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High Pressure Treatment of Grape Seed to Enhance the Yield of Oil Extraction

Ana L. Magalhães[1], C P. Passos[1], Jorge A. Saraiva[2,*], Manuel A. Coimbra[2], Carlos.M. Silva[1]

[1]CICECO, Department of Chemistry, University of Aveiro, 3810-193 Aveiro, Portugal
[2] Department of Chemistry, University of Aveiro, 3810-193 Aveiro, Portugal

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Abstract

The combination of pre-treatments with natural matrices processing may induce higher extraction yields and/or reduce the treatment times involved. In this essay, the application of a high pressure treatment prior to the supercritical fluid extraction of grape seed (*Vitis vinifera*) oil has been investigated.

Experiments show that, under the operating conditions adopted, the final extraction yield slightly decreases with increasing processing pressure. Up till 3000 bar such effect is negligible, though a variation of 15.5% was measured at 5000 bar.

With respect to the supercritical fluid extraction results, the comparison between cumulative curves achieved with treated and untreated grape seed evidenced faster kinetics in the former case, despite final yields were approximately the same. Therefore, combining a high pressure treatment with supercritical fluid extraction may certainly reduce CO_2 consumption to reach the same extractability under optimized operating conditions.

1. Introduction

Increasing consumer demand for high quality, freshlike, safe foods that are minimally processed, incited the industry to continuously improve the existing technologies and gave birth to many research efforts on novel technologies (Ludikhuyze et al., 2003), such as high-pressure processing. In addition to food preservation, high-pressure treatment (HPT) may result in food products acquiring novel structure and texture, and hence can be used to develop new offers or increase the functionality of certain ingredients (Saraiva et al., 2002; Castro et al., 2006). The application of high pressure leads to rearrangement in the tissue architecture, which may result in increased extractability, even at ambient temperature. The combination of high pressures and lower temperatures could become a viable alternative to some current industrial practice (Rastogi et al., 2007). The inclusion of HPT as a preceding step of other unit operations, such as solid-liquid extraction, is being considered as one of the emerging possibilities in the path leading to novel products and new process development opportunities (Garcia et al., 2001). When applied as a pre-treatment, it may promote higher solute extractability and increase extraction kinetics, which ultimately reduces processing times.

* Corresponding author: Tel. +351 234 370 716; E-mail: jorgesaraiva@ua.pt.

Grape seed (*Vitis vinifera*) is a well known oilseed crop and a major by-product resultant from the wine industry, containing typically between 7-15% (w/w) of oil (Gomez, et al. 1996). It is also an appealing product due to its large availability, as a major by-product resultant from the wine industry. The aim of this study is to investigate and improve the yield of grape seed oil extraction by applying an HPT prior to supercritical fluid extraction (SFE) with CO_2.

2. Experimental Procedure

2.1. Processing of solid plant material

Seeds were collected from grapes (*Vitis vinifera*) of the red variety *Touriga Nacional* harvested at technological maturity, provided by a local wine industry (*Caves* Messias, Anadia, Portugal) during September 2007. Seeds were collected during transfer of the musts in wine fermentation, and separated from pulp and skins by decantation and sieving. A first wash removed immature grains floating at water surface. Subsequently, the seeds were submitted to several washes with water (200 g/L) under gentle stirring with a magnetic bar at 4 °C during a minimum of three days, with two water exchanges per day, until a minimum constant turbidity was observed. The purified seeds were finally washed with ethanol, air dried at room temperature, and stored at 4 °C until use. Finally, milling was carried out on a domestic coffee mill, and the particles classified in a standard sifter with several mesh sizes.

In this work, to ensure the biomass used in different runs had the same size distribution, the extractor load was prepared by combining fixed masses of crushed seed from each mesh interval. Specifically, a mean particle diameter $\bar{d}_p = 1.3$ mm was adopted, being calculated by Sauter's equation (Povh et al., 2001) to a set of fractions within [0.71;1.4]:

$$\bar{d}_p = \frac{1}{\sum_{i=1}^{k} \frac{\Delta m_i}{d_{p_i}}} \qquad [1]$$

where d_{p_i} is the size of sieve i, which retains a mass of solid Δm_i.

2.2. High pressure treatment

Prior to both conventional Soxhlet and supercritical fluid extractions of grape seed oil, the HPT was applied. It has been accomplished in a Hydrostatic press (Unipress Equipment, Model U33, Poland). Experiments were conducted at several pressures during 15 min at 22°C. The milled seed was previously subjected to an overnight water floating (2 mL_{water}/g_{seed}) and only then subjected to the HPT. After the samples were freeze dried.

2.3. Soxhlet extraction

The yield of grape seed oil extraction was assessed by conventional Soxhlet carried out with 150 mL of *n*-hexane in a Soxhlet apparatus (50 mL capacity; 23×100 mm cartridge) during 4 h. The mass oil was determined gravimetrically after solvent evaporation. Furthermore, to ensure that the resultant oil carries no water, the extracted samples were passed over sodium sulfate anhydrous under vacuum in a G1 sintered glass filter, and evaporated in a rotary evaporator at 30 °C. The oil was then transferred to speed-vacuum tubes and dried by centrifugal evaporation. The yield of the process is expressed as the mass of oil extracted from 100 g of dried grape seed. The results thus obtained were assumed as reference values, being used for comparison with those achieved by SFE. The yield of the process $(\eta,\%)$ was expressed as the mass of oil extracted from 100 g of dried grape seed.

2.4. Supercritical fluid extraction

The experiments of SFE of grape seed oil were carried out in a semi-batch extraction apparatus built/assembled at the University of Aveiro. It has been operated with nearly 65 g of crushed grape seed, a CO_2 flow rate of 0.6 kg h^{-1}, at 180 bar and 40°C. Two sets of experiments were carried out, using treated and untreated samples for comparison.

3. Results and discussion

The results obtained by conventional Soxhlet confirmed that the average particle diameter has a strong effect on the yield of the grape seed oil extraction. Figure 1 shows an increase in the oil recovery of 128% when d_p is reduced from range 1-1.4 mm $(\eta = 6.7\%)$ to $d_p < 0.5$ mm $(\eta = 15.3\%)$. These determinations evidenced it is fundamental to ensure that the same size distribution is being processed to allow comparisons between them. Therefore, the procedure based on Eq.1 and described in section 2.1 was always carried out in this essay, and the average value $\bar{d}_p = 1.3$ mm has been adopted and fixed since then.

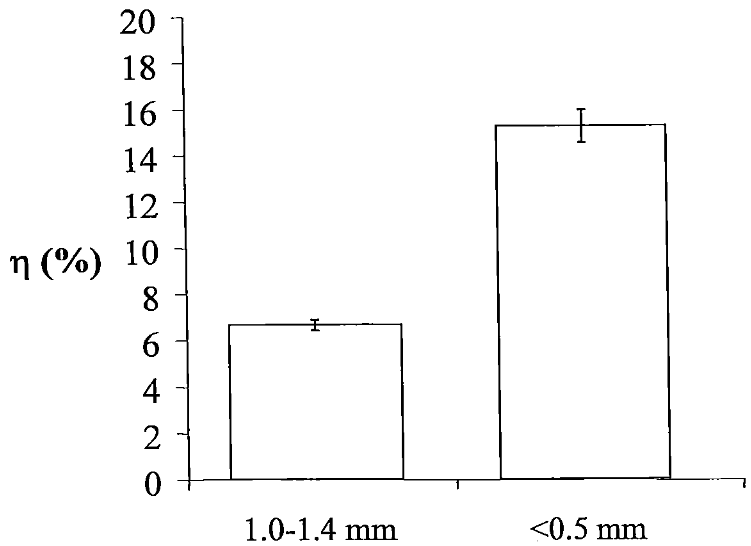

Figure 1. Extraction yield (%) against granulometry of crushed seed particles.

Introducing HPTs prior to conventional Soxhlet extractions, slightly lower recoveries were obtained by increasing the operating pressure, P, (Figure 2). The lowest pressures applied (1000 and 3000 bar) produced nearly the same yields, $\eta = 6.5\%$ and 6.4%, respectively, while $P = 5000$ bar decreases oil removal to 5.8%. These observations correspond to a global variation of 15.5%, and suggesting that pressure treatment change the oil availability, givin rise to lower yields.

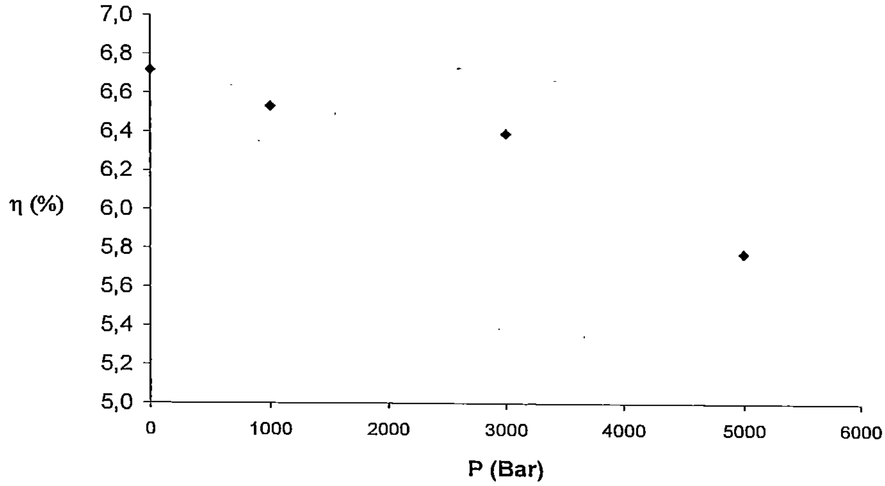

Figure 2. Extraction yield (%) as function of the operating pressure of several high pressure treatments, for $\bar{d}_p = 1.3$ mm and $t = 15$ min.

In Figure 3 cumulative curves for grape seed oil extraction with supercritical CO_2 are plotted, for the operating conditions of 180 bar and 40°C, and $\bar{d}_p = 1.3$ mm. In abscissas the ratio of solvent consumption to seed charge $\left(m_{CO_2}/m_{seed}\right)$ is presented. The two cumulative curves correspond to measurements achieved with untreated samples and with high pressure pre-treated biomass at $P = 1000$ bar during 15 min. These results exhibit the characteristic behaviour associated to seed oils (e.g., Sovova et al., 1994): a first equilibrium and film controlled period approximately linear, followed by a second diffusional controlled period with more asymptotic behaviour.

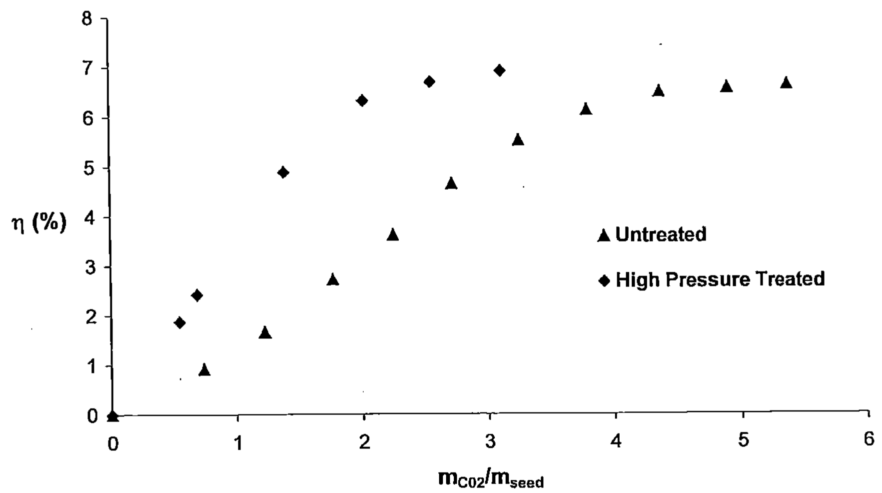

Figure 3 – Effect of HPT ($P = 1000\,\text{bar}$; $t = 15$ min) upon the cumulative extraction curves obtained by SFE of grape seed oil at 40°C and 180 bar, for $\bar{d}_p = 1.3$ mm.

Figure 3 corroborates previous finding by Soxhlet experiments, *i.e.* HPT does not increase the final yield, as both curves tend nearly for the same plateau. Nonetheless, a distinct behaviour was observed during extraction, since the curves are different for the intermediate conditions. Such fact shows that the extraction kinetic is faster for the treated samples. It is worth noting that the CO_2 consumption necessary to achieve complete oil removal almost doubles from pre-treated $\left(m_{CO_2}/m_{seed} \cong 3\right)$ to untreated $\left(m_{CO_2}/m_{seed} \cong 5.5\right)$ samples. In terms of processing time, it additionally implies that only half extraction time is need to accomplish total oil removal.

4. Concluding remarks

In this work, the effect of a high pressure pre-treatment upon the yield and efficiency of grape seed oil extraction has been evaluated.

Experiments show a slight impact of HPT on the final yield up till 3000 bar performed during 15 min. Nonetheless a weak yet consistent decrease has been observed with increasing operating pressure, which may suggest possible changes on the oil availability from the biomass.

The comparison between cumulative curves of the SFE of treated and untreated grape seed evidenced faster kinetics in the former case, despite final yields are approximately the same. Therefore, combining HPT and SFE we may reduce CO_2 consumption to reach the same extractability under optimized operating conditions.

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