OPTIMIZATION OF CHARANTIN RICH POWDER FROM *MOMORDICA CHARANTIA* USING EXTRACTION AND SPRAY DRYING PROCESSES

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...Special Dedication to my late father, Zaini bin Mohd Yasin...
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ABSTRACT

Momordica charantia (M. charantia) fruits have a lot of health benefits through its antitumor, antimicrobial, antiviral, immunotoxin, antifertility and antimutagenic properties. Besides, M. charantia has the potential of controlling glucose levels in hyperglycemic states in Asian countries. The target compound in this fruits is charantin. Thus, a research on the production of charantin rich powder from M. charantia was carried out using a water-based extraction and spray dryer. The objectives of this research are to determine the optimum conditions for the extraction of charantin from M. charantia, to investigate the optimum condition of total solid content of maltodextrin for the feedstock of the spray drying process, and to optimize the parameters of the M. charantia powder spray drying process to produce higher quality charantin encapsulation. The process conditions of this study were particle size (250 ≤ particle diameter ≤ 300) µm, extraction time (6 hours) and ratio sample:solvent (1:20). The optimum temperature for maximum extraction was 80 °C which yielded 8.238 mg/mL of charantin. The optimum feedstock solution level was 10° brix of total solid content of maltodextrin which yielded 6.948 mg/mL of charantin. The spray drying was run at an inlet temperature ranging from 150 °C to 190 °C and a feed flow rate ranging from 2 to 4 mL/min. The maximum charantin concentration during spray drying process condition was achieved at 170 °C and 3 mL/min with the concentration of 2.400 mg/mL, powder yield of 2.9 g, encapsulation efficiency of 29.1% and moisture content of 2.3%. The optimization of spray drying conditions were evaluated in term of inlet temperature and feed flow rate with respect to responses of powder yield, concentration of charantin and moisture content. The results showed that a temperature of 175.96°C and feed flow rate of 2.41 mL/min produced the highest powder yield, moisture content and concentration of charantin with 3.51 g, 2.5% and 1.563 mg/mL, respectively. The regression coefficient, $R^2$ for powder yield, concentration of charantin and moisture content were 0.8741, 0.7579 and 0.7651, respectively. This shows that the higher value of $R^2$ is consistent with the data predicted using an experimental data model. The results of this study have a potential for commercialization as the powder contains rich encapsulation of charantin.
ABSTRAK

Buah peria katak memberi banyak kesan yang baik kepada kesehatan menerusi antitumor, antimikrob, antivirus, imunotoksin, antikesuburan dan antimutagenik. Selain itu, peria katak juga berpotensi dalam mengawal paras glukosa pada keadaan hiperglisemik di negara-negara Asian. Sasaran komponen dalam buah peria katak ialah charantina. Oleh itu, satu kajian tentang pengeluaran serbuk charantina daripada peria katak telah dijalankan dengan menggunakan kaedah penyarian berasaskan air dan pengeriting sembub. Objektif dalam kajian ini termasuklah menentukan keadaan optimum bagi proses penyarian charantina daripada peria katak, mengkaji tahap keadaan optimum terhadap jumlah kandungan pepejal maltodekstrin bagi suapan proses pengeritingan sembub dan menentukan keadaan optimum parameter bagi proses pengeritingan sembub buah peria katak dalam menghasilkan pengkapsulan charantina yang berkualiti tinggi. Keadaan proses yang digunakan dalam kajian ini adalah saiz zarah (250 ≤ diameter zarah ≤ 300) µm, masa penyarian (6 jam) dan nisbah sampel:pelarut (1:20). Suhu optimum bagi penyarian maksimum ialah 80 °C yang menghasilkan 8.238 mg/mL charantina. Paras larutan suapan optimum ialah 10° brix jumlah kandungan pepejal maltodekstrin yang menghasilkan 6.948 mg/mLcharantina. Pengeritingan sembub dijalankan pada suhu masukan dalam julat 150 °C hingga 190 °C dan kadar aliran suapan dalam julat 2 mL/min hingga 4 mL/min. Kepekatan charantina maksimum yang dicapai ketika proses pengeritingan sembub pada keadaan 170 °C dan 3 mL/min dengan kepekatan 2.400 mg/mL, hasil berat serbuk 2.9 g, kecekapan pengkapsulan 29.1% dan kandungan kelembapan 2.3%. Pengoptimuman keadaan pengeritingan sembub dinilai berdasarkan kepada suhu masukan dan kadar aliran suapan terhadap gerak balas hasil berat serbuk, kepekatan charantina dan kandungan kelembapan. Hasil keputusan menunjukkan bahawa suhu 175.95°C dan kadar aliran suapan 2.41 mL/min menghasilkan berat serbuk yang tinggi, kepekatan charantina dan kandungan kelembapan masing-masing adalah 3.51g, 2.457% dan 1.563 mg/mL. Pekali regresi, $R^2$ untuk hasil berat serbuk, kepekatan charantina dan kandungan kelembapan masing masing adalah 0.8741, 0.7579 dan 0.7651. Ini menunjukkan nilai $R^2$ yang tinggi adalah konsisten dengan data ramalan menggunakan model data eksperimen. Keputusan kajian ini berpotensi untuk dikomersialkan sebagai serbuk yang kaya kandungan pengkapsulan charantina.
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LIST OF ABBREVIATIONS

DE    Dextrose Equivalent
RSM   Response Surface Methodology
HPLC  High Performance Liquid Chromatography
PLE   Pressurize Liquid Extraction
SD    Spray Drying
CTD   Cast Tape Drying
SC-SO₂ Supercritical Carbon Dioxide
E-PVC Emulsion Polymerization
S-PVC Suspension Polymerization
ANOVA Analysis of Variance
SSR   Sum of Squares Regression
SSE   Sum of Square Error
SST   Sum of Square Total
QSRR  Quantitative Structure-Retention Relationship
ADMI  American Dry Milk Institute
IDF   International Dairy Federation
PY    Powder Yield (g)
EE    Encapsulation Efficiency (%)
TCE   Total concentration of charantin in extraction process (mg/mL)
TCS   Total concentration of charantin in powder after spray dried (mg/mL)
MC    Moisture Content (%)
LIST OF SYMBOLS

Mt  Metric Tons
$R^2$  Regression Coefficient
$W_{after}$  Mass of the bottle after spray drying (g)
$W_{before}$  Mass of the bottle before spray drying (g)
$M_a$  Mass crucible after drying (g)
$M_b$  Mass crucible before drying (g)
$W_1$  Mass of extracted oil (g)
$W_2$  Mass of sample used (g)
$Y$  Peak area (mAU*S)
m  Slope of the equation line
$x$  Concentration of $\beta$-sitosterol glucoside/stigmasterol glucoside
c  Intercept line
$Y$  Predicted responses
$\beta_0$  Intercept regression coefficient
$\beta_1, \beta_2$  Linear regression coefficient
$\beta_{12}$  Interaction regression coefficient
$\beta_{11}, \beta_{22}$  Quadratic regression coefficient
$Y_u$  Experimental data
$\bar{Y}$  Mean for experimental data
$\hat{Y}_u$  Predicted data
$p$  Number of terms in model/equation
$N$  Number of experiment
CHAPTER 1

INTRODUCTION

1.1 Background of Study

The study of natural products like plants can be a very thought-provoking subject for scientists to explore further. For decades, plants have been inevitably powerful sources of vast amount of compounds such as vitamins, phenolics, and metabolites that are rich in bio-activities like antioxidant, anticarcinogenic, antibacterial, and antiinflammatory properties. Hence, plants have become a major source for researchers in identifying and developing natural colorants, biodiesel, new drugs and so forth with no or less side effect. With this intention, the demand and requirement of plant studies have increased among natural product researchers.

*Momordica charantia* (M. charantia), a plant commonly known as bitter melon, bitter gourd, kugua, karela or balsam pear which belongs to the Cucurbitaceae family was selected in this study. Physically, *M. charantia* looks like a climber and bears oblong fruits similar in shape to the cucumber (Raina *et al.*, 2016). *M. charantia* is a plant with great nutritional value and has been commonly used as medicine. Traditionally, *M. charantia* is famous as a method for controlling glucose levels in hyperglycemic people in Asian countries who consume this fruit’s juice in the early morning on an empty stomach (Raina *et al.*, 2016; Zhang *et al.*, 2016). Besides being antidiabetic, others medicinal properties such as antitumor, antimicrobial, antiviral, immunotoxin, antifertility and antimutagenic, have been clarified for *M. charantia*. 
Researchers from United Kingdom found that *M. charantia* fruits contain biologically active chemicals such as steroids, protein, saponins, alkaloids, triterpenes and fixed oils (Raman and Lau, 1996). *M. charantia* contains several phytochemicals that have been isolated which include charantin, charine, cryptoxanthin, goyasaponins, gypsogenin, gentisic acid, momordicilin, momodicsins, momordenol and so forth (Grover and Yadav, 2004).

Basically, *M. charantia* extracts in concentrated and powder form is the main commercial source of charantin. Charantin consists of a mixture of two compounds which are stigmasteryl glucoside and sitosteryl glucoside (Pitipanapong et al., 2007). According to Belinda (2000), aside from its usage to treat diabetes, charantin can also be used as a substitute for existing insulin injection treatments that are used to treat diabetes patients by stimulating their pancreas to reduce sugar content in the blood. As they are free from side effects, this compound is now broadly accepted as a surrogate medicine for diabetes mellitus (Sharma et al., 1996).

In the food and pharmaceutical industries, the microencapsulation technique has been widely used to protect food ingredients against volatile losses, deterioration, or premature interaction with other ingredients (Fang and Bhandari, 2011). Research by Jafari et al. (2016) defined microencapsulation as a process of seizing active substances within other materials to produce particles with dimensions of a few micrometers to a few nanometers. Besides the encapsulation technique, another factor that plays an important role in feed and powder characteristics is the selection of carrier agent. There are several types of encapsulation agents that are commonly used by researchers, including maltodextrin (Fongin et al., 2017; Matsuura et al., 2015; Oberoi and Sogi, 2015), the combination of maltodextrin and gum arabic (Idham et al., 2012; Nguyen et al., 2017; Premi and Sharma, 2017), sucrose and glucose (Bayram et al., 2005), and skim milk powder (Shamaei et al., 2017). Maltodextrin is low cost, has low viscosity at higher temperatures according to level of dextrose equivalent (DE) (Matsuura et al., 2015; Wangsakan et al., 2003) and can protect active compounds from oxidization. Thus, maltodextrin was used in this research as an encapsulation agent followed by spray drying which is used as an encapsulation technique for charantin.
Many studies have been conducted by previous researchers on the extraction of active compounds from *M. charantia* fruits. The most common methodologies used for the extraction of active compound include solvent extraction (Horax *et al.*, 2010; Virdi *et al.*, 2003), pressurized liquid extraction (Pitipanapong *et al.*, 2007; Syahariza *et al.*, 2017), heat reflux extraction (Budrat and Shotipruk, 2008; Shan *et al.*, 2012) and water-based extraction (Jain and De, 2016; Wang *et al.*, 2014). The hindrance of using solvent extraction is that it contains high toxicity of extracted yield and is costing when pressurized liquid extraction is applied. Thus, the feedstock of *M. charantia* for spray drying was prepared using water-based extraction method.

Spray drying is a method used to dehydrate fluids such as milk, coffee, and eggs, and it is also used widely in the pharmaceutical and chemical industries (Thirugnanasambandham and Sivakumar, 2015). This is the most important technology used to change the physical properties and remove water content in food. Spray drying is also a method of producing dry powder from a slurry or liquid by rapidly drying it with hot gas. The hot drying gas can be passed as a counter-current or co-current flow to the atomizer direction. The particle separator is typically a cyclone device that operates competently as the co-current flow allows particles to have a lesser residence time within the system. The particles in the chamber which are usually paired with a fluidized bed system enable for greater residence time if the counter-current flow method is used (Møller *et al.*, 2009).

All spray dryers use a spray nozzle or atomizer to disperse the slurry or liquid into a controlled drop size spray. The most common nozzles used are the single fluid pressure swirl nozzle and the rotary nozzle, while the two-fluid or ultrasonic nozzles are used in some applications too. The drying process is core, very rapid and is generally heated to temperatures lower than 100 °C, thus, spray drying is recommended for the encapsulation of heat-sensitive food ingredients. (Fang and Bhandari, 2011).

The optimization and assessment of the extraction process using mathematical and intelligent modeling seem to be essential for industrial applications (Sodeifian *et al.*, 2016). An experimental design has been used to optimize the number of
experiments. To support the interpretation of experimental design result which is discussed mainly in terms of powder yield, concentration of charantin and moisture content, response surface methodology (RSM) was used in this present study. RSM is an effective statistical tool for optimization as it consists of multiple variables that influences powder yield. Study by Sablania and Bosco (2018) state that, the equation and statistical analysis showed an effect on all the responses while the optimized conditions of spray drying validated the model.

1.2 Problem Statement

There are a lot of studies regarding the encapsulation of potential herbs using the spray drying process. However, there are some limitations on the spray drying methodology itself. For example, the drying process takes place without knowing the concentration of the active compound before and after process, as well as the active compound loss. In addition, feedstock preparation for the spray drying process is one of its weaknesses as the preparation take places without the extraction process and with unknown concentrations of the active compound. Besides that, proximate analysis is more appropriate to be used to determine the physical characteristics on a powder, for example its particle size, moisture content, bulk density and ash.

Thus, to overcome this limitation, the extraction of M. charantia fruits using water based extraction was implemented in order to quantify its active compounds such as charantin so to prepare the feedstock for the spray drying process. In addition, the powder produced was analyzed in terms of physical (moisture content) and chemical (concentration charantin) characteristic in order to confirm that the product contains antidiabetic properties.
1.3 Objectives of Research

The objectives of the research are:

i. To determine the optimum condition for the extraction of charantin from *M. charantia*.

ii. To investigate the optimum condition of total solid content of maltodextrin for the feedstock of the spray drying process.

iii. To optimize the parameters of the *M. charantia* powder spray drying process to produce high quality charantin encapsulation.

1.4 Scopes of Research

The scopes of this research include:

i. Water based extraction

Feedstock preparation was performed with a particle size $250 \leq dp \leq 300 \mu m$ and run using a water-based extraction for 6 hours at 60 °C, 80 °C, and 95 °C with ratio sample: solvent (1:20).

ii. Encapsulation process of charantin

Total solid content of maltodextrin which ranged from $5^\circ$ – $20^\circ$ Brix was determined according to the suitable amount of carrier agent and later was set as a constant parameter along with the best condition of extraction temperature. Suitable nozzle sizes in the range of 0.5 – 1.5 mm was set as a constant parameter before the encapsulation studies were conducted at inlet temperature in ranging from 150 °C to 190 °C and a feed flow rate ranging from 2 to 4 mL/min.
iii. Optimization of Spray Drying Process

The operating conditions of the spray drying process such as the inlet temperature, $X_1$ (150 – 190) °C and feed flow rate, $X_2$ (2 -4) mL/min with respect to responses such as powder yield, concentration of charantin and moisture content were optimized using respond surface methodology and contour plot.

iv. Analysis of *M. charantia* extracts and powder

The concentrations of charantin in the Soxhlet extraction, water-based extraction and spray dried powder were quantified using High Performance Liquid Chromatography (HPLC) analysis and the physiochemical characteristic of the dried powder was studied in terms of moisture content.

1.5 Significance of Research

*M. charantia* can be commercialized as it is rich in bioactive compounds especially charantin as charantin is one of the sources for the treatment of the diabetic mellitus disease. Using this treatment, the number of diabetic patients can be reduced in the future. Furthermore, the use of the spray dry method provides a promising alternative in the pharmaceutical industry as an encapsulation technique as it distribute the particles with control shelf life.

Lastly, the experimental procedures of this research may be used by third parties and also give benefits for future study. Besides that, other parties can decide on a suitable parameter based on this research’s experimental data and adopt it according to their uses.
1.6 Limitation of Study

The limitation of this study is the water-based extractor which is, a shaking water bath equipment used in the laboratory. The maximum operating temperature of the equipment is only up to 95°C. In addition, the up-scaling of the feed flow rate pump to spray drying was 1mL/min and not in decimal places.

1.7 Thesis Outline

Overall, this thesis consists of 5 chapters. Chapter 1 began with an introduction to the research project. This chapter included the background of the research, problem statement, objective of research, scope of research, significant of research and limitation of research. Three research objectives were stated within the scope of research.

In Chapter 2, the literature review describes the fundamental theory and application used in this study. This includes previous studies on the extraction process and spray drying process on a wide range of samples. In addition, it also provides an overview on the characteristics of the material which is the M. charantia fruit and the factors affecting the Soxhlet extraction. Besides that, the chapter also describes an overview on optimization using Response Surface Methodology (RSM).

Chapter 3 discusses in detail the research methodology used for the extraction process and spray drying process. The flow of this research includes the pretreatment of sample, preparation of feedstock using the water-based extraction, determination of the total solid content of maltodextrin and extraction temperature, encapsulation using the spray drying technique, and optimization of the drying process. The method for the quantification of charantin which is High Performance Liquid Chromatography and the moisture content analysis are also discussed in this chapter.
In Chapter 4, the results and discussion obtained from the process conducted in Chapter 3 are described briefly. The results from the Soxhlet extraction are presented first in order to determine the exact amount contained in the *M. charantia* fruits. Next, the extraction and pre-encapsulation processes are discussed further in terms of both extract yield and powder yield concentration of charantin. Then, the spray drying results are illustrated and conversed in terms of the effect of spray drying conditions towards powder yield, concentration of charantin and moisture content. Lastly, the optimization of data is computed using response surface methodology (RSM).

The conclusion in Chapter 5 answers all the objectives stated in Chapter 1. Recommendations are also provided for future work and improvement.
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