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The use of a plug-flow model for scaling-up of spray drying bioactive orange peel extracts

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ABSTRACT

The spray drying of orange peel extracts was scaled up from a laboratory-scale spray dryer to a pilot-scale spray dryer using two methods to predict the parameters of the spray-drying process. The mass and energy balance model predicted the outlet gas temperature, absolute humidity and the final moisture content of the particles with 5%, 2% and 74% errors, respectively whereas, the plug-flow model successfully predicted these parameters with <1.5%, 1% and 15% error, respectively. The total phenolic content and the antioxidant capacity of the powders were retained to the extent of 88 ± 7% and 95 ± 8%, respectively, after the scale-up process. Therefore the plug-flow model was found to be a useful method in the scale-up process as a rapid estimation method for predicting the key parameters in spray drying with good accuracy, in order to keep the quality of the products within the required range.

Industrial relevance: Peels and seeds of citrus fruits, such as oranges, contain bioactive phenolic compounds that have demonstrated cancer-inhibition properties. However, due to their bitterness, they are not consumed and are considered to be waste material. The compounds can be extracted and turned into concentrated powder supplements. In powder form, these compounds can be easily incorporated in controlled and concentrated dosages into food formulae in order to create functional foods while masking the unpleasant taste. However, due to the heat sensitivity of these compounds, the minimum amount of heat treatment is required in the process. Spray drying, which is one of the fastest drying techniques available, can be used to convert these extracts into powders. However, spray drying these extracts is very challenging due to the presence of large amounts of sugars that cause stickiness and product loss during the process. In general, adding significant amounts of carriers to sugar-rich foods to overcome their stickiness during spray drying has been used in industry, compromising the purity of the final products. Moreover, in order to mass produce the powders, the process needs to be scaled-up. However, spray drying is extremely difficult to scale up using dimensional analysis (Oakley, 1994; Zlokarnik, 2003b). Computational fluid dynamic (CFD) simulations have been suggested as a powerful tool in this scale up of spray drying, but they are very time consuming. Therefore, if simpler models that do not require CFD simulations can adequately predict the process parameters, they can be used as fast estimation techniques for scale up. The results from this study guide the use of a plug-flow model for scaling up the process of spray drying orange-peel extracts while keeping an acceptable quality for the final product.

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1. Introduction

Phenolic bioactive compounds are abundant in the peels and seeds of fruits and vegetables, which can be extracted and transformed into nutraceutical compounds for consumption. Since the concentrated powders have many advantages, such as ease of handling, transportation, storage and longer shelf life under proper packaging conditions (Main, Clydesdale, & Francis, 1978), microencapsulation by spray drying has been one of the techniques used by many researchers to produce powders from these extracts at the laboratory scale. In many studies, significant quantities of carriers and encapsulating agents have been used, decreasing the final product purity. Moreover, the scaling up of the process and any potentially undesirable effects on the quality of the extract powders during the scale-up process have not been studied yet.

Many processes in which a chemical or microbiological conversion is taking place along with heat, mass or momentum transfer are scale-dependent (Zlokarnik, 2003a). Spray drying is a thermal unit operation where simultaneous heat and mass transfer occurs, and the complexity of the process makes it very difficult to scale up using dimensional analysis. The particle and droplet motion inside a spray-drying chamber depends on the flow patterns of air inside the chamber. The difficulty and complexity of modeling the gas and flow patterns (Oakley, 1994) and the non-linearity of the physical processes (Kerkhof, 1994) makes the

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direct scale-up of spray drying challenging. Several essential physical properties of the system change simultaneously, due to the continuous changes of drying conditions during the spray-drying process (Kerkhof, 1994; Zlokarnik, 2003b). Therefore, spray dryers have been designed and built based on rules derived from experience with existing plants for many years (Masters, 1994; Oakley, 1994; Zlokarnik, 2003b). Computational fluid dynamic (CFD) simulations have been suggested as a powerful tool in spray-dryer design. CFD simulations and predictions of flow patterns in spray dryers have been studied previously by many researchers (Fletcher et al., 2006; Fletcher & Langrish, 2009; Langrish, Williams, & Fletcher, 2004). However, the CFD simulations are very time consuming and require very large computational resources (Fletcher & Langrish, 2009).

On the other hand, simpler models have been used to study the spray-drying process, such as plug-flow ones. Plug-flow models have been used by many researchers (Keey & Pham, 1976; Langrish, 2009b; Truong, Bhandari, & Howes, 2005; Zbicinski, Grabowski, Strumillo, Kiraly, & Krzanowski, 1988). In this approach, the drying chamber is divided into small regions along the vertical axis of the drying chamber (basically one-dimensional modeling). This model generates a system of eight "non-linear first-order ordinary differential equations as a function of the axial distance of the drying chamber" (Truong et al., 2005). This model generally considers the overall process to be running at steady state, although the drying kinetics of the particles are clearly unsteady state. Often, a characteristic drying curve has been used to represent the drying kinetics of the particles (Huang, Kurmar, & Mujumdar, 2004; Langrish & Kockel, 2001; Zbiciński & Li, 2006). The plug-flow model considers the particles and gas to be flowing parallel to each other, and it is mostly suitable for tall-form dryers (Keey & Pham, 1976; Langrish, 2009b). When Truong et al. (2005) validated the predictions of the plug-flow model with experimental work on a short form dryer consisting of a cylinder on top of a cone, they noticed that the predicted air and wall temperatures were the same in the conical part of the drying chamber and only 2–4 °C lower than the measured values in the cylindrical part of the dryer. The moisture-content predictions were 37% different to those found experimentally (Truong et al., 2005). Truong used a rotary atomizer and assumed that the distribution of droplet sizes followed a log-normal distribution function. Ali et al. (2014) also used this model to predict the gas temperature and moisture content of particles in a counter-current spray dryer, and their predicted results for gas temperature were also in good agreement with the experimental measurements (1% error). However, the observed moisture content was predicted within 57% due to simplifying assumptions that were used in the drying kinetics models (Ali et al., 2014). Ali (2014) also mentioned that the plug-flow model approach produced similar trends in results to the more advanced CFD models in terms of the final gas temperature and moisture content of the particles. Although the plug-flow model is not able to capture the complex interaction between the particles and the gas flow and to predict the wall-deposition pattern of particles, it is much simpler than advanced CFD models and can be used as a rapid estimation for the performance of a spray dryer (Ali et al., 2014). However, this approach has not been reported very often for scale-up purposes.

Therefore, the aim of this study was to scale-up the spray drying of bioactive orange-peel extracts using both the simple heat and mass-balance approach and the plug-flow model approach using a characteristic drying curve. The study has also aimed to compare the accuracy of these models with experimental data. Also, the antioxidant activity and therefore the bioactivity of the orange-peel extracts have been studied because these parameters may be affected by scale-up.

2. Materials and methods

2.1. Materials

Fresh oranges were purchased from a local supermarket, and their peels were used for Soxhlet extraction. Pure whey protein isolate (WPI), ultra-filtered ion exchanged and unflavored, from Vitaco Health Australia Pty Ltd was used. The rest of the chemicals used in the experiments were purchased from Sigma-Aldrich; Gallic acid 97.5–102.5% (titration), sodium carbonate *ReagentPlus*®, ≥99.5%, Folin Ciocalteu's reagent (FCR) and 2, 2-Diphenyl-1-picrylhydrazyl (DPPH).

2.2. Sample preparation and extraction

Washed oranges were wiped to dry and then peeled. Then by using a Homemaker-brand food processor, the peels and white skins were chopped into small pieces (<1 mm). Then a 30 × 100 mm–1.5 mm thickness Whatman cellulose extraction thimble was filled with 25 g of the chopped peels. The thimble was placed in a conventional Soxhlet extraction apparatus, and the extraction was performed for 4 h using de-ionized water as solvent. A 9:1 solvent to solid ratio was used for extraction experiments. The extracts were then used for spray drying after 18 h refrigeration.

2.2.1. Spray drying

A Buchi-B290 spray dryer (Buchi, Switzerland) at a main air flow rate of approximately 38 m^3/h, and an atomizing air flow rate of 536 NL/h was used for the small-scale spray drying experiments. For scale-up purposes, the experiments were carried out in a pilot-scale spray dryer system in the School of Chemical and Biomolecular Engineering at the University of Sydney fitted with a Buchi two-fluid nozzle. The pilot-scale spray dryer that has been used here is a modified Niro short-form dryer with an internal diameter of 0.8 m and a height of 2 m, consisting of an upper cylindrical part, 1.3 m in height and a lower conical bottom, 0.63 m in height (Langrish, Chan, & Kota, 2007). The drying airflow rate and drying air temperatures were adjusted according to the results from small-scale trials and the heat and mass balance calculations. The powders were placed in air-tight plastic bags and were stored at −18 °C for further analysis.

2.3. Powder characterization

2.3.1. Yield of spray drying process

The yield of the spray-drying process was calculated by dividing the amount of the powders collected as the product by the amount of the total solids in the feed (reported as a percentage). When calculating the yield for the pilot-scale spray dryer, the powders collected both in the collection vessel of the cyclone and at the bottom of the spray dryer were considered as the product in the calculations.

2.3.2. Moisture content

About 2 g of powders were placed on a Petri dish and were oven-dried at 85 °C for 24 h using a fan-forced drying oven (Islam, Edrisi, & Langrish, 2013).

2.3.3. Particle size measurements

The particle size of the powders were measured using a laser diffraction instrument (a Malvern Mastersizer 3000, Malvern Instruments, UK) with a dry powder feeder unit. Three measurements have been carried out, and the average mean particle size has been reported.

2.3.4. Morphology of the particles

The morphology of the powders was studied by a Zeiss EVO 50 scanning electron microscope (Carl Zeiss, Germany) fitted with a LaB6 filament. A small amount of the powders was placed on a carbon tape mounted on an aluminum stab and was coated with gold to achieve a standard thickness of 25 nm. The operating voltage of the instrument was set to be 10 kV for all the samples.

2.3.5. Total phenolics content and antioxidant activity

The method described by Singleton et al. (1999) was used to assess the total phenolic content (TPC) of the extracts. The powders were first

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