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Supercritical extraction of oil seed rape: Energetic evaluation of process scale

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ABSTRACT

This study focuses upon the supercritical CO_2 extraction of oil seed rape (*Brassica napus*) and the energy consumption associated with it in two different scales (1 L and 2 × 16 L). Experiments were carried out to determine the influence of pressure, temperature and flow rate on the extraction yield. The yield varied from 37 to 97% of the one for *n*-heptane. The energy consumption in both extraction plants allowed an analysis of the different components involved in the extraction process, namely pumping, heating and cooling. This energy consumption was analysed depending on the amount of CO_2 used, so the calculations can be extrapolated to any supercritical fluid extraction process undertaken in those plants. In the particular case of the supercritical extraction of oil seed rape, the best conditions in our experimental range were achieved at 55 MPa and 35 °C, yielding 100.3 g of oil per kW h. This yield was comparable to that obtained in the pilot plant of 97.4 g oil/kW h. An accurate energetic evaluation of the extraction process at different scales has provided further evidence to encourage the change to supercritical fluid extraction as an economically viable industrial process.

1. Introduction

The supercritical fluid extraction of oil seed rape is potentially a large scale application of this technology that has been thoroughly studied [1–6] and modelled [7,8], but still has not been industrially applied.

Boutin et al. [1] studied the extraction at pressures ranging from 15 to 45 MPa and temperatures of 35–75 °C during 20–120 min with 8–19 kg/h CO_2 flowrate in a 3 L extractor. Their yields varied from 0.3% to 89.8% compared to an organic solvent extraction, concluding that the main parameter that influenced the extraction was the pressure, while temperature had a small influence (beyond the retrosolubility point). Boutin et al. [8] also developed the modelling of the extraction in different conditions (30–34 MPa, 45–70 °C, 5–25 kg/h of CO_2).

The traditional extraction process includes mechanical pressing extraction and extraction with organic solvents, while the solid residual matrix is used as animal feed. The organic solvent is normally hexane, leading not only to toxicity problems in both the residual matrix and in the extract, but also needing a further purification step. Hexane has been recognised as a hazardous air pollutant by the US EPA and it has been reported by the EPA Toxic Release Inventory that more than 20,000 t of hexane are released to the atmosphere each year from the extraction of vegetable oils [9]. Although extraction with hexane has a lower unit cost than extraction with supercritical CO_2 ($scCO_2$), production costs increase as extra refining steps and energy input is needed to reduce solvent residues to meet legally enforced maximum residue levels and to recover the solvent [10].

The advantages of supercritical fluid extraction against conventional extraction methods are clear: shorter extraction times, avoidance of toxic solvents and solvent residues and purity of the final product. In particular, $scCO_2$ is ideal due to its non toxic, non flammable nature and affordable processing conditions. On the other hand, since its critical temperature is low, the thermal degradation of thermolabile compounds can be avoided. Several oleaginous plants have been extracted with $scCO_2$ such as soybean, corn, wheat germ, sunflower seeds, safflower seeds or peanuts [11]. Some other supercritical extractions have been even economically evaluated with supercritical fluids [12]. However, in those economic evaluations, the energy is implemented as a variable, being simulated rather than experimentally gathered [13–15]. Furthermore, the energy consumption is often treated as a whole, being a black box where only a final figure is obtained. Being able to divide the energy consumption into the different components of a system will allow a better understanding of it, leading to an easier optimisation of the process. Rodríguez-Meizoso et al. [16] also include the electricity divided into the different components of the

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supercritical process, but based on the theoretical consumptions depending on the specifications of each of the components.

The main industrial drawback of the supercritical extraction is the reluctance of industry to adopt new technologies, mainly in an area involving fluids at high pressure. This technophobia combines with the current economic constraints on capital investment creating a difficult barrier to surpass. However, an accurate energetic evaluation of the extraction process at different scales could provide the needed information to encourage the change to supercritical technologies.

If the consumed energy per kg of CO_2 is used as a measuring unit, the energetic evaluation of any supercritical extraction in a certain extraction plant can be taken into account. Furthermore, if the yield is then introduced as a variable for a specific set of parameters, the energetic viability of such process can be investigated.

The aim of this work is to evaluate the supercritical CO_2 extraction of rape seed oil and its energy consumption at two different scales. The influence of pressure (25–55 MPa), temperature (35–75 °C) and flow rate on the extraction yield and energy consumption was analysed.

2. Materials and methods

2.1. Raw material

Oil seed rape seeds, *Brassica napus* (Syngenta variety NK Grandia), were harvested from farms in north Wales (Flintshire, Denbighshire and Anglesey) and west Cheshire and supplied by Blodyn Aur (Corwen, Wales, UK). Seeds were crushed twice with a roller mill (BDC Systems Limited, United Kingdom) with a roller gap of 1 mm. The seeds were freshly crushed prior to each of the extractions to avoid oil degradation. CO_2 was provided by BOC gases.

2.2. Extraction equipment

The experiments were carried out in the CO_2 laboratory of the Biocomposites Centre in Bangor University. The optimisation of the process was accomplished in the 1 L extraction plant while the scaling up was accomplished in the 2 × 16 L pilot plant.

The optimisation of the extraction was carried out in a 1 L plant extractor (Thar Technology, Pittsburgh, PA, USA) with two separators (0.5 L and 0.25 L capacity) and computerised control of temperature and pressure. Its working limits are 60.0 MPa and 90 °C. The heating in the system is accomplished by electric heating elements while the cooling is accomplished by a Neslab RTE10 bath. The sapphire piston pump (P-200A) works with a maximum CO_2 flow rate of 12 kg/h. The energy consumption is recorded by two systems: plug-in monitors (2000MU, Prodigit Electronics) and a real time energy data capture system. The latter is provided by Enistic Energy Management Systems, which uses individual current clamps and sensors in conjunction with a smart box and controller. The controller passes the data to the Enistic servers where it can be accessed online [17] and further analysed.

The scaling up of the extraction was conducted in a 2 × 16 L extractor pilot plant (Separex, France) with two separators (1 L capacity) with computerised control of temperature and pressure and automatic sampling. Its working limits are 70.0 MPa and 80 °C and it has a recirculating system for the CO_2. The heating in the system is accomplished by a water recirculating system powered by a heater (Vulcatherm H2100). The cooling is accomplished by a chiller (Hitema C2000). The metallic piston pump (P200 LGP D26) works at a maximum flow rate of 50 kg/h. The energy consumption and metrics of the different components is collected by the software.

Pressures ranged from 25.0 MPa to 55.0 MPa and temperatures from 35 °C to 75 °C. CO_2 flows ranged from 2.4 kg/h to 7.2 kg/h for the small plant pump and 40 kg/h for the pilot plant pump. The separators conditions in the small plant were: separator 1, 1.0 MPa and 45 °C; separator 2, atmospheric pressure and 25 °C. The separator conditions in the pilot plant were: separator 1, 8.0 MPa, 45 °C; separator 2, 5.0 MPa, 25 °C. The separator conditions are set to obtain a maximum recovery of the oil in the first separator, achieved by a marked decompression. While a total oil recovery in separator 1 was achieved in the small scale plant, a small amount of oil was recovered in separator 2 in the pilot plant. The separator conditions in the pilot plant are different than in the small scale to allow a recirculation of the CO_2 (a minimum of 5.0 MPa are required at the exit of separator 2). However, these conditions avoid any recirculation of the oil through the system.

The experimental procedure is the following. The extractor is packed with crushed seeds (0.66 kg and 9.78 kg in the small and the pilot plant, respectively). $ScCO_2$ enters the extractor in supercritical condition due to the chiller that allows pressurisation of the liquid in the pump and due to the inline pre heater before entering the extractor. Once the pressure and working temperatures are reached in the extractor and separators, the extraction time begins. Pressure in the vessels is controlled by the automatic back pressure regulators. The extracted oil is collected every hour (in the small scale) or every 60 s (in the pilot plant, thanks to the automatic sampling). The extraction was halted when the experiment was entering in the asymptotic phase, coinciding with a recovered amount of oil in the hourly fraction smaller than 5% of the extractable oil.

Soxhlet extractions with *n*-heptane were carried out as a comparison.

3. Results and discussion

3.1. Extraction experiments

A set of 14 experiments was planned in the range of 25–55 MPa, 35-–75 °C and 2.4–7.2 kg/h in the 1 L extraction plant to determine the best extracting conditions. The experiments can be seen in Table 1 together with their yields.

The yields for the supercritical experiments, ranging from 38 to 96%, have been expressed as percentage of yield of a Sohxlet *n*-heptane extraction. This *n*-heptane yield was 46 g oil/100 g of crushed seeds. The supercritical extraction curves are shown in Fig. 1, where the cumulative extraction yields are plotted against the amount of CO_2 used.

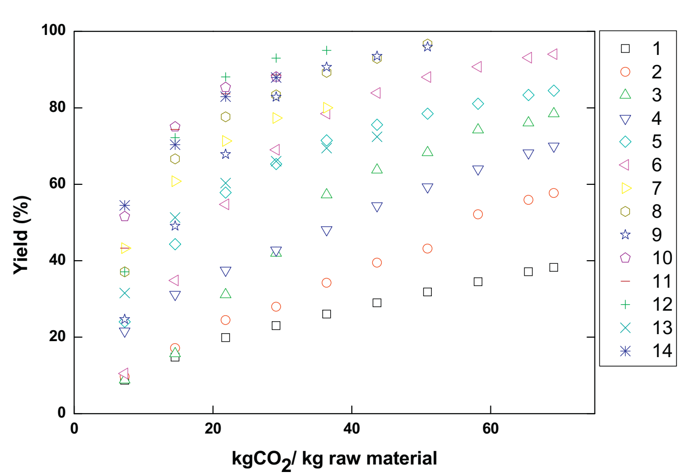

Fig. 1. Extraction curves for the lab scale experiments. kg CO_2/kg raw material vs yield (%). Numbers correspond to the experimental conditions in Table 1.