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Valorization of grape pomace: Drying behavior and ultrasound extraction of phenolics

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ABSTRACT

The wine industry generates huge amounts of grape pomace as an industrial waste. Recently, there has been much interest in the phenolic compounds of grape pomace, because of their health benefits, such as antioxidant activity, acting as free radical scavengers, and inhibition of lipoprotein oxidation. Thus, the objective of this work is to optimize a new method for grape pomace application in food industries based on the ultrasound-assisted extraction of phenolics compounds. Dehydration of grape pomace is the first step before extraction. Thus, another subject of this work is to study the drying behavior of this winery by-product and the kinetics of total phenolics degradation during the drying process. Drying (moisture vs time) data were obtained on slabs of grape pomace in an air dryer operating at 60–85 °C. The effective diffusivity was determined using the method of slopes of the drying curve taking into account the effect of moisture content on diffusivity. The combined effect of moisture content and temperature on effective diffusivity was expressed by an empirical model. In addition, each moisture loss curve was fit to empirical simplified drying models. Finally, the effects of solvent type, extraction temperature, solvent/solid ratio, amplitude level, and pulse duration/pulse interval ratio on the yield of phenolics extraction were studied.

1. Introduction

Grape pomace is the winery waste originated during the production of must by pressing whole grapes. Nine million tons of this waste are produced per year in the world, which constitutes about 20% w/w of the total grapes used for wine production (Teixeira et al., 2014). Grape pomace constitutes a source of phenolic compounds. Its phenolic content mainly include anthocyanins (e.g., malvidin, peonidin), flavan-3-ols (e.g., catechin, proanthocyanidins), flavonols (e.g., quercetin, myricetin), stilbenes, and phenolic acids (Negro et al., 2003; Makris et al., 2007). Based on its polyphenolic content, several studies have reported its high antioxidant activity (Boussetta et al., 2009; Rockenbach et al., 2011a,b). Therefore, the recovery of phenolic compounds from grape pomace is of great importance, not only because of their significant properties, but also because it could exploit a large amount of the wine industry wastes, which are mainly used today as cattle feed or for soil conditioning or they are trucked away to disposal sites (Louli et al., 2004).

Grape pomace is highly perishable due to its high moisture content and water activity. The pomace could be frozen until required, but large volumes are involved here and an extract would need to be prepared from the pomace, or it would need to be dried in any event before use in value-added or functional food product formulations (Vashisth et al., 2011). Thus, dehydration of wet grape pomace is a first step before developing further applications. According to Milczarek et al. (2011), the high moisture content of grape pomace makes it costly to transport in its original wet form and must be reduced to

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about 5–8% (wet basis) to extract. Drosou et al. (2015) reported that dried grape pomace extracted with water exhibited higher yield than the wet pomace, since drying causes breakage and destruction of cell walls and consequently large cavities and intercellular spaces are formed allowing to the cellular substances to be easily extracted. Hartley et al. (1990) suggested that because phenolic acids are mainly bonded to carbohydrate and proteinaceous moieties, during drying their release might occur due to breakdown of cellular constituents and covalent bonds. Therefore, the released polyphenolic constituents become more amenable for extraction.

Several researchers have described the advantages of mathematical models to develop a better understanding of controlling the parameters of a drying process (Perez and Schmalko, 2009). Thin layer drying models have found wide application among mathematical models and these kinds of models have three categories. While theoretical thin layer models take into account internal resistance to moisture transfer, semi-theoretical and empirical models consider external resistance to moisture transfer between product and heated air (Akgun and Doymaz, 2005). Numerous studies have investigated the drying kinetics of different agricultural products in order to determine the water diffusivity coefficient and the suitable mathematical model of the specific drying process. As far as the grape pomace is concerned, limited research has been achieved with regard to its drying behavior (Sui et al., 2014; Drosou et al., 2015).

Drying of grape pomace can result in significant degradation of the polyphenolics and also affect antioxidant and free-radical scavenging capacities (Vashisth et al., 2011). Generally, the problem of chemical conversions during drying is extremely complicated. The rate constants of the reactions depend on temperature, concentration of the reactants and concentration of water (water activity) (Sokhansanj and Jayas, 1995). The influence of the water activity is important but insufficiently understood. Although various oxidation reactions show a minimum rate of reaction at a certain water activity (Pan et al., 1999), in general, chemical reactions are slower as the water activity decreases (Karel, 1979). However, limited research has been achieved with regard to the phenolics involvement in product quality changes during grape pomace drying (Khanal et al., 2010; Vashisth et al., 2011; Tseng and Zhao, 2012).

Several studies have been published in the past to extract phenolic compounds from grape pomace. Grape pomace phenolics can be extracted with various extraction methods, such as normal stirring, Soxhlet extraction, microwave-assisted extraction, supercritical fluid extraction, and accelerated solvent extraction (Ahn et al., 2004; Louli et al., 2004; Fang and Bhandari, 2010; Marqués et al., 2013; Rajha et al., 2014). Shortcomings of existing extraction technologies, like increased consumption of energy, high extraction time, possible degradation of bioactive compounds, and high consumption of harmful chemicals, have forced the food and chemical industries to find new separation “green” techniques, such as ultrasound extraction (Chemat et al., 2011). Extraction enhancement by ultrasound has been attributed to the cavitation, which generates high shear forces and microbubbles that enhances surface erosion, fragmentation, and mass transfer (Awad et al., 2012). In recent years, ultrasonic irradiation has been used to recover phenolic compounds from different plant food materials and their by-products, such as green tea, wheat bran, jatoba bark, arecanut, *Stevia rebaudiana* Bertoni, citrus peel, black chokeberry waste, coconut shell, orange peel, onion waste, and olive mill wastewater (Xia et al., 2006; Rodrigues and Pinto, 2007; Rodrigues et al., 2008; Wang et al., 2008; Ma et al., 2009; Khan et al., 2010; Jerman-Klen and Mozetic-Vodopivec, 2011; Galanakis et al., 2013; Jang et al., 2013; Veggi et al., 2013; Galvan D'Alessandro et al., 2014; Barba et al., 2015). However, limited research has been achieved with regard to the ultrasound-assisted extraction of phenolics from grape pomace (Corrales et al., 2008; Delgado-Torre et al., 2012; Drosou et al., 2015). In addition, the multitude of process dimensions which present themselves when they are combined with conventional process variables such as temperature, time, solid/liquid ratios should be further studied in order to optimize the whole process for a future industrial implementation.

One objective of this work was to study the phenomena governing the diffusion process during grape pomace drying, determining the effects of process parameters on diffusivity and expressing these effects with empirical models. These models can be used to control or optimize the variables of grape pomace drying process, which is a first step before developing further applications of this important food industry waste. Another objective of this work was to study the kinetics of total phenolics degradation during the drying process. In addition, the ultrasound-assisted extraction of phenolics from grape pomace was optimized.

2. Materials and methods

2.1. *Plant material*

Agiorgitiko (*Vitis vinifera*) grape pomace, harvested in 2013, was kindly provided from Epanomi region (North Greece) after red vinification. The pressed pomace was received containing 81.7 ± 1.4% moisture content and immediately stored at −30 °C to avoid enzymatic degradation of the polyphenols until further use.

2.2. *Drying of grape pomace*

Flat grape pomace (slabs) was prepared in 30 cm × 19 cm dishes. The pomace samples were spread in the dishes applying light pressure to form slabs of uniform thickness of about 6 mm. The drying experiments were performed at temperatures of 60, 65, 70, 75, 80, and 85 °C in a tray dryer with an air velocity of 1.2 m/s. The drying data were obtained by periodic weighing of the samples with a balance (PCB 1000-2, KERN & Sohn GmbH, Germany), placed on the top of the dryer. The initial moisture content was determined by the vacuum oven method at 70 °C for 24 h. Dried pomace samples were drawn periodically and analyzed for total phenolics. Grape pomace drying and thermal degradation kinetics were replicated twice at the six product temperatures.

2.3. *Extraction of phenolics from grape pomace*

Grape pomace was dried at 60 °C for 20 h and kept at −30 °C until use. The pomace was ground in a laboratory mill (Type A10, Janke and Kunkel, IKA Labortechnik, Germany) immediately prior to extraction. A 130 W of maximum nominal output power, 20 kHz of frequency VCX-130 Sonics and Materials (Danbury, CT, USA) sonicator equipped with a Ti–Al–V sonoprobe (13 mm) was used for ultrasound-assisted extraction in pulsed mode. The solvents used were ethanol, water, 50% aqueous ethanol, 70% aqueous ethanol, and 70% aqueous