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Assessment of phenolic compounds stability and retention during spray drying of *Orthosiphon stamineus* extracts

Sook Fun Pang [a], Mashitah M. Yusoff [b], Jolius Gimbun [a,c,*]

[a] Faculty of Chemical & Natural Resources Engineering, Universiti Malaysia Pahang, 26300 Gambang, Pahang, Malaysia
[b] Faculty of Industrial Sciences & Technology, Universiti Malaysia Pahang, 26300 Gambang, Pahang, Malaysia
[c] Centre of Excellence for Advanced Research in Fluid Flow (CARIFF), Universiti Malaysia Pahang, 26300 Gambang, Pahang, Malaysia

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ABSTRACT

This paper presents a microencapsulation study using whey protein isolate and maltodextrin of polyphenol obtained from *Orthosiphon stamineus* leaves. Polyphenol content was analysed using ultra-performance liquid chromatography. Higher solid concentration leads to higher solution viscosity, bigger particle size, lower moisture content and less dented surface, which may improve particle flowability. Microencapsulation using a least amount of protein (0.05 wt.%) yielded better retention of romarinic acid (82.08%), sinensetin (79.57%) and eupatorin (81.08%) than those with higher protein concentration. Meanwhile, 5.33 wt.% of maltodextrin provide the highest polyphenol retention of rosmarinic acid (82.67%), sinensetin (82.24%) and eupatorin (80.19%). The results suggest that eupatorin and rosmarinic acid are more susceptible to thermal degradation than sinensetin during spray drying. Formulation using 5.33% maltodextrin provide a better preservation of polyphenols compared to other formulations.

1. Introduction

Orthosiphon stamineus (vernacular name: *'misai kucing'*) is consumed widely as a herbal tea among the Southeast Asian population. *O. stamineus*is traditionally used in Malaysia for treatment of bladder inflammation, eruptive fever, oedema, hepatitis, jaundice, hypertension, diabetes mellitus, gout, rheumatism, diuretic and influenza (Ho, Ismail, Shaida-Fariza, & Ahmad, 2010). Previous studies revealed that extract of *O. stamineus* contained many medically useful bioactive compounds such as terpenoids, polyphenols and sterols that poses a diuretic (Arafat et al., 2008), antidiabetic (Mohamed, Yam, Ang, Mohamed, & Asmawi, 2013), antiangiogenic and antiproliferative properties (Dolečková et al., 2012).

Extraction of polyphenol from *O. stamineus* has been subjected to many intensive studies (e.g. Akowuah & Ismail, 2010; Akowuah, Ismail, Norhayati, & Sadikun, 2005; Chew et al., 2011; Olah et al., 2004; Pouralinazar, Yunus, & Zahedi, 2012;). However, no previous work related to production of powder-based product from *O. staminues*. A powder-based product is desirable for convenience of consumption besides having longer shelf life and ease of handling. Spray drying is a common method of producing powder, but it requires very high temperature, which may adversely affect the quality of product. Furthermore, the polyphenol from *O. stamineus* extracts is prone to thermal degradation (Akowuah & Ismail, 2010). Thermal degradation of other bioactive compounds such as vitamin E and vitamin A has been reported by Xie, Zhou, Liang, He, and Han (2010). The thermal degradation is undesirable because the degraded product is of low nutritional value and consequently, hampers the intention to produce a functional food or nutraceutical product. However, very limited study concerning drying and preservation of polyphenol in *O. stamineus* extract is available in the literature. The microencapsulation technique via spray drying is an effective way to preserve the bioactive compounds from thermal degradation. Microencapsulation is defined as a process in which small particles are enclosed by a coating, or embedded in a homogeneous or heterogeneous matrix by wall material or encapsulating agent. The choice of wall material is one of the main concerns for microencapsulation process. Common microencapsulation agent such as whey protein isolate (WPI) and maltodextrin are often used for herbal or plant-related product. Betz et al. (2012) for instance used WPI for microencapsulation of phenolic compounds from bilberry extract, while Şhahin-Nadeem et al. (2013) used maltodextrin for microencapsulation of

* Corresponding author. Faculty of Chemical & Natural Resources Engineering, Universiti Malaysia Pahang, 26300 Gambang, Pahang, Malaysia. Tel.: +60 95492899; fax: +60 95492889.
E-mail addresses: jolius@ump.edu.my, j.gimbun@gmail.com (J. Gimbun).

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phenolic compounds from herbal tea (*Salvia fruticosa*). Many studies related to microencapsulation of fruit and plant extracts using different wall materials have been reported (e.g. Krishnaiah, Sarbatly, & Nithyanandam, 2012; Krishnan, Kshirsagar, & Singhal, 2005), but none on *O. stamineus*. Therefore, this work aims to minimise the degradation of bioactive compounds from *O. stamineus* extract during spray drying using microencapsulation technique using encapsulating agents such as whey protein isolate and maltodextrin.

2. Materials and methods

2.1. Chemicals and plant material

The HPLC grade solvents such as the acetonitrile (ACN) and methanol were purchased from Merck (Darmstadt, Germany) and trifluoroacetic acid (TFA) were purchased from Fisher Scientific (Leicestershire, UK). The dimethyl sulfoxide (DMSO), standard of rosmarinic acid, eupatorin and sinensetin were obtained from Sigma Aldrich (St. Louis, MO). The lactose-free whey protein isolate powder was obtained from Ultimate Nutrition (Fleetwood, Lancashire, UK) with 99% of undenatured proteins meanwhile Maltodextrin DE10 was purchased from San Soon Seng Food Industries (Malaysia). Leaves were collected in Gambang, Pahang, Malaysia from a white flowered *O. stamineus* similar to one that has been deposited at the Forest Research Institute, Malaysia (voucher no. ZAS1113). Freshly collected leaves were washed with deionised water and dried at 37 °C for 3 days before crushed to powder. Drying the leaves at 37 °C will not significantly affect the phenolic content in the leaves since most degradation occurs at temperature above 60 °C. Drying is unavoidable because the exact mass of the plant (dry weight) used for each run of the experiment to ensure similar amount of plant material used throughout this work. Prior to use the powder was kept in an air-tight plastic bag in a desiccator at room temperature to prevent moisture absorption.

2.2. Ultrasonic assisted extraction

O. stamineus extracts were prepared using ultrasonic-assisted extraction at 50 °C for 90 min at 45 kHz. The extraction time of 90 min was chosen from the initial study on the effect time to the concentration of phenolic compounds, which indicate 90 min as optimum extraction time (Pang, 2013). Total of 8 g of powdered *O. stamineus* leaves was added to 100 ml of 50% methanol (8 wt.%). The aqueous 50% methanol enabled a simultaneous extraction of both hydrophilic (hydroxylated, i.e. rosmarinic acid) and lipophilic (methoxylated, i.e. sinensetin and eupatorin) phenolic compounds (Pang, 2013). The supernatant was then separated from the residue by filtration using Whatman no. 1 filter paper. Extract was concentrated by evaporating out excessive methanol from the extract at 40 °C in vacuo. The total solid content for each sample was determined by evaporating the liquid from 5 ml solution completely in an oven.

2.3. Microencapsulation by spray drying

The extracts were encapsulated by two types of wall material which are whey protein isolate and maltodextrin with a dextrose equivalent of 10. Initially, 80 ml of stock encapsulation agent solution containing 0.1 wt. %, 1 wt.%, 10 wt.% and 20 wt.% of either WPI or maltodextrin were prepared. A 50 ml *O. stamineus* extract (1.05 wt.% solid content) was added to the stock encapsulation agent solution and further diluted with ultrapure water to make the total volume of 150 ml. Subsequently, the solution was mixed by magnetic stirring at 40 °C for 30 min to obtain a homogeneous solution. The feed concentration contain 0.05 wt.%, 0.53 wt.%, 5.33 wt.% and 10.67 wt.% of either WPI or maltodextrin. The resultant solution was spray dried using a lab scale spry dryer (Lab Plant SD06A, UK) fitted with 0.5 mm atomizer and air velocity of about 4.1 m/s was set constant throughout the experiment. The inlet air temperature was set at 180 °C and maintained at ±1 °C by the proportional–integral–derivative controller. Feed was metred into the dryer by means of a peristaltic pump at 407.1 ml/h. Similar setups were employed for all experiments to ensure a fair comparison. Dried powder samples were collected from a Schott bottle attached at the bottom of the cyclone separator.

2.4. Analysis of polyphenols content

The total solid content from *O. stamineus* extract was determined by evaporating the liquid from 5 ml solution completely in an oven. Moisture content for all dried powder samples is determined using a moisture analyser, and the water content is subtracted during preparation of solution for UPLC analysis of polyphenol after spray drying. The same dry weight of solid (bioactive compounds) is set for the initial solution (extract) and after drying the solution to ensure a fair comparison of polyphenol retention. The predetermined amount of dried powder was dissolved in 60% aqueous methanol with aid of vortex mixer to ensure dissolution of less polar compounds. The stock solution of rosmarinic acid (10 mg/ml) was prepared in methanol, whereas eupatorin (10 mg/ml) and sinensetin (5 mg/ml) were dissolved in DMSO. The three analytical standards were further diluted until 0.08 μg/ml to develop an eight points calibration curve. Qualitative and quantitative determinations of *O. stamineus* extract major constituents (rosmarinic acid, sinensetin and eupatorin) were performed on a Waters Acquity UPLC H-Class (Milford, MA) fitted with Acquity UPLC HSS T3 column (2.1 × 75 mm, 1.8 μm) and an Acquity UPLC HSS T3 VanGuard column guard (2.1 × 5 mm, 1.8 μm). The UPLC system is equipped with photodiode array detector and connected to a computer running Waters Empower 2 software. The mobile phase consists of solvent A:water:TFA (20:0.001; v/v) and solvent B: ACN:TFA (20:0.001; v/v) and the following gradient elution: 0–2.0 min, 26% B; 2.0–3.9 min, 26–50% B; 3.9–6.9 min, 50–95% B and finally washing the column with 95% B for 0.6 min and reconditioning the column with 26% B isocratic for 1.4 min. The temperature was maintained at room temperature (24 °C), with injection volume of 2 μl and flow rate at 0.17 ml/min. The sample was filtered with 0.2 μm PES membrane filter before injected to the UPLC system. The peaks for rosmarinic acid (3.10–3.30 min), sinensetin (5.50–5.60 min) and eupatorin (5.65–5.75 min) were detected at 340 nm.

2.5. Viscosity determination

The viscosities of the prepared samples were determined at 20 °C with the aid of a viscometer (DV-III Ultra, Brookfield, USA) fitted with spindle SC4-18. Samples were placed in the measurement cell of the viscometer and allowed to equilibrate at 20 °C. The viscosity of the sample was measured at shear rate 26.4 s^{-1}. Viscosity readings were taken after subjecting the sample to shear for 1 min. The viscosity (η) was obtained in terms of millipascal-second (mPa s).

2.6. Particle size distribution

The particle size distribution of spray dry powder was measured using laser diffraction particle size analyzer (Malvern 2000 mastersizer, Malvern Instruments Co., Worcestershire, UK) equipped with an automated dry powder dispersion unit (Scirocco 2000). The