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Influence of canola seed dehulling on the oil recovery by cold pressing and supercritical CO_2 extraction

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ABSTRACT

Dehulling canola seeds provides an enriched cake in oil and proteins leading to an improved meal quality after oil extraction. The aim of this work was therefore to explore the impact of dehulling pre-treatment on both oil recovery and quality, using cold pressing followed by supercritical CO_2 extraction. For this purpose, whole canola seeds, dehulled seeds, and reconstituted mixtures of 5% and 10% hulls were used to recover canola oil. After 1 h pressing, the obtained results showed that the lowest oil yield ($\approx$37%) was obtained using dehulled seeds, indicating that dehulling adversely affects the efficiency of cold pressing. After pressing, remaining oil in press cake was extracted by supercritical CO_2. After process optimization, the optimal parameters providing the maximum extraction yield ($\approx$74%) were found at 40 °C temperature, 35 MPa pressure, 8.5 kg/h CO_2 flow, and 10 min of conditioning. Using these conditions for grinded seeds, the combined process allowed a quasi-total oil recovery (95 ± 3%) and showed similar oil quantities extracted from either whole seeds, dehulled seeds, or reconstituted mixtures, demonstrating thus the efficiency of the proposed process. On the other hand, total phenolic compounds content in oil was reduced upon dehulling, and results show that the oil obtained by supercritical CO_2 extraction was about twice enriched in phenolic compounds when compared to that obtained by mechanical pressing.

1. Introduction

Canola seeds (rapeseeds with low erucic acid and glucosinolate contents) contain high amounts of oil (38–50%) and proteins (36–44%) (Xu and Diosady, 2012). Canola oil is characterized by its low content of saturated fatty acids, whereas the residual meal represents a valuable source of proteins mainly used for animal feed (Rempel and Scanlon, 2012). The industrial oil seeds transformation process involves pre-treatment (crushing/flacking and cooking), mechanical pressing, and *n*-hexane extraction to recover the residual oil.

Replacing *n*-hexane with non-toxic solvents (*i.e.* supercritical CO_2 (sc-CO_2)), for oil extraction from rapeseed has been widely investigated (Cvjetko et al., 2012; Dunford and Temelli, 1996; Moquin et al., 2006; Pederssetti et al., 2011; Przybylski et al., 1998; Uquiche et al., 2012). This interest is mainly related to the absence of toxicity and explosion risks, low operational temperature, cheapness and the renewable character of CO_2 (Kazmi, 2012). Sc-CO_2 extraction can be used directly after rapeseed cruching (Martin et al., 2015) or flacking (Boutin and Badens, 2009). However, the energy consumption for sc-CO_2 extraction from rapeseed is rather important (10.2 kWh/kg of oil) (Martin et al., 2015), and the global process should be optimized. Cold pressing has important industrial advantages (i.e. low energy consumption and low equipment cost). Therefore, it is advantageous to express the major quantity of canola oil mechanically with low energy consumption and extract the remaining part of oil using sc-CO_2.

Dehulling can be used to remove the fibrous envelopes of the seeds in order to obtain "kernels"; the fraction concentrated in oil and proteins, and "hulls"; the fraction containing mostly fibers. Thus, dehulling leads to a meal of high proteins content highly sought for animal feed (Usha et al., 2012). Hulls can be used as fuel for the energy supply of the oil mill (Kazmi, 2012). The growing global demand for proteins and the recent success of dehulled sunflower meals lead to reconsider the potential utilization of dehulled canola seeds, containing 10–18% hulls. Few studies have been cited in literature concerning oil extraction from dehulled canola seeds. In fact, Schneider (1979a,b) showed that rapeseeds

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dehulling before pressing has the advantage of keeping the screw press temperature below 40 °C, thus limiting the seed enzyme activities, and reducing the transfer of undesired compounds from seeds to the oil (Schneider, 1979a,b). More recently, it has been reported that the oil obtained from dehulled seeds had better sensory characteristics and lower amount of waxes compared to whole seed expressed oil (Gupta, 2012). This statement concurs with previous studies showing that fats extracted from Canadian and Australian rapeseed hulls contain waxes and polar compounds that can precipitate during cold storage (Liu et al., 1996). Other advantages of dehulling reported in literature are the reduction of refining costs, and the improvement of the taste and smell, generating milder oil than that of conventional virgin, cold-pressed canola oils (Usha et al., 2012). This behavior was demonstrated by Zhou et al. (2013), who studied the effect of dehulling on the flavor characteristics of cold-pressed rapeseed oil. Interest has been also extended to the generated meal from dehulled seeds. In fact, the nutritive value and applicability of dehulled protein-rich rapeseed meal diet for piglets was studied by Danielsen et al. (1994). Moreover, the effect of dehulling on the composition of antinutritive compounds in various cultivars of rapeseed was studied (Matthäus, 1998). More recently, Carré and co-workers (2015) showed that oil extraction from dehulled rapeseeds has advantages comparatively to the conventional process if most of the oil containing in kernels is recovered. In fact, the impact of dehulling on oil extraction is still insufficiently explored and should therefore be investigated.

In this work, whole canola seeds, grinded seeds, dehulled seeds, and reconstituted mixtures of dehulled seeds and either 5% or 10% hulls were defatted using cold pressing followed by sc-CO_2 extraction. The impact of seeds dehulling and grinding on the yields of mechanical expression and sc-CO_2 extraction were evaluated. Consolidation coefficient b for pressing, and diffusion coefficient D for sc-CO_2 extraction were estimated based on simplified mathematical models. The influence of sc-CO_2 extraction parameters (pressure, temperature, CO_2 flow rate and conditioning time) was studied and the optimal process conditions were determined. Phenolic compounds were evaluated for oils obtained after cold pressing and sc-CO_2 extraction of whole and dehulled canola seeds.

2. Materials and methods

2.1. Chemicals

Hexane, methanol, Folin-Ciocalteu reagent and sodium bicarbonate (Na_2CO_3) were obtained from Fisher Scientific (Illkirch, France). Tween 20 was purchased from Sigma–Aldrich (Saint-Quentin Fallavier, France).

2.2. Canola seeds and hulls

Whole canola seeds, dehulled seeds and hulls were provided by the Technical center for oilseeds "CETIOM" (Pessac, France). According to the supplier, the tested canola seeds contain an average of 13.5% hulls (w/w). Reconstituted mixtures of 5% (w/w) and 10% (w/w) hulls, mixed with dehulled seeds, were prepared. Seeds were grinded using a coffee grinder, then sifted through 630 μm sieve, in order to study the influence of this pre-treatment on the oil extraction yields. The average particle size was determined using a "Malvern Mastersizer 2000" granulometer.

2.3. Oil quantification

Oil content in samples was determined using Soxhlet's method as previously reported by AFNOR (1981). Briefly, 5 g of grinded plant materials were washed with 250 mL hexane for 24 h. Oil content was determined by measuring the difference of weight before and after the extraction. Oil extraction yield (Y) was determined as follows:

$$Y,\% = \frac{\textit{mass of recovered oil}}{\textit{oil content in sample}} * 100 \tag{1}$$

2.4. Protein quantification

Proteins content in the different plant materials was determined as described by Dumas (1831).

2.5. Oil recovery

Canola oil was first mechanically expressed from whole, grinded or dehulled seeds using cold pressing. The residual oil was extracted from obtained press cake using sc-CO_2.

2.5.1. Cold pressing

Hydraulic press (Creusot-Loire, France) was used for expression of canola oil. 300 g of whole or pre-treated seeds were pressed under a constant pressure of 10 MPa for 1 h. The mass of oil was continuously monitored using electronic balance (Mettler Toledo), and the press cake thickness (h) was recorded during the experiment. The resulting press cake was stored at 4 °C until proceeding to sc-CO_2 extraction.

2.5.2. Supercritical CO_2 extraction

The remaining oil in press cake was extracted using sc-CO_2 pilot scale equipment (SEPAREX) (Fig. 1). The extraction protocol was adapted from Koubaa et al. (2015a) with modifications. The operational conditions were first optimized in order to maximize the oil extraction yields. 50 g of kernels press cake (bed diameter = 10 cm, height ≈ 5 cm) were introduced into the extraction vessel (**Ev**), which was first pre-heated at the desired temperature (two levels of extraction temperatures were tested: 40 °C and 60 °C). All exit valves as well as the recirculation valve (**Rv**) were closed then the **Ev** was pressurized at the desired pressure (four pressures were tested: 15 MPa, 25 MPa, 35 MPa and 45 MPa) using the CO_2 pump. The CO_2 pressure was manually maintained in the **Ev** by a backpressure valve (**Bv**) located between the extractor and the cyclonic separators (**S_1** and **S_2**). The influence of conditioning time (0 and 10 min) with sc-CO_2 was also studied. After reaching the desired pressure in the **Ev**, the **Bv** and **Rv** were either directly opened (without conditioning), or maintained closed for 10 min conditioning time. The extraction was then maintained for 6 h, under a continuous flow of CO_2 (three CO_2 flow rates were tested: 2 kg/h, 8.5 kg/h and 15 kg/h). **S_1** and **S_2** pressure was maintained at 6 MPa with 40 °C temperature. The extracted oil was collected periodically during the different experiments from **S_1** and **S_2**, then stored at −20 °C until analysis. The extraction kinetics were determined and the diffusion coefficients were calculated. After determining the optimal extraction conditions with sc-CO_2, using non-grinded dehulled seeds, the selected parameters were tested for the other seed samples.

2.5.3. Press cake porosity

The porosity of the cold press cakes was determined using a Micromeritics AccuPyc 1330 pycnometer.

2.5.4. Consolidation coefficient

The consolidation performance of mechanical expression has been successfully evaluated using Eq. (2) (Leclerc and Rebouillat,