UNIVERSITI PUTRA MALAYSIA

PROCESSING AND CONTROL STRATEGIES FOR THE IMPROVEMENT OF SPRAY-DRIED COCONUT MILK QUALITY

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By

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Spray drying of liquid containing hydrophobic material such as coconut milk fats depends entirely on its feed formulation and process operating conditions. Good encapsulation of fats can only be achieved by spray drying of a stable emulsion. However, an excessive amount of additives is usually used to stabilize the emulsion without considering the natural quality of coconut milk. The main goal of this research is to propose process and control strategies for the improvement of the quality of spray-dried coconut milk by minimizing the use of additives. In order to achieve this goal, the effect of feed formulation and inlet drying temperature on the spray dried powder is evaluated. It also focuses on maintaining product quality by the application of a nonlinear model-based inferential control strategy.

Different sonication amplitude setting (60, 80 and 100%) and sodium caseinate (SC) concentration (0 to 2% w/w) were used in the emulsification process to evaluate the effects on the degree of stability of the emulsion. The effect of sodium caseinate concentrations and drying temperatures (140, 160 and 180ºC) to spray dried coconut milk was assessed based on the physical and functional properties of the powder. The one-dimensional model with the integration of reaction engineering approach (REA) model was used to predict the dynamic behaviour that relates operating temperatures to the moisture content of powder produced. The empirical model, i.e., nonlinear autoregressive with exogenous input (NARX) model and neural network (NN) model, were used in the inferential control system as system identification and soft sensor estimator, respectively.

High stability and good properties of coconut milk emulsion were achieved with the addition of SC concentration ≥ 1% w/w and ultrasonic amplitude setting of ≥80%. The emulsification process significantly reduced the effective average particle size

to lower than half (<5 µm) of its original size. Increasing the SC concentration (≥ 1% w/w) reduced both the creaming index and free fat content to 0% and <10%, respectively without significant change in apparent viscosity. This indicates that the emulsion has achieved high stability. High ultrasonic amplitude setting (≥80%) leads to the formation of stabilized emulsion with submicron range size (<1 µm) of droplets.

For spray dried coconut milk, good physical and functional properties of powder were achieved with the addition of SC concentration ≥ 1% w/w and inlet temperature of ≤160°C. Higher the SC concentration (≥ %1 w/w) produced smaller-sized powder particles (<30 µm) which leads to low particle density and bulk density of the powder. High stability emulsion with 1% w/w of SC was unstable during the atomisation process due to re-coalescence of fat as the size of droplet increased to >2µm after the spray drying process. Adding SC to emulsion reduced the moisture content of powder to less than 5% without significant change due to SC concentration. The lowest moisture content (<4%) was obtained at the inlet temperature of 180°C. The highest free fat content, insolubility and droplet size were obtained at the inlet temperature of 180°C regardless of SC concentration. The presence of fleck was also noticed in the powder.

The reaction engineering approach (REA) model is used to represent the drying kinetics of a single droplet of coconut milk. Integration of the REA model into the one-dimensional model enables accurate prediction of dynamic moisture content of spray dried coconut milk. The developed REA model accurately predicts the drying behavior of the coconut milk droplet with R^2 value of 0.9786 obtained during model validation. Integration of REA model into one-dimensional model leads to a high accuracy model to predict the variables of the spray drying process with the mean absolute percentage error (MAPE) was found to be 17.1% for moisture content and 6.2% for outlet temperature. Good control performance was achieved by the nonlinear inferential control system in controlling the moisture content of coconut milk powder. Minimal offset (<0.0003 kg/kg) of the responses at various set points were obtained which indicates the accuracy of the neural network estimator. Less overshoots were obtained by TL PI inferential control during setpoint tracking. On the other hand, good control performance was obtained by ZN PI inferential control during disturbance rejection.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

STRATEGI PEMPROSESAN DAN KAWALAN UNTUK PENAMBAHBAIKAN KUALITI SANTAN KERING SEMBURAN

Oleh

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Pengeringan semburan bagi cecair yang mengandungi bahan hidrofobik seperti lemak santan bergantung sepenuhnya kepada formulasi suapan dan keadaan operasi prosesnya. Pengkapsulan lemak yang baik hanya boleh dicapai dengan pengeringan semburan terhadap emulsi yang stabil. Walaubagaimanapun, jumlah bahan tambahan yang berlebihan selalunya digunakan untuk menstabilkan emulsi tanpa mempertimbangkan kualiti semulajadi santan. Tujuan utama penyelidikan ini adalah untuk mencadangkan strategi proses and strategi kawalan untuk penambahbaikan kualiti santan kering semburan dengan penggunaan pengemulsi yang minimum. Untuk mencapai tujuan ini, kesan formulasi suapan dan suhu pengeringan masukan terhadap serbuk kering semburan adalah dinilai. Penyelidikan ini juga memberi tumpuan untuk mengekalkan kualiti produk dengan menggunakan strategi kawalan taabir berasaskan model tidak linear.

Penetapan amplitud ultrasonik yang berbeza (60, 80 dan 100%) dan kepekatan natrium kaseinat (SC) yang berbeza (0 hingga 2% b/b) digunakan dalam proses pengemulsian untuk menilai kesannya terhadap tahap kestabilan emulsi. Kesan kepekatan natruim kaseinat dan suhu pengeringan (140, 160 dan 180ºC) kepada santan kering semburan ditaksir berdasarkan sifat fizikal and sifat fungsi serbuk. Model satu dimensi dengan integrasi model pendekatan kejuruteraan tindak balas (REA) digunakan untuk meramalkan tingkah laku dinamik yang menghubungkan suhu operasi dengan kandungan lembapan serbuk yang dihasilkan. Model empirik iaitu model auto mundur tak lelurus dengan masukan luar kawalan (NARX) dan model rangkaian saraf (NN) masing-masingnya digunakan dalam sistem kawalan taabir sebagai pengenalpastian sistem dan penganggar penderia lembut.

Kestabilan yang tinggi dan sifat-sifat yang baik emulsi santan telah dicapai dengan penambahan kepekatan SC ≥ 1% b/b dan penetapan amplitud ultrasonik ≥80%. Proses emulsifikasi mengurangkan saiz zarah purata berkesan dengan ketara hingga lebih rendah daripada separuh saiz (<5 µm) dari saiz asalnya. Meningkatkan kepekatan SC mengurangkan kedua-dua indeks pengkriman dan kandungan lemak bebas masing-masingnya kepada 0% dan <10%, tanpa perubahan ketara dalam kelikatan ketara. Ini menunjukkan bahawa emulsi telah mencapai kestabilan yang tinggi. Penetapan amplitud ultrasonik yang tinggi (≥80%) membawa kepada pembentukan emulsi yang stabil dengan titisan dengan saiz julat submikron (<1 µm).

Untuk santan kering semburan, sifat fizikal dan sifat berfungsi yang baik telah dicapai dengan penambahan kepekatan SC ≥ 1% b/b dan suhu salur masuk ≤160°C. Kepekatan SC yang lebih tinggi (≤1 % b/b) menghasilkan zarah serbuk yang bersaiz lebih kecil (<30 µm) yang menyebabkan kepadatan zarah dan kepadatan pukal serbuk yang rendah. Emulsi kestabilan tinggi dengan 1% b/b SC adalah tidak stabil semasa proses atomisasi kerana tautan semula lemak berdasarkan saiz titisan yang meningkat sehingga >2 µm setelah proses pengeringan semburan. Menambah SC ke emulsi mengurangkan kandungan lembapan serbuk menjadi kurang dari 5% tanpa perubahan ketara disebabkan oleh perbezaan kepekatan SC. Kandungan lembapan terendah (<4%) diperoleh pada suhu masukan 180°C. Kandungan lemak bebas tertinggi, ketakbolehlarutan tertinggi dan saiz titisan tertinggi diperoleh pada suhu salur masuk 180°C tanpa mengira kepekatan SC. Kehadiran flek juga diperhatikan dalam serbuk.

Model pendekatan kejuruteraan tindak balas (REA) digunakan untuk mewakili kinetik pengeringan satu titisan santan. Integrasi model REA ke dalam model satu dimensi memungkinkan ramalan tepat kandungan lembapan dinamik santan kering semburan. Model REA yang dibangunkan dengan tepat meramalkan tingkah laku pengeringan titisan santan dengan nilai R^2 0.9786 yang diperoleh semasa pengesahsahihan model. Integrasi model REA ke dalam model satu dimensi membawa kepada model ketepatan tinggi untuk meramalkan pembolehubah-pembolehubah dalam proses pengeringan semburan dengan purata peratusan ralat mutlak (MAPE) sebanyak 17.1% untuk kandungan lembapan dan 6.2% untuk suhu salur keluar. Dalam sistem kawalan taabir, ofset tindak balas yang minimum (<0.0003 kg/kg) pada pelbagai titik set diperoleh yang menunjukkan ketepatan penganggar rangkaian saraf. Sedikit lajakan diperolehi oleh kawalan taabir TL PI semasa penjejakan titik set. Sebaliknya, prestasi kawalan yang baik diperolehi oleh kawalan taabir ZN PI semasa penolakan gangguan.

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LIST OF SYMBOLS

α	constant	-
A_P	surface area of the particle	m^2
C_p	specific heat capacity of particle	J/(kg.K)
$C_{p,b}$	specific heat of air	J/(kg.K)
$C_{p,solid}$	specific heat of dried particle	J/(kg.K)
$C_{p,v}$	specific heat of vapour	J/(kg.K)
$C_{p,water}$	specific heat of water	J/(kg.K)
D	diameter of the chamber	m
$D_{4,3}$	volume mean diameter	µm
D_{50}	volume median diameter	µm
d_s	sphere diameter	m
D_v	vapor-air diffusivity	m^2/s
e	error between the process and set point value	-
E_b	enthalpy of the hot air	J/kg
f	nonlinear estimator	-
g	acceleration due to gravity	m/s^2
F_p	mass flowrate of particle	kg/s
G	mass flowrate of drying air	kg/s
$H_{emulsion}$	total height of the emulsion	cm
h_m	mass transfer coefficient	m/s
h_h	heat transfer coefficient	$J/(s.m^2 \cdot K)$
$H_{transparent}$	height of the transparent serum layer	cm
k_b	thermal conductivity of air	W/(m.K)
Kc	controller gain	-

Kcu	Ultimate gain	-
L	length of the chamber	m
m_s	mass of dry matter	kg
m_p	mass of particle	kg
m_w	mass of water	kg
M_{water}	molecular weight of water	kg/kmol
n	Number of data	-
Nu	Nusselt number	-
P_{sat}	saturated vapour pressure	Pa
Pr	Prandtl number	-
Pu	Ultimate period	Cycle/s
Q_{loss}	heat loss to the surrounding	W
r	droplet's radius	m
Re	Reynolds number	-
R_G	specific gas constant	J/(kg.K)
RH	relative humidity	%
Sh	Sherwood number	-
Sc	Schmidt number	-
T_{amb}	ambient temperature	K
T_b	hot air temperature	K
T_b'	hot air temperature	°C
T_{in}	inlet temperature	°C
T_p	particle temperature	K
T_p'	particle temperature	°C
T_{out}	outlet temperature	°C
T_s	droplet interface temperature	K

u	input	-
U_p	heat loss coefficient	J/(s.m^2.K)
v	velocity of droplet	m/s
v_a	velocity of air	m/s
v_p	velocity of particle	m/s
W_f	weight of flask with extractable fat	g
W_0	weight of empty flask	g
W_s	weight of the sample	g
X	moisture content of the product	kg/kg, db
X_b	equilibrium moisture content	kg/kg, db
X_i	input values of the neural network estimator	-
y	output	-
$\bar{X}$	mean values of output data.	-
Y	absolute humidity	Kg/kg, db
$\bar{Y}$	mean values of output data.	-
$\bar{Y}_{ave}^{exp}$	average of experimental data	-
Y_i	output values of the neural network estimator	-
Y_i^{exp}	experimental outputs	-
Y_i^{pred}	model output	-

Greek Letters

ρ_c	density of continuous phase	kg/m^3
ρ_o	density of dispersed phase	kg/m^3
$\rho_{v,s}$	water vapor concentration at droplet interface	kg/m^3
$\rho_{v,b}$	vapor concentration of drying air	kg/m^3
$\rho_{v,sat}$	saturated vapor concentration	kg/m^3

η_c	viscosity of continuous phase	m^2/s
ϑ_G	kinematic viscosity of air	m^2/s
θ	number of particles per second	particles/s
λ	latent heat of water evaporation	J/kg
τ_D	derivative time	s
τ_i	integral time	s
μ_b	viscosity of bulk gas	Pa.s
ΔE_v	apparent activation energy	J/mol
$\Delta E_{v,b}$	equilibrium activation energy	J/mol

LIST OF ABBREVIATIONS

ANOVA	One-Way Analysis of Variance
AOAC	Association of Official Analytical Chemists
ASEAN	Association of Southeast Asian Nations
CDRC	Characteristic Drying Rate Curve
CFD	Computational Fluid Dynamic
DSC	Differential Scanning Calorimetric
EMC	Equilibrium Moisture Content
IAE	Integral Absolute Error
ITAE	Integral Time Absolute Error
LQG	Linear–Quadratic–Gaussian
MC	Moisture Content
MPC	Model Predictive Control
MSE	Mean Squared Error
NARX	Nonlinear Autoregressive Model with Exogenous Input
NN	Neural Network
NN-PID	Neural Network Proportional Integral Derivative
ODE	Ordinary Differential Equation
PI	Proportional-Integral
PID	Proportional-Integral-Derivative
PLS	Partial Least Squares
R	Correlation Coefficient
R^2	Coefficient of Determination
REA	Reaction Engineering Approach
RH	Relative Humidity

R-ZN	Relaxed-Ziegler Nichols
RSM	Response Surface Methodology
SC	Sodium Caseinate
SDS	Sodium Dodecyl Sulphate
SEM	Scanning Electron Microscope
TL	Tyreus-Luyben
ZN	Ziegler-Nichols

CHAPTER 1

INTRODUCTION

1.1 Research Background

Coconut milk has received great attention, especially as functional foods, pharmaceuticals, nutraceuticals, and cosmetics. This is due to recent research that has suggested coconut milk is a highly nutritious food. It is rich in fibre, vitamins, minerals and significant amounts of medium chain saturated fatty acids particularly, lauric acid. Lauric acid is predominant in coconut milk which covers almost 50% of the total saturated fat. It is a powerful antimicrobial agent (Huang et al., 2014; Nakatsuji et al., 2009) and has the greatest antibacterial activity in medium-chain triglycerides (fatty acids) (Batovska et al., 2009).

Coconut is the fourth important industrial crop in Malaysia in terms of total planted area (Abu Dardak & Quoquab, 2016). Coconut milk is oil or fat emulsion in water obtained from manual or mechanical extraction of grated coconut meat. However, this emulsion is highly unstable and prone to phase separation to two distinct layers i.e. cream layer and serum layer. Due to its health benefits, the global demand for coconut milk in food industries is increasing tremendously. Dried coconut milk is the best solution for easier transportation and storage with higher microbiological stability compared to in the liquid phase. The spray drying process has been identified as an effective drying process which preserves the nutritional value of the food powder (Naik et al., 2014).

In the production of food powder, the end-product must meet particularly high-quality properties. For the spray drying process, the dried product properties are highly dependent on the dryer operation and properties of the feed (Mujumdar, 2014; Patel, Patel, & Suthar, 2009). For emulsion-based powder produced from the spray drying process, fat and protein are the two major components that determine the powder stability. This is because fat is prone to oxidation without proper encapsulation, meanwhile protein is easily denatured due to temperature treatment. Emulsifier needs to be introduced to promote the stability of both, emulsion and powder (Jafari et al., 2007b). However, for a fat-filled product such as coconut milk powder which has fat content higher than 50%, the emulsifier is needed to allow the conversion of the liquid to powder phase (Seow & Gwee, 1997). Otherwise, it will remain in a liquid state when spray dried due to the high-fat content (Hassan, 1985).

The emulsification process i.e. incorporation of homogenization process and addition of emulsifier to emulsion is widely used to prepare high stability emulsion (Simuang et al., 2004). The homogenization process will reduce the size of the fat globules while the emulsifier lowers the interfacial tension between the oil and water phases, which will also prevent the fat globules from coalescing. In terms of operating conditions during the spray drying process, the inlet temperature has a significant impact on the properties of the powdered emulsion as it is directly related to the drying rate of the process (Gharsallaoui & Chambin, 2007).

The assessment of the product quality is crucial to assure a high-quality product is delivered to the consumer. The quality needs to be maintained by providing a tight control strategy to the process. In food production, the product quality such as moisture content can only be measured after the process has completed and cannot be measured in real-time due to the low reliability of the measuring device as well as consideration of the hygienic issue of the food product. Therefore, inferred variable isw usually used as the control variable in the control system. In the spray drying process, outlet drying temperature has been widely selected as a controlled variable as it is directly correlating to the final moisture content of products (Maas et al., 2011). Model-based control is a promising technology in providing a better control system even though it involves the unmeasured variable. The performance of model-based control is highly reliant on the presence of an accurate process model which is used to provide a nonlinear correlation between the process-dependent variables to the independent variables. The fundamental process model provides fundamental knowledge which can predict the overall behaviour of the spray drying process which allows prediction of unmeasured process variables. The fundamental model can be used to generate dynamic time-series data for formulating an empirical model of the process to make it feasible to be used in designing inferential control. This will benefit the industry to produce high-quality coconut milk powder at optimum conditions with high nutrient retention.

1.2 Problem Statement

Coconut milk is one of the most popular plant-based milk alternatives to dairy milk. The increasing demand for coconut milk worldwide is contributed by several main factors i.e. increasing in veganism, increasing demand for lactose-free milk and, the discovery of health benefits of coconut milk fats and vitamins (Market Research Future, 2019). Currently, the coconut milk is sold in liquid and powder form with the liquid formulation dominating the market. However, in the future, it is predicted that the powder form will conquer the market due to the increased use of coconut milk powder in food production worldwide (Market Research Future, 2019).

Attempts to prolong the shelf life of coconut milk to powder form is usually conducted using a spray drying process, but this process is aggravated by high-fat content of coconut milk emulsion. The existing natural coconut milk protein is insufficient to emulsify the high amount of fat in the emulsion in its natural state and to withstand high shear during the atomization process. Therefore, prior to the spray drying process, a high amount of additive is added to stabilize the coconut milk emulsion in order to reduce the effect of fat content that causes fouling in the spray dryer. Previous studies have also found out that the stability and feed properties of emulsion affecting the quality of spray dried powder (Carneiro et al., 2013; Jafari et al., 2007a). However, this leads to a reduction in the nutritional quality of the dried powder. Commercial coconut milk powder produced in Asia has been found to consist of high concentration (>50%) of maltodextrin to stabilize the fat (Viet Delta, 2015).

Spray drying of liquid at high temperature will lead to a low moisture content of powder, but high temperature treatment to coconut milk emulsion not only removes the moisture from the droplet but also leads to protein denaturation due to the heat sensitive behaviours of proteins (Tangsuphoom & Coupland, 2005). Thus, it negatively affects the properties of the powder.

In order to maintain the powder quality, an efficient control strategy is needed. Most of the product quality can only be measured at the end of the process, therefore controlling the inferred variables becomes common practice in the food industry. However, variation of product quality due to the presence of disturbances and parameter variations might not be similar to the effect to inferred variable and this might harm the controller performance (Zaror & Pérez-Correa, 1991). Due to that, inferred based control system is insufficient to maintain the properties of the powder, however, real-time measurement of the properties is unfeasible. The fundamental model of the spray drying process can be used to obtain the fundamental knowledge about the quality and properties of the powder, but the properties of spray dried powder can only be predicted if the drying kinetics of the selected material is available.

1.3 Research Objective

The goal of this research is to improve the quality of spray dried coconut milk with a minimum usage of emulsifiers. The four main objectives that have been determined for this research are summarized as follows:

- To evaluate the effect of sodium caseinate concentration and sonication amplitude on the stability and physical properties of coconut milk emulsion prior to the spray drying process.
- To study the effect of sodium caseinate concentration and drying temperature on the physical and functional properties of the spray dried coconut milk powder.
- To predict the moisture content of coconut milk powder via one-dimensional model with the integration of drying kinetic model.
- To design the nonlinear model-based inferential control to control moisture content of coconut milk powder.

1.4 Research Scope

The research scope of this work focuses on improving the quality of spray dried coconut milk produced by a co-current spray dryer. This involves the formulation of coconut milk emulsion via ultrasonic homogenization with the addition of a minimum amount of sodium caseinate (≤ 2% w/w). The properties of the emulsion are characterized based on its stability and physical properties. The emulsion with different levels of stability is fed to the co-current spray dryer. The effect of different feed formulations with the inlet temperatures on the physical and functional quality of the coconut milk powder is evaluated. The functional properties were selected based on their functionality in the reconstitution and storage of powder.

The modelling part consists of formulation of a fundamental model that integrates drying kinetic of coconut milk i.e., reaction engineering approach (REA) model with a one-dimensional model of co-current spray dryer, which is mainly used to predict the dynamic of moisture content of the powder. The fundamental model is used to generate nonlinear time-series data for empirical model development i.e., nonlinear autoregressive with exogenous input (NARX) model and neural network (NN) estimator. The empirical models are embedded in an inferential control system which functioned to control the moisture content of the spray dried coconut milk. The closed loop tuning rules are used to determine the controller parameters. The performance of the controller is evaluated based on the set point tracking and disturbance rejection.

1.5 Research Hypothesis

It is expected that several aspects need to be considered in order to produce a good quality coconut milk powder in terms of its properties while maintaining its nutritional value. Firstly, to address the issue of the effect of feed formulation on the quality of coconut milk powder, the stability and physical properties of the coconut milk emulsion are evaluated after the emulsification process. It is expected that both, homogenizer power and the amount of emulsifier may affect the properties of the emulsion. Secondly, to understand the effect of the spray drying process on the quality of the powder, the functional and physical properties of the powder are evaluated after the spray drying process. It is hypothesized that inlet drying temperature may affect the powder properties as this is the main variable to allow evaporation to occur and, the stable emulsion may protect the fat globules from disruption during the spray drying process.

To address the issue of the measurement of powder moisture content to be used as a control variable in the control system, the one-dimensional model with the integration of drying kinetic model is developed to produce a highly accurate dynamic time-series process model. Finally, the application of nonlinear model of the process in an inferential control strategy is expected to provide good control of powder moisture content.

1.6 Thesis Outline

This thesis is organized into five main chapters. The introduction and objectives are stated in Chapter 1, whilst Chapter 2 deals with a literature review on emulsion-based powder produced from the spray drying process and the control strategy of the spray drying process. In Chapter 3, the general material and methods involved in the formulation of emulsion to be fed to the spray dryer and the analysis of the emulsion and powder properties are explained in detail. Likewise, the procedures involved in designing the inferential control based on the fundamental and empirical approaches are discussed in detail in this chapter. Chapter 4 showed experimental results of the physical stability of coconut milk as well as the physical and functional properties of spray-dried coconut milk. This then proceeds with simulation results of drying kinetic model, one-dimensional model, empirical models, and inferential control system which elaborated in Chapter 5. Conclusions and recommendations for further work are presented in Chapter 6.

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LIST OF PUBLICATIONS

Abdullah, Z., Taip, F. S., Kamal, S. M. M., & Rahman, R. Z. A., 2020. The effect of drying temperature and sodium caseinate concentration on the functional and physical properties of spray-dried coconut milk. Journal of Food Science and Technology, 1-9.

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