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Optimization of oil yield and oil total phenolic content during grape seed cold screw pressing

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ABSTRACT

Grape seed oil being industrially obtained by solvent extraction, in this study we investigate oil extraction from grape seeds by cold screw pressing, as an alternative extraction process.

Effect of raw material and process parameters were evaluated using a 12 experiments Taguchi experimental design. Variables were (1) type of grape seeds, (2) preheating temperature (90 and 120 °C), (3) screw rotation speed (40 and 70 rpm) and (4) die diameter (10 and 15 mm). The type of grape seed was the most influencing parameter on the studied responses. Screw rotation speed and die diameter only affected the oil yield. Maximum oil yield was observed for type 1 grape seeds (64.3%, o/o). Total oil polyphenol content was also maximal for type 1 seeds with up to 121 mg GAE/kg of oil. Maximum oil yield was 57.3% and 58.8% (o/o) for type 2 and type 3 grape seed respectively and total polyphenol oil content was below 90 mg GAE/kg for these two types of seeds. Additional experiments were carried out on type 1 seeds to enhance oil yield and oil polyphenol content. Maximal oil yield achieved was 73% (o/o) and total oil polyphenol content was increased up to 26% (153 mg/kg of oil) on the maximum yield obtained with Taguchi design.

1. Introduction

Worldwide production of grape (*Vitis vinifera* L.) is estimated to 66 million tons in 2009 (FAOSTAT, 2011). The major use of grape is wine making (from 70 to 80%), the rest being used for fruit consumption and nutraceutical or pharmaceutical uses. Grape pomace, the residue of wine processing, accounts for 20% of grape (w/w). It is composed of seeds, 38–52% on a dry matter basis, but also of stems, pulp and skin (Maier et al., 2009). The high oil and phenolic content of grape seeds offers alternative valorization pathways for these by-products (Baydar et al., 2007).

Grape seed contains from 8 to 20% of oil (dry basis). Linoleic (58–78%, C18:2*n*−6) and oleic (10–20%, C18:1*n*−9) fatty acids are the two major fatty acids present in this oil (Crews et al., 2006; Lutterodt et al., 2011). Saturated fatty acids (C16:0, C18:0, C20:0) accounting for 10% of total fatty acids, lead to an unusual high smoking point (190–230 °C, according to Morin (1996)). Such fatty acid profile renders this oil suitable for edible purposes. Additionally, this oil is reported to contain minor components such as phenolic compounds (between 59 and 360 mg gallic acid equivalent (GAE)/kg) (Maier et al., 2009; Bail et al., 2008). Polyphenols identified in grape seed are catechin, epicatechin, trans-resveratrol and procyanidin B1 (Maier et al., 2009; Pour Nikfardjam, 2001). These phenolic compounds are reported to be involved in a wide range of biological activities (Siger et al., 2008) but are mostly known for their antioxidant properties. Given the unsaturation level of grape seed oils, those compounds are beneficial for oil conservation, by increasing oil oxidative stability (Siger et al., 2008).

After seed separation from grape pomace (by drying and screening), oil recovery from grape seeds is achieved. At an industrial scale, grape seed oil is extracted by continuous mechanical expression (screw pressing) and/or by a solvent process (Laisney, 1996). To ensure a higher oil quality, mechanical pressing is preferred (lower process temperature, no solvent) although lower

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yields are achieved (from 55 to 95% according to the processed raw material (Singh and Bargale, 2000)). Screw pressing has been studied for a large variety of oilseeds (linseed, canola, crambe, chiaseeds (Savoire et al., 2013)). Considering oil yield, the studies demonstrated that factors contributing to increase pressure and temperature in the screw barrel have a major positive influence on oil yield. Those factors can be modulated by impacting on the diameter of the restriction die located at the meal discharge and by screw rotation speed (the lower the speed and the smaller the restriction opening, the higher the yield) (Savoire et al., 2013; Vadke and Sosulski, 1988). Suitable pretreatments of seeds can also enhance oil yield: flaking, cooking, moisture conditioning. The latter has a key role in expression as an optimal moisture content regarding oil yield can be found depending on seed variety (e.g. linseeds 9–11% (db), rapeseed 5% (db), crambe seed 3–4% (db) (Savoire et al., 2013)). Considering oilseed thermal treatment such as cooking, although it allows an increase in oil yield, it could be responsible for oil degradation. So cold-pressing is usually preferred to retain more health beneficial components such as antioxidants increasing oil nutritive value (Maier et al., 2009; Lutterodt et al., 2011; Siger et al., 2008).

Few studies report the effect of screw pressing parameters on oil quality (free fatty acids content, peroxide value, oxidative stability). However relatively high temperatures can be reached during the process: 60–68 °C according to Maier et al. (2009) which can impact the oil composition and quality.

The aim of our work was firstly to evaluate the impact of process parameters such as die diameter, preheating temperature of the barrel and screw rotation speed, on oil yield and total polyphenol content of cold pressed oil. Process performances were also investigated in terms of press capacity and oil temperature achieved during pressing. Another objective of our work focused on the effect of the raw material on oil yield and polyphenols content of oil obtained by cold screw pressing: three types of grape seeds were thus processed. Finally, a special attention was given to the influence of seeds moisture content. Aiming at optimization, an approach based on a combination of several experimental designs was used.

2. Experimental procedures

2.1. Grape seeds processing

2.1.1. Grape seeds

Grape seeds (three types according to their harvest time and the duration between grape pressing and seed drying) were provided by the Distillerie Jean Goyard (Aÿ, France). Type 1 grape seeds were harvested and stored for 2 weeks before being dried and separated from grape pomace. For types 2 and 3, duration between harvesting and drying was less than 2 days. The grape seeds also differ as they were not harvested at a same date. So the cultivars are different for types 1, 2 and 3 but the grape was harvested in the same region (Champaign area near Epernay, France).

After harvest, seed moisture content was lowered to about 7% (db) by air drying at the Distillerie. Material was kept in a closed bag, at room temperature until screw pressing. Their characteristics are presented in Table 1. Seeds were processed upon reception except for a minor part of the experiments which were carried out on seeds that were pre-equilibrated to various moisture contents as follows: (1) the seeds were first reduced to minimal water content in a ventilating oven at 40 °C. (2) The moisture content was then adjusted to predefined values by sprinkling adequate amounts of water on them in a polyethylene bag, and the bag was agitated by hand. (3) The closed bags were stored at 5 °C for more than 5 days for equilibration. (4) Moisture content was determined after equilibration. 4 kg of seeds were prepared per batch.

Table 1
Characteristics of the three types of grape seeds processed (GAE*: gallic acid equivalent).

	Type 1	Type 2	Type 3
Harvest time (2009)	Week 41	Week 38	Week 40
Duration between pressing and drying	15 days	2 days	2 days
Water content (% dry basis)	7.4 ± 0.3	6.4 ± 0.2	7.1 ± 0.2
Oil content (% dry basis)	13 ± 2	13.5 ± 0.6	15 ± 2
Seed total polyphenol content (% dry basis GAE*)	5.8 ± 0.6	9.5 ± 0.9	9.6 ± 0.3

2.1.2. Screw pressing experiments

Oil expression was carried out on a Komet screw press (S87G model, IBG Monforts, Germany). The length of the press barrel was 95.8 mm, and had a constant internal diameter of 75.1 mm. Four die diameters could be chosen: 8, 10, 12 and 15 mm. An R6 (198 mm length, inner diameter of 36 mm, flight-to-flight distance of 15.5 mm) screw was used for all the experiments. The screw rotation speed could be varied from 20 to 110 rpm. An acquisition software (UTC electronic services, France) was used to monitor oil temperature, masses of crude oil and meal as well as power consumption (power-meter PX120, Metrix, France). Oil temperature was measured (1 °C precision) using a type K thermocouple placed in the closest hole from the screw head.

Before screw pressing experiments, the press head was pre-heated at the desired temperature for 20 min using a temperature-regulated heating ring. Pressing experiments were conducted without external heating (cold pressing). During pressing, grape seeds were fed in the press on demand by gravity through the hopper and seeds level was maintained constant to ensure a constant press behavior. Steady state was assumed as oil and meal flows remained constant (steady state was achieved after 5 min pressing for all experiments). Sampling (oil and meal) was continuously done during the steady state (oil and meal were collected during the 30–50 min of the experiment, then representative samples were further analyzed). Press capacity was determined by addition of oil and meal flows (precision 0.1 g/s). Mass balance was checked by seed weighting before expression and comparison with cumulated oil and meal masses obtained. Power consumption was monitored using a Metrix PX120 power meter. Data obtained were converted into specific mechanical energy (SME) by dividing the average power consumption recorded during experiment by the press capacity.

Crude oils were centrifuged (10 min, 3000 g, room temperature) to separate oil from sediments. Samples of clarified oil, sediments and meal were stored at −20 °C until assessment of total polyphenol content. Oil extraction yield is defined as the ratio of clarified oil recovered from expression to the amount of oil originally present in the seeds. Cake oil content was calculated from oil mass balance.

2.2. Analytical procedures

2.2.1. Chemicals

N-hexane, ethanol, methanol and Na_2CO_3 as well as Folin–Ciocalteu reagent (VWR, France) were used. Water used was obtained from the milli-Q water purification system (Millipore Corporation, USA). Gallic acid (Sigma–Aldrich, France) standard served as standard for quantification of total polyphenol.

2.2.2. Determination of water content

Water content of grape seeds was determined by dehydration at 103 °C according to the French standard procedure (AFNOR, 2010). Analysis was made on 5 g of grinded sample, weighted with an accuracy of 0.1 mg. Results are expressed as the ratio of water loss per gram of dried sample. Determination of water content was performed in triplicate.