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Compressional-puffing pretreatment for enhanced antioxidant compounds extraction from *Aloe vera*

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ABSTRACT

Compressional-puffing pretreatment is an efficient pretreatment process that can enhance extraction efficiency of bioactive compounds from plants. In this study, raw and pre-dried *Aloe vera* at moisture content 0–42.47 g/g dried biomass was puffed at 200 °C for 20 s and was extracted using 60% v/v ethanol solution at 70 °C for 1 h. Pre-drying with 80 °C thermal drying or −50 °C freeze-drying produced mediate sized pores which do not release the targeted bioactive compounds from *Aloe* biomass. Conversely, the puffing yielded numerous 0.1–5 μm pores that contributed to the enhanced extraction efficiencies. There presents a moisture content (1 g/g dry material) that maximized the extraction yield of bioactive compounds, which denoted the compromise between thermal inertia to heat up at too much water and explosion expansion strength to be produced at too few water in biomass. The even distribution of water in biomass subjected to puffing was proposed essential to efficient puffing treatment of the biomass.

1. Introduction

Phenolic compounds such as phenolic acids, flavonoids, tannins and lignans in various plant tissues are scavengers to free radicals so are assistants to the reproduction and growth of the plants [1–5]. Antioxidant activity of phenolic compounds is attributable to the capability of donating hydrogen atom to free radicals or by removing the unpaired electrons from the radicals [6]. The flavonoids yield antioxidant capability by delocalizing electrons with the involved hydroxyl groups to the free radicals [7,8].

Transfer of the antioxidants such as phenolic compounds from plant tissues to solvents provides a commercial product form for market sale. Pretreatments are generally applied to overcome the transfer resistance for enhanced extraction efficiency and yield [9]. Puffing process offers high temperature to build up pressure and then suddenly releases the pressure to explore out the formed superheated vapor inside the sample [10]. Compressional-puffing pretreatment is an efficient and simple puffing process compared to the rotary puffing process [11,12] by applying a high mechanical pressure on material. Huang et al. [11] used the compressional-puffing process to enhance extraction yields of fucoidan from *Sargassum glaucescens*. Chiang et al. [13,14] applied the compression-puffing pretreatment to pretreat *Pinus morrisonicola* and proposed the optimal operational conditions for maximum yields of phenolic compounds and the associated antioxidant [13,14].

This study applied the compressional puffing-assisted ethanol extraction process on *Aloe vera* for recovering involved antioxidants inside the biomass matrix, using *P. morrisonicola* as the comparison reference. The focus of the present study is on the effects of sample moisture content on the extract antioxidant activities, with too much moisture the heating would be insufficient to vaporize all water while with too few moisture no sufficient vapor would be produced for subsequent explosion action. We noted herein the presence of a critical moisture content at which the compressional puffing ethanol extraction would yield the best extract performance.

2. Materials and methods

2.1. Materials

A. vera is widely cultivated for agricultural and medicinal uses because of its abundance of phytochemicals [15–18]. *P. morrisonicola*, as studied in [13,14], is used as control [19–21]. The *A. vera* sample was purchased from Jianguo Holiday Flower Markets in Taipei City, Taiwan (Fig. S1a). The plant was 2 years old. The samples were cut and washed by deionized water (DI water) before use. After drying and compressional-puffing treatment, the sample was extracted in 60% v/v ethanol. *P. morrisonicola* was purchased from Guguan city, Taiwan. Pine needles were cut into 1.5–3 cm long for further tests. After drying and compressional-puffing

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pretreatment, the sample was extracted in 60% v/v ethanol and the extract was stored in a refrigerator at 4 °C until used.

2.2. Treatments

The samples were tested with drying + puffing + extraction at identical conditions in triplicate to assure the data reproducibility.

2.2.1. Drying protocols

To provide appropriate initial moisture contents for compression-puffing treatment, the samples were dried by two different drying protocols. The first protocol used an oven (model NDO-450ND, Eyela, Japan) at prescribed temperature. The *Aloe* pieces at 1 cm × 1 cm × cm size were placed onto a weighing plate (10 cm × 10 cm) in the oven. The sample weights were recorded over testing time. Some samples were dried at 100 °C to reveal the possible effects of drying temperature on extraction performance.

The second protocol used a freeze-drier (model FD45-12P, Kingmech, Taiwan) to dry the samples. The weights for 1 cm × 1 cm × 1 cm sized *Aloe* samples were packaged by aluminum foil and were put it into the glass bottle. The working condition for the freeze-drying machine was at −50 °C and 20 mm torr.

2.2.2. Puffing protocol

The raw samples (42.47 g/g dried material) and the pre-dried samples at 3, 1, 0.33 or 0 g/g moisture content were placed into the compressional-puffing machine (model R2-L, Yuan Chuang Food Machinery Co. Ltd., Taiwan) consisting of two circular plates, upper plate and bottom plate, forming a cylindrical chamber (65 mm diameter, 6 mm deep) [11]. The puffing process basically contains three stages. The first one is that when it starts, upper plate comes down to the bottom plate. Second stage is that upper plate gives a mechanical pressure of 5 atm on bottom plate and maintains for a period of time. Final stage means that the puffing finishes and the upper plate goes back to original place suddenly to release high steam pressure. Furthermore, the temperatures of upper and bottom plates were set to 200 °C before puffing. The operating time for puffing process in second stage was 20 s. After pretreatment, the samples were packed into PE bags and stored at −20 °C until used.

2.2.3. Extraction protocol

The untreated and treated samples were extracted in 60% ethanol solvent, 70 °C extraction temperature, 1 h extraction time and 50 mL/g liquid–solids ratio, with parameters being identified optimal ones for *P. morrisonicola* tests [13]. Restated, *Aloe* were weighted by analytical balance to a controlled value and then put into a 50 mL vessel (Fisher Scientific, USA). And the liquid–solids ratio was fixed to 50 mL solvent per gram solid. After then, batch solvent extraction was conducted at 70 °C in a thermostatic bath (Biobase Co., Ltd, China). Finally, the extract was filtered through 0.2 μm membrane filter (Merck Millipore, USA) and the solution was stored at 4 °C until used.

2.3. Analyses

2.3.1. Extract composition

The total flavonoid content (TFC) of sample was measured using the method of [13,14] with modifications. 0.5 mL extract was mixed with 5 mL of ethanol, 0.25 mL 0.5 M sodium nitrite and 0.25 mL of 0.3 M aluminum chloride for 5 min. Then, 1.5 mL of 1 M sodium hydroxide was added. The absorbance at 510 nm was measured with an UV–vis spectrophotometer (DR2700™, HACH, Colorado, USA) with quercetin as the model compound.

Total phenolic content (TPC) was measured using the method modified by Adefegha and Oboh [22]. Briefly, 0.1 mL extract was mixed with 0.5 mL Folin–Ciocalteau's reagent and 5.9 mL DI water for 2 min. Then 1.5 mL 20% (w/w) sodium carbonate was added and mixed at 40 °C for 30 min. The absorbance at 765 nm using gallic acid as model compound was measured.

2.3.2. Antioxidant activity

The DPPH method and the ABTS method were applied for quantifying the antioxidant activity of samples. The quenching capability of free radicals was measured by using 2,2′-diphenyl-1-picrylhydrazyl with Trolox as reference [23]. The ABTS method is similar to the DPPH method with working solution prepared by 2,2′-azino-bis(3-ethylbenzothiazoline-6-sulfonic acid) diammonium salt and potassium persulfate [24]. The measured activity was listed as specific Trolox equivalent (TE) in unit of μmol/g.

2.3.3. Other analyses

The SEM pictures of samples were obtained by using Nova™ NanoSEM 230 (FEI, USA). The specimens were first cut into small pieces and pasted onto a carbon conductive tape. Second, these specimens were coated by sputtering platinum on it to increase the conductivity. And then putting it into the chamber, the pictures could be taken by the detector.

The mercury intrusion porosimetry (MIP) instrument, Micromeritics Autopore IV 9520 mercury porosimeter (Norcross, USA) was applied to probe the internal pores of samples. The samples were cut into about 5 mm × 5 mm size with 1 g of weight. By applying the pressure from the outside, mercury would penetrate into pores of the sample with the required equilibrated pressure was related to the pore size, so the porous properties could be obtained. In this study, the minimum pore access diameter is about 6 nm with a maximum 300 MPa injection pressure.

3. Results and discussion

3.1. Characteristics of dried samples

Fig. S2 in Supplementary Materials shows the drying kinetics of *Aloe* samples under 80 °C thermal drying and −50 °C freeze-drying. Different mathematical forms, Henderson–Pabis model, Newton's model, Logarithmic model and Wang–Singh model were used to fit the experimental data. Table S1 lists the results. From the results, the Logarithmic model best fits the experimental data so it was used herein for controlling the initial sample moisture contents before puffing.

Thermal drying led to significant volume shrinkage and color change of the samples (Fig. S1b). Conversely, freeze-drying had thickness change and minor color change (Fig. S1c). A common assumption is that freeze-drying can prevent major deterioration of heat-sensitive substances in the samples subjected to drying [25] while thermal drying would produce skin layers by inhomogeneous structure rupture so increases the moisture transfer to the surroundings [26].

The raw *Aloe* samples had principally the pores of size about 300 μm (Fig. 1) with total pore area of 0.17 m^2/g. The thermally dried sample had a similar 300 μm peak with some emerging 1–2 μm peaks, giving an increase of total pore area to 2.16 m^2/g. The freeze-dried sample exhibit 30–40 μm and 0.1–5 μm pores. The corresponding pore area was increased to 2.39 m^2/g. The mechanisms for thermal drying are very different from freeze-drying, but are not the focus of the present study.

3.2. Puffed samples

3.2.1. Appearance, porosity and moisture content changes

After puffing, owing to the vapor expansion the volumes of tested samples were increased (Fig. S3). The puffing tends to cre-

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