DEVELOPMENT OF A SPIRAL FINNED CRYSTALLIZER FOR PROGRESSIVE FREEZE CONCENTRATION PROCESS

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DEDICATION

This thesis is especially dedicated to my ever loving husband, Muhamad Fadli Samsudin and my beautiful daughter, Fayra Madeena. A special feeling of gratitude also goes to my loving parents, Samsuri Zakaria and Atikuoh Ibrahim who have supported me all the way since the beginning of my studies, as well as my parent-in-law.

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ABSTRACT

Progressive freeze concentration (PFC) has emerged as a viable technology for concentration of liquid solution. For this present research, a new spiral finned crystallizer was designed and fabricated as the main component in the PFC system. The spiral finned crystallizer was designed with the aim of increasing productivity and quality of product. Further analysis on its performance, a process optimization and modelling study were carried out after the completion of the design. For the performance analysis, glucose solution was used as a liquid food model solution. The performance of the crystallizer was analysed through the system efficiency assessed in parallel with the effect of operating conditions. It was found that the effective partition constant (K) was satisfactorily low at intermediate coolant temperature, high circulation flowrate, intermediate circulation time and intermediate shaking speed. A low K value and a high solute recovery (Y) value represent the best performance of the PFC system. In terms of Y, the highest achieved was approximately 0.98 g of glucose obtained per 1 g of initial glucose. A mass validation was successfully obtained from the experimental results. The evaluation of the crystallizer in terms of ice production, fluid mechanic and heat transfer characteristics was also carried out. 0.64 g/m^2s^1 of maximal ice production was attained, reflecting a good function of the spiral fin. A process optimization employing Response Surface Methodology (RSM) in Statistica software was applied to study the relationships of coolant temperature, circulation flowrate, circulation time and shaking speed on K and Y. The optimum conditions to produce the best K and Y were found to be 10.30 °C of coolant temperature, 3097.50 mL/min of circulation flowrate, 64 minutes of circulation time and 29.53 ohm of shaking speed. The best K predicted was 0.25 and 0.99 for Y. A heat transfer model was also successfully developed in order to study ice crystal mass formation.

ABSTRAK

Pemekatan pembekuan progresif (PFC) telah muncul sebagai teknologi yang berdaya maju untuk pemekatan larutan cecair. Untuk kajian ini, satu penghablur bersirip lingkaran baru telah direka dan dibentuk sebagai komponen utama dalam sistem PFC. Penghablur bersirip lingkaran telah direka dengan tujuan untuk meningkatkan produktiviti dan kualiti produk. Analisa lanjut mengenai prestasinya, proses pengoptimuman dan kajian pemodelan telah dijalankan selepas selesai proses mereka bentuk. Untuk analisa prestasi, larutan glukosa telah digunakan sebagai larutan model makanan cecair. Prestasi penghablur dianalisa melalui kecekapan sistem yang ditaksirkan selari dengan kesan keadaan operasi. Telah didapati bahawa pemalar pemisahan berkesan (K) adalah rendah pada suhu penyejuk yang sederhana, kadar aliran peredaran yang tinggi, masa peredaran yang sederhana dan kelajuan gegaran yang sederhana. Nilai K yang rendah dan nilai dapatan bahan larut (Y) yang tinggi mewakili prestasi terbaik bagi sistem PFC. Dari segi nilai Y, nilai yang paling tinggi dicapai adalah lebih kurang 0.98 g glukosa diperolehi bagi setiap 1 g glukosa awal. Keseimbangan jisim telah berjaya mengesahkan keputusan ujikaji yang diperolehi. Penilaian bagi penghablur dari segi penghasilan ais, mekanik bendalir dan pemindahan haba telah dijalankan. 0.64 g/m^2s^1 pengeluaran ais maksimum telah dicapai, mencerminkan fungsi yang baik oleh sirip lingkaran. Proses pengoptimuman menggunakan kaedah gerak balas permukaan (RSM) dalam perisian Statistica telah digunakan untuk mengkaji hubungan antara suhu penyejuk, kadar aliran peredaran, masa peredaran dan kelajuan gegaran pada K dan Y. Keadaan optimum untuk menghasilkan K dan Y yang terbaik adalah pada suhu penyejuk 10.30 °C, kadar aliran peredaran 3097.50 mL / min, masa peredaran 64 minit dan kelajuan gegaran 29.53 ohm. Nilai K yang diramalkan adalah 0.25 dan 0.99 untuk Y. Model pemindahan haba juga telah berjaya dibangunkan untuk mengkaji pembentukan kristal ais.

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF SYMBOLS AND ABBREVIATIONS

RO - Reverse osmosis
FC - Freeze concentration
SFC - Suspension freeze concentration
PFC - Progressive freeze concentration
RSM - Response surface methodology
ANOVA - Analysis of variance
CCD - Central composite design
SS - Sum of squares
SST - Total sum of squares
DF - Degree of freedom
MS - Mean square
DOE - Design of experiment
K - Effective partition constant
C_S - Solute concentration in the ice
C_L - Solute concentration in the solution
V_L - Volume of the liquid phase
Vi - Volume of ice phase
V_O - Initial volume to be concentrated
C_O - Initial concentration of the solution
Y - Solute recovery
CT - Coolant temperature
CF - Circulation flowrate
TM - Circulation time
SS - Shaking speed
m_L - Mass of concentrated solution

m_O - Mass of initial solution

T - Temperature

V - Volume

dS/dt - Entropy change with time

Θ - Entropy production per unit time and volume

J_q - Flux of heat

J_{H2O} - Mass fluxes of water

J_s - Mass fluxes of solute

$\nabla \ln T$ - Thermodynamic force for heat transport

c_S - Concentration of solute

c_{H2O} - Concentration of water

$\nabla \mu_{i,T}$ - Concentration dependent part of the chemical potential gradient

υ_{ice} - Ice velocity (ice growth rate)

V_{ice} - Molar volume of ice

V_{H2O} - Molar volume of water

l - Phenomenological coefficients in flux equations

Δ - Film thickness

$\Delta_f H$ - Enthalpy of freezing

R - Universal gas constant

ρ_{liq} - Density of liquid

c_i - Concentration of interface between ice and solution

ρ_{ice} - Density of ice

k_c - Mass transfer coefficient

k_i - Kinetic coefficient

$(dc/dt)_{eq}$ - Slope of phase diagram

h - Heat transfer coefficient

T_b - Bulk temperature

c_b - Bulk concentration

F - Feed flow

C_p - Specific heat of the feed

ΔT - Temperature difference between inlet and outlet in the feed

$d\theta$ - Differential increase in time

dW - Amount of ice crystallized in $d\theta$

H - Heat fusion of water

U - Overall heat transfer coefficient

A_m - Mean area for heat transfer

ΔT_{log} - Mean logarithmic temperature difference between feed and refrigerant

H_{amb} - Heat transfer coefficient for losses to the environment

ΔT_{amb} - Temperature difference between the outside surface of the freezer and the environment

A_i - Area of the aluminium tube

A_e - Area of the aluminium tube plus the ice layer

A_m - Mean area for heat transfer

h_s - Heat transfer coefficient on the falling film side

k_{ice} - Thermal conductivity of ice

h_R - Heat transfer coefficient on the refrigerant side

R - Tube radius

L - Length

Δx - Thickness of the ice later formed

dx - Differential thickness increase

ρ_h - Ice density

$\%f$ - Percentage of fructose

$\%g$ - Percentage of glucose

$\%s$ - Percentage of sucrose

m_f - Molecular weight of fructose

m_g - Molecular weight of glucose

m_s - Molecular weight of sucrose

M_{S-S} - Molecular weights of sucrose

M_{S-G} - Molecular weights of D-glucose

M_{S-m} - Molecular weights of juice

$T_{S,Bx}$ - Freezing points of sucrose

$T_{G,Bx}$ - Freezing points of glucose

$T_{m,Bx}$ - Estimation of the freezing point at the concentration (°Brix) of interest

M_p - Mass of particle

U_{p-h} - Horizontal component of particle velocity
ε - Bed porosity
d_p - Particle diameter
$m_{crystallized}$ - Mass of ice crystallized
$\bar{m}_u$ - Ice production per unit of surface area and time
M - Net mass of ice
A - Surface area
t - Time of each experiment
N_{Re} - Reynolds number
D - Diameter
υ - Average velocity of the fluid
ρ - Fluid density
μ - Fluid viscosity
f - Friction factor
c_p - Specific heat of solution
t_1 - Time at point 1
t_2 - Time at point 2
T_{b1} - Bulk temperature of solution at point 1
T_{b2} - Bulk temperature of solution at point 2
T_c - Temperature of coolant
T_{c1} - Inlet temperature of coolant
T_{c2} - Outlet temperature of coolant
ΔH - Fusion heat of freezing
τ - Time taken
t_3 - Time at point 3
t_4 - Time at point 4
θ - Freezing point temperature of solution
T_c - Temperature of coolant
C_3 - Solute concentration of unfrozen solution at time t_3
C_4 - Solute concentration of unfrozen solution at time t_4
k - Thermal conductivity
η_f - Fin efficiency
m - Molarity
$\omega_{s.ice}$ - Solute mass fraction in ice

x - Distance

u - Growth

R^2 - R-squared

LIST OF APPENDICES

CHAPTER 1

INTRODUCTION

1.1 Research Background

Increasing a solution concentration is necessary in terms of transportation, conservation and manufacturing in food industry, desalination and wastewater treatment. There are several methods in concentration and water removal process such as evaporation, reverse osmosis (RO) and freeze concentration (FC). Many efforts have been committed to develop improved methods for concentration and water removal process. In evaporation, higher level of concentration can be obtained compared to FC and RO. However, there are impairment of sensory (colour, taste and aroma) and nutritional value of the finished product such as vitamin because of the heat induced (Gulfo *et al.*, 2014b). Thus, the quality of the product is poor. Moreover, high energy is demanded in vacuum evaporation which can contribute to higher cost (Randall *et al.*, 2011).

In RO, water is removed as permeate through the membrane without the phase change and heat is not supplied for separation of water. Thus, energy requirement is low, thermal damage to the product is low thus resulting in better flavour and colour retention compared to evaporation. The performance of RO depends on the types of membrane (Prerana Dayasagar, 2004) and the cost of membrane is quite high (Sánchez *et al.*, 2011b). A single stage of RO system cannot achieve a concentration higher than 25 – 30° Brix, which is much lower than 45 – 65° Brix for products obtained from evaporation (Jiao *et al.*, 2004). In addition, one major problem of RO is fouling which can affect the quality of the water produced.

Water may also be removed by freezing out water as ice crystal. FC is the process of concentrating a solution by freezing out the water content into ice crystals (Sánchez *et al.*, 2011b). The aim of FC is to form very pure ice crystal where there is only water without any solids retained in the ice crystal (Hernández *et al.*, 2010). The resulting solid and liquid phase are then subsequently separated as ice and concentrated solution (Berk, 2009). During ice crystal formation, solutes are rejected by the nature of ice crystal lattice formation which is formed by pure water (Jusoh, 2010). Water solidification process forming the small dimension ice crystal lattice makes the inclusion of any impurities impossible except for fluorohydric acid and ammonia, thus there is no solute contaminants in ice (Lorain *et al.*, 2001).

As compared to evaporation and reverse osmosis, FC has some benefits in producing high quality concentrated solution because the use of cold temperatures can reduce the losses caused by volatility and chemical reactivity (Kobayashi and Lee, 1964). There is no loss of volatiles in FC because no high temperatures are used and no vapour-liquid interface exists (Petzold and Aguilera, 2013). Therefore, higher flavour and quality of freeze concentrated products are produced. FC has been applied for various industrial requirements involving fruit juices, milk products, sugar solutions, brackish water and wastewater. However, practical application of FC is limited due to the complexity of its equipment and the high capital investment (Zhang and Hartel, 1996; Rodríguez *et al.*, 2000; Roos *et al.*, 2003; Miyawaki *et al.*, 2005; Habib and Farid, 2006).

FC can be divided into suspension freeze concentration (SFC) and progressive freeze concentration (PFC) (Muller and Sekoulov, 1992; Wakisaka *et al.*, 2001;

Miyawaki *et al.*, 2005; Kawasaki and Matsuda, 2008; Aider and de Halleux, 2009). The more conventional SFC consists of steps including nucleation, crystal growth and separation of ice crystals, involving a scraped-surface heat exchanger (SSHE) and a recrystallizer (Lemmer *et al.*, 2001; van Nistelrooij, 2005; Otero *et al.*, 2012). Small ice crystals are produced in the SSHE after being pumped from a feed tank. SSHE is the most expensive processing unit in an FC plant where 30% of the total investment costs comes from SSHE (Habib and Farid, 2006). SSHE outflow containing small ice crystals is fed to the recrystallizer where they are mixed with larger crystals. The crystal growth takes place in the recrystallizer as a result of the Gibbs-Thomson effect (Ostwald ripening) (Miyawaki *et al.*, 2016). From the recrystallizer, a slurry flow is transported to a separation device where the ice crystals are separated from the concentrated liquid (Lorain *et al.*, 2001). The separation and washing of the ice crystals is important in SFC because the solutes present in the solution will contaminate the ice crystals by adhering to the ice crystals. The efficiency of SFC is found to be high but it is difficult to separate the ice crystals from the concentrated liquid because of the large surface area. As a result, this method needs very complicated system which makes the SFC process the most expensive method among other concentration methods (Moreno *et al.*, 2013).

Nowadays, many developments of FC are associated with PFC because of the simpler separation step (Moreno *et al.*, 2014a). PFC is a type of FC that progressively produces ice crystal layer by layer on a cooled surface until it forms a large and single ice crystal block (Miyawaki *et al.*, 1998). Large ice crystal has fewer impurities or amount of solutes than small ice crystal (Shirai *et al.*, 1987; Kobayashi *et al.*, 1996; Widehem and Cochet, 2003). So, the purity of ice crystals produced from PFC is much higher than purity of ice crystals produced from SFC. Since there is only a single block of ice crystal, it would be much easier to separate the ice crystal from the mother solution which in turn resulting in low operation and maintenance cost (Jusoh *et al.*, 2008a).

Although PFC has been proven to yield quality of the product of better than SFC, its productivity is still lower than SFC. Productivity is a measure of the efficiency of the FC system to produce ice crystal and it is calculated from the amount of the ice crystal produced by the system. Various studies have been done so that the quantity of the product can be increased. Different kinds of design have been investigated in order to obtain high quality and higher efficiency. In 1997, Liu *et al.* had developed a vertical PFC system which is composed of a cylindrical sample vessel of stainless steel, a cooling bath and a driving system to move the sample vessel into the cooling bath at a constant speed. This PFC system was applied to a solution containing glucose and/or blue dextran as a model liquid food. As a result, only a single ice crystal grows and the ice crystal separation from the concentrated solution is relatively easy.

In order to increase productivity and to get high yield of product, tubular ice system was developed by Miyawaki *et al.* (2005). The apparatus of the tubular ice system was composed of two straight pipes, bent pipes at the top and bottom and pump for circulation. The tubular ice system has concentrated coffee extract, tomato juice and sucrose solution to high concentrations with excellent yields. Nakagawa *et al.* (2010) employed a batch crystallizer in order to use solute elution from a frozen matrix as a concentrating operation. A batch crystallizer was made up to freeze solution with a jacket cooler and a rod heater was set at the centre of the crystallizer in order to make the solution contact with the frozen zone. Almost all the solute could be recovered from the original solution. In 2012, Rich *et al.* developed a dynamic layer crystallizer for freezing desalination of sea water. The crystallizer consists of a stainless steel tube immersed in a cylindrical double jacketed tank. As a result, an ice layer was formed on the external surface of the tube during the freezing step.

1.2 Problem Statement

Since a long time ago, many efforts have been made to improve the PFC system where it is proven that it can be applied to concentrate fruit juices, wastewater, pharmaceutical and sea water. However, some of the previous PFC system produces low productivity of the product because of small surface area of the cooling surface (Liu *et al.*, 1997), consists of many equipments where the system have seven different types of tank, ice maker, compressor unit, pumps and pipes (Wakisaka *et al.*, 2001), have too long of process time which is more than 10 hours and cause the process becomes costly (Ramos *et al.*, 2005), has irregular flowrate because of the bent pipes (Miyawaki *et al.*, 2005) and have the formation of foam in the process that obstructed the movement and distribution of the solution which reduced the heat transfer (Sánchez *et al.*, 2010). All of this has motivated researchers in this field to build up method for obtaining higher quality of ice crystal with higher productivity. At the same time, the system must be simple and easy to operate. The PFC system is recognized as the good alternative if it can produce high quality of product with high productivity.

High quality of product can be defined by high purity of ice crystal and high concentration of concentrated solution. The solutes must be expelled from the ice crystal interface to increase concentration of the solution (Rodrigues *et al.*, 2011). The major difficulty of PFC operation is how to control the freezing front velocity because the solutes can be easily trapped in the ice crystal when the freezing front velocity is too fast. Thus, in order to exclude solutes from the ice crystal, ice crystal growth rate must be lower than a limit value during the whole PFC process. The limit is recognized as a critical growth rate that determines the quality of ice crystal produced. The ice crystal growth rate must be controlled with the suitable operating conditions where its value must not be too low or too high.

As an alternative to overcome all the weaknesses presented by the previous designs, a new design of PFC system has been developed in this study. The design of the new PFC system, which is a new crystallizer with spiral fins as the main part, provides highest contact surface area and optimum flow characteristic. The increase of the contact surface area enhances the heat transfer between solution and coolant. The ice crystal is grown on the inside surface that is being cooled by the coolant. The productivity can be increased with the increase of contact surface area between the coolant and the solution through the cooling surface. The improvement in concentration efficiency of the PFC process assisted by the additional spiral fins has been experimentally examined. By providing increased contact surface area by the additional spiral fins, this crystallizer attempted to improve the concentration performance by increasing the heat transfer between solution and coolant. Although the theory and practice of fin has been widely studied by many researchers for different applications of heat exchangers, the efficiency of fin in a crystallizer has not been studied. In addition, the improvement in concentration efficiency of the PFC process also has been assisted by shaking of the solution. A shaker has been added as an assisted technique for this PFC system.

Nowadays, crystallization process has been abundantly used in the industry. In chemical industry, crystallizers are often used to achieve liquid-solid separation and generate high purity products, for example in production of sugar. The developments of the technology in industrial crystallization is becoming more exciting and challenging in the world of chemical engineering.

1.3 Objectives

The main objective of this research is to design and develop a new crystallizer for the use in a PFC system. Specific objectives are to:

i. Design, fabricate and characterize a new crystallizer with spiral fin as the main component in the PFC system.
ii. Study the effect of several operating conditions for the newly designed PFC system using glucose as a model solution.
iii. Study ice crystal mass formation using heat transfer model.
iv. Conduct a case study in fruit juice concentration by using the new PFC system to find the optimum operating conditions.

1.4 Scope of Research

There are four major scopes of this study.

i. A new crystallizer with a spiral fin was made of stainless steel. Fin efficiency calculation was made in order to ensure the suitability of the fin to the crystallizer. The cooling jacket has been equipped on the crystallizer and insulated by polyurethane foam. The geometrical characteristics in terms of ice production, fluid mechanic and heat transfer of the PFC system was determined.
ii. The performance of PFC system for concentration process of glucose solution was studied according to the effect of circulation flowrate, circulation time, coolant temperature and shaking speed. The effect of these operating parameters was evaluated through the value of effective partition constant (K) and solute recovery (Y). These operating parameters have been investigated through the following ranges:
    - Circulation flowrate from 2600 mL/min to 3400 mL/min
    - Circulation time from 40 minutes to 80 minutes
    - Coolant temperature from -8 °C to -16 °C
    - Shaking speed from 20 ohm to 60 ohm
iii. The case study of PFC system has been conducted on apple juice where the application of PFC system on the real solution can be known. Optimization process has been applied to determine optimum conditions for the apple juice concentration and to optimize the operating conditions of the process using Response Surface Methodology (RSM).
iv. The model was developed based from the fundamental equation of heat transfer. Experimental values of ice crystal mass were obtained at different coolant temperature and were compared with the values estimated by the model.

1.5 Significance of Research

The spiral finned crystallizer for the PFC system has never been engaged in any application. This is a novel idea and this study would be beneficial to measure the opportunity of a new attractive method for any suitable application. This alternative is simpler and can reduce the operation time.

In addition, this study can provide an understanding on how to design the PFC system with high quality of product and high productivity. This study also can be a reference for any other applications in the industry.

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