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Study of the supercritical drying of wet Okara

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Abstract: This study investigates the drying of okara (wet soybean pulp) using supercritical carbon dioxide at a low temperature of 40 deg C. The okara used in this work is obtained directly from a soy bean milk processing factory in Singapore and the samples were dried using supercritical carbon dioxide at supercritical pressure of 103 bar and temperature of 40 deg C, with a CO₂ flowrate of 20ml/min. The drying time was varied from 10 min to 60 min to investigate the drying kinetics and water extraction efficiency of supercritical carbon dioxide at the conditions used. The drying tests were carried for low sample mass loading (<500mg) and high sample mass loading (>1000mg) to determine the effect of sample loading on the drying. The results show that the water uptake efficiency of supercritical CO₂ is higher at high mass loading (36.9%) as compared to a lower efficiency at low mass loading (18.6%). This suggests that a packed bed configuration where the extraction vessel is filled with wet okara will be favourable for the extraction. A pilot scale supercritical drying unit is proposed using the experimental results obtained in this study and calculations for pressure drop using Ergun’s equation showed that the pressure drop a 4m tall extraction column will be approximately 7 bar. The low pressure drop is attributed to the liquid-like density (638 kg/m³) and gas-like low viscosity (0.049 mPa.s) of carbon dioxide at supercritical conditions. This study demonstrates the feasibility of drying high moisture particulates such as okara using supercritical carbon dioxide even at low temperatures.

Keywords: Supercritical CO₂, Okara drying, Sustainable process, Packed Bed
1. INTRODUCTION

In Singapore, soy products in the form of soy milk, soy bean curd and vegetarian food are an important part of our people’s diet and protein intake. The local soy processing industry produces large amounts of okara (soy bean pulp) daily. Okara contains more than 80% moisture (wet basis) and can become a useful source of protein for animal feed. However, the high moisture content in the okara produced poses a great problem in the food safety as it deteriorates easily (Aguirre et al., 1981), especially in Singapore’s humid climate. One of the most common treatment of okara is drying to at least 8% moisture (wet basis) before transportation and processing into animal feed. Many studies of drying of okara has been carried out using conventional methods such as hot air and superheated steam impinging steam dryer (Choicharoean et al., 2011), pneumatic flash drying (Grizotto et al., 2011), direct rotary dryer (Luz et al., 2010), jet spouted dryer (Wachiraphansakul et al., 2005) and even using electrohydrodynamic (EHD) drying (Li et al., 2006).

Supercritical fluid processing has been applied to several applications in the pharmaceutical (Lee et al., 2008) (Lee et al., 2009) and food industry (Weidner, 2009). Carbon dioxide ($\text{CO}_2$) is the preferred supercritical fluid used for such applications. This is due to the favourable properties of $\text{CO}_2$ being non-toxic, non-flammable and having abundant supply. $\text{CO}_2$ has a critical temperature and pressure of 31.1 deg C and 73.8 bar respectively. It is also known to inactivate microbes and viruses (Perrut 2012), which makes it an ideal solvent in the food processing industry.

Under supercritical conditions, $\text{CO}_2$ also has an increased affinity for water (Sabirzyanov et al., 2012). This makes it an ideal candidate for drying aqueous solutions and wet samples which otherwise cannot be dried using conventional drying techniques due to heat sensitivity. Supercritical $\text{CO}_2$ drying can be applied to produce dried fruit, vegetables, herbs and spices as an alternative to freeze drying (Khalloudi et al., 2010). Supercritical $\text{CO}_2$ drying offers low temperature water extraction operations at energy efficient levels. Previous studies of supercritical $\text{CO}_2$ processing of okara focus only on the extraction and recovery of oil components in okara (Quitain et al., 2006). In this work, the use of supercritical $\text{CO}_2$ to dry okara is demonstrated and a preliminary scale-up configuration for the supercritical drying plant for okara and similar wet food residue product is proposed.

2. MATERIALS AND METHODS

2.1. Materials

The Okara (Soybean pulp) was a generous contribution from F&N Foods Pte Ltd. The okara samples collected were stored in small freezer bags in a -23 deg C freezer within 1 hour of collection. Before each experiment, the frozen samples will be removed and thawed at room temperature before analysis and drying. Compressed carbon dioxide cylinder was purchased from Singapore Oxygen Air Liquide Limited (SOXAL). Refrigerating liquid (Thermal G) was purchased from JULABO GmbH.

2.2. Measurement of moisture content

The free moisture content (wet basis) of the okara samples before and after drying were determined by a mass balance of the samples before and after undergoing a drying cycle of 48 hours in a bench-top freeze dryer to remove almost all moisture in the samples. The moisture content of wet okara samples were found to be consistently in the range of 79 – 84% moisture (wet basis).

2.3. Morphology analysis of samples

The morphology dried okara samples used in this study was analysed using a Scanning electron microscope (SEM, JEOL JSM-5600LV). The samples were coated with platinum (Autofine Coater, JEOL JFC-1300) prior to analysis.

2.4. Supercritical Carbon Dioxide Drying

A schematic of the experimental setup used in supercritical drying process is shown in Figure 1. Wet okara samples were weighed and placed in a filter bag before transferring to the high pressure extraction vessel (V1). Compressed $\text{CO}_2$ from cylinder (C1) was first liquefied by heat exchange with cooling liquid (Thermal G) at -20 deg C from a refrigerated circulator (E1). The liquefied $\text{CO}_2$ was then delivered to the high pressure extraction vessel (V1) using a high pressure liquid pump (P1, reciprocating pump, Eldex BBB-4-2). The temperature of the extraction vessel (V1) was controlled and maintained using a water bath heater (E2) at 40 deg C. The pressure in the extraction vessel was controlled via an automatic back pressure regulator (P2). The supercritical $\text{CO}_2$ with moisture is then vented off to a fume cupboard.
3. RESULTS AND DISCUSSION
3.1. Drying kinetics of okara using supercritical CO2

SEM analysis of the surface morphology of freeze dried okara is shown in Figure 2. The sizes of the samples ranged between 200 – 500 µm and a closer look into the surface morphology shows a thin corrugated structure on the sample surface.

In this work, the supercritical CO2 drying of wet okara was carried at 103 bar and 40 deg C with a CO2 flowrate of 20ml/min at pump P1. 2 sets of experiments were carried out to evaluate the effects of a high mass loading (>1000mg) of okara and low mass loading (<500mg) of okara on the drying efficiency and kinetics of the supercritical CO2 drying process.

The solubility of water in supercritical CO2 at 103 bar and 40 deg C is 0.00414 moles water/ moles CO2 (Sabirzyanov et al., 2011) which is equivalent to 1.693mg water/ g CO2 as shown in Figure 3. The experimental result for water extraction at high sample loading (> 1000mg wet okara) and low sample loading (<500mg wet okara) is shown in Figure 3. The extraction of water from okara in higher mass loading experimental runs is approximately 0.6251mg water /g CO2, yielding a water uptake efficiency of 36.92%. Similarly, the extraction of water from okara in the lower mass loading experimental runs is approximately 0.3155mg water / g CO2, giving a water uptake efficiency of 18.63%. Possible reason for the low water uptake efficiency observed is that the wet samples were loosely packed into the extraction vessel and only a fraction of supercritical CO2 introduced into the extraction vessel actually passes through the matrix of the wet sample.
The drying kinetics of the okara was studied by determination of the rate of drying vs. the drying time. In Figure 4, the normalized moisture content left in the okara matrix vs drying time is shown for experimental runs carried out at low and high okara mass loadings in the extraction vessel. Interestingly, at higher moisture content, it was observed that the reduction in moisture content follows a linear relationship with drying time which corresponds to a period of constant rate drying. For the low sample loading experiments, it was observed that as the remaining moisture content approaches 30%, the rate of drying seemed to decrease with time, corresponding to a period of falling rate drying.

### 3.2. Possible scale-up of drying unit using supercritical CO2

Our preliminary results show that the amount of wet okara loaded into the extraction vessel has an influence on the water extraction efficiency of CO2. The water extraction efficiency of water by supercritical CO2 will likely be improved if the okara is loaded into the extraction vessel in the form of a packed bed. Packed beds are usually associated with problems of uneven fluid distribution and also high pressure drop. However, supercritical CO2, having a gas-like viscosity and liquid like density, is likely to overcome these obstacles. Based on an average production of 3 tonne of okara per day in a typical soy-milk production facility in Singapore, the parameters summarized in Table 1 were used to calculate the pressure drop, supercritical CO2 flow rate and volume of the extraction equipment. A simplified flow diagram of a scale-up supercritical drying unit is shown in Figure 5.
Based on the experimental results of water extraction at 0.6251 mg water/g CO\(_2\), the amount of CO\(_2\) required to process 3 tonnes of wet okara (81% moisture, wet basis) to dry okara (8% moisture, wet basis) daily will be 3808 tonnes. However, with a CO\(_2\) regeneration system in place, the actual amount of CO\(_2\) required daily will be much lesser since the CO\(_2\) will be recycled back to the extraction unit.

Ergun’s equation (equation 1) for calculations of pressure drop in flow through a packed bed (Ergun, 1952) was used to calculate the difference pressure drop using different CO\(_2\) flow rate. The assumptions for the pressure drop calculations are: (1) 6 water extraction vessels operating in parallel; (2) Height to diameter ratio of 5:1 for the extraction vessels; (3) 22-hour steady-state continuous drying operation daily; (4) Complete removal of water from CO\(_2\) after extraction using the scCO\(_2\) regeneration unit.

\[
\frac{-\Delta P}{L} = 150 \frac{(1-\varepsilon)^2}{\varepsilon^3} \frac{\mu}{\rho} + 1.75 \frac{(1-\varepsilon) \rho \mu^2}{\varepsilon^3} \frac{1}{\rho D}
\]

Where:
- \(-\Delta P/L\) = pressure drop per unit length of the bed (Pa/m)
- \(\rho\) = density of supercritical CO\(_2\) (kg/m\(^3\))
- \(\mu\) = viscosity of supercritical CO\(_2\) (Pa.s)
- \(\varepsilon\) = porosity of okara in the packed bed
- \(\psi\) = sphericity of okara
- \(D\) = average diameter of okara particles (m)

Based on the assumptions listed, the inner diameter and height of extraction vessels required is 0.778 m and 3.891 m respectively. Flowrate of CO\(_2\) through the extraction vessel will be 48.1 kg/s (0.0754 m\(^3\)/s). Using equation 1, the pressure drop across the bed can be calculated as 1.68 bar/m and the pressure drop across each vessel will be 6.55 bar. It should be noted that the pressure drop across the bed estimated in this work is only 6.36% of...
the operating pressure and the actual pressure drop is expected to be lower as the volumetric flow rate of CO$_2$ was estimated at a lower value of water extraction efficiency using the experimental results.

4. CONCLUSIONS AND FUTURE WORK

The removal of water from high-moisture containing particulates using supercritical CO$_2$ drying process was demonstrated in this work using wet okara obtained from the soymilk manufacturing industry. The scale-up calculations indicate that the pressure across the packed bed is reasonable low due to the very low viscosity of supercritical CO$_2$. This drying process can be applied to many other useful food residues, fruits, vegetables which require drying at low temperatures. The amount of water extraction per unit mass of CO$_2$ from the wet matrix of okara and other food by-products can be improved by increasing the temperature and pressure of the supercritical fluid. Further experimental studies will be carried out to determine to optimal conditions for drying of okara and other similar food residues using the experimental setup illustrated in this work.

To improve the cost and energy efficiency of a scale-up development unit for supercritical CO$_2$ drying, Different CO$_2$ regeneration options are available. Typical supercritical drying process uses a zeolite packed bed to remove water from water-laden supercritical CO$_2$ for recycle back into the extraction vessel (Almeida-Rivera). Other options include the use of a flash separator at reduced pressure to separate gaseous CO$_2$ and liquid water. Current research on the comparison of different regeneration options for CO$_2$ in a large-scale supercritical drying plant design is now on-going.

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