

OPTIMIZATION OF EXTRACTION AND SPRAY DRYING PARAMETERS ON
FICUS DELTOIDEA EXTRACT

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Dedicated to my beloved husband, Mohd Nasrol Shaman

To my beloved uni and baba

To everyone who believe in my abilities

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ABSTRACT

Vitexin, a marker compound present in *Ficus deltoidea* (*F. deltoidea*) exhibits various phytochemical properties valuable for human health. The previous study has reported lower process yield from the production of *F. deltoidea*. Thus, this study aimed to obtain the highest yield whilst maintaining the vitexin quality by optimising the processing parameters for water extraction and spray drying of *F. deltoidea* using the Box Behnken Design. In the extraction process, the effect of extraction temperature, extraction duration and solvent to raw material ratio on total solid content and vitexin were investigated. Whereas, for the spray drying process using spray dryer with drying capacity of 1000 ml/h, the effect of the inlet air temperature, feed flow rate, air pressure and feed temperature were determined based on process yield, vitexin and moisture content. In addition, the thermal effect of spray drying process on four bioactive compounds of *F. deltoidea*; vitexin, total saponins, total protein, and total polysaccharides were identified. The experimental data obtained from the extraction and spray drying processes were analysed using the analysis of variance. The optimum conditions of the extraction process was achieved at 87 °C of extraction temperature, 2.71 hours of extraction duration and 19.12:1 g/g of solvent to raw material ratio with 8.64 mg/g of solid content and 0.62 % vitexin. The yield of the extract at this condition was 17.39 %. Whereas, the optimum condition for spray drying process was achieved at 191 °C of inlet air temperature, 2 ml/min of feed flow rate, 28 psi of air pressure and 25 °C of feed temperature with 10.01 % of process yield, 0.42 % of vitexin and 4.33 % of moisture content. The spray drying heat does not affect the total saponins and total polysaccharides. However, it led to more than 30 % and 50 % reduction of vitexin and total protein. Therefore, response surface methodology was successful in the optimization of the processing parameters for the extraction and spray drying processes to produce the highest yield and maintain the quality of *F. deltoidea* extract.

ABSTRAK

Vitexin, sebatian penanda dalam *Ficus deltoidea* (*F. deltoidea*) mempamerkan pelbagai sifat fitokimia yang baik untuk kesihatan manusia. Kajian sebelum ini melaporkan hasil proses yang rendah daripada pengeluaran *F. deltoidea*. Oleh itu, kajian ini bertujuan mendapatkan hasil proses yang tinggi dan mengekalkan kualiti *vitexin* dengan mengoptimumkan parameter pemprosesan pengekstrakan air dan pengeringan semburan untuk *F. deltoidea* menggunakan *Box Behnken Design*. Dalam proses pengekstrakan, kesan suhu pengekstrakan, tempoh pengekstrakan dan nisbah pelarut kepada bahan mentah terhadap jumlah bahan kering dan *vitexin* telah disiasat. Manakala, untuk proses pengeringan semburan menggunakan pengering semburan dengan kapasiti pengeringan 1000 ml/h; kesan suhu udara masuk, kadar aliran masukan, tekanan udara dan suhu masukan ditentukan berdasarkan hasil proses, *vitexin* dan kandungan kelembapan. Tambahan lagi, kesan pemanasan pengeringan semburan ke atas kualiti empat sebatian bioaktif dalam *F. deltoidea*; *vitexin*, jumlah saponin, jumlah protein, dan jumlah polisakarida telah dikenal pasti. Data eksperimen yang diperoleh daripada pengekstrakan dan pengeringan semburan *F. deltoidea* dianalisis dengan menggunakan analisis varians. Keadaan optimum untuk proses pengekstrakan telah dicapai pada 87 °C suhu pengekstrakan, 2.71 jam tempoh pengekstrakan dan 19.12:1 g/g nisbah pelarut kepada bahan mentah dengan 8.64 mg/g jumlah ekstrak dan 0.62 % *vitexin*. Hasil ekstrak pada keadaan ini adalah 17.39 %. Manakala, keadaan optimum untuk proses pengeringan semburan dicapai pada 191 °C suhu udara masuk, 2 ml/min kadar aliran masuk, 28 psi tekanan udara dan 25 °C suhu masukan dengan 10.01 % hasil proses, 0.42 % *vitexin* dan 4.33 % kandungan kelembapan. Haba pengeringan semburan tidak menjejaskan jumlah saponin dan jumlah polisakarida. Walau bagaimanapun, ia membawa lebih daripada 30% dan 50% pengurangan pada *vitexin* dan jumlah protein. Oleh itu, kaedah permukaan gerak balas berjaya mengoptimumkan parameter pemprosesan pengekstrakan dan pengeringan semburan untuk menghasilkan hasil proses yang tertinggi dan mengekalkan kualiti ekstrak *F. deltoidea*.

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LIST OF ABBREVIATIONS

ANOVA	-	Analysis of Variance
BBD	-	Box Behnken Design
C	-	Concentration
CCD	-	Central Composite Design
DF	-	Dilution factor
F	-	Fisher
<i>F. deltoidea</i>	-	<i>Ficus deltoidea</i>
HPLC	-	High Performance Liquid Chromatography
MS _A	-	Means of squares for factor A
MS _E	-	Means of squares for error or residual
N	-	Normality
P	-	Probability
RSM	-	Response Surface Methodology
SS	-	Sum of squares
SS _E	-	Sum of squares of residual
SS _R	-	Sum of squares of regression
SS _T	-	Sum of total squares
TLC	-	Thin Layer Chromatography
UV	-	Ulva Violet
UV-Vis	-	Ultraviolet-Visible
V	-	Volume
Var.	-	Varieties
W	-	Weight

LIST OF SYMBOLS

%	-	Percentage
°C	-	Degree Celsius
µg	-	Microgram
µl	-	Microliter
µm	-	Micrometer
b ₀	-	Constant term
e	-	Residual
F ₀	-	Calculated F value
g	-	Gram
H _A	-	Alternative hypothesis
H ₀	-	Null hypothesis
h	-	Hour
mg	-	Milligrams
min	-	Minutes
ml	-	Mililiter
mm	-	Milimeter
nm	-	Nanometer
ppm	-	Part per million
psi	-	Pounds per square inch
R ²	-	Coefficient of multiple determination
rpm	-	Revolution per minutes

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CHAPTER 1

INTRODUCTION

1.1 Background of Study

The search for herbal remedies has become one of the main approaches in the curative of common ailments in most developing countries. Malaysia is known as one of the countries possessing a wide variety of plant species and the existence of traditional medicinal remedies which includes *Ficus deltoidea* (*F. deltoidea*). *F. deltoidea* or locally known as Mas Cotek is recognized as a Malaysian herbal plant (Nasir *et al.*, 2014). This plant has begun to acquire an international recognition and has entered the pharmaceutical market. The dried leaves of *F. deltoidea* have been formulated and commercialized as a tonic drink, capsule, tea and also has been sold as a slimming aid (Woon *et al.*, 2014). The capsule was commercialized as a herbal supplement (Omar *et al.*, 2011). Meanwhile, the tea was packed with 1-2 g of *F. deltoidea* per sachet, where it was recommended to infuse in 1L of hot water (Choo *et al.*, 2012).

F. deltoidea plant has been used as a traditional herbal medicine to regain energy, improve blood circulation, recover and enhance sexual desire, reduce cholesterol and joint pains. Moreover, it's also been taken by women after childbirth for the purpose of strengthening the uterus (Sulaiman *et al.*, 2008; Oh *et al.*, 2010). Currently, researchers have found that *F. deltoidea* plant exhibits other medicinal properties such as antinociceptive, anti-melanogenic, enhancing wound healing and anti-photoaging (Sulaiman *et al.*, 2008; Oh *et al.*, 2010; Abdulla *et al.*, 2010; Hasham *et al.*, 2013).

There are various bioactive compounds in secondary metabolites important for the human's body which can be found in *F. deltoidea* such as flavonoids, tannins and phenol (Oh *et al.*, 2010). There are two main chemical compounds considered as marker compounds found in *F. deltoidea* known as vitexin and isovitexin. However, the amounts of these compounds were found different depending on the variety of *F. deltoidea*. For example, isovitexin was identified as the main compound in *F. deltoidea* var. *terengganuensis* and var. *angustifolia*, while vitexin was found as the main compound in *F. deltoidea* var. *deltoidea* (Abdullah *et al.*, 2009). As this study conducted on *F. deltoidea* var. *deltoidea*, and isovitexin amount were less, therefore the isovitexin was omitted.

Flavones C-glycosides such as vitexin from the flavones group found in *F. deltoidea* leaves extract was believed to possess biological properties including anti-inflammatory, anti-diabetic and antioxidant (Abdullah *et al.*, 2009; Farsi *et al.*, 2011; Choo *et al.*, 2012; Farsi *et al.*, 2014). The other secondary metabolites compound found in the *F. deltoidea* is total saponins. Meanwhile, compounds like total protein and total polysaccharides which are called as primary metabolites were found also in the *F. deltoidea*. All these three compounds were believed to give medicinal properties towards human's health.

Total saponins has been reported to exhibit pharmacological effects including hypo-cholesterolemic activity, antimicrobial, anti-inflammatory, antidermatophytic, antitussives and antiglycation activity (Chen *et al.*, 2010; Chen *et al.*, 2011). The primary metabolite such as total protein is the major source of energy essential to human's health. Protein may contain essential amino-acids which the body cannot synthesize such as tryptophan, methionine, leucine, isoleucine, valine and lysine. Meanwhile, polysaccharides were also found to have several pharmacological activities like anti-bacterial (Lee *et al.*, 2006), anti-tumor, anti-virus and immune activating (Nakamura *et al.*, 2009). Since vitexin, total saponins, total protein and total polysaccharides were beneficial on human's health, therefore it is important to apply good processing techniques which lead to a high amount of these compounds.

The production of *F. deltoidea* water extract was mainly carried out through conventional method such as boiling, soaking or maceration. The capability of selected solvents to extract solutes in the *F. deltoidea* plant is shown by the amount of dry residue or total solid content obtained. Higher amount of dry residue obtained from selected extraction conditions, usually can be found at low consumption of solvent. Besides affecting the properties of the final product, the amount of dry residue also affects the yield of the drying process. Higher dry residue can reduce the amount of water to be evaporated in the drying process (Fernandes *et al.*, 2012). Hence, it is important to obtain high dry residue of *F. deltoidea* extract in this study.

Spray drying is established and was widely used as a method of transforming *F. deltoidea* water extract into a powder form by decreasing the water content. The operational cost for the spray drying of extract is cheaper compared to freeze drying. The spray drying process produced good quality of powder by having properties such as stable in microbiological activity, lower in water activity, ease of handling and also longer shelf life (Gharsallaoui *et al.*, 2007; Tonon *et al.*, 2008). The physicochemical properties of spray dried powders are rely on the processing variables like type of atomizer, drying air temperature, drying air pressure and properties of liquid feed (viscosity, flow rate, particle size). A quality of spray dried powder is characterized by physical properties (moisture content, water activity, rate of dissolution, porosity, particle size distribution, hygroscopicity, bulk density, wettability, solubility and flowability) and by its nutritional composition such as protein, fats, mineral, water and carbohydrates (Tonon *et al.*, 2008).

1.2 Problem Statement

The extraction process is the most crucial part in the preparation of herbal extract. The batch solid liquid aqueous extraction and spray drying technology have been applied for the processing of *F. deltoidea*. The current production process of *F. deltoidea* using water extract has a low yield of 8 % (Oh *et al.*, 2011; Hasham *et al.*, 2013). Most of the studies conducted only focus on the yield of extract, total polyphenols and the effect of *F. deltoidea* extract towards diseases. However, less attention was given to the effect of

extraction processing parameter on the quality of marker compound present in *F. deltoidea* such as vitexin. Only studies by Abdullah *et al.* (2009) and Farsi *et al.* (2013) have reported the amounts of vitexin in water extract and methanol extract respectively. However, the result collected was not considered as the optimum result since the extraction process occurs at fixed operating parameters. In addition to that, Wahid *et al.* (2010) has conducted a study on effect of operating parameters of *F. deltoidea* extract on total phenolic content and antioxidant activity by varying the parameters such extraction temperature, extraction duration and ratio of water to raw *F. deltoidea* leaves. However, this was also not an optimum result since there was no optimization method considered either the classical one. Therefore, the effects of extraction operating parameters need to be identified in order to achieve the highest extraction yield and vitexin. Among the operating parameters affecting the extraction process, the extraction temperature, extraction duration and ratio of solvent to raw material are the most important parameters considered for the analysis of extraction efficacy such as yield and quality (Hinneburg and Neubert, 2005).

Drying process is another important process in producing a solid form of the *F. deltoidea* extract. The common practice by previous researchers in producing the solid form of the *F. deltoidea* was using the freeze dryer (Sulaiman *et al.*, 2008; Abdullah *et al.*, 2009; Nurdiana *et al.*, 2012). However, the existing freeze dry process was associated with longer drying process, higher maintenance and cost. Alternatively, some researchers have used spray dry process for the production of *F. deltoidea* (Adam *et al.*, 2007; Oh *et al.*, 2011; Hasham *et al.*, 2013). Spray drying powders however, have some problems with its properties, such as stickiness, solubility and hygroscopicity. At elevated drying temperature, the atomized particle tends to stick at the wall or at the bottom of dryer and thus leading to low process yield (Tonon *et al.*, 2008). Hence, it is important to study the effect of processing parameters of spray drying process in order to recover the highest process yield with better degradation of phytochemical compounds of the spray dried product.

The quality of phytochemicals of *F. deltoidea* extract from spray drying process were also need to be considered due to the use of elevated temperature during the drying process. High operating temperature may contribute in degradation of phytochemicals in

F. deltoidea powder which will diminish its biological activity. Therefore, the thermal effects of spray drying process on the spray dried of *F. deltoidea* extracts were investigated by comparing the quality of the compounds in the extract and also the spray dried powder.

1.3 Hypothesis

Optimization of processing parameters in the extraction and spray drying processes of *F. deltoidea* is able to produce better processing conditions in order to produce highest yield and also maintain the quality of bioactive compounds in *F. deltoidea*.

1.4 Objective of Research

The objective of this research is to identify the optimum processing parameters for the extraction and spray drying processes on the quality of *Ficus deltoidea*.

1.5 Scope of Research

In order to achieve the objective of this research, there are three major scopes of the study which comprised of:

- i) Optimization of the extraction process of *F. deltoidea* on total solid content and amount of vitexin with three different parameters which includes extraction temperature, extraction duration and solvent to raw material ratio.

- ii) Optimization of the spray drying process of *F. deltoidea* on process yield, amount of vitexin and moisture content with four different parameters which includes inlet air temperature, feed flow rate, air pressure and feed temperature.
- iii) Analysis of the thermal effects of spray drying process on the quality of bioactive compounds in *F. deltoidea* like vitexin, total saponins, total protein and total polysaccharides.

1.6 Significance of Study

The research regarding the extraction of flavone in *F. deltoidea* is expected to contribute valuable experimental data for the phytochemical and herbal processing research technology. Besides, this study would also help to identify the main and interaction effects between the independent variables of processing parameters towards total solid content, amount of vitexin, process yield, and moisture content. In addition, the determination of the correlation between response variables is useful to provide the optimized processing parameters for better production of *F. deltoidea* and at the same time to retain the quality of bioactive compounds in *F. deltoidea*.

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