proven adequate by good R-squared, residual and Analysis of Variance (ANOVA) analysis. Interactions between process variables according to the model agree well with the fundamental theory of FC and finally optimum conditions for each response have been identified. The best K predicted was 0.17, 78.5 % for ΔV and 0.05 mg/ml for IP. In the subsequent heat transfer study standard lines for overall heat transfer coefficient (U_o) were plotted. The U_o lines then facilitate in generating a model to predict ice crystal mass produced, originated from a heat balance analysis. Error analyses have proven the model's reliability with R-squared of 0.997 and Absolute Average Relative Deviation (AARD) of 10.6% between experimental and model data, with the highest predicted mass of 973.2g.

ABSTRAK

Pemekatan pembekuan (FC) telah dikaji untuk tujuan penggunaan di dalam olahan air sisa industri. Tujuan kajian adalah untuk merekabentuk penghablur yang lebih produktif, sebagai alat utama di dalam pemekatan pembekuan lapisan (PFC) yang lebih ekonomi di mana hablur ais terbentuk sebagai satu lapisan di atas permukaan yang disejukkan. Analisis ke atas prestasi sistem, optimasi proses dan kajian pemindahan haba juga perlu dilakukan sejurus setelah rekabentuk siap. Akhirnya, sebuah penghablur kuprum berbentuk pilin telah berjaya direkabentuk, dinamakan penghablur gulungan, yang mana boleh memberikan luas permukaan yang tinggi, memudahkan proses pembesaran skala dan praktikal. Di dalam analisis prestasi yang dilakukan menggunakan larutan glukosa sebagai air sisa buatan, pemalar pemisahan berkesan (K) didapati rendah pada kadar alir kitaran yang tinggi, kepekatan asal larutan yang rendah dan suhu cecair penyejuk medium. Suhu cecair penyejuk yang rendah pada -10°C didapati menyebabkan pertumbuhan hablur ais yang tinggi, yang mana menyebabkan penjeratan zat terlarut yang tinggi di dalam ais, mempengaruhi ketulenannya. Kadar alir kitaran yang tinggi pada 1000 ml/min pula menyebabkan penjeratan zat terlarut yang rendah, memberikan ketulenan ais yang tinggi. Dalam konteks penurunan isipadu, nilai tertinggi dicapai adalah 76.65% dan suhu cecair penyejuk dilihat paling banyak mempengaruhi di mana suhu rendah mengakibatkan penurunan yang tinggi disebabkan oleh pertumbuhan ais yang tinggi. Pengoptimuman proses menggunakan kaedah tindakbalas permukaan (RSM) dalam perisian.Statitica untuk memberikan keadaan optimum bagi menghasilkan K, ketulenan ais (IP) dan penurunan isipadu larutan (ΔV) terbaik telah menerbitkan tiga model regresi, yang telah dibuktikan kesahihannya berpandukan nilai R kuasa dua, sisa dan analisa varian (ANOVA) yang baik. Interaksi antara pembolehubah proses berpandukan model tersebut juga didapati selari dengan teori asas pemekatan pembekuan dan akhirnya keadaan yang optimum telah dikenalpasti. Ramalan nilai terbaik pada keadaan optimum untuk K adalah 0.17, 78.5 % untuk ΔV dan 0.05 mg/ml untuk IP. Dalam kajian pemidahan haba, garis-garis piawai untuk pemalar pemindahan haba keseluruhan (U_o) telah diplot. Garis-garis U_o ini membantu dalam menerbitkan satu model ramalan jisim hablur ais yang terhasil, yang mana berasal daripada satu analisa keseimbangan haba. Model yang diterbitkan telah dibuktikan kesahihannya dengan nilai R kuasa dua 0.997 dan Purata Sisihan Mutlak Relatif (AARD) 10.6% di antara data eksperimen dan model, dengan nilai ramalan jisim tertinggi 973.2 g.

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LIST OF SYMBOLS AND ABBREVIATIONS

IWW	-	Industrial wastewater
SSFE	-	Scrapped surface heat exchangers
FC	-	FC
PFC	-	Progressive Freeze Concentration
DOE	-	Design of experiment
CCD	-	Central composite design
RSM	-	Response surface methodology
CC	-	Coil Crystalliser
COD	-	Chemical oxygen demand
RI	-	Refractive index
DSC	-	Differential scanning calorimetric
IP	-	Ice purity
RO	-	Reverse osmosis
K	-	Effective Partition constant
FL	-	Flowrate
CT	-	Circulation time
ISC	-	Initial solution concentration
CTemp	-	Coolant temperature
ΔV	-	Volume reduction
U_o	-	Overall heat transfer coefficient
C_L	-	Concentration of liquid phase
VOC	-	Volatile organic compounds
FBHE	-	Fluidized bed heat exchanger
TSS	-	Total suspended solids
UV	-	Ultra violet

BOD	-	Biochemical oxygen demand
BTEX	-	Benzene, toluene, ethylbenzene and xylene
TCE	-	Trichloroethylene
PCE	-	Perchloroethylene
μ	-	Chemical potential
μ_1	-	Chemical potential of the liquid
μ_s	-	Chemical potential of the solid
T	-	Temperature
C_S	-	Concentration of ice/solid phase
C_L	-	Concentration of liquid phase
V_L	-	Volume of liquid phase
V_o	-	Initial volume of glucose concentration
C_o	-	Initial solution concentration
$\bar{v}_{ice}$	-	Average ice growth
m^2	-	Meter squared
kg	-	Kilogram
ρ_{ice}	-	Mass of melted ice
τ	-	Length of time taken for ice growth
A	-	Area covered by ice on the plate surface
$\omega_{s,\infty}$	-	Mass fraction of the solute
w/w	-	Weight/weight
μms^{-1}	-	Micrometer per second
$u_{s,\infty}$	-	Velocity of ice front
ppm	-	Part per million
F	-	Feed flow
Cp	-	Specific heat of the feed
$d\theta$	-	Differential increase in time
dW	-	Amount of ice crystallized
ΔH	-	Heat of fusion of water
U	-	Overal heat transfer coefficient
A_m	-	Mean area for heat transfer
ΔT_{log}	-	Mean logarithmic temperature difference between feed and refrigerant

h_{amb}	-	Ambient difference between the outside surface of the freezer and the environment
ΔT_{amb}	-	Temperature difference between the outside surface of the freezer and the environment
R_F	-	Freezing ratio
V_S	-	Volume of the solid produced
V_i	-	Initial solution volume
ID	-	Immersion distance
IR	-	Immersion rate
t	-	Time of process
Bx	-	Brixx
Re	-	Reynolds number
D_e	-	Representative diameter
u	-	Average flowrate
u_o	-	Fluid viscosity
De	-	Diameter
S	-	Cross sectional area of the flow
l_p	-	Wetted perimeter
ANOVA	-	Analysis of variance
$K_2Cr_2O_7$	-	Potassium dichromate
Y	-	Predicted response value
B	-	Regression coefficient
X	-	Experimental factor influencing the process
α	-	Extreme values
R	-	Total thermal resistance from inside to outside flow $^oC/W$
h_i, h_o	-	heat transfer coefficient for inside and outside flow, respectively $W/(m^2.^oC)$
k	-	Thermal conductivity of tube material $W/(m.^oC)$
x	-	Thickness of medium wall
K_{exp}	-	K experimental
K_p	-	Model predicted K
ΔV_{exp}	-	Experimental value for ΔV
R^2	-	R-squared
Twi	-	Temperature of the wall at the inlet

Tsi	-	Temperature of the solution at the inlet
Twm	-	Temperature of the wall at the middle point
Tsm	-	Temperature of the solution at the middle point
Tcm	-	Temperature of the coolant at the middle point
Two	-	Temperature of the wall at the outlet
Tso	-	Temperature of the solution at the outlet
Tco	-	Temperature of the coolant at the outlet
A_o	-	Outside surface area
A_i	-	Inside surface areas of tube
h_g	-	Heat transfer coefficient for glucose solution
h_o	-	Heat transfer coefficients for ethylene glycol (50%)
k_i	-	Thermal conductivity of ice
x	-	Thickness of ice layer
A_m	-	Logarithmic mean area
R	-	Radius of copper tube for the CC
T_b	-	Bulk solution temperature
ΔT_b	-	Bulk solution temperature difference, ˚C
T_c	-	Bulk coolant temperature
L	-	Total length of the CC
dw/dt	-	Mass of ice formed in time t
ΔH	-	Heat of fusion of water
FPD	-	Freezing point depression

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1 CHAPTER 1

INTRODUCTION

1.1 Introduction

Solution concentration is a process that would normally separate relatively pure water and leaves behind concentrates in a smaller volume. There are basically three methods available which are evaporation, reverse osmosis (RO) and freeze concentration (FC). Evaporation, a concentration method which produces pure water in terms of condensed vapour would leave behind a concentrated solution by applying heat to reach the solvent boiling point (Geankoplis, 1993). It is the simplest and commonest method but large amount of energy is needed to achieve the boiling point of solvent (in most cases is water) and consuming a large amount of fuel (Ramteke et al., 1993, Keshani et al., 2010).

RO separates the water molecules and the solutes in a solution by using water selective membrane. It can produce almost pure water and uses the least amount of energy as the process involves no phase change (Rodriguez et al., 2000). However, the membrane can easily be clogged during operation by the content of the solution which affects the cost highly when the membrane has to be replaced (Miyawaki et al., 2004). The process efficiency is also limited by the chemical compatibility of the dissolved substances with the membrane. The high osmotic pressure involved is also very expensive to attain thus making RO not favourable to be applied in solution concentration (Rodriguez et al., 2000).

FC, a process that freezes or crystallises water molecules out as ice crystals and leaves behind a highly concentrated solution has become more and more favourable these days. The purging of highly pure water is enabled by the nature of the crystal lattice build up by pure water from an aqueous solution or suspension at temperatures lower than its melting point, rejecting all impurities which remain in the mother liquor (Halde, 1980). Approximately 80% of the dissolved compounds can be concentrated in 25% of the original volume through FC (Maurer et. al, 2006) and the process uses less energy compared to evaporation as the heat of crystallization of water (330 kJ/kg) is lower than heat of vaporisation (2200 kJ/kg) (Lorain et al., 2001).

FC could be engaged in many fields of applications, and among them is in liquid food concentration. Liquid food such as fruit juices are semi finished products for use in further production of fruit juice beverages and powder. The concentration process provides microbiological stability, and also leads to economical packaging, transportation and distribution of final products (Keshani et al., 2010). Because FC does not involve heating, it could successfully provide a concentration process with low loss of volatile components in the juice, thus preserving its original taste and aroma. This dewatering nature makes it the best concentration method in avoiding quality loss of liquid food and dairy products (Nakagawa et al., 2009).

This study however is focusing on another important area that could benefit from FC, which is treatment of wastewater. All life depends on water, ultimately the most valuable liquid on earth. Reputed as the universal solvent as it dissolves more substances than any other liquid, wherever it travels either through the ground or through human bodies, it takes along valuable chemicals, minerals, and nutrients. With this nature, water could easily be contaminated during consumption in a variety of applications on earth, where the polluted and/or contaminated fractions termed wastewater (Mark, 2004).

Although some kinds of water pollution can occur through natural processes, this phenomenon is mostly and frequently a result of human activities. Apart from generation sourced by unavoidable utilisation domestically, the industries are playing a major role in generating and increasing the amount of wastewater generated. Plants and factories are continuously expanded, established and operated as urged by modern living, to produce goods and materials to equip for the lifestyle. In most of the operations water is used as a very important utility, which is consequently converted into industrial wastewater (IWW) usually tainted with various kinds of substances. IWW should rightfully be treated according to the local municipal council or environmental department standard before being disposed off to the environment (Tchobanoglous et al., 1991).

IWW can contain various types of pollutant ranging from chemicals to suspended matters and the type of treatment depends on the type of the pollutants. Wastewater treatment can be divided into three major categories, which are biological, physical and chemical. In short, biological treatments involve the usage of microorganisms in the treatment, chemical treatments involve addition of chemicals to the wastewater to treat it and physical treatments usually engaged mechanical means (Tchobanoglous et al., 1991).

Regardless of the type of treatment afterwards, it is such an advantage if the volume of IWW could be reduced extensively, which in return would result in a reduction in operation cost in terms of the respective necessary utility. Hazardous IWW is frequently treated by incineration. But incineration of an aqueous solution with a solid content of less than 10% requires tremendous power to 'burn' the water and maintain the high temperature necessary to destroy the hazardous compound (Holt, 1999). In addition, the combustion gas produced contributes to the emissions from the process and can rapidly exceed local limits.

The three concentration methods mentioned earlier can provide a treatment for IWW through water removal which would yield concentrated wastewater in a smaller volume and almost pure water that can be readily disposed off to the environment. Despite the simplicity of evaporation process, it is very dangerous when hazardous volatile organic compounds (VOCs) constitute the IWW, (Rogers, 1999) which is one of the major setbacks to the method. With high operation and maintenance cost for RO, FC is seen as an appealing alternative to other wastewater treatment by water removal. It is safe to be applied to wastewater containing hazardous VOCs as it does not involve heating.

1.2 Problem Statement

FC could be carried out in two different methods; suspension freeze concentration (SFC) and progressive freeze concentration (PFC). SFC produces ice crystals as a suspension in the wastewater (Figure 1.1 (a)) and the subsequent separation of this solid-liquid mixture at this freezing temperature is difficult, involving filtration of the ice crystals to separate them from the mother liquor. This involves other unit operations, which are filtration unit at a near freezing temperature and also a washing tower, therefore increasing the capital and operating cost. The size of the ice crystals is also required to be as big as possible because small crystals will retain a large amount of concentrate on its surface and in the voids. This is not easy to be accomplished and require a close attention during operation. SFC also involves a high capital investment cost as it involves the usage of a scraped surface heat exchanger (SSHE) to generate ice crystal seeds, which is very expensive and accounts for roughly 30% of the total investment costs in a FC process (Habib and Farid, 2006).

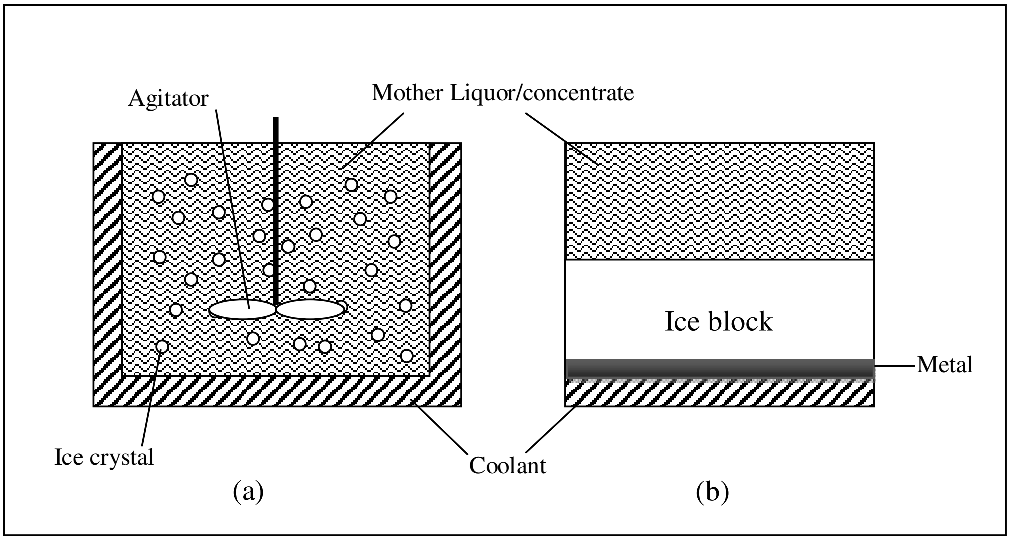

Figure 1.1: Different type of FC: (a) SFC and (b) PFC (Miyawaki et al., 2005)

Researches have been carried out on FC in order to tackle all the limitations of SFC mentioned above and eventually PFC was discovered, which produces ice crystals in forms of a layer or a block on cooled surface. This ice block is not produced as a suspension in its mother liquor but formed at the wall where refrigeration is supplied as illustrated in Figure 1.1 (b). This eliminates the need of a complicated separation mechanism to remove the ice crystal block because the mother liquor can simply be drained out of the system to separate the two phases. It also eliminates the usage of SSHE that is used in producing small ice crystal and only utilizes the refrigeration system to obtain ice layers on the crystalliser surface.

Many kinds of design have been investigated for PFC other than the basic conventional design of PFC, which is just a cylinder with metal bottom supplied with refrigeration and the ice is formed layer by layer vertically. Other designs include ice maker channels in square pillars by Wakisaka et al. (2001), tubular PFC system by Miyawaki et al. (2004) and a multi-plate cryoconcentrator by Raventos et al. (2006) and Hernandez et al. (2009) to name a few.

PFC was proven to be a promising alternative to be applied in treating and concentrating wastewater based on its capability of lowering the capital cost involved in SFC through elimination of several unit operations such as the scraped surface heat exchanger (which is the ice crystal producer and usually contributes 30% of the capital cost) and the washing column. However, as previous related researches revealed, the productivity of such a system is very much lower compared to the conventional SFC. This is mainly due to the nature of the formation of ice layer which is normally very slow. The slow ice layer formation is a vital obstructive factor in making the PFC favourable commercially. Even with the lower capital cost that can be proposed by such a system, it would not be the choice for the industry if the process would not yield a good productivity and efficiency.

With the elimination of the ice crystal producer (SSHE) in PFC, the crystalliser as the equipment where the main heat transfer to occur must be of the right design and carefully outlaid. The ice layer is to be produced at a high rate and simultaneously resulting in ice block with high purity, thus requiring a high surface area per coolant volume ratio. A careful selection process of the suitable non-corrosive metal with high thermal conductivity to be used as the crystallisation surface has to be carried out in order to ensure high productivity. The shape of the crystalliser should also be competent in facilitating future scale of the process. Several heat transfer aspect of the crystallisation chamber design should be investigated as well as the operating condition of the process. An investigation leading to construction of a PFC system of satisfactory productivity and efficiency in order to compete with costly SFC system would be very much beneficial.

1.3 Objective of Study

This study is dedicated to design and develop a new crystalliser, the main component of a PFC process, to enable layer crystallisation of ice for the purpose of wastewater treatment. The objectives of the research are as listed below:

i. To design/develop a new crystalliser for PFC of wastewater;

ii. To study the effect of circulation flowrate on the performance of the crystallisation chamber;

iii. To investigate the effect of coolant temperature on the partition constant (K), solution volume reduction and ice purity;

iv. To explore the efficiency of the crystallisation chamber design by varying the initial concentration of solution;

v. To study the effect of circulation time/period on the performance of the crystallisation chamber;

vi. To determine the optimum process conditions (parameters) using Response Surface Methodology (RSM);

vii. To profile the temperature distribution in order to calculate the overall heat transfer coefficient, U_o, for the PFC process;

1.4 Scope of Research

With the intention of achieving the above-mentioned objective, a series of scopes was implemented. The scopes determine the extent of investigations in achieving all objectives mention in the previous section. Below is the listing of the scopes involved:

i. The designing of the new crystalliser took into account new material of construction, shape to increase surface area and equipped with all relevant features for a successful PFC process;

ii. The range of circulation flowrate studied was 400 to 1000ml/min, to investigate its effect on K, ice purity and solution volume reduction;

iii. The coolant temperature range applied was -4 to -10°C, investigated on the K, solution volume reduction and ice purity;

iv. Initial solution concentration was investigated in the range of 4 to 10mg/ml to explore the efficiency of the crystalliser design;

v. The effect of circulation time/period on the performance of the crystalliser was investigated between 5 to 20 minutes of circulation;

vi. RSM was carried out using STATISTICA software to generate an experimental design, followed by generation of a prediction model with subsequent investigations on the model's adequacy. Interactions between the process variable were also observed and the optimum conditions for the process were generated;

vii. Overall heat transfer coefficient, U_o, was calculated for the PFC process according to different coolant temperature and time, followed by a development of a model predicting the ice crystal mass produced during operation.

1.5 Significance of Research

As this study is focusing on designing a new crystalliser to be used in a PFC process for the purpose of wastewater treatment, it provides an alternative for IWW treatment by water removal. This PFC system should highly be in demand in industries producing IWW with high VOCs, where heating would cause an alarming danger. It is also suitable for removing water from IWW with high total suspended solids (TSS) and heavy metals as most particles are rejected during ice crystal lattice formation, making it a more advantageous solution to RO that would have a high rate of fouling in this situation. This research would also provide an understanding in how to design a PFC system with high productivity and also provide references for future application of PFC in concentration of food and pharmaceutical compounds.

1.6 Outline of the Thesis

Below is how the thesis is arranged in order to report the research carried out:

Chapter 1 introduces the research and highlights the objective and the scopes implemented in order to achieve it.

Chapter 2 titled Theories and Literature Review elaborates the various wastewater treatment methods available today and the advantages and the disadvantages of each method. In this chapter, previous work of researchers all over the world is also reviewed, which consists of researches on suspension FC, performance of various design of PFC FC crystalliser or freezer, evaluation and analysis aspect of the researches, including the ground work by ancient researchers in this field. This chapter also stated and elaborated the crystallisation theories, which is very important in understanding the process itself. The Design of Experiment, Response Surface Methodology (RSM), optimum condition determination and ANOVA were also discussed at the end of the chapter.

Chapter 3 provides explanations of the methodology of the experiment. A research methodology flow chart attached in the beginning of the chapter provides an overview of how the research is undertaken. Later in the chapter, the methods or experimental steps taken through the research are elaborated. Operating parameters investigated are circulation flowrate, coolant temperature, initial concentration of glucose solution and circulation time/period.

Chapter 4 presents the resulted design of the new crystalliser, which is termed, multi-layer crystalliser (CC). Material used and the geometry of the design is elaborated, equipped with all necessary chemical and physical characteristics. The designing work is carried out cautiously, considering the necessary aspects to result in good FC process. It also includes justifications of why each materials or equipment is used in the experimental rig that were used in the investigation.

Chapter 5 reports on the findings and theories to explain the effects of all the operating parameters investigated, which are the process fluid circulation flowrate, coolant temperature, initial concentration of solution and circulation time, on the performance of the PFC system. The performance was indicated by three variables, which are the partition constant, K, ice purity and volume reduction of the process fluid.

Chapter 6 focuses on the optimisation process carried out in order to obtain the optimum value for each operating condition, in order to give maximum performance of the system. The dependant variables investigated were partition constant, K, ice purity and volume reduction. The optimisation process was carried out using Response Surface Methodology, incorporated in Statistica Software. The models generated in order to obtain the optimum conditions corresponding to each respond are properly explained and justified.

Chapter 7 reports on the heat transfer activity occurring during the FC process. The overall heat transfer coefficient, U_o, was calculated for each coolant temperature employed, generating standard lines as reference. A model was also proposed in order to predict the crystal mass produced at any time of the FC operation.

Chapter 8 concludes the research work and summarises all relevant findings generated from the study. Some suggestions to improve the design and to generate more data in the future are also given.