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EFFECTS OF SOAKING TECHNIQUES ON PHYSICAL PROPERTIES AND WATER QUALITIES DURING RETTING PROCESS OF PEPPER BERRIES (Piper nigrum L.)

PUTERI NURAIN BINTI MEGAT AHMAD AZMAN

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By

PUTERI NURAIN BINTI MEGAT AHMAD AZMAN

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Chair : Prof. Ts. Rosnah Shamsudin, PhD
Faculty : Engineering

The pepper berry *(Piper nigrum L.)* is referred to as the 'King of Spices' and is one of the most widely used spices. The retting process of pepper berries is a necessary method to produce white pepper. The conventional retting process of pepper berries that encounters the long period for softening the pericarp and also, resulting the bad smell and low quality of white pepper produced during the rainy season. This study aims to determine the effects of soaking techniques on the physical properties and water qualities during the retting process of pepper berries. In this study, the physical properties of pepper berries from the Kuching variety at different maturity levels were measured. Their mass was predicted using four models: linear, quadratic, s-curve, and power. The results showed that mass of ripe pepper berries based on the volume (Quadratic model) is recommended equation, as the nonlinear form $M = 0.828 - 0.015V + 7.376 \times 10^{-5}V^2$ had the highest R^2, 0.995, at the 1% probability level compared to others. Next, the retting process was conducted for stagnant water by having three soaking conditions of 1 kg (A), 2 kg (B), and 3 kg (C). Meanwhile, the retting process using flowing water at a rate of 70 $\mathrm{L/min}$ was carried out by having soaking conditions of 5 kg (F) and 10 kg (G). Daily samplings were done for 7 consecutive days. The results reveal that the soaking condition F had the lighter colour of soaked pepper berries (70.79%). Soaking condition G had the highest reduction in diameter (25.27%) and weight (36.36%) of soaked pepper berries compared to the soaking conditions A, B, C and F. According to the results, the soaking water qualities during the retting process using stagnant water is the most significantly $(p < 0.001)$ affected due to the organic matter and bioactive compounds leached out from pepper berries. An electrocoagulation was evaluated in accelerating the retting process of pepper berries using stainless steel and aluminium electrodes. The retting process using an electrocoagulation was conducted by having 166.67 g pepper berries soaked in 1000 mL of water for 7 days' data. The findings indicate that the soaked pepper berries during the

retting process using electrocoagulation with stainless steel proved the rapid reduction in dimensions (major axis: 21.23%, medium axis: 23.63%, minor axis: 21.61%, diameter: 22.07%) and volume (44.05%) of soaked pepper berries compared to the soaked pepper berries in the retting process using stagnant and flowing water. The experimental data were analysed by performing correlations. Thus, these findings are considered useful for predicting the physical properties and soaking water qualities changes as well as provide a novel insight into the acceleration of the retting process of pepper berries.

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

KESAN TEKNIK-TEKNIK RENDAMAN TERHADAP SIFAT-SIFAT FIZIKAL DAN KUALITI-KUALITI AIR SEMASA PROSES PERENDAMAN BERI LADA (*Piper nigrum* L.)

Oleh

PUTERI NURAIN BINTI MEGAT AHMAD AZMAN

Mei 2022

Pengerusi : Prof. Ts. Rosnah Shamsudin, PhD
Fakulti : Kejuruteraan

Beri lada (*Piper nigrum* L.) dikenali sebagai 'Raja Rempah' merupakan salah satu daripada rempah yang paling popular digunakan. Proses perendaman beri lada adalah kaedah yang diperlukan untuk menghasilkan lada putih. Proses perendaman beri lada secara konvensional mengambil tempoh yang lama untuk melembutkan perikarpa dan juga, ia mengakibatkan bau busuk dan kualiti rendah lada putih yang yang dihasilkan semasa musim hujan. Kajian ini bertujuan untuk menentukan kesan teknik rendaman terhadap sifat fizikal dan kualiti air semasa proses perendaman beri lada. Dalam kajian ini, sifat fizikal beri lada daripada varieti Kuching pada tahap kematangan yang berbeza telah diukur. Jisim mereka telah diramalkan menggunakan empat model: linear, kuadratik, lengkung-s, dan kuasa. Keputusan menunjukkan bahawa jisim beri lada masak berdasarkan isipadu (model kuadratik) adalah persamaan yang disyorkan kerana bentuk bukan linear, $M = 0.828 - 0.015V + 7.376 \times 10^{-5}V^2$ mempunyai R^2 tertinggi, 0.995, pada tahap kebarangkalian 1% berbanding yang lain. Seterusnya, proses perendaman dijalankan untuk air bertakung dengan mempunyai tiga keadaan rendaman iaitu 1 kg (A), 2 kg (B), dan 3 kg (C). Manakala proses perendaman menggunakan air mengalir pada kadar 70 L/min dijalankan dengan mempunyai keadaan rendaman 5 kg (F) dan 10 kg (G). Persampelan harian dilakukan selama 7 hari berturut-turut. Hasil kajian menunjukkan bahawa beri lada dalam keadaan rendaman F mempunyai warna yang lebih cerah (70.79%). Keadaan rendaman G mempunyai pengurangan paling tinggi dalam diameter (25.27%) dan berat (36.36%) beri lada yang direndam berbanding keadaan rendaman A, B, C dan F. Mengikut keputusan, kualiti air rendaman semasa proses perendaman menggunakan air bertakung adalah paling ketara ($p < 0.001$) terjejas disebabkan oleh bahan organik dan sebatian bioaktif yang terlarut lesap daripada beri lada. Elektrokoagulasi telah diuji dalam mempercepatkan proses perendaman beri lada menggunakan elektrod keluli tahan karat dan aluminium. Proses perendaman menggunakan elektrokoagulasi telah dijalankan dengan merendam 166.67 g beri lada dalam

1000 mL air selama 7 hari. Dapatan kajian menunjukkan bahawa beri lada direndam semasa proses perendaman menggunakan elektrokoagulasi dengan elektrod keluli tahan karat telah membuktikan pengurangan dengan cepat dalam dimensi (paksi utama: 21.23%, paksi sederhana: 23.63%, paksi kecil: 21.61%, diameter: 22.07%) dan isipadu (44.05%) berbanding beri lada yang direndam dalam proses perendaman menggunakan air bertakung dan mengalir. Data eksperimen dianalisis dengan melakukan korelasi. Oleh itu, penemuan ini dianggap berguna untuk meramalkan perubahan sifat fizikal dan kualiti air rendaman serta pendekatan baru dalam mempercepatkan proses perendaman beri lada.

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This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfilment of the requirement for the degree of Master of Science. The members of the Supervisory Committee were as follows:

Rosnah binti Shamsudin, PhD
Professor, Ts
Faculty of Engineering
Universiti Putra Malaysia
(Chairman)

Hasfalina binti Che Man, PhD
Professor, Ir
Faculty of Engineering
Universiti Putra Malaysia
(Member)

Mohammad Effendy bin Ya'acob, PhD
Senior Lecturer, Ir
Faculty of Engineering
Universiti Putra Malaysia
(Member)

ZALILAH MOHD SHARIFF, PhD
Professor and Dean
School of Graduate Studies
Universiti Putra Malaysia

Date: 13 October 2022

Declaration by Members of Supervisory Committee

This is to confirm that:

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

Signature: ______________________________
Name of Chairman of Supervisory Committee: Professor Ts. Dr. Rosnah binti Shamsudin

Signature: ______________________________
Name of Member of Supervisory Committee: Professor Ir. Dr. Hasfalina binti Che Man

Signature: ______________________________
Name of Member of Supervisory Committee: Ir. Dr. Mohammad Effendy bin Ya'acob

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LIST OF ABBREVIATIONS

ΔE	Total colour change
A	Soaking condition of a jute sack filled with 1kg filled for retting process using stagnant water
*a**	Redness or greenness
AR	Aspect ratio
B	Soaking condition of 2 jute sacks filled, each jute sack with 1kg filled for retting process using stagnant water
*b**	Yellowness or blueness
BI	Browning index
BOD	Biochemical oxygen demand
C	Soaking condition of 3 jute sacks filled, each jute sack with 1kg filled for retting process using stagnant water
*c**	Chroma
COD	Chemical oxygen demand
CPA	Criteria projected area
DC	Direct current
D_g	Geometric diameter
DO	Dissolved oxygen
DOE	Department of Environment
F	Soaking condition of 5 kg of pepper berries soaked in the flowing water during the retting process
FAQ	Fair average quality
G	Soaking condition of 10 kg of pepper berries soaked in the flowing water during the retting process
*h**	Hue angle
HA	Humic acids

HCl	Hydrochloric acid
L	Major axis
*L**	Lightness or darkness
MPB	Malaysian Pepper Board
PA_L	Projected area of major axis
PA_T	Projected area of medium axis
PA_W	Projected area of minor axis
PC1	First component
PC2	Second component
PC3	Third component
PCA	Principal component analysis
R^2	Coefficient of determination
SA_{sp}	Surface area of spheroid shape
SEE	Standard error of estimate
SIRIM	Malaysian Industrial Research and Standards Institute
T	Medium axis
TDS	Total dissolved solid
TSS	Total soluble solid
V	Volume
W	Minor axis
w/w	Weight per weight

CHAPTER 1

INTRODUCTION

1.1 Background of study

Piperaceae is a family of flowering plants or trees cultivated for their fruits. *Piper nigrum* L., or pepper, is an economically and ecologically important species in the Piperaceae family. Pepper is used in most food preparations and is characterised by its pungent smell, spicy taste, and health-friendly properties. Pepper has a variety of applications in the culinary industries. There are four different types which comprise green pepper, red pepper, black pepper, and white pepper. According to Nair (2011), the growing consumption of pepper for culinaries in Eastern countries and the technical advancement of preservation in the food sector have driven global demand for the spice to higher levels over the forecast period. Increased governmental support for the production of pepper due to its antioxidant and anti-inflammatory properties, combined with increased consumption of fast-food products and fried products, expected to further boost demand for pepper.

Given the market potential of pepper the processing method in production should be monitored in order to obtain higher quality products. In the production of white pepper, soaking is an essential and necessary method in the water retting process to soften the pericarp of mature pepper berries. Retting is defined as a process to break chemical bonds by using natural microorganisms which allows for the loosening and separation of the fibre bundles from non-fibre fractions or woody core (Collins, 1934; Akin, 2010; Kuhad & Singh, 2013; Aziz et al., 2019). Water retting is also commonly used for bast plants such as hemp, jute, flax, and kenaf to produce long fibres (Tahir et al., 2011; Aziz et al., 2019). In Sarawak, the conventional retting used in the production of white pepper is to soak the fresh berries for 12-14 days under running water, such as rivers, after harvesting and threshing (Rosnah & Chan, 2014). In Johor, retting is carried out in stagnant water, which is changed regularly. According to Aziz et al. (2018) and Mazaheri & Mozaffari (2019), there are possibilities that during prolonged retting organic matter and bioactive compounds naturally present in pepper may leach out into the soaking water. Conversely the soaking water may also contains inorganic matter and microorganisms (Mazaheri & Mozaffari, 2019). These possibilities during retting need further investigation.

Firstly, the quality or characteristic of pepper both chemical and physical produced in retting can be determined through analysis. Pepper quality is an important factor in production to conserve its taste and flavour. Physical properties are measured and observed on both processed and unprocessed pepper. Processing such as drying, soaking, and others may change these properties. Additionally the properties may also be affected during machine processing which include handling, cleaning, conveying and storing (Lorestani

& Ghari, 2012). The most crucial properties in the design of processing machine are its dimensions, weight, volume, and projected area (Mohsenin, 1986; Feizollah & Satar, 2014). Basic data on the physical properties of bio-materials are useful to all engineers, industrial processors, scientists, pharmacists, crop breeders, researchers, and others who may use the information for designing advanced machine. (Pradhan et al., 2010; Pathak et al., 2019). Analysis of regression, such as mass modelling, produces an equation that may describe and predict the statistical relationship between one or more predictor factors and variable reactions. Mass modelling can be analysed by regression relationships, such as Linear, Quadratic, S-curve, and Power as utilised in previous studies (Lorestani & Ghari, 2012; Nur Salihah et al., 2015; Jaiswal et al., 2017; Pathak et al., 2019). The statistical significance in the regression relationship shows that the modifications in the dependent variables are correlated. A high coefficient of determination (R^2) indicates that the model may explain most of the percentage of variability in the dependent variable. The correlation determination technique between mass and its corresponding physical properties is more specific for automatic classification of most berries and fruits. Furthermore, pepper is categorized in the berries group and is similar to cherries, raisins and others. The differences between berries and other fruits can be observed. Berries are fleshy fruits produced from a single ovary, while non-berries are produced from single or multiple ovaries. Berries are edible when the entire ovary wall is ripened. This may not be the same for all fruits. The physical properties are important parameters to evaluate the changes affected by processing. Some earlier publications dealt with the physical properties of pepper berries, (Murthy & Bhattacharya (1998) and Rosnah & Chan (2014)). Other studies reported similar research related to other berries such as raisins (Karimi et al., 2011) and trans-himalayan seabuckthorn (Jaiswal et al., 2017).

Secondly, the qualities of soaking water should be monitored and well-managed. According to Safwat (2020) and Chindaprasirt & Rattanasak (2020), wastewater from industrial processes should be managed under controlled conditions, that may include the reduction in chemical oxygen demand (COD) or neutralising the PH before being discharged. The determination of water qualities such as turbidity, pH, dissolved oxygen (DO), chemical oxygen demand (COD), total dissolved solid (TDS), and colour are essential for further processing.

Electrocoagulation is a promising method in reducing pH, total soluble solid (TSS) and metal content, as well for removing organic matter, colloidal particles, colour, and microorganisms (Niazmand et al., 2020; Amri et al., 2022). It is also effective for organic and inorganic pollution treatment because it takes advantage of coagulation-flocculation, flotation, and electro-oxidation or electro-reduction (Holt et al., 2005; Dura, 2013; Rodriguez et al., 2020; Adou et al., 2022). The prolonged retting process of pepper berries may cause the leaching out of bioactive compounds and organic matters from pepper pericarp into the soaking water upon degradation of the pericarp. (Aziz et al., 2018). Due to its reduction abilities, electrocoagulation is the chosen method for accelerating the retting process of pepper berries in white pepper production. Electrocoagulation is commonly used to treat wastewater from industries and less for other purpose. The process is carried out in a reactor in the presence of a pair of metal electrodes (anode-cathode). Based on electrochemical principles, the cathode is oxidized (loses electrons) which the metals cations are released into the water through dissolving metal electrodes, while a sacrificial anode dissolves itself with the passage of electric current through it (Kabdaşlı et al., 2012; Vepsäläinen, 2012; Hedes et al., 2019; Bakshi et al., 2019). In electrocoagulation, the ions (heavy metals) and colloids (organic and inorganic) in wastewater are mainly contained in an electrically charged solution. According to Tetreault (2003), colloidal mechanisms may be destabilised by adding charged ions to the opposite side of the colloid solution. The destabilised colloids can then be aggregated and isolated from the wastewater. According to Jawad et al. (2021), stainless steel, aluminium and iron electrodes were used to remove acidic yellow dye and chemical oxygen demand from textile wastewater. In this technique dark coloured wastewater changes into clear medium.

1.2 Problem Statement

Pepper is a very well-known spice as it is widely used in the food and pharmaceutical industry for centuries. It has a unique taste, and flavour and endorsed with various benefits. In the harvesting process, the selection of pepper berries depends on the maturity levels, either immature, mature, and ripe. The physical properties and relationships in mass modelling of pepper berries should be pre-determined in order to design and optimise the machine for handling, cleaning, conveying, and storing (Lorestani & Ghari, 2012). However, past research on mass prediction using model equations is rather limited for pepper berries compared to other berries or fruits. Mass modelling of pepper berries has not been adequately carried out in past studies.

The production of white pepper requires a soaking technique for the retting process. Farmers in Sarawak still use the conventional retting process, which include soaking the jute sack containing the berries in a flowing river. A recurrent problem faced is the lengthy retting process which requires about 10-20 days for pericarp removal. During rainy season, the strong river current often sweep away the jute sacks. The churning river water carries higher suspended load and is more contaminated thus affecting the retting process. Rotting berries often turn into dark grey mash with strong odour. The colour of peppercorn is also affected leading to less amount of high-quality produce generated which is creamy white shade. The quality should thus be improved by accelerating the retting process to ensure more creamy white peppers produced. This problem needs to be investigated.

Studies on the on the physical properties of fresh pepper berries are also lacking. Such studies are however common for other fruits including orange, gumbo, apple, pomegranate, fava bean, persimmon, pomelo, dried Terminalia chebula and banana. There is insufficient investigation reported in the literature related to the properties of fresh pepper berries and soaking water during the retting process. In comparison many studies are recorded on other food materials such as beans, soybean, cowpea, wheat kernel, peanuts, rice, chickpea, maize and others. One previous study however documented the effects on physical properties caused by enzymatic retting of green pepper berries in white pepper production (Rosnah & Chan, 2014). However, the effects of the retting process on the physical properties of mature pepper berries particularly in the processing of white pepper are poorly documented.

1.3 Objectives

The main objective of this study was to determine the influences of soaking techniques on the physical properties of pepper berries during the retting process. In this study, the changes in the physical properties of soaked pepper berries and soaking water qualities are elucidated. The specific objectives are as follows:

1. To determine the physical properties of pepper berries at different maturity levels and its mass modelling.
2. To analyse the influence of two soaking techniques, stagnant and flowing water, on the physical properties of pepper berries and soaking water qualities during the retting process.
3. To evaluate the effectiveness of electrocoagulation in treating the pepper wastewater as well as accelerating the retting process of pepper berries using two types of electrodes, stainless steel and aluminium.

1.4 Scope of study

This study focuses on effects of different soaking techniques on the physical properties and soaking water qualities during the retting process of pepper berries. Herein, the scope of the study is described in a chronological order, beginning with sample preparation and ending in the acceleration of the retting process using electrocoagulation.

Firstly, pepper berries were obtained from a farm located in the south of Malaysia and the Kuching variety were chosen. The berries were sorted according to different maturity levels (immature, mature and ripe). The physical properties (dimensions, aspect ratio, sphericity, weight, volume, density, moisture content and colour) were then determined at the different maturity levels. The physical properties recorded were used in the model equations (linear, quadratic, s-curve and power) to formulate the mass modelling. For further analysis the mature pepper berries were selected. The retting process was conducted in stagnant and flowing water to demonstrate the conventional technique. For stagnant water, three different soaking conditions were used: 1 kg of pepper berries (A), 2 kg of pepper berries (B) and 3 kg of pepper berries (C) were soaked in 18L of water. The same water volume was used for each soaking. Additionally, two soaking conditions: 5 kg of pepper berries (F) and 10 kg of pepper berries (G) were soaked in flowing water. The physical properties of soaked pepper berries and soaking water qualities were subsequently analysed with different soaking techniques used in the retting process.

Furthermore, the effect of accelerated retting process was evaluated by examining the results of electrocoagulation on the physical properties of soaked pepper berries and the soaking water qualities. The electrocoagulation was conducted using 2 different types of electrodes (stainless steel and aluminium). Changes in physical properties of soaked pepper berries and soaking water qualities due to electrocoagulation under stagnant and flowing water conditions were compared.

1.5 Thesis overview

Chapter 1 highlights the general overview of pepper berries background, conventional retting process, and objectives of this study. The scope of the study is also described.

Chapter 2 presents a literature review related to the pepper varieties, benefits, white pepper production, electrocoagulation, physical properties, and soaking water qualities. This chapter reviews the past studies reported in the literature.

Chapter 3 explains in detail the sample preparation and methodologies used in the study. This chapter detailed the procedure for different soaking techniques during the retting process. The experimental and statistical analyses were also explained.

Chapter 4 discusses the experimental results obtained from the study as related to previous studies reviewed in the literature.

Chapter 5 concludes the overall results of the study. The recommendations for possible future work are also stated.

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