



Preparation of inulin and phenols-rich dietary fibre powder from burdock root

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ABSTRACT

The simultaneous ultrasonic/microwave assisted extraction (UMAE) of inulin and production of phenols-rich dietary fibre powder from burdock root was studied. It was found that UMAE required a much shorter extraction time than conventional stirring extraction. And the recovery of inulin increased with the increase of microwave power as well as solvent to solid ratio. A comparison of scanning electron microscopy images of raw and simultaneous ultrasonic/microwave treated burdock root indicated microfractures and disruption of cell walls in burdock root flakes. The dietary fibre powder prepared from the residue of burdock root after inulin extraction was rich in phenols (302.62 mg GAE/100 g powder). The results showed that drying temperature in the selected range did not significantly affect the hydration properties. The relatively high water retention capacity, swelling capacity and oil-holding capacity of the dietary fibre powder indicated its broad use as a modifier of properties of formulated products.

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1. Introduction

Inulin, a non-digestible oligosaccharide, stimulates the growth of bifidobacteria, which is believed to have health-promoting functions. Many other health enhancing aspects of inulin are concerning diabetes, lipid metabolism, and cancer prevention (Flamm, Glinsmann, Kritchvsky, Prosky, & Roberfroid, 2001; Roberfroid, Gibson, & Delzene, 1993). These qualities considered to its ability to improve rheological characteristics and nutritional properties of food allow inulin to be classified among functional foods (Niness, 1999). One source of inulin is burdock, which is a popular vegetable in Japan, has been extensively analysed for its components due to their antioxidant properties (Chow, Wang, & Duh, 1997) as well as for its extractable components given antimicrobial activity (Duh, 1998; Lin, Lin, Yang, Chuang, & Ujji, 1996).

Several works about inulin preparation have been reported, including stirring extraction (Laurenzo et al., 1999) and UAE (Wei et al., 2007). However, most studies focuses on inulin as a product, how to treat a large amount of residue generated by inulin extraction has not been reported. Currently, the health benefits of dietary fibre have led to increased consumption of fibre-rich products. For instance, the demand for by-products from fruits and vegetables as sources of dietary fibre keeps increasing (Fuentes-Alventosa et al., 2009).

Ultrasonic and microwave radiation could accelerate the extracting process and improve bioactive compound extraction (Gergely Csiktusna'di Kissa, Esther Forga'cs, & Tibor Cserha'ti,

2000; Hoang, Sharma, Susanto, Di Maso, & Kwong, 2007; Salisova, Toma, & Masor, 1997; Sueli Rodrigues, Gustavo & Fabiano, 2007). The increasing interest on applying sonochemistry to natural product extraction lies in its advantage on reducing extraction time, saving in energy, increasing yield, etc. Microwave assisted extraction heats the extracts quickly and accelerates the extraction process for adsorption and desorption of the targeted compounds from matrix (Zhang & Liu, 2008). Hence, coupling microwave with ultrasonic extraction is a complementary technique and may present many advantages.

The aim of this study was to investigate the effect of simultaneous ultrasonic/microwave treatment on the extraction of inulin and the microstructure of the burdock root. Also physicochemical properties of dietary fibre powder resulting from extraction residue were evaluated.

2. Materials and methods

2.1. Materials

Burdock root, provided by Xuzhou Wangda Farm and Sideline Products Co., Ltd., was stored in cold storage.

All other chemicals were of analytical grade.

2.2. Stirring extraction technique

Burdock root (5 g), which was previously grounded was mixed with 75 ml water and stirred with an electric mixing paddle in the water bath of 50 °C during the entire extraction process. The extract was filtered through a Whatman No. 1 filter paper and

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the filtrate was concentrated to 30 ml using a rotary evaporator at 60 °C under vacuum. The proteins in the extract were removed using the Sevag reagent (Navarini et al., 1999).

2.3. Simultaneous ultrasonic and microwave assisted extraction of inulin

Simultaneous ultrasonic/microwave assisted extraction (UMAE) experiment was carried out with an ultrasonic and microwave extracting apparatus (CW-2000, Shanghai Xintuo Microwave Instrument Co. Ltd., China). The schematic diagram of UMAE apparatus is shown in Fig. 1.

An open microwave with maximal power of 800 W at a frequency of 2450 MHz, and an ultrasonic transducer with a fixed power of 50 W at a frequency of 40 kHz were used simultaneously to extract inulin from burdock root. Burdock root (5 g), which was previously grounded, was put into a 250 ml Erlenmeyer flask with 75 ml distilled water. Then the flask was moved into the chamber of the apparatus. The extraction process was performed in the chamber with simultaneous different microwave power and ultrasonic power of 50 W. The post-treatment of the extracts was the same as that mentioned in the stirring extraction.

2.4. Preparation and determination of total dietary fibre

The wet fibrous residue of burdock root after inulin extraction was dried in an oven at 60 and 80 °C. Dried fibre was ground in a hammer mill to a particle size lower than 1 mm and stored at 4 °C prior to analysis.

Total dietary fibre content was determined by enzymatic gravimetric method (AOAC, 2000). Triplicate samples were gelatinized with heat stable α -amylase, then digested with protease and amyloglucosidase to remove the protein and starch present in the samples (Al-Farsi & Lee, 2008). Determination of undigested protein and ash contents was carried out for corresponding corrections. Total dietary fibre was calculated as the sum of soluble dietary fibre and insoluble dietary fibre. Total dietary fibre was expressed as milligram per 100 g of burdock root residue on a dry weight basis.

2.5. Determination of inulin content

Total carbohydrate was determined by phenol–sulphuric acid method (Dubois, Gilles, Hamilton, Rebers, & Smith, 1956) using glucose (Sigma) as standard. Reducing sugar was determined by the dinitrosalicylic acid method using D-Fructose (Sigma) as standard (Miller, 1959). The inulin content was measured with the difference between total carbohydrate and reducing sugars (Wei

et al., 2007). The recovery of inulin was expressed as mg per gram of initial burdock root on wet weight basis.

2.6. Determination of total phenols contents

The total phenols contents were determined using Foline–Ciocalteu reagent (Al-Farsi, & Lee, 2008; Yoo, Lee, Park, Lee, & Hwang, 2004). Dry burdock root residue (2 g) was extracted with 100 ml of acetone–water solution (7:3, v/v) for 20 h at room temperature. After filtration, the acetone extracts were kept in the dark at 4 °C until further analysis. One milliliter of extract was mixed with 1 ml of Foline–Ciocalteu's phenol reagent and allowed to react for 5 min. Then, 10 ml of 7% sodium carbonate solution (w/v) were added, and the final volume was made up to 25 ml with deionised water. After 1 h of reaction at ambient temperature, the absorbance at 750 nm was measured by a Spectrophotometer (Unico Instrument Co., Ltd.). Measurements were calibrated to a standard curve of prepared gallic acid solution, and the total phenols concentration was expressed as mg gallic acid equivalent (GAE) per 100 g of sample (dry weight).

2.7. Functional properties

2.7.1. Water retention capacity and oil-holding capacity

Water retention capacity (WRC) was determined by centrifugation as described elsewhere (Jiménez et al., 2000; Robertson et al., 2000) with slight modification. Samples (0.30 g \times 3) were suspended in water (20 ml). After 20 h of equilibration at room temperature, the suspension was centrifuged at 4000g for 20 min. Supernatants were discarded and the hydrated fibres were weighed. WRC was expressed as ml of water/g of dry fibrous residue powder. Oil-holding capacity (OHC) was determined under the same conditions as WRC using soybean oil (0.925 g/ml density), and was expressed as ml oil/g of dry fibrous residue powder.

2.7.2. Swelling capacity (SWC)

For SWC determination, 0.30 g of sample was hydrated in 10 ml of distilled water in a calibrated cylinder (5 cm diameter) at room temperature (about 20 °C). After equilibration for 20 h, the bed volume was recorded and expressed as volume/g of original sample (dry weight).

2.8. Scanning electron microscopy (SEM) analysis

In order to investigate the influence of ultrasound and microwave during extraction on the structure of the materials and to understand the extraction mechanism, the residue after extraction of inulin was collected and dried in air for the SEM analysis (Li, Wang, Wang, Chen, & Mao, 2007; Zhang et al., 2008). Sample particles were fixed on the silicon wafer and sputtered with gold to a thickness of about 100 nm. The shape and the surface characters of the samples were observed and recorded on the scanning electron microscope (Quanta-200, FEI Ltd., Holand).

3. Results and discussion

3.1. Effect of simultaneous ultrasonic and microwave treatment and time on inulin extraction

In simultaneous ultrasonic/microwave assisted extraction, burdock root was added with a ratio of solid(g):solvent(ml) of 1:15, in a beaker and treated in an ultrasonic and microwave extracting apparatus with a microwave power of 400 W and a fixed ultrasonic power of 50 W for a period of 60 s. Conventional solvent extraction at 50 °C under the same condition of other factors was also per-

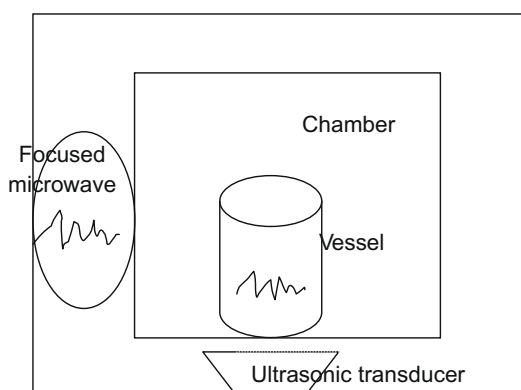


Fig. 1. Simple schematic diagram of UMAE apparatus.

formed. The effect of simultaneous ultrasonic/microwave treatment and its extraction time on the extraction recovery of inulin was shown in Fig. 2. The extraction rate of inulin was very high during the first 60 s of the UMAE. After that, the recovery of inulin decreased from 99.03 to 83.78 mg/g with the increasing extraction time. The decrease may be due to the degradation of some inulin fractions caused by the combined intense action of microwave and ultrasonic. According to the reports of Ishimaru, Kagoroku, Chachin, Imahori, and Ueda (2004) and Wei et al. (2007), inulin of burdock is a polydisperse carbohydrate, and a part of the inulin fractions may be easily degradable. Unlike to conventional heating methods, internal heating of microwave in UMAE may cause partial hyperthermia, coupled with the intensely mechanical shaking of ultrasonic, so the easily degradable inulin fractions may be degraded to reducing monosaccharide, which caused the decrease of inulin recovery.

The recovery of inulin by UMAE for 900 s was compared to that of conventional stirring extraction (CSE) (Fig. 2). The highest recovery of inulin by CSE was 99.78 mg/g, which was appreciably higher than that by UMAE (99.03 mg/g). But on the other hand, the amounts obtained by UMAE in 60 s were comparable to those by CSE for 300 s, indicating a significant reduction of extraction time.

Ultrasonic could dramatically improve the extraction of target component mainly through the phenomenon of cavitation. The mechanical effect of ultrasonic promotes the release of soluble compounds from the plant body by disrupting cell walls, enhancing mass transfer and facilitating solvent access to the cell content. Meanwhile, microwave heats the whole sample volumetrically, it disrupts weak hydrogen bonds and promotes the rotation of molecular dipoles. At the same time, electric field induced movements of dissolved ions increase solvent penetration into the matrix and thus facilitate analyte solvation. And the heating effect causes the solvent temperature to rise and increase the solubility of target compound. That is why the extraction efficiency of inulin was further improved by simultaneous ultrasonic/microwave assisted extraction.

3.2. Effect of microwave power

The microwave power had a significant effect on the extraction of inulin from burdock (Fig. 3). By 60 s simultaneous ultrasonic/microwave assisted extraction, when the microwave power was increased from 150 to 400 W coupled with a fixed ultrasonic power of 50 W, the recovery of inulin was increased fast from 87.5 to 99.89 mg/g.

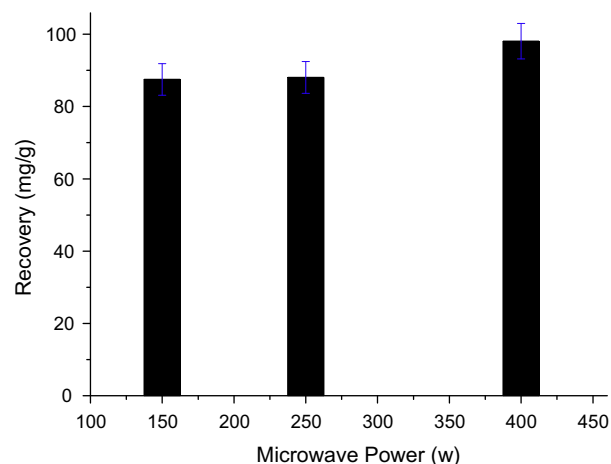


Fig. 3. Effect of microwave power on inulin extraction.

The reason for the increase of inulin recovery in the case of UMAE may be due to the high pressure gradient formed inside the plant material. Microwave absorption results in significant internal heating thus creating significantly high internal pressures which enhance inulin extraction from the burdock. The cell walls can swell and burst because of internal heating and thus further accelerate the intracellular product release into the solvent. Therefore an appropriate increase in microwave power resulted in a rise in yield. Thus, the microwave power of 400 W was chosen as the output microwave power in UMAE.

3.3. Effect of solvent to solid ratio

As shown in Fig. 4, the recovery by UMAE were found to increase with the increase of the ratio of solvent to solid and then fall down at the high ratios. The larger liquid (solvent) to solid ratio means a larger concentration difference which favors mass transfer. But in microwave assisted extraction, a higher solvent volume may give lower recovery (Eskilsson & Bjorlund, 2000). As shown in Fig. 4, the inulin recovery increased with the increase of solvent before the solvent to solid ratio reached 15, and then it fell down. This was probably due to the inadequate stirring of the solvent when the microwave was applied at larger volumes. Hence, the liquid to solid ratio of 15:1 (mL/g) seems to be appropriate for UMAE.

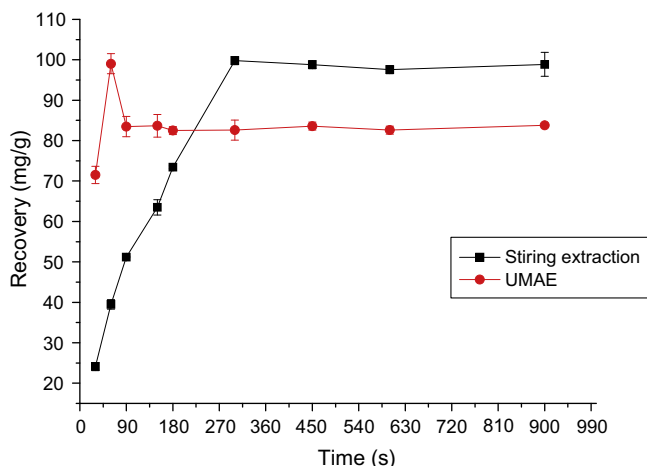


Fig. 2. Effects of simultaneous ultrasonic and microwave treatment and time on the extraction of inulin.

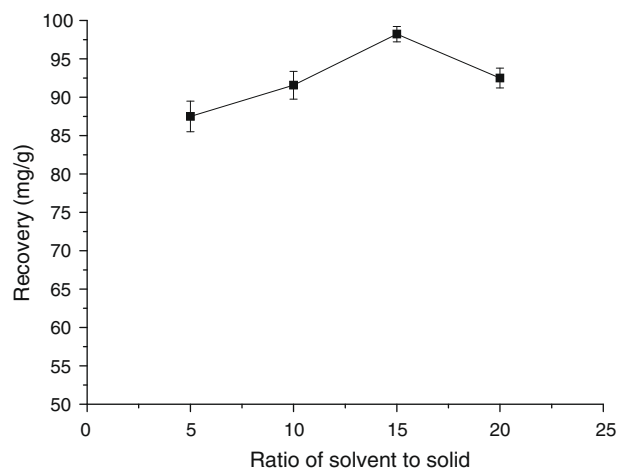


Fig. 4. Effect of solvent to solid ratio on inulin extraction.

3.4. Mechanism of simultaneous ultrasonic and microwave assisted extraction

In order to elucidate the mechanism of simultaneous ultrasonic/microwave assisted extraction of inulin from fresh burdock root, the microstructure of the untreated burdock samples, simultaneous ultrasonic/microwave assisted extraction samples and stirring extraction samples were examined by scanning electron microscopy (Fig. 5).

After 20 s of UMAE, the cells could not be distinguished and the microstructure of the sample was destroyed. There is more destruction to the root microstructure in Fig. 5b than that in Fig. 5c. This was the result of hitting and cavitating by ultrasound's intense shaking coupled with the heating and expansion of microwave. On one hand, large instantaneous energy generated by the ultrasound system leads to disruption of burdock root cells and quick dissolution of inulin from burdock cells into solvent without a permeation process. Meanwhile, water molecules in the cells, because of their higher dipole moment, absorb microwave energy strongly, leading to efficient heating of the sample. The selective interaction between the internal free water molecules and microwave leads to rapid increasing of temperature and causes expansion with subsequent rupture of the cell walls. Such systems undergo a dramatic expansion, and subsequent rupture of the cell walls, allowing the release of the compounds into the solvent (Wang, Xiao, & Li, 2008). Therefore the rupture of cell walls and migration of compounds out of the cells into solvent in UMAE was both easier than that in CSE. UMAE causes more cell wall damage than CSE (Fig. 5c and b). In the CSE process, the solvent transfer into the matrix, extract the inulin by solubilization, therefore little destruction of the microstructure of sample occur and long extraction time are needed, as is the case in our study (Fig. 2).

3.5. Total dietary fibre content and total phenolic content of the burdock root residue powder

Table 1 shows the content of total dietary fibre (TDF) and the content of moisture in powder from burdock root by-product. It could be seen that the content of total dietary fibre was high in the powder, indicating good physicochemical properties of the dietary fibre powder.

As burdock root have been reported to be a rich source of phenolic compounds (Chen, Wu, & Chen, 2004), presence of phenolic compounds in powder of burdock root residue was investigated.

The total phenolic content (PC) in powder of burdock root by-product was approximately 302.62 mg GAE/100 g dry weight. Peels of several other vegetables and fruits such as carrot, apple, pear, pomegranate, mango and peach have been found to contain higher amount of phenolics than the edible flesh parts (Garau, Simal, Rosselló, & Femenia, 2007; Zhang & Hamauzu, 2004).

A little decrease in PC was observed after drying at higher temperature (80 °C). Higher drying temperature may lead to more degradation of phenolics. However, shorter drying time required to dry the sample at higher temperature also shortened the degradation reaction. Therefore, there were no significant differences in total phenolic content at different drying conditions.

3.6. Functional properties of powder of burdock root by-product

The results obtained for (water retention capacity) WRC and (oil-holding capacity) OHC are presented in Table 2. The WRC is the quantity of water that remains bound to the hydrated fibre following the application of an external force (pressure or centrifugation) (Raghavendra et al., 2006). WRC is an important property of dietary fibre from both physiological and technological points of view. Drying at high temperatures may cause a reduction in this

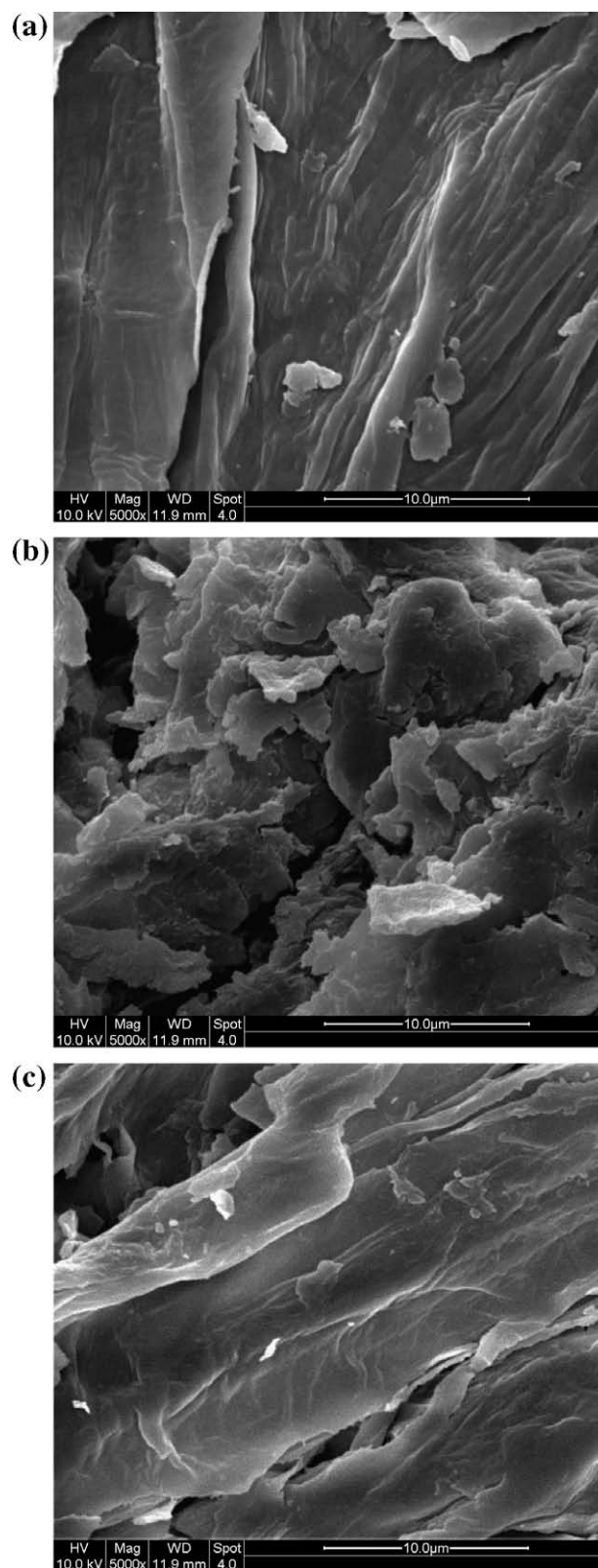


Fig. 5. Scanning electron microscope pictures of burdock root before (a) and after UMAE (b), CSE (c).

capacity (Garau et al., 2007), thus fibre prepared at high temperature (80 °C) had a bit lower WRC (Table 2) in this work. This might be caused by the degradation of some dietary fibre components leading to the loss of ability to retain water in the powder.

Table 1

Compositions of powder of burdock root by-product generated by inulin extraction (mg/100 g dry powder).

Drying temperature (°C)	Total dietary fiber	Water	Total phenols
60	43209.57 ± 521.21	5220.66 ± 69.72	302.62 ± 7.28
80	43151.37 ± 783.39	5202.51 ± 98.75	285.96 ± 4.96

Table 2

Functional properties of fibre-rich powder from burdock root by-products.^a

	WRC (ml water/g powder)	OHC (ml oil/g powder)	SWC (ml/g powder)
60 °C	12.72 ± 0.34	8.50 ± 0.36	16.10 ± 0.57
80 °C	11.92 ± 0.42	8.28 ± 0.30	15.95 ± 0.61

^a Values are means of triplicate assays.

As shown in table 2, fibre-rich burdock root powder had a WRC (12.72 ml water/g) similar to the results reported previously (Fuentes-Alventosa et al., 2009; Grigelmo-Miguel & Martín-Belloso, 1999). These WRC values are in the range of most reported fibres, e.g. 12.6 ml water/g for peach pulp fibre and around 11 ml water/g for lemon fibre (Grigelmo-Miguel & Martín-Belloso, 1999; Lario et al., 2004). Other by-products had lower values than those mentioned above, e.g. cocoa husks (Lecumberri et al., 2007), with a WRC value of about 5 ml water/g fibre. Basing on the values above, this fibre-rich powder could be promoted as a modifier of viscosity and texture of formulated products as well as promoting the decrease in calories.

Results for OHC of burdock root powder (around 8.50 ml oil/g) are presented in Table 2. High drying temperatures caused a little reduction in this capacity. Values reported in the documents were much lower than those for burdock root residue powder, in other researchers findings, e.g. 0.6–1.8 ml oil/g for apple pomace and citrus peel (Figueroa, Hurtado, Estévez, Chiffelle, & Asenjo, 2005). The highest reported level was about 6 ml oil/g of carrot sarcocarp (Garau et al., 2007). Fibre-rich burdock root powder had higher values (8.50 ml oil/g), so this fibre powder may be appropriate to products in which emulsifying properties are required.

The (swelling capacity) SWC of burdock root residue powder are presented in Table 2. The results showed that the powder from burdock root by-product possessed high SWC. These values are of similar order with those of citrus residues (Figueroa et al., 2005). As shown in table 2, drying temperature makes little effect on this capacity. Higher drying temperature resulted in little reduction of WRC.

4. Conclusion

Inulin was extracted by simultaneous ultrasonic/microwave assisted extraction (UMAE) and phenols-rich dietary fibre powder was produced using burdock root residue generated by inulin extraction. The effects of simultaneous ultrasonic/microwave treatment on inulin extraction and the physicochemical properties of dietary fibre powder prepared were first evaluated. The results indicated that coupling microwave with ultrasonic technique presented more advantages. The suitable condition for UMAE of inulin was under a simultaneous ultrasonic power of 50 W and a microwave power of 400 W, at a ratio of 1 g of solid material to 15 ml of water for an extraction time of 60 s. Although the recovery of inulin by UMAE (99.03 mg/g) was appreciably lower than that by conventional stirring extraction (99.7899 mg/g), the extraction time was significantly shortened from 300 to 60 s by UMAE. Phenols-rich dietary fibre powder was produced using the burdock root residue in which the inulin had been extracted. The water retention capacity,

swelling capacity and oil-holding capacity of the dietary fibre powder prepared were 12.72 ± 0.34 ml water/g, 8.50 ± 0.36 ml oil/g and 16.10 ± 0.57 ml/g, indicating its prospect as a functional food ingredient.

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