UNIVERSITI PUTRA MALAYSIA

KINETICS OF FOULING DEPOSIT REMOVAL OF PINK GUAVA PUREES IN A CLEANING-IN-PLACE TEST RIG

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By

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Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfilment of the Requirements for the Degree of Master of Science

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Chairman : Norashikin Binti Ab. Aziz, PhD
Faculty : Engineering

Cleaning-in-place (CIP) is an important process in food factories, to maintain a hygienic processing environment. The development of economic CIP requires comprehensive studies of the removal kinetics of the fouling deposit. This work was carried out to investigate the removal kinetics of pink guava puree (PGP) fouling deposits, which to the knowledge of the author has not yet been reported anywhere. This work is divided into three parts which are: (1) design of the cleaning test rig and simulation validation of the design, (2) development of in-situ and ex-situ methods to prepare the PGP fouling deposit, and (3) investigation of the removal kinetics of the PGP fouling deposits by using the cleaning test rig under different cleaning parameters. The design of the lab-scale cleaning test rig was based on the standard design of a recirculating water flow channel. The entry length of 1.016m was determined from computational fluid dynamics (CFD) simulation, which was performed to simulate the cleaning environment in the rig, and to ensure the functionality of the rig was in order before the rig was fabricated. Both methods on developing the physical model of PGP fouling deposit was compared and results have shown than an ex-situ method is a practical method to apply. An ex-situ method was able to form reproducible samples of PGP fouling deposit with low production time and minimal consumptions on raw materials. In part three, only alkaline cleaning stage was considered in this study. The cleaning study was performed at different parameters: temperatures (35-70 °C), fluid velocities (0.6-1.5 m/s) and NaOH concentrations (0-2.0 wt%). Cleaning profiles have shown two stages: rapid and gradual stages. Cleaning response in both stages was investigated by employing an effective removal rate constant, k_2. The findings suggested that alkaline rinse can be divided into two stages with the following conditions: (1) conditions for rapid stage are 70 °C, 1.2 m/s, 1.5 wt% NaOH, with t_{rapid}=2 minutes; and (2) conditions for gradual stage are 35 °C, 1.5m/s, water (0wt% of NaOH) and with $t_{gradual}$=10 minutes. The results of the cleaning time suggest that the shortest cleaning time (less than 12 minutes) can be found at 1.5 m/s, 70 °C and with a NaOH concentration of 1.0, 1.5 and 2.0 wt%. Findings from this work suggest two cleaning schemes for alkaline cleaning stage, which classified as 1) economical cleaning scheme and 2) fast cleaning scheme. In economical cleaning scheme, the industries need to identify the rapid and the gradual stage for their cleaning process and this cleaning scheme will reduce the cost on chemicals and utilities. While for fast cleaning scheme, the application of excessive cleaning parameters is needed. However, the cleaning cost is expected to increase significantly.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Master Sains

KINETIK PENYINGKIRAN UNTUK MENDAPAN KOTORAN DARI PURI JAMBU BATU MERAH JAMBU DALAM KELENGKAPAN UNTUK UJIAN PEMBERSIHAN

Oleh

NURUL IZZAH BINTI KHALID

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Pembersihan setempat adalah suatu proses yang penting di kilang makanan, bagi mengekalkan persekitaran pemprosesan yang bersih. Pembangunan pembersihan setempat yang ekonomi memerlukan kajian komprehensif berkenaan kinetik penyingkiran untuk mendapan kotoran. Kerja penyelidikan ini dijalankan untuk mengkaji kinetik penyingkiran untuk mendapan kotoran dari puri jambu batu merah jambu (PJBMJ), untuk pengetahuan penulis belum pernah dilaporkan di mana-mana sahaja. Kerja penyelidikan ini terbahagi kepada tiga bahagian iaitu: (1) reka bentuk kelengkapan untuk ujian pembersihan dan pengesahsahihan reka bentuk melalui penyelakuan, (2) pembangunan kaedah penyediaan mendapan kotoran PJBMJ secara di situ dan eksitu, dan (3) kajian mengenai kinetik penyingkiran untuk mendapan kotoran PJBMJ dengan menggunakan kelengkapan ujian pembersihan pada parameter pembersihan yang berbeza. Kelengkapan ujian pembersihan yang berskala makmal direka berdasarkan reka bentuk piawai bagi saluran aliran air yang beredar semula. Panjang masukan 1.016m telah ditentukan daripada penyelakuan perkomputeran dinamik bendalir, yang telah dijalankan bagi menyelakuankan persekitaran pembersihan di dalam kelengkapan ujian pembersihan, dan untuk memastikan kelengkapan ujian pembersihan ini dapat berfungsi dengan tertib sebelum kelengkapan ujian pembersihan itu dibikin. Kedua-dua kaedah bagi membangunkan model fizikal untuk mendapan kotoran PJBMJ telah dibandingkan dan keputusan telah menunjukkan bahawa kaedah eksitu adalah kaedah yang praktikal untuk digunakan. Kaedah eksitu dapat membentuk sampel boleh ulang semula untuk mendapan kotoran PJBMJ dengan masa pengeluaran yang rendah dan penggunaan minimum ke atas bahan mentah. Dalam bahagian ketiga, hanya peringkat pembersihan beralkali telah dipertimbangkan dalam kajian ini. Kajian pembersihan telah dijalankan pada parameter yang berbeza: suhu (35-70°C), halaju bendalir (0.6-1.5m/s) dan kepekatan NaOH (0-2.0wt%). Profil pembersihan telah menunjukkan terdapat dua peringkat pembersihan iaitu: peringkat deras dan peringkat beransur. Tindak balas pembersihan bagi kedua-dua peringkat ini telah disiasat dengan menggunakan pemalar kadar penyingkiran berkesan, k_2. Hasil penemuan mencadangkan bahawa pembersihan beralkali boleh dibahagikan kepada dua peringkat khususnya daripada keadaan ini: (1) keadaan peringkat deras ialah 70 °C, 1.2 m/s, 1.5 wt% NaOH, dengan t_{deras}=2 minit; dan (2) keadaan peringkat beransur ialah 35°C, 1.5m/s, air (0wt% NaOH) dengan $t_{beransur}$=10 minit. Manakala berdasarkan keputusan masa pembersihan, dicadangkan bahawa masa pembersihan yang paling singkat (kurang daripada 12 minit) boleh didapati di 1.5m/s, 70°C dan dengan kepekatan NaOH 1.0, 1.5 dan 2.0wt%. Hasil penemuan daripada kerja-kerja ini

mencadangkan dua skim pembersihan untuk peringkat pembersihan beralkali, yang diklasifikasikan sebagai 1) skim pembersihan yang ekonomi dan 2) skim pembersihan yang cepat. Dalam skim pembersihan yang ekonomi, industri perlu mengenal pasti peringkat deras dan peringkat beransur untuk proses pembersihan dan skim pembersihan ini akan mengurangkan kos pada bahan kimia dan utiliti. Manakala bagi skim pembersihan yang cepat, penggunaan parameter pembersihan yang lebih diperlukan. Walau bagaimanapun, kos pembersihan dijangka meningkat dengan ketara.

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I certify that a Thesis Examination Committee has met on (January 2015) to conduct the final examination of Nurul Izzah Binti Khalid on her thesis entitled "Kinetics of Fouling Deposit Removal of Pink Guava Purees in a Cleaning-In-Place Test Rig" in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The Committee recommends that the student be awarded the Master of Science.

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Declaration by graduate student

I hereby confirm that:

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This is to confirm that:

- the research conducted and the writing of this thesis was under our supervision;
- supervision responsibilities as stated in the Universiti Putra Malaysia (Graduate Studies) Rules 2003 (Revision 2012-2013) are adhered to.

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TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF ABBREVIATIONS

CIP	Cleaning-in-place
PGP	Pink guava puree
PGJ	Pink guava juice
WPC	Whey protein concentrate
PHE	Plate heat exchanger
THE	Tubular heat exchanger
PI&D	Process and instrumentation diagram
FDG	Fluid dynamic gauging
JIS	Japanese Industrial Standards
NaOH	Sodium hydroxide
KOH	Potassium hydroxide
UHT	Ultra High temperature
CFD	Computational fluid dynamics
PDEs	Partial differential equations
SS	Stainless steel
HTC	Heat transfer coefficient

NOMENCLATURES

C_D	Drag coefficient	dimensionless
F_D	Drag force	N
B	Width of the flat plate	m
L	Length of the flat plate	m
ρ	Density of the fluid	kg/m^3
v	Fluid velocity	m/s
τ	Shear stress	Pa
A	Area of the flat plate	m^2
L_e	Minimum entry length	m
D_n	Nominal diameter of pipe	m
D	Diameter of the pipe	m
μ	Dynamic viscosity of the flow	Pa.s
Re	Reynolds number	dimensionless
m_c	Mass of cleaning chemical	g
c	Desired concentration of NaOH	wt %
z	Volume of cleaning chemical	L
a	Concentration of NaOH	wt %
X_f	Fouling thickness	m
X_{fa}	Measured fouling thickness	m
X_{ft}	Fouling-resistance thickness	m
A_f	Area of fouling deposit	m^2
m_f	Mass of fouling deposit	kg
ρ_f	Density of fouling deposit	kg/m^3

ρ_{H_2O}	Density of water (1000 kg/m^3).	kg/m^3
d	Diameter of the inner concentric tube-fouling rig	m
L_f	Length of the concentric tube-fouling rig	m
R_{fa}	Measured fouling resistance	m^2°C/W
R_{ft}	Fouling resistance	m^2°C/W
k_f	Thermal conductivity of fouling deposit	W/m°C
V_f	Volume of fouling deposit	m^3
ΔT_1	Temperature oil out – temperature product in	°C
ΔT_2	Temperature oil in – temperature product out	°C
ΔT	Temperature changes of product	°C
ΔT_{lm}	Log mean temperature difference	°C
Q	Heat transfer	J/s
$\tilde{V}$	Volumetric flow rate	L/min
Q	Flow rate	L/h
m	Mass flow rate of product	kg/s
c_p	Specific heat of fouling deposit	J/kg°C
U	Overall heat transfer coefficient	W/m^2°C
U_o	Initial overall heat transfer coefficient.	W/m^2°C
$T_{oil,in}$	Oil input temperature	°C
$T_{oil,out}$	Oil output temperature	°C
$T_{PGP,in}$	PGP input temperature	°C
$T_{PGP,out}$	PGP output temperature	°C
T_{in}	Input temperature	°C
T_{out}	Output temperature	°C

$T_{inter,out}$	Intermediate outlet temperature	°C
$T_{UHT,out}$	UHT section outlet temperature	°C
T_{wall}	Wall temperature	°C
t	Time	hour

CHAPTER 1

INTRODUCTION

Fouling formation is a particularly severe problem, especially in the food and milk industries where frequent cleaning is needed (Visser and Jeurnink, 1997). Fouling deposit is an unwanted by-product of most process industries, such as food, petroleum and water treatment industries. In the petrochemical industry, it is common practice to clean only once a year or less, whereas daily cleaning is applied in the food industry (Visser and Jeurnink, 1997). The formation of food fouling deposit is rapid in a heat exchanger which affects the heat transfer and develops resistance to the fluid flow. In earlier days, manual cleaning was practiced where all the process equipment was dismantled one by one to be cleaned by hand. Nowadays, most of the industry has shifted their cleaning method to cleaning-in-place (CIP) to save cost and reduce downtime. This chapter gives a brief background of CIP, and pink guava puree (PGP).

1.1 Cleaning-in-place (CIP)

The main reasons for cleaning are appearance, safety, plant efficiency and to prevent microorganism contamination (Tamime, 2008). A clean appearance gives confidence in the quality of the products and also provides a dirt-free tidy working environment for the workers. Pipe leakage due to frequent dismantling of equipment can cause slippery floors and can be very dangerous. An accident in the work place can cause expensive repercussions. Other than that, clean equipment can provide a more efficient work system and avoid energy wastage.

In the food industry, daily cleaning is practiced compared to the petroleum industry, which cleans only once a year. Food processing equipment such as pipelines and pumps which have direct contact with food products can provide a suitable place for bacterial growth which can cause contamination in food products. Product residue accumulated after processing can be one of the factors that initiate bacterial growth. Furthermore, for high temperature processes such as pasteurisation and sterilisation, fouling deposit can easily attach to the hot surface of the processing equipment. Frequent cleaning and inspection are important to prevent attachment of fouling and bio-fouling deposit and to ensure that food product can be pasteurised correctly. In the market, there are many cleaning procedures and detergents which provide different cleaning effects for all kinds of fouling deposit. Different kinds of food products generate different characteristics of fouling deposit. Thus, each food plant should have a formulated CIP process to efficiently remove the fouling deposit. The selection of suitable and optimum cleaning methods is very important to avoid any chemical wastage and to minimise the downtime.

The most common cleaning methods applied are two-stage cleaning and single stage cleaning (Christian and Fryer, 2004). In two-stage cleaning, two different types of detergent, namely alkaline and acidic detergents are used. Alkaline detergent is used in the first stage and this is followed by the acidic detergents in the second stage. Commonly, sodium hydroxide liquor is used and followed by nitric acid. At every stage, the chemical solution is cycled for about 15 minutes to 1 hour depending on experimentation and degree of experience. Between the stages, water is used to remove all traces of detergent which is also called rinsing. Lastly, before the final rinse using water, a disinfection cycle is initiated. A chemical such as sodium hypochlorite is used in this cycle. Details of the cleaning process are explained in Section 2.1.

For single stage cleaning, a formulated detergent is used. This type of cleaning only needs one stage of circulation of the cleaning chemicals. Although a formulated detergent can save cleaning time, the cost is greater compared to individual alkaline and acidic detergents. A formulated detergent is usually used for a fouling deposit that is very difficult to remove. Thus, suitable cleaning chemicals must be selected considering the type of fouling deposit and also the cost of the cleaning.

1.2 Pink Guava Puree

The scientific name for guava is *Psidiumguajava* L. Guava come from the *myrtaceae* family. Guava is also known as guayaba, guayabo, arazá-puitá, goyavier, and gobiabiera. The major producers of guava in the world are India, Brazil and Mexico. Since 1987, Malaysia has also started to become one of the major producers of guava. In Malaysia, guava is planted in Perak, Johor, Selangor and Negeri Sembilan (Lim and Khoo, 1990). Pink guava has a high demand in the world because it is highly nutritious and good for the health (Lim and Khoo, 1990). PGP is well accepted by beverage and food manufacturers in Europe, the United States, Australia, Japan, Korea, Singapore and also local manufacturers in Malaysia (Sime Darby, 2014).

Pink guava is among the favourite food ingredients that are used for producing baby food, beverages, juices, ice cream, frozen desserts, yoghurt, fruit jelly and confectionery products (Sime Darby, 2014). The average amount of ascorbic acid in pink guava is three to six times higher compared to oranges at 50-300 mg/100g fresh weight (Thaipong et al., 2006). PGP has anti-hypertensive properties which are suitable for patients with hypertension (Ayub et al., 2010). PGP is also rich in antioxidants that help to reduce the incidence of degenerative diseases such as arthritis, arteriosclerosis, cancer, heart disease, inflammation and brain dysfunction (Lim et al., 2006).

Since 2006, almost nine million kilograms of pink guava are harvested annually in Malaysia and the Sime Darby plantations produce 15 % of the world's pink guava puree. The PGP process involves several unit operations such as pasteurisation, UHT, and cooling. The critical area for rapid fouling deposit formation is in the high temperature operations such as pasteurisation and the UHT process. The fouling deposit also forms in the low temperature area (i.e. the cooling area). However, this research work is only focused on the high temperature condition.

1.3 Objective of the Study

Pink guava puree fouling deposit was used as the physical fouling deposit model in this work. To the knowledge of the author, this type of deposit has not been studied previously by any other researcher. PGP fouling deposit is classified as a carbohydrate-based fouling deposit which is considered easier to clean when compared to protein-based fouling deposit. This work focuses on alkaline cleaning stage for which sodium hydroxide was used in the cleanability experiments. For cleaning kinetics studies, a lab-scale cleaning test rig was designed and was utilised for the cleanability experiments.

The objectives of this study are:

- To perform simulation validation of the lab-scale cleaning test rig before it can be utilised for the cleanability experiments.
- To develop a suitable physical model of the PGP fouling deposit.
- To investigate the cleaning kinetics during the alkaline cleaning stage by using the lab-scale cleaning test rig.

1.4 Significance of the Study

In Malaysia, there is no standard CIP process that was formulated for all food industries. Most of the food industries applied the standard CIP process designed for dairy-based fouling deposit. Every type of food-based fouling deposit requires a different formulated CIP process to achieve effective cleaning. Short CIP process is favorable to food industries as food industries incur downtime when cleaning is performed. Thorough investigation of CIP performance on different fouling deposits is a must to obtain effective and economical cleaning.

PGP processing plants in Malaysia are referring to cleaning program for dairy industries for their cleaning program. This can be considered as excessive cleaning parameters for carbohydrate-based fouling deposit like PGP fouling deposit. Research into cleaning was performed to find the best CIP process for the specific problem of PGP fouling deposit removal under high temperature conditions. Most of the previous researchers focused on dairy production (Visser & Jeurnink, 1997; Robbins et al., 1999; Grijspeerdt et al., 2004; Nema & Datta, 2005; Sahoo et al., 2005; Simmons et al., 2007; Rosmaninho et al., 2007; Mahdi et al., 2009) instead of tropical resources such as pink guava that has a very good market demand, as mentioned above. Dairy is one of the staple foods in western countries and fouling research first began in these western countries. However, it is still important to explore the fouling characteristics of other sources of local food such as juices and purees which are not as critical as the protein-based deposits. PGP was chosen to be the physical model for fouling deposit in this study due to it being considered an ill-defined fouling fluid model compared to milk. There is currently no guidance for industry to control and clean PGP fouling deposit as no related reference has been published as far as is known. So it is important to conduct this study as it can provide knowledge for the industry in order to optimally clean PGP fouling deposit.

There are several types of equipment available for cleaning research. However, most of this equipment does not consider the CIP environment and does not allow on-line monitoring. Chapter 2 provides information on the existing equipment for cleaning research. By considering these two main challenges (the CIP environment and on-line monitoring), a lab-scale cleaning test rig was designed to study the cleaning process. The cleaning test rig enabled an investigation into the cleaning kinetics during the alkaline cleaning stage, and was designed with a test section which allowed video recording of the removal of the PGP fouling deposits. Furthermore, this rig was designed to closely mimic the typical industry flow environment in food piping, whereby different cleaning parameters could be manipulated to study the CIP performance.

Shear stress has generally been considered as one of cleaning parameters that contribute to cleaning efficiency. The findings from this work of research can provide basic knowledge of development stage of the design for the cleaning test rig to investigate experimental shear stress. The findings from this study can also provide fundamental data for optimising CIP in future work. Moreover, the findings can benefit the PGP industry in Malaysia whereby a suitable CIP process can be implanted to benefit the industry.

1.5 Structure of the Thesis

The description of this work is arranged into six chapters in this thesis. The following chapters will provide a specific explanation concerning this research.

Chapter 1 gives an introduction to the subject of Cleaning-in-place (CIP). The chapter continues with a brief introduction to Pink Guava Puree (PGP) and states the Objectives and the Significance of the study.

Chapter 2 describes the previous studies and their findings in related areas of fouling and cleaning studies, which involves CIP process, cleaning parameters, cleaning mechanisms, methods of development of physical fouling deposit models, cleaning test rig applications, shear stress, modelling of cleaning kinetics, and modelling and simulation by a simulation software, COMSOL Multiphysics.

Chapter 3 describes the method and the equipment used for this work. At the beginning of the chapter, the design requirements and design work of a lab-scale cleaning test rig is explained in detail. The procedure for developing Computational Fluid Dynamic (CFD) models for simulating the flow inside the lab-scale cleaning test rig by using COMSOL Multiphysics is explained. Simulation validation is performed to determine the location for the test section and to prove the fully developed flow inside the rig. Then, the procedure for developing the PGP fouling deposit is described by using two different methods namely, ex-situ (by using an oven) and in-situ (by using a concentric tube-fouling rig). A suitable physical model for the fouling deposit is chosen to be used in the cleanability experiments. Procedures for the cleanability experiments of the removal of PGP fouling deposit using different sets of parameter combinations (temperature, fluid velocity and chemical concentration) are also included in this chapter. The performance of a Lab-scale cleaning test rig for PGP fouling deposit removal was tested.

Chapter 4 presents the specifications of the design and results analysis from the simulation validation of the lab-scale cleaning test rig by using COMSOL Multiphysics. The results from calculation and simulation were used to determine the fully developed flow zone after pipe bending. This is to determine the location of the test section. The flow behaviour of the water flow inside the cleaning test rig and the test section is discussed. The flow is simulated for different cleaning parameters (fluid velocity, fluid temperature and chemical concentration).

Chapter 5 discusses the physical model fouling deposit development method that was used in this work. Two methods were used to prepare the PGP fouling deposit for the experiments, which were the ex-situ method (using an oven) and the in-situ method (using a concentric tube-fouling rig). The concentric tube-fouling rig was validated before it was used for preparing the fouling deposit. The ex-situ method was chosen as to develop the physical model of the PGP fouling deposit for this study. The results from the cleanability experiments which study the effects of cleaning parameters (temperature, fluid velocity and chemical concentration) on the fouling deposits are discussed here.

Chapter 6 concludes the study and gives some suggestions for future studies in cleaning and fouling of PGP and other food-based fouling deposits. In this chapter, some suggestions to modify the lab-scale cleaning test rig are also provided.

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