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A novel method for food particle production using subcritical water extraction: *Ganoderma* mushroom as a case example

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ABSTRACT

In recent years, supercritical fluid-based technologies have been used for the production of food particles as well as the extraction. These methods are used in particle generation as well as extraction due to properties of solvent such as having good dissolving and diffusing power, moderable conditions, capability to produce particles with a regular particle size distribution of the solvent and being environmentally friendly. Recently, three different techniques named rapid expansion of supercritical fluid (RESS), supercritical anti solvent application (SAS) and particle formation with gas-saturated solvent (PGSS) are used for particle generation. In this study, a novel system capable of doing subcritical water extraction and particle formation was briefly introduced. The system was based on first hydrothermal extraction of target compound, then transition of saturated extract solution into fine particles at underflow in hot air assistance. *Ganoderma lucidum* mushroom was used as working material to investigate process parameters. Also, extract composition and structure were identified.

1. Introduction

In recent years, conventional extraction methods have been replaced by new techniques like supercritical fluid technology [1–5]. Environmental pollution and high cost originating from products and by products of traditional methods in which organic solvents are used can be stated as significant reasons of this transformation. So, supercritical fluid technology has been an important alternative being new and available for improvement in various food material applications [6–11]. Other important advantages of supercritical fluids come from their diffusivity power being close to gases and being at least two orders of magnitude larger than liquids [11–17]. The technique discussed in this study basically adopted the principle of subcritical water extraction to the air assisted particle formation technology. Examples of different particle formation methods have arisen from the use of carbon dioxide as an extraction solvent about 20 years ago [18–20]. So, carbon dioxide is a commonly used supercritical fluid for obtaining food particles and is also applied for different fields such as extraction, crystallization and sterilization [21–25]. Depending on the nature of the process applied, polarity of supercritical fluids can be changed using co-solvents in comparison with the polarity of target compound to be extracted [26–28].

Generally, particles with small size and narrow size distribution (i.e., large surface area per unit volume) are desirable in many chemical processes such as adsorption, reaction and catalysis. Tendency to use supercritical fluids for extraction and particle generation has been increased because of their capability for producing fine particles rich in valuable components. Conventional methods for particle size reduction include milling, grinding, jet milling, crushing, and air micronization, or recrystallization from solution like liquid anti-solvent and spray-drying. But these methods have some difficulties in accomplishing desired particle properties and in operating mostly organic chemicals for applications of temperature and pressure sensitive materials. When these factors are considered, it has been found that traditional methods to form food particles can be replaced with new particle formation techniques using supercritical fluid technology. Rapid expansion of supercritical solutions (RESS), supercritical anti-solvent process (SAS) and particle generation from gas-saturated solutions (PGSS) methods are the most preferred ones for particle production [29–32]. Basic principle in these methods is lowering the pressure of saturated solution soon after the extraction process and obtaining fine particles [33].

In this study, extraction and particle formation processes are described, the novel system design formed by combining two techniques is introduced and post processes and analyze methods are

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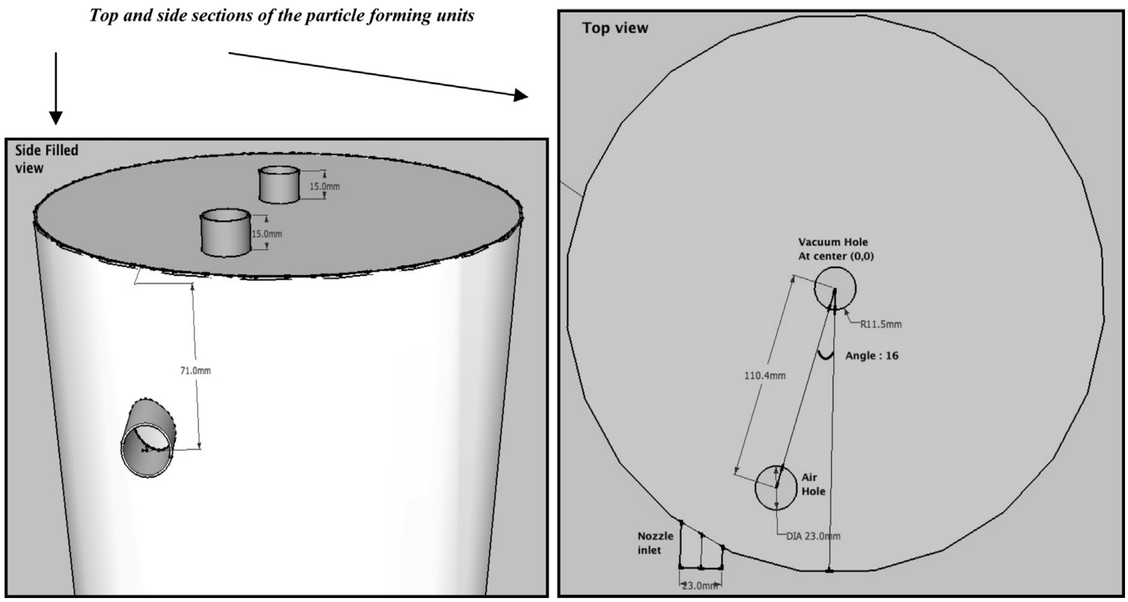

Fig. 1. Pre-designed particle formation drying unit.

explained by selecting *Ganoderma lucidum* as example. *Ganoderma* mushroom is well known with the names of Reishi or Lingzhi being produced in Asian countries and has been farmed in reasonable number of countries. Being a member of Polyporaceae family which is a fungal species traditionally consumed nearly 4000 years of self-medication in liquid or powder used in Chinese medicine, it is assimilated as the "mushroom of immortality" among people and has recently become one of the strongest foods that has applicability for new technological improvements. *Ganoderma* species are widely researched due to their reported potent bioactive properties. Among these constituents, glucans and polysaccharides have been reported to exhibit anti-tumor and anti-inflammatory effects and have been supported by US Food and Drug Administration (FDA). Starting from all these positive effects, in this study, water soluble organic compounds, beat glucans and polysaccharides have been extracted from ground form of *Ganoderma lucidum* using subcritical water. Following extraction, the extracts were used to form particles in combined system for extraction-particle production. Particles in powdered form have been mostly utilized in food industry areas such as functional food production, fruit juice production, etc.

The description of process steps used in this study could be summarized as following:

- Subcritical water was used as critical solvent.
- The process utilized high solvating power of supercritical fluids.
- After loading SCF with the solute, a phase change from the supercritical to the gas-like state took place during the expansion in the supersonic free jet. This lead to high super saturation, and subsequently to particle formation.
- Finally, while particles were collected in sampling chamber, air was purged out of the container through a valve.
- The most interesting applications of SCFs occurred in range; $1 \leq P/P_{\mathrm{critical}} \leq 3$ for pressure and $1 \leq T/T_{\mathrm{critical}} \leq 1.2$ for temperature (in the region slightly above the fluids' critical point). Thermo physical properties exhibited a huge variation with respect to pressure and temperature.
- The final product obtained was analyzed using DLS (Dynamic light scattering) method to determine the size of stabilized particles; using SEM (scanning electron micrographs) method to investigate morphological structure. Also, extract composition was investigated using chromatographic analyses of HPLC (High Performance Liquid Chromatography) and GC/MS (Gas chromatography–mass spectrometry).

2. Materials and methods

2.1. Materials

Ganoderma lucidum was used as sample material in 3 mm ground form. It contains 6.52% hydrogen, 1.20% nitrogen, 50.26% oxygen and 42.02% carbon. *Ganoderma* samples were stored at room temperature keeping away from humidity until they used and did not undergo any further pretreatment.

Polystyrene standard kits were acquired from Shodex Company (Shodex STANDARD S Series- SM 105, Shodex Ltd., Munich, Germany). Tetrahydrofuran (THF) was purchased from Wako Chemicals (Germany) GPC solvent. HPLC-grade solvents including acetonitrile and acetic acid were purchased from Wako Chemicals (Neuss, Germany).

2.2. Methods

A schematic representation of the combined extraction/particle production experimental apparatus that was used to produce *Ganoderma* powders was shown in Figs. 1 and 2, respectively. This system has exhibited a novel property of being a combination of two different physical operation systems. It could make the operator save energy, time and money with products being better in quality.

The whole particle design apparatus was divided into three sections:

(a) A supercritical fluid delivery unit (a water source, a circulating heater, an HPLC pump),
(b) A solute dissolving and extracting unit (a pre-heater, an extraction vessel and an oven),
(c) A crystallizing-separating unit (nozzle and collector).

The apparatus used for the experiments was showed in Fig. 2. Experimental design could be successfully operated for temperatures between 25 °C and 350 °C. In each experiment, 10 mL