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# Spray drying of orange peel extracts: Yield, total phenolic content, and economic evaluation

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#### 1. Introduction

The consumption of antioxidants, which are abundant in fruits and vegetables, has been suggested because of their benefits for health (Liu, 2003). Antioxidants, such as vitamin C, vitamin E, carotenoids, and phenolic compounds, have the ability to scavenge reactive oxygen and nitrogen species and prevent oxidative damage to important biological macromolecules, such as DNA, lipids, and proteins (Carr and Frei, 1999). In the food industry, antioxidants are used to maintain nutritional quality and avoid undesirable changes in the color, flavor, and texture (Finley and Given, 1986). Several synthetic antioxidants, such as butylated hydroxytoluene (BHT) and butylated hydroxyanisole (BHA), are commonly used as food additives (Ajila et al., 2007). However, these antioxidants have been reported to be potentially toxic and carcinogenic (Ito et al., 1997). On the other hand, natural antioxidants have been found to be safer than many synthetic ones and to provide additional nutrition value (Ajila et al., 2007). This has led to increasing development of natural antioxidants, especially from fruits and vegetables.

Citrus fruits have been explored as one of the sources for natural antioxidants (Bocco et al., 1998). Orange, as one of the main citrus fruits grown in Australia, had a total production amount of 507,232 tonnes in 2008, which was 78% of the total produced citrus fruits in Australia (Keogh et al., 2010). Peel and seed are the major by-products of orange fruit processing, which are sometimes

#### ABSTRACT

Orange peel extract powders were produced using two steps: microwave-assisted extraction and spray drying. The extraction solvent-to-solid ratio has been found to significantly affect the level of total phenolic compounds in the extract and powder. Under the outlet air temperatures between 43 and 79 °C, spray drying of orange peel extracts resulted in a peak yield trend (between 75% and 92%) and high TPC (total phenolic content) recoveries. An evaluation of economics has also been conducted, suggesting that this process is economically feasible, with profits of 6.1 USD/kg and 8.8 USD/kg for solvent to solid ratios of 2 and 14, respectively. The uncertainty analysis of the economics showed that the selling price, the labor cost and the orange peel cost are the three most important parameters compared with the costs of electricity, natural gas, and water.

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used as animal feed and often simply sent to waste. This agricultural waste may cause major environmental problems due to high associated chemical and biological oxygen demand (COD and BOD) (Lin et al., 2013). There is also a potential problem with citrus extract in waste water treatment, which can kill composting microorganisms. Therefore, the utilization of orange peel to produce valuable products is expected to lower its environmental impacts. Bocco et al. (1998) have reported that orange peel extract contains phenolic compounds, such as phenolic acids and flavonoids, so the extract has significant antioxidant activity.

Drying is a method that can be used to reduce the moisture content of food products and prolong their shelf-life. Compared with liquid extracts, the production of dried extracts could result in lower storage and transportation costs, and also better storage stability (Fang and Bhandari, 2011). Spray drying is a commonly-used method to convert feed from a liquid state into a powder form (Masters, 1979). In the spray-drying process, the moisture content of the feed is removed very quickly. This process usually occurs with the temperature of the product being much lower than 100 °C, so it may be suitable for the drying of heat-sensitive materials, such as phenolic compounds in orange peel extracts.

Several studies have been conducted in the area of spray drying for natural extracts. Ersus and Yurdagel (2007), for example, found that higher air inlet temperatures resulted in higher anthocyanin losses, due to degradation, during the spray drying of black carrot extracts. Another study was carried out by Fang and Bhandari (2011) on the spray drying of bayberry juices. They found good retention of total phenolics and anthocyanins content after spray drying, which were 96% and 94%, respectively. Kha et al. (2010)







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studied spray drying of Gac fruit aril extract and found a significant decrease in the total carotenoid content as the spray-drying temperature increased from 120 °C to 200 °C. This behavior may occur due to thermal degradation and oxidation. However, the economic evaluation of the production of phenolic powder, by considering its properties, has not been explored yet.

The objective of this study was to evaluate the effect of different drying conditions on the total phenolic content recovery and the yield from spray drying the orange peel extract. Moreover, the effect of extraction solvent to solid ratio on the total phenolic content of spray-dried powder was assessed. An evaluation of economics was also performed to analyse the feasibility of these processes.

#### 2. Materials and methods

#### 2.1. Chemicals

Gallic acid (G7384), sodium carbonate (S2127), and Folin Ciocalteu's reagent (FCR) (F9252) used in this study were obtained from Sigma Aldrich.

#### 2.2. Extraction of phenolics from orange peels

Navel oranges were obtained from the local supermarket (Coles Broadway, Sydney). The sample fruits were washed with tap water and peeled using gloves. The peels were then cut into small sizes (1–3 mm) and stored in a refrigerator for the experiments.

The prepared orange peel samples, together with deionized water, were added to the microwave vessel. The solvent to solid ratios were adjusted to 2 and 14, with the total amount of solvent and peels used for each solvent to solid ratio being kept constant (15 g) to provide the same heat load per unit mass during extraction. The temperature of the system was increased to 135 °C for 7 min. The extraction was then carried out using a microwave system (ETHOS SEL Microwave Solvent Extraction Labstation) at constant temperature (135 °C) for 3 min. The extraction temperature was chosen to be high enough to yield an extract with high antioxidant activity, but not too high to reduce the possibility of Maillard reactions occurring during the extraction (Ahmad and Langrish, 2012). After the extraction was completed, the microwave vessel was cooled down to the ambient temperature. Solid residue was then separated from the liquid extract using filter paper, and the liquid extract was stored in a cool place for the determination of the total phenolics content.

#### 2.3. Determination of total phenolics content (TPC)

The TPC in the extracts was assessed according to the method described by Singleton et al. (1999). The sample was prepared by mixing 0.1 mL of orange peel extract with 7.9 mL of deionized water and 0.5 mL of FCR. The mixture was left in the dark for 5–8 min, before 1.5 mL of 20% sodium carbonate solution was added to the sample. The mixture was then left again in the dark for 2 h. The absorbance value of the sample was then read using a UV/VIS Spectrophotometer (Cary 50, Varian, USA) at a wavelength of 765 nm. Gallic acid was used to produce a standard calibration curve. The TPC was expressed as mg of gallic acid equivalents per g of dry matter (mg GAE/g DM).

#### 2.4. Spray drying

The orange peel extract was dried using a Buchi B-290 mini spray dryer with a high performance cyclone. The operating conditions were: drying air inlet temperature varying from 100 to 200 °C, atomization air flow rate of 50 mm (1052 L/h), liquid feed

pump rate of 4 mL/min (10%), and aspirator rate of  $38 \text{ m}^3/\text{h}$  (100%). All the inlet and outlet air temperatures for each run are shown in Table 1. The yield (or recovery) from spray drying has been calculated using Eq. (1).

Yield (%) = 
$$\frac{\text{mass of solid in the collecting vessel}}{\text{mass of solid in the feed}} \times 100\%$$
 (1)

#### 2.5. Statistics

Data in this study were obtained from two replicates for each experiment and are presented as means  $\pm$  standard deviation. For statistical analysis, differences were tested for significance by using the ANOVA method, using a significance level of  $P \leq 0.05$ .

#### 2.6. Evaluation of the economics

The evaluation of the economics for the process was carried out using data from the extraction and spray-drying experiments, as explained in the previous sections. The required experimental data include the yield from spray drying, the total phenolic content and moisture content of the powder, the optimum extraction time, the mass fraction of solvent (water) remaining in the residue, and the humidity of the ambient air. The manufacturing cost of orange peel extract powder was calculated using the approach proposed by **Turton et al.** (2009), as shown in Eq. (2). The cost of manufacturing (COM) is a function of the fixed capital cost for investment (FCI), the cost of operational labor (COL), the cost of utilities (CUT), the cost of water treatment (CWT), and the cost of raw material (CRW).

$$COM (USD/year) = 0.280 \times FCI + 2.37 \times COL + 1.23 \times (CUT + CWT + CRM)$$
(2)

The FCI was calculated by predicting the purchased equipment costs using the cost correlation method (Towler and Sinnott, 2013; Turton et al., 2009). The flowsheet used to calculate the FCI in this study consists of three 100 L microwave-assisted extractors with integrated mesh filters (Li et al., 2012), one solvent reservoir tank, two centrifugal pumps, one compressor, one indiret heater, and one spray dryer. The process flow diagram is shown in Fig. 1.

It was assumed that the industrial-scale unit would work with the same performance as the lab-scale unit at the same solvent to solid ratio, temperature, pressure, and extraction time (Rosa and Meireles, 2005). The plant was assumed to run 24 h per day, 330 days per year, resulting in 7920 h per year. The efficiency of the pumps, the fan, and the indirect heater were assumed to be 75%, 70%, and 85%, respectively (Baker and McKenzie, 2005; Turton et al., 2009).

Turton et al. (2009) proposed a method to calculate the number of operators required per shift ( $N_{OI}$ ), which is shown in Eq. (3):

Table 1

The inlet and outlet air temperatures from the spray drying experiments at atomization air flow rate of 1052 L/h, liquid feed pump rate of 4 mL/min, and aspirator rate of 38 m<sup>3</sup>/h.

Run	Inlet temperature (°C)	Outlet temperature (°C)
1	100	43
2	100	43
3	125	52
4	125	50
5	150	66
6	150	64
7	175	75
8	175	73
9	200	79
10	200	79

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