



**UNIVERSITI PUTRA MALAYSIA**

***OPTIMISATION AND RHEOLOGICAL MODELLING OF THERMOSONICALLY  
EXTRACTED TROPICAL FRUIT JUICE CONCENTRATES***

**NORAZLIN BINTI ABDULLAH**

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THERMOSONICALLY EXTRACTED TROPICAL FRUIT JUICE  
CONCENTRATES**

**By**

**NORAZLIN BINTI ABDULLAH**

**Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia,  
in Fulfilment of the Requirements for the Degree of Doctor of Philosophy**

**April 2015**

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Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment  
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**April 2015**

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This study aims to investigate the extraction of tropical fruit juice with assisted thermosonication treatment through optimisation and rheological modelling approach. Juice from the pink-fleshed guava, pink-fleshed pummelo and soursop was extracted using direct and indirect thermosonication methods by varying the intensity, time and temperature and compared to those extracted using water bath incubation. The main effects, and the 3D or 4D surface plots for each response were developed based on the factors that influenced the responses, where 3D surface plot contains of three axes for two variables and a response, while 4D surface plot explains three variables simultaneously with coloured surface to represent response values. The results indicated that the best extraction method for guava and pummelo juices were using the indirect thermosonication method with parameters of 1 kW, 55°C and 30 minutes and 2.5 kW, 54°C and 23 minutes, respectively. The direct thermosonication method at 10% amplitude and 55°C for 2 to 10 minutes was more suitable for the soursop juice.

The steady-state flow test of pink-fleshed guava, pink-fleshed pummelo and soursop was studied for combination of different temperature and concentration. All the rheological data were then superimposed into a master curve using shear rate-temperature-concentration superposition technique to predict rheological behaviour at wider range of shear rate. The final equations show shear-thinning behaviour of pink-fleshed guava, pink-fleshed pummelo and soursop with flow behaviour index of 0.2217, 0.7507 and 0.6347, respectively.

The influence of concentration on rheological behaviour of pink-fleshed guava, pink-fleshed pummelo and soursop juice concentrates was evaluated using existing models and their individual lines were shifted into a master curve using concentration-temperature principle to predict rheological parameters at wider

concentration values. A new mathematical model could be applied to describe the effect of temperature on consistency coefficient and flow behaviour index of pink-fleshed guava, pink-fleshed pummelo and soursop. An expression for new mathematical model, which combined the Arrhenius and logistic sigmoidal growth models is proposed to alleviate problems of negative region of modelling the effect of temperature on consistency coefficient and flow behaviour index using the Arrhenius model alone, and due to the improvement of curve fitting ( $R^2 \geq 0.99$ ).

In conclusion, tropical fruit juice extraction can be done efficiently with thermosonication treatment, where the combination of mild heat and cavitation effects of ultrasound functions to reduce juice yield, ascorbic acid content and total soluble solids content loss.



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

**PENGOPTIMUMAN DAN PERMODELAN REOLOGI BAGI PATI JUS  
BUAH-BUAHAN TROPIKA YANG DIEKSTRAK SECARA TERMOSONIK**

Oleh

**NORAZLIN BINTI ABDULLAH**

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Kajian ini bertujuan untuk menyiasat pengekstrakan jus buah-buahan tropika dengan dibantu oleh rawatan termosonik melalui pendekatan pengoptimuman dan permodelan reologi. Jus daripada jambu berisi merah jambu, limau bali berisi merah jambu dan durian Belanda diekstrak menggunakan kaedah termosonik secara langsung dan tidak langsung dengan mempelbagaikan keamatan, masa dan suhu dan dibandingkan dengan yang diekstrak menggunakan pengerasan rendaman air. Plot bagi kesan-kesan utama, 3D atau 4D bagi setiap gerak balas dibangunkan berdasarkan factor-faktor yang mempengaruhi gerak balas, di mana plot permukaan 3D mengandungi tiga paksi untuk dua pembolehubah dan satu response, manakala plot permukaan 4D menerangkan tiga pembolehubah secara serentak dengan permukaan berwarna untuk mewakili nilai respon. Keputusan menunjukkan bahawa kaedah pengekstrakan terbaik untuk jus-jus jambu dan limau bali menggunakan kaedah termosonik secara tidak langsung dengan parameter masing-masing ialah 1 kW, 55°C dan 30 minit, dan 2.5 kW, 54°C dan 23 minit. Kaedah termosonik secara langsung pada amplitud 10% dan 55°C selama 2 hingga 10 minit lebih sesuai untuk jus durian Belanda.

Ujian aliran keadaan mantap bagi jambu berisi merah jambu, limau bali berisi merah jambu dan limau bali dikaji untuk gabungan suhu dan kepekatan yang berbeza. Model hukumkuasa disesuaikan kepada data dan semua data reologi kemudian ditindih menjadi satu lengkung induk menggunakan teknik pertindihan kadar ricihan-suhu-kepekatan sama ada dengan anjakan mendatar tunggal atau dua dimensi (mendatar-menegak) ke satu suhu rujukan dan kepekatan untuk langkah pertama dan kedua bagi penganjakan, masing-masing, untuk meramal sifat reologi pada julat kadar ricihan yang lebih luas. Persamaan-persamaan akhir menunjukkan sifat penipisan ricih bagi jambu berisi merah jambu, limau bali berisi merah jambu dan limau bali dengan indeks sifat aliran masing-masing ialah 0.2217, 0.7507 dan 0.6347.

Pengaruh kepekatan ke atas sifat reologi bagi pati jus jambu berisi merah jambu, limau bali berisi merah jambu dan durian Belanda dinilai menggunakan model-model sedia ada dan garis-garis individu dianjak menjadi satu lengkung induk menggunakan prinsip kepekatan-suhu untuk meramal parameter reologi pada nilai-nilai kepekatan yang lebih luas. Satu model matematik yang baru boleh digunakan untuk menjelaskan kesan suhu ke atas pekali kekonsistenan dan indeks sifat aliran bagi jambu berisi merah jambu, limau bali berisi merah jambu dan durian Belanda. Satu ungkapan untuk model matematik yang baru, yang menggabungkan model-model Arrhenius dan pertumbuhan logistik sigmoidal dicadangkan untuk mengurangkan masalah rantau negatif bagi permodelan kesan suhu ke atas pekali kekonsistenan dan indeks sifat aliran menggunakan model Arrhenius sahaja, dan disebabkan peningkatan penyuaian lengkung ( $R^2 \geq 0.99$ ).

Kesimpulannya, pengekstrakan buah-buahan tropika boleh dilakukan secara berkesan dengan rawatan termosonik, di mana gabungan haba antara haba yang sederhana dan kesan kavitasi bagi fungsi ultrabunyi untuk mengurangkan kehilangan hasil jus, kandungan asid askorbik dan kandungan jumlah pepejal larut.

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## LIST OF ABBREVIATIONS

3D	Three-dimensional
4D	Four-dimensional
$\sigma$	Shear stress
$\mu$	Newtonian viscosity
$\dot{\gamma}$	Shear rate
$K_B$	Bingham consistency coefficient
$\sigma_o$	Yield stress
AA	Ascorbic acid
TSS	Total soluble solids
$K$	Consistency coefficient
$n$	Flow behaviour index
$R^2$	Regression coefficient
RMSE	Root mean square error
$SSE$	Sum of square error
$SST$	Total sum of squares
$\bar{y}_{\text{experimental}}$	Mean of experimental value of dependent variable
$y_{\text{experimental}}$	Experimental value of dependent variable
$y_{\text{model}}$	Model value of dependent variable
$N$	Number of data
$V$	Line slope
$W$	$y_{\text{model}}$ -intercept
RSM	Response surface methodology

$Y$	Response
$\beta_0$	Constant for intercept
$\beta_{01}$ to $\beta_{09}$	Constant for blocks
$\beta_j$	Linear coefficient
$\beta_{jj}$	Quadratic coefficient
$\beta_{ij}$	Interaction coefficient
$x_i$ and $x_j$	Independent variables ( $i$ and $j$ are in the range of 1 to $k$ )
$k$	Number of independent variables ( $k=3$ ).
$S$	Standard deviation
$R^2_{\text{adj}}$	Adjusted regression
ANOVA	Analysis of variance
DMRT	Duncan's multiple range tests
$JY$	Juice yield
$g$	Guava
$c$	Control
$ds$	Direct thermosonication
$is$	Indirect thermosonication
$M$	Motion frequency
$t$	Time
$T$	Temperature
$A$	Amplitude
$P$	Power
$a_T$	Shear rate-temperature shift factor
$\dot{\gamma}_T$	Shear rate at temperature $T$

$\dot{\gamma}_{T_{ref}}$	Shear rate at reference temperature
$a_C$	Shear rate-temperature-concentration shift factor
$\dot{\gamma}_C$	Shear rate at concentration C
$\dot{\gamma}_{C_{ref}}$	Shear rate at reference concentration
$b_C$	Concentration shift factor
$\sigma_C$	Shear stress at concentration C
$\sigma_{C_{ref}}$	Shear stress at reference concentration
$a_T$	Shift factor
$C_T$	Concentration at defined temperature
$C_{T_{ref}}$	Concentration at reference temperature
$k_T$	Constant
$n_T$	Constant
$E_a$	Activation energy
$R$	Universal gas constant
$H$	Maximum value
$D$	Constant
$r$	Growth parameter
$k_c$	Constant
$n_c$	Constant
$b$	Constant
$N$	Number of population

## CHAPTER 1

### INTRODUCTION

#### 1.1 Background of Study

Fruits are perishable and some of them are seasonal. The demand for fruit juice is growing due to advantages of it being available all year round. Fruit juice is convenient for consumer without having to peel and cut fruits. People have been educated through mass media on the health benefits of fruit juice consumption. The processing of tropical fruit juice started in many countries during the last decades (Sharoba and Ramadan, 2011). Malaysia has increased by 13% in sales volume of ready-to-drink juice by litres in less than a decade (Anonymous, 2014b). The Star newspaper (Anonymous, 2014b) reported that 47% of those interviewed wanted to look cool and fashionable by drinking juice, while 65% of them love to drink juice for its goodness. Malaysians are also concerned about the taste and the quality of fruit juice. Processing into fruit juice can also help to reduce post-harvest wastage. Fruit production worldwide records over 360 million metric tons (Sharoba and Ramadan, 2011).

The pink-fleshed guava, pink-fleshed pummelo and soursop fruits juice are the main subject in this research because these fruits have a lot of medical benefits. There is limited and/or no information and published data found on extracting juices from these three fruits as well as preserving them into juice concentrates (Brasil *et al.*, 1995; Quek *et al.*, 2012). By consuming these fruits, cancer cells can be attacked safely and effectively naturally without causing extreme side effects like nausea and hair loss. The pink-fleshed guava (*Psidium guajava* L.) contains lycopene, which can prevent skin damage from UV rays and offer protection from prostate cancer (van Breemen and Pajkovic, 2008). It is also rich in carotene to protect from lung and oral cavity cancers. The high vitamin C content in pink-fleshed pummelo (*Citrus Maxima* M.) helps to strengthen and maintain the elasticity of arteries (Sarvamangala *et al.*, 2013). Besides good for digestive system, the pummelo can aid in weight loss process because the fat burning enzyme in pummelo can help to absorb and reduce starch and sugar in the body. The pink-fleshed pummelo is slightly sweeter and more nutritious than other pummelos because of the darker pigment. The soursop (*Annona muricata* L.) contains annonaceous acetogenins, which owns prostate cancer chemopreventive compounds (Atawodi, 2011). This fruit is good as a remedy for urethritis, haematuria and liver ailments (Badrie and Schauss, 2010) and also widely used as anticancer folk therapies in the North, Central and South America, and Southeast Asia (Ko *et al.*, 2011b).

The soursop fruit is hardly found, while the fresh pink-fleshed guava fruit cannot be found in Malaysian market. The nation's only pink guava producer is Sime Darby Beverages Sdn. Bhd. and the company does not sell the pink-fleshed guava fruits except for special request. The harvested fruits are processed into puree immediately



or not more than six hours after being harvested everyday and the puree is exported. Pink-fleshed guava ready-to-drink juice and pink-fleshed guava juice concentrate, which is branded as GoFresh, are produced in small amount, and only sold in selected local supermarket and served in-flights by the Malaysia Airlines (Anonymous, 2015b). The pink-fleshed pummelo is only available twice a year, i.e., during Chinese New Year and Mid-Autumn Festival. Since the fruits are highly perishable and seasonal, they should be processed into juices to make their nutrition and freshness available all year around.

## 1.2 Statement of the Problem

Generally, fruit juice extraction is done by pressing the pulp to expel the juice. However, there are many fruit-specific ways to extract the juice (Bates *et al.*, 2001). Juice from firm and seed inedible fruits like apple is pressed mechanically, where hot press is a common process to get higher juice yield. For firm fruits with inedible skin and seeds such as citrus fruits, their fleshes are pressed mechanically to extract the juice. A tough adhering skin and seeds like soursop is hand peeled and its juice is extracted using a pulper finisher, which use heat to soften the tissue and inactivate enzymes. A steam extraction system is a current practice to extract juice from soft and readily extractable fruits such as grapes, where juice is flow out from the fruits and leave the skin empty.

The pink-fleshed guava and soursop fruits are difficult-to-juice produce and perishable almost immediately after they have ripened, while the pink-fleshed pummelo fruit is a free-run juice (Bashir and Abu-Goukh, 2003; Anonymous, 2009; Quek *et al.*, 2012; Davies and Mohammed, 2013). As the pink-fleshed guava and soursop juices are difficult to be extracted, a more advanced extraction process is needed to increase the volume of juice produced. Extraction methods which produce high quality juices will fulfil consumers' demand on nutritious, convenient and minimally processed fruit juices for health reasons. Thermosonication treatment, which is a combination of mild heat and ultrasound aids in juice extraction process through high extraction rate with minimum adverse effects of heating. Although ultrasonically assisted extraction process is known of its capability of releasing contents such as sugar, medicinal compounds and protein from biological materials by disrupting the cell walls (Mason *et al.*, 1996), it has not been used for extracting pink-fleshed guava, pink-fleshed pummelo and soursop juices. Sin *et al.* (2006) and Lee *et al.* (2006) applied hot water extraction method to extract sapodilla and banana juice, respectively, while Cendres *et al.* (2011) extracted juice from grape, plums and apricots using microwave.

The fruit juice industry deals with variety of temperatures and unit operations such as extraction, size reduction, fluid flow and heat transfer in its production process. The knowledge on rheological behaviour of the tropical fruit juice concentrates as affected by concentration and temperature are necessary for food and process engineering, for scientific applications, and to evaluate juice-processing equipments. A proper design of extraction operations could help to optimise processing, prevent

over-dimensioned of facilities and reduce wasteful use of economic resources (Falguera *et al.*, 2010). It also leaves less fouling thus fruit juice may flow easily without being stuck in a pipe. Optimisation and modelling approach is important in helping to design a proper tropical fruit juice extraction with a systematic and accurate approach. A better understanding of the optimisation technique and rheological behaviour of the tropical fruit juice concentrates provide opportunities of maximizing the capacity of fruit juice production without adverse effects to the nutrients content. The preservation of nutrients content is definitely apprehending the exclamation of Malaysian government urging the public to eat healthy (Anonymous, 2014a; Anonymous, 2015a).

### 1.3 Aims and Objectives

The aim of this study is to investigate the extraction of tropical fruit juice with assisted thermosonication treatment through modelling and optimisation approach. Tropical fruits in this context refer to the pink-fleshed guava, pink-fleshed pummelo and soursop. This research aims to provide a best and efficient thermosonic-assisted juice extraction method to increase the volume of difficult-to-juice produce with minimum adverse effects on vitamin C of the juice. Besides that, this research also provides model for correlating critical processing parameters, i.e., temperature and juice concentration with its rheological behaviour for optimisation of juice flowability during juice production process, handling and transportation.

The specific objectives are:

- i. to compare the direct and indirect thermosonic-assisted extraction methods and conditions for producing higher juice yield with maximum ascorbic acid and total soluble solids levels of the tropical fruit juices,
- ii. to model rheological behaviour of juice concentrates using the power law model and obtain the rheological parameters,
- iii. to investigate the applicability of shear rate-temperature-concentration superposition technique for modelling rheological data,
- iv. to model the effect of concentration on rheological behaviour of the tropical fruit juice concentrates using existing models, and concentration-temperature principle for developing a master curve, and
- v. to develop and analyse a proposed new mathematical model, which combines the Arrhenius and logistic growth models for modelling the effect of temperature on the rheological behaviour of the tropical fruit juice concentrates.

### 1.4 Research Novelty

The novelties of this study are:

- i. Extracting pink-fleshed guava, pink-fleshed pummelo and soursop fruit juices using thermosonication-assisted treatment.
- ii. Blocked face-centered central composite design for optimised the tropical fruit juice extraction where four-dimensional (4D) surface plot has helped to explain

- simultaneously the optimum juice extraction conditions affected by combination of 3 input factors.
- iii. Application of vertical shift of rheological data of pink-fleshed guava to make a smooth master curve.
  - iv. A new mathematical model proposed from the combination of Arrhenius and logistic sigmoidal growth models to alleviate limitation of using Arrhenius model which trends to negative values of  $K$  and  $n$  due to positive value of  $K$  and low  $n$  magnitude between 0 and 1.

## 1.5 Scope of Thesis

The research performed in this thesis for studying fruit juice extraction and modelling of its rheological behaviour in concentrate form is meant to increase juice production capacity and improve juice processing efficiency. Chapter 2 reviews previous studies on fruit juice production and mathematical modelling. The groups of tropical fruit juice and its processing technologies are summarised. A full description about thermosonication treatment and its effects on fruit juice quality and safety are reviewed. The mathematical modelling for process optimisation, existing rheological models and superposition principle are reviewed and presented.

Chapter 3 provides the materials, set-up and experimental procedures in performing this research. Fruit juice concentrates preparation is reported and the design of each experiment is illustrated by process flow chart. The methods used for analysing each sample and the optimisation steps are described. The statistical analysis used is explained at the end of the section.

A suitable juice extraction method is necessary to minimise the adverse effects cause by overheating during juice processing. Chapter 4 offers results of the best juice extraction method for fruit type studied. The characteristics of fresh fruit pulp and mixture pulp for each materials involved are reported. The last section in Chapter 4 gave experimental verification on the precision of predicting equations on models.

Chapter 5 presents the flow behaviour of juice concentrates by the power law model fitting. This chapter shows the development of a master curve using shear rate-temperature-concentration superposition principle via single horizontal and horizontal-vertical shifting.

Chapter 6 presents the correlations between juice concentration and consistency coefficient or flow behaviour index by the power law and exponential models. The master curve was constructed to explain the effect of concentration on consistency coefficient or flow behaviour index of all rheological data in one curve line.

The Arrhenius model has been widely used to describe the effect of temperature. Chapter 7 proposes new mathematical model to predict the consistency coefficient and flow behaviour index at temperature increase. This chapter presents the discovery of a new mathematical model, a cross product of the Arrhenius and logistic sigmoidal growth models.

Finally, Chapter 8 summarises the findings and overall significance of this study. Suggestions for future work are made.



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