THE PERFORMANCE OF PROGRESSIVE FREEZE CONCENTRATION FOR WATER PURIFICATION USING ROTATING CYLINDRICAL CRYSTALLISER WITH ANTI-SUPERCOOLING HOLES

FARAH HANIM BINTI AB.HAMID

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DEDICATION

Specially dedicated to
my best partner, my wonderful husband, ***Amiro Iqbal Mohd Aminudin****,*
my beautiful and fabulous mother, ***Rohana Sulaiman****,*
forever in memory, my adored late father, ***Ab.Hamid Mohamed****,*
my baby boo, ***Aakif Uthman Amiro Iqbal***
my beloved siblings,
my cute overloaded nephews and nieces,
and not to forget my family in laws.
Thanks for your continuous love, support and prayers.

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ABSTRACT

Progressive freeze concentration (PFC) is a freeze concentration method which forms ice crystals as a layer or a block on a cooled surface, which can be applied in purification of seawater to obtain pure water in ice form and leave the impurity behind. The aim of this study is to design a rotating cylindrical crystalliser with anti-supercooling holes in order to prevent supercooling phenomenon as well as to improve the solution movement in the crystalliser to increase productivity of the PFC system. Prevention of supercooling is important as it will affect the purity of ice produced. The performance analysis was carried out by using saline solution as simulated seawater, and was evaluated by the value of effective partition constant (K), desalination rate (R_d), efficiency (E %) and solute recovery (R_s). The system performance was found to be at its best at 300 rpm of rotation speed, four hours of rotation time, coolant temperature at -8 °C and 35 g/L of initial concentration with K value, E%, R_d and R_s of 0.376, 62.37%, 35.71% and 0.672, respectively. Low K value, high E%, high R_d and high R_s represent the best performance due to higher purity of ice crystal produced. Response surface methodology (RSM) in STATISTICA software was employed for optimisation process to obtain the optimum conditions in producing the best K value and R_d. Due to the limitation of optimization process by RSM offered by STATISTICA, a hybrid Artificial Neural Network and Genetic Algorithm in MATLAB was implemented for multiple response optimisations, where the best K value and R_d predicted were 0.26 and 49%, respectively. A mathematical heat transfer model in predicting ice crystal growth at different coolant temperature was successfully developed with an Average Absolute Relative Deviation of 5.56% and R^2 value of 0.873. This newly designed crystalliser was found to be capable of producing ice crystals with high efficiency and productivity.

ABSTRAK

Pemekatan pembekuan progresif (PFC) adalah satu kaedah pemekatan pembekuan yang membentuk kristal ais sebagai satu lapisan atau bongkah di atas permukaan sejuk, yang mana telah digunakan dalam penulenan air laut bagi mendapatkan air tulen dalam bentuk ais dan menyisihkan bendasing. Tujuan kajian ini adalah untuk membangunkan satu silinder penghablur berputar bersama lubang anti-penyejukan lampau bagi menangani fenomena penyejukan lampau dan juga memperbaiki pergerakan larutan di dalam penghablur berkenaan untuk meningkatkan produktiviti sistem PFC. Pencegahan masalah penyejukan lampau adalah penting kerana ia akan menjejaskan ketulenan ais yang terhasil. Analisa prestasi telah dijalankan dengan menggunakan larutan garam sebagai air laut simulasi, dan telah dinilai oleh nilai pemalar pemisahan berkesan (K), kadar penyahgaraman (R_d), kecekapan (E%) dan dapatan bahan larut (R_s). Prestasi sistem yang terbaik ditemui pada kelajuan putaran 300 rpm, masa pusingan selama empat jam, suhu penyejuk pada -8 °C dan kepekatan larutan awal pada 35 g/L dengan nilai K, E%, R_d dan R_s masing-masing adalah 0.376, 62.37%, 35.71% dan 0.672. Nilai K yang rendah, E% yang tinggi, R_d yang tinggi dan R_s yang tinggi mewakili prestasi terbaik kerana ketulenan kristal ais yang dihasilkan adalah lebih tinggi. Kaedah gerak balas permukaan (RSM) dalam perisian STATISTICA telah digunakan untuk proses pengoptimuman bagi mendapatkan keadaan optimum dalam menghasilkan nilai K dan R_d yang terbaik. Disebabkan oleh keterbatasan proses pengoptimuman oleh RSM yang dapat diberikan oleh STATISTICA, satu gabungan Rangkaian Neural Tiruan dan Algoritma Genetik dalam MATLAB telah dilaksanakan untuk pengoptimuman beberapa tindak balas untuk mendapatkan keadaan optimum, di mana nilai K dan R_d terbaik yang diramalkan masing-masing adalah 0.26 dan 49%. Satu model matematik pemindahan haba dalam meramalkan kadar pertumbuhan kristal ais pada suhu penyejuk yang berbeza telah berjaya dibangunkan dengan Purata Sisihan Mutlak Relatif adalah 5.56% dan R^2 adalah 0.873. Penghablur dengan reka bentuk baru berkebolehan menghasilkan kristal ais yang mempunyai keberkesanan dan produktiviti yang tinggi.

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LIST OF SYMBOLS

K - effective partition constant

R_d - desalination rate

Y - solute yield

E % - efficiency of concentration

U_o - overall heat transfer coefficient

C_L - solute of concentrations in the solution phases

C_i - concentration of ice

C_S - solute concentrations in the ice

C_o - the solute concentration at the beginning in the solution phase

V_L - concentrate volume

V_o - initial volume of the solution

m_s - mass in the concentrated

m_o - mass in the initial solution

$m_{s\,liq}$ - solute mass in the liquid phase

$m_{s\,0}$ - initial solute mass

Q - amount of heat transferred

A - area for heat transfer

ΔT_{min} - effective temperature difference

R - total resistance

A_i - inside surface area of tube

A_o - outside surface area of tube
h_i - heat transfer coefficient for solution
h_o - heat transfer coefficient for coolant
x - thickness of ice layer
K - thermal conductivity
A_m - logarithmic mean area
K_o - limiting partition coefficient
Y - kinematic viscosity
μ - dynamic viscosity
P - density of fluids
R_e - Reynolds number
V - velocity
D - internal diameter of pipe
h_f - head loss
F - appropriate dimensionless factor
G - acceleration of gravity
L - length of straight pipe
R_s - solute recovery
S_o - initial salinity of the solution
S_i - salinity of ice produced
S_l - concentration of NaCl in the concentrated solution
T_i - temperature of the ice layer interface
T_o - temperature of inside wall
T_w - temperature of outside wall
λ_i - heat conductivity coefficient of the ice
δ - thickness of the wall
λ_w - heat conductivity coefficient of the wall
L - latent heat of fusion, J/kg

dm_i/dt - ice growth rate

α_s - heat transfer coefficient of seawater solution

N_{Nu} - Nusselt number

N_{Pr} - Prandtl number

μ_b - fluid viscosity at bulk average temperature

μ_w - viscosity at wall temperature

LIST OF ABBREVIATIONS

RO - reverse osmosis
FC - freeze concentration
SFC - suspension freeze concentration
PFC - progressive freeze concentration
COD - Chemical oxygen demand
RSM - Response Surface Methodology
ANN - Artificial Neural Network
MSF - multi stage flash
HTS - heat transfer surface
FBHE - fluidized bed heat exchanger
SSHE - scraped surface heat exchanger
STC - stirred tank crystalliser
ANOVA- analysis of variance
MOO - multi objective optimization
GA - genetic algorithm
CCD - central composite design

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CHAPTER 1

INTRODUCTION

1.1 Research Background

Water purification is categorized as an extremely important process to ensure that the water is safe to use and free from any contaminants that can give bad effect to health. Water purification can be achieved through concentration method. Several concentration methods have been developed since the last few decades in order to meet the growing demand in the industry. Among these, three methods have found their commercial applications which are evaporation, reverse osmosis (RO) and freeze concentration (FC).

In evaporation method, due to high latent heat, a vast amount of fuel and thermal energy is consumed in order to evaporate water. As a result, this process has higher operating costs and environmental problems. Meanwhile, in RO process, the membranes are sensitive to pH, oxidizers, a wide range of organics, algae, bacteria and particulates (Miller, 2003). As a consequence, the cost of RO is high due to the maintenances of membrane. In addition, the osmotic pressure applied in this method also leads to high energy consumption which contributes to operation costs increment (Rodriguez *et al.*, 2000).

However, among all the technologies introduced before, freeze concentration is thought to present a splendid opportunity to be applied because in terms of energy, it is more favourable than evaporation (Englezos, 1994). Freeze concentration is one of the methods to concentrate solution by producing pure ice in the solution. It crystallises out the water from liquid solution in the form of ice crystals and leaves a highly concentrated solution behind (Okawa *et al*., 2009). According to Melak *et al*. (2016), freeze desalination has a lower energy requirement compared to other thermal processes.. Moreover, the latent heat of water solidification (334 J/g) is much lower than heat of vaporization (2257 J/g) (Nakagawa *et al.*, 2010), where the quantity of latent heat of solidification is about 1/7 of the latent heat of vaporization in the same condition (Gao *et al.*, 2014). Due to a low temperature operation, where corrosion is not significant, the freeze concentration process has lower production costs, requires less maintenance and with less precipitation (Luo *et.al.*, 2010). There are two methods in freeze concentration, which are suspension freeze concentration (SFC) and progressive freeze concentration (PFC).

SFC is a method to produce small ice particles. Eventhough it achieves high efficiency of concentration, but the ice particles formed in this method are difficult to be separated from the concentrated solution (Okawa *et al*., 2009). In addition, the size of ice crystal is still limited in this conventional method (Gu *et al.*, 2005). This process is unfavourable because it needs many equipments in order to complete the process, involving separation of ice and mother liquor. The steps involved make the whole process very expensive.

PFC has been introduced in order to amend the weaknesses of SFC. In this process, the ice will be formed as a large single ice crystal instead of small particles. The ice crystal will be formed on the surface of the heat conducting material where the cooling is supplied. The separation of ice crystal from its mother liquor is much easier to be handled compared to SFC as only a single crystal is formed. Due to this reason, this process requires a lower cost since it needs no series of equipments. However, despite of it being easy to handle, the productivity of PFC is discovered to be much lower than the conventional SFC.

Therefore, there are several designs of the crystalliser that have been developed in order to improve the productivity of freeze concentration system and in the meantime, to reduce the high investment costs. Several designs have been introduced, designed, constructed and operated under different conditions in order to amend the weaknesses and reduce the cost from previous design. Tubular ice system was introduced in 2005 by Miyawaki *et al.* (2005) while cylindrical vessel in vertical was introduced by Ramos *et al*. (2005) which is altering the previous design from Liu *et al*. (1998). From time to time, the crystalliser design has been developed in order to adapt with the demand of quality and quantity of the product.

1.2 Problem Statement

In PFC, solution movement is very important because it will affect the product quality and productivity. The problem of low productivity is basically due to low distribution of heat from the solution to the coolant which is essentially influenced by solution movement. Therefore, solution movement plays a main role to provide optimum heat distribution. From previous studies, several problems of solution movement have been detected. For instance, a typical stirrer introduced by Liu *et al*. (1998) and Ramos *et al*. (2005) face the problem of topical movement, where the solution will only have a strong and fast circulation near the rotating blade. In other words, the movement is only focused at one place, resulting in uneven distribution of the solute, thus will affect the quality of ice produced. For falling film introduced by Sánchez *et al.* (2011) and Moreno *et al*. (2014a), the disadvantage of this type of solution movement is it is affected by ambient temperature. Meanwhile, for pumping type (Jusoh *et al.*, 2009; Miyawaki *et al.*, 2005), both designs face a problem of flow pattern since it has bends, which contribute to producing uneven flow. This phenomenon can give an affect to the consistency of ice purity produced. Therefore, it is important to come up with a new approach to provide solution movement.

Apart from the disadvantages of existing designs of PFC which include low productivity and poor ice quality, the phenomenon of supercooling is a serious problem that should be concerned on (Liu *et al.*, 1998). Ice seeding process needs to be done to address this problem if there is no special design feature introduced to the crystalliser. The main problem of the ice seeding process is it consumes longer operation time. In addition, with this extra requirement, the procedure will involve too many steps which will lead to the process becoming more complicated. Therefore, it is essential to develope a new design of crystalliser with a feature to overcome the supercooling problem.

1.3 Objective of Study

In order to complete this research successfully, several objectives have to be achieved. The objectives of this research are as follows:

1. To design a rotating cylindrical crystalliser with anti-supercooling holes as the main component in PFC system.
2. To evaluate the performance of the newly designed crystalliser for PFC system at various operating conditions which are coolant temperature, rotation time, rotation speed and initial concentration, and to determine the optimum conditions.
3. To develop a mathematical heat transfer model on prediction of ice crystal growth.

1.4 Scope of Study

To achieve the objectives of this study, these following scopes were applied:

1. A new crystalliser with cylindrical shape was designed taking into consideration the aspects of construction material, solution movement and design feature in preventing supercooling problem. The additional features for ice sampling and temperature profiling purpose were also introduced. The study of fluid mechanic in term of flow pattern and friction study of the PFC system was also investigated. In addition, the holes capability to freeze out water molecules was determined through some additional experiments. These experiments were carried out to examine the influence of holes numbers towards ice purity produced.

2. The efficiency of PFC system was determined based on the effect of operating parameters which are rotation speed of crystalliser, rotation time, initial concentration and coolant temperature. The range for rotation speed is 150 rpm to 350 rpm. Meanwhile the range for rotation time is an hour to five hours, initial concentration at 25g/L to 45g/L and coolant temperature at -11°C to -7°C. The performance of the system was measured by observing the changes in effective partition constant, (K), desalination rate (R_d), efficiency of concentration (E%), water recovery and solute recovery (R_s). Saline solution was used as target liquid. Optimization for this system was analysed by using Response Surface Methodology (RSM) via Statistica software and Artificial Neural Network (ANN)

3. A new model of heat transfer was developed in order to predict the growth of ice formed based on a modification of convective heat transfer coefficient according to the system condition.

1.5 Significance of Study

In this research, this newly developed crystalliser design is believed to be able to improve the quality and productivity of the PFC system. The cylindrical shape is equipped with anti-supercooling holes to prevent supercooling phenomenon and improvement of the productivity could also be attained by introducing a new approach for solution movement. This research is able to provide the fundamental in designing a crystalliser with high performance and better quality and productivity. In addition, this crystalliser is focused to be applied for water purification process which could be an important new source of water supply, since water demand is dramatically increasing over the world recently in areas with fresh water supply scarcity. High quality water supply is expected to be produced, which is safe and compatible with the standard of World Health Organisation. Furthermore, this research can also be a reference for the industries since PFC can also be applied in other industrial processes such as food concentration, wastewater treatment and pharmaceutical.

1.6 Thesis Outline

This thesis is written in 7 chapters and the chronology for each chapter is briefly elucidated below.

The first chapter is Chapter 1 which presents the research background of research, the problem statement and the research objectives. The scopes of the study are also introduced as a guideline in order to achieve the objectives of the study.

This is followed by Chapter 2 where a review on previous studies on freeze concentration and specifically on progressive freeze concentration is elaborated. The previous technology on water purification is reviewed and the advantages and disadvantages for each method are listed. Furthermore, the design for PFC setup is also discovered where the improvement for each design can be seen from times to times. A brief review about optimisation process via Response Surface Methodology (RSM) and multi response optimisation via Artificial Neural Networks (ANN) is also presented in this chapter. Finally, a fundamental of heat transfer model in PFC is explicated at the end of this chapter.

Then, the procedures involved which starts with designing the crystalliser, experimental set up, experimental works including evaluation method are demonstrated in Chapter 3. A flow chart of research methodology is also provided in this chapter. Elaboration on the details in developing a crystalliser named rotating crystalliser with anti-supercooling holes is presented. This includes the procedures in material selection, determination of dimension, and introduction of special features. The procedure of optimisation process is elucidated in detail, followed by the development of heat transfer model in predicting ice crystal growth.

All the results and discussion on the geometrical analysis such as fluid mechanics, friction study and holes test and the effect of all the four parameters including rotation speed, coolant temperature, rotation time and initial concentration is covered in Chapter 4. Five determinant parameters were employed in order to determine the performance of the system which are effective partition constant (K), desalination rate (R_d), solute recovery (R_s), water recovery and concentration efficiency (E%).

Chapter 5 expounds the optimisation procedures in order to determine the optimum condition for the rotating crystalliser with anti-supercooling holes system by implementing Response Surface methodology via Statistica software. Furthermore, the optimum level of each variable and the effect of their interactions on the response which is K value and desalination rate (R_d) as a function of two variables were studied by plotting three dimensional response surface curves known as contour plots. In addition, the contour plots were employed in aiding visualization of the interaction. However, a set of optimum condition is required to simultaneously obtain a low value of K and a high value of desalination rate (Rd). Therefore, a multiple objective optimisation was implemented in order to find out an experimental optimal point for both responses. For this reason, a hybrid of ANN-GA was used to achieve the optimisation objective.

The development of heat transfer model is explained in Chapter 6. A new model was developed in order to predict the ice crystal growth for the system. By considering the new system of rotating crystalliser with anti-supercooling holes, a modification of convective heat transfer coefficient is done according to the condition of the new system such as type of flow and heat transfer area. A fundamental heat transfer model and several assumptions were employed in developing the new model. Furthermore, the validation of the model is also provided at the end of the chapter in order to determine the model appropriateness.

Finally, Chapter 7 presents the conclusion for each of the study objective. A brief summary for the results and discussions has been made and presented in this chapter. Furthermore, several suggestions and recommendations are advocated for future research study.

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