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Original Research Paper

Grinding kinetics of red grape seed residue in stirred media mill

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ABSTRACT

The main objective of the present paper was to experimentally investigate the grinding kinetics of red grape seed which is the by-product of winery and juice industry. Stirred media mill was used as a high energy density mill to improve the raw grape seed fineness, i.e. mean particle size approx. 10 μm using various rotor circumferential velocities under dry condition. The effect of stress intensity and stress number on the particle size distribution of ground grape seed was investigated. Optimum conditions (rotor velocity and residence time) were determined, mean particle size close to 10 μm and 5000 cm^2/g geometric specific surface area were reached within the studied variables. Additionally, concerning the material structure, FTIR measurements of the ground grape seed samples were carried out which demonstrated that no structural changes were detected. Furthermore, the specific grinding work was measured for each test, in this way energy utilization, efficiency was determined.

1. Introduction

Grape pomace is the byproduct of juice industry and winery, approximately it counts for 25% of the grape weight. Various utilization possibilities of the components of this material are known worldwide – agriculture, food industry, pharmaceutical industry, cosmetics; i.e. functional foods (dietary fiber + polyphenols), food processing (biosurfactants), cosmetics (grapeseed oil + antioxidants), pharmaceutical/biomedical (pullulan) and supplements (grape pomace powder) [1].

Grape pomace was also used as raw material for biogas production experiments carried out by Hajdú [2], who found that 250–270 m^3/t biogas productivity was achieved. However, one of the most prosperous components is the grape seed which can be produced from grape pomace by different separation technologies. Grape seeds are mainly composed of lipids, proteins, carbohydrates and polyphenols. The main polyphenols isolated from grape seeds are catechins and their polymers [3].

Jayaprakasha et al. [4] established that grape seed extracts may be exploitable for the preservation of food products and for health supplements and nutraceuticals.

The study of Molva and Baysal [5] highlighted the potential use of grape seed extract of the fruit juice/beverage industry as natural antimicrobial to inhibit the growth of A. acidoterrestris bacteria.

Polyphenols from grape-derived products have been associated with the prevention of numerous diseases including cardiovascular and neurodegenerative diseases such as Alzheimer's disease, as well as several forms of cancers [6–8].

Bagchi [9] demonstrated that grape seed powder extract provides excellent protection against oxidative stress and free radical-mediated tissue injury. The antioxidants are potent scavengers of free radicals and serve as inhibitors of neoplastic processes.

The grape seed structure can be divided into five parts: 1 - cuticle and epidermis, 2 - outer integument or soft seed coat, 3 - medium integument or hard seed coat, 4 - inner integument and 5 - endosperm and embryo. Cadot et al. [10] found that polyphenols have been observed mainly in the epidermis and in the outer integument.

Boussetta et al. [11] investigated the polyphenols extraction conditions of grape seed. Three different pre-treatments were applied on grape seeds: (1) pulsed electric fields (8–20 kV/cm, 0–20 ms), (2) high-voltage electrical discharges (10 kA/40 kV, 1 ms) and (3) grinding in a small scale cutting mill (180 W, 40 s) in order to increase the polyphenols yield. The ground samples were sieved to select particles finer than 1000 μm and were immediately used for diffusion experiments. All the pre-treatments could enhance the extraction kinetics.

Bucić-Kojić [12] studied the kinetics of a batch solid–liquid extraction of total phenolic compounds (PC) from milled grape seed using 50% ethanol at different extraction temperatures (25–80 C). They concluded that the maximum yield of PC was

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0.13 kg_{GAE}/kg_{db} after 200 min of extraction in agitated vessel at 80 °C.

The above technologies are based on mainly chemical processes (i.e. extraction). However, there are numerous application in which mechanical preparation by (grinding) is appropriate to produce a valuable final product in powder form or to enhance the extraction efficiency. The grinding process has a very important role in the industry. Beside the traditional applications, such like metallurgical, chemical, cement and mineral processing industry, it is more and more widely used in the cosmetics, food, pigment and pharmaceutical industry (active materials) as well as treatment of biomass and different kinds of waste.

Smekal [13] defined the term of "mechanical activation" as processes effected by mechanical energy, which increase the chemical reactivity of the system without altering chemical composition. However, there are several states of mechanical activation and the following situations can be distinguished: (i) mechanical dispersion, (ii) surface activation and (iii) mechano-chemical (structural) activation [14–16].

There are only limited papers reported the process of grape seed grinding. One of the known and published technology [17] using air jet milling, they reached 10 μm median particle size. Jet mills are widely used in the food industry [18–21]. However, the disadvantage of this kind of mill that it requires ancillary air compressors to assure high flow rates, pressure can exceed 10 bars [22].

Recently Zhao et al. [23] reported that superfine grinding decreased the bulk density and fluidity, and improved the solubility of grape seed. However, this paper is dealing only with the material properties of the ground and classified samples and not the grindability characteristics.

According to the literature the following mills are reported as micro grape seed manufacturing apparatuses: air jet mill, impact mill, cutter mill, disc mill [23,24].

Based on the fact, that only very limited number of papers are dealing with grape seed grinding (micronization), in spite of the relevance of the size reduction process during its utilization, the goal of the research reported in this paper is to investigate the grinding kinetics of grape seed in a high energy density mill (stirred media mill) and study the particle morphology and material structure during the grinding process.

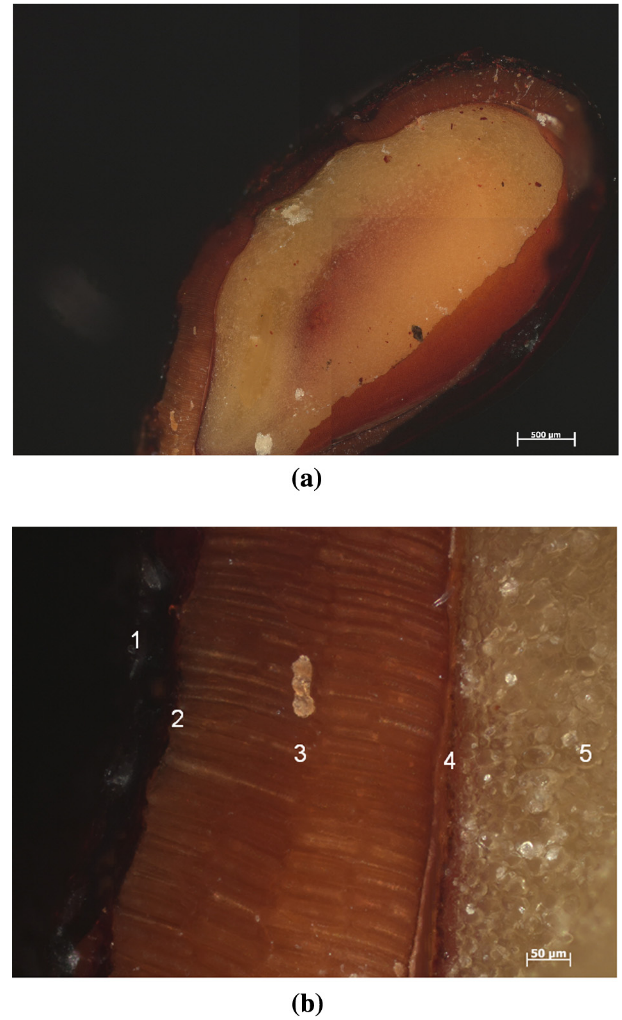

Fig. 1. Optical microscopy images of raw grape seed, a – cross section, b – detail of cross section 1 - cuticle and epidermis, 2 - outer integument or soft seed coat, 3 - medium integument or hard seed coat, 4 - inner integument and 5 - endosperm and embryo.

2. Material and methods

2.1. Material

The red grape seed pellet sample originated from Verimpex 2000 Co. Ltd. Hungary was generated during the grape oil manufacturing after cold pressing treatment. The maximum particle size was 15 mm and the density of the grape seed was determined by pycnometer method using alcohol as media, it was found to be 1.52 g/cm^3. Moisture content was measured in drying oven at 105 °C until constant mass overnight, which was 7.47%.

Fig. 1a and b show the optical microscopy images of the fracture surface of a raw grape seed. The main zones of the seed structure [10] can be detected clearly in Fig. 1b, which are as follows from the left side: 1 - cuticle and epidermis, 2 - outer integument or soft seed coat, 3 - medium integument or hard seed coat, 4 - inner integument and 5 - endosperm and embryo.

The cuticle and epidermis are very thin layers on the outer surface, followed by the outer integument or soft seed coat (Fig. 1b). This latter one is dark brown, having a 100–200 μm layer thickness. The next layer is a light brown, orange colored one, with size of 200–500 μm, which is ridged, ribbed toward the center of the core. The biggest part in volume of the seed is a more or less transparent light colored material, so called endosperm and embryo consisting of rounded and spherical shaped primary particles with a median size of 20–30 μm (Fig. 1b). Between the two latter ones there is the inner integument in the size range of 10–20 μm.

2.2. Preparation methods

The raw grape seed pellet was pre-ground in a hammer mill and a Retsch centrifugal ZM 200 type impact mill prior to the systematic grinding experiments in stirred media mill. The hammer mill was used with 2 mm sieve and the Retsch ZM 200 with 0.5 mm sieve and 10.000 1/min revolutional number.

The grinding of the grape seed was carried out in a batch stirred media mill with 530 cm^3 grinding chamber volume. Stirred media mills are widely used in various fields, i.e. pharmaceutical, ceramic, mineral processing and chemical industry [25]. The chamber is filled with fine size grinding media (mostly wear resistant ceramics) which are put into relative motion by the rotating agitator, therefore high energy density can be reached compared with a conventional tumbling mill.

The material of the liners and the five stirrer discs made of high wear resistant Al_2O_3 ceramic. The perforated stirrer discs have a triangular shape. The mill was operated in horizontal position. During the grinding experiments water cooling was applied using