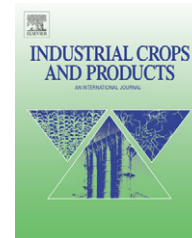


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Aqueous extraction of residual oil from sunflower press cake using a twin-screw extruder: Feasibility study

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ABSTRACT

The objective of this study was to evaluate the feasibility of an aqueous process to extract the residual oil from sunflower press cakes using a co-rotating twin-screw extruder. Two different configurations were tested: the expression from whole seeds followed by the aqueous extraction, in two successive apparatus or in the same one. For the aqueous extraction stage, the oil yield depended on the operating conditions including screw rotation speed, screw profile, and inlet flow rates of press cakes and water. Liquid/solid separation required the addition of a lignocellulosic residue (wheat straw), upstream from the filtration zone. However, even with maximum fiber inlet flow (around 20% of the inlet flow rate of the solid matters for the highest amount of wheat straw), drying of the cake meal did not improve. The lixiviation of the material was also incomplete. Oil yield was better when the expression and the aqueous extraction were conducted in the same extruder. For all the trials carried out using such a configuration, the corresponding cake meal contained less than 10% residual oil, and the total oil yield was 78% in the best operating conditions. Nevertheless, the contribution of the aqueous extraction stage was extremely limited, less than 5% in the best trial, partly due to a ratio of the water to the press cake too low. For the aqueous extraction stage, the oil was extracted in the form of an oil-in-water emulsion whose stability was minimized because of its low proteins content due to their thermo-mechanical denaturation during the expression stage.

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1. Introduction

Sunflower (*Helianthus annuus* L.) is cultivated for its seeds' high oil content. Oil represents up to 80% of its economic value. The industrial processes for oil production consist of four successive stages: trituration, pressing, extraction of the residual oil using hexane and refining (Isobe et al., 1992; Rosenthal et al., 1996). The extraction yields are close to 100% with very good oil quality. However, the use of hexane to remove oil from the press cake is an increasingly controversial issue and

could be prohibited due to its carcinogenicity (Galvin, 1997). Consequently numerous solvents have been considered.

Water is an interesting alternative medium (Hagenmaier, 1974; Southwell and Harris, 1992). In the aqueous extraction process, the oil, being immiscible with water, separates readily from the extract. The fine crushing of the seeds is the first stage in cell disruption. It facilitates the diffusion of the soluble compounds and the release of the oil. Liquid/solid separation by centrifugation produces three fractions: the hydrophobic phase (oil-in-water emulsion), the hydrophilic phase and the

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insoluble phase (Rosenthal et al., 1996; Mechling, 2002; Evon et al., 2007). The oil is then recovered after demulsification by alcohol extraction. Besides, aqueous extraction of oil can be regarded as a process primarily aimed at solubilizing proteins which results in the release of the oil (Rosenthal et al., 1996).

The oil yield and the protein yield are 86% and 85%, respectively, when the aqueous extraction is carried out in the next operating conditions: dispersion of the ground seeds in water inside a batch reactor, extraction by simple stirring at room temperature, 45 min for the extraction duration, 1:10 for the ratio of the seeds to the water, 10 for pH (Hagenmaier, 1974).

Because all the cotyledon cells are not efficiently ruptured during the extraction process, a fraction of the oil remains trapped in the cellular matrix and oil yield is so limited. Extraction efficiency can be improved by mechanical lysis of the cells in a twin-screw extruder (Isobe et al., 1992; Bouvier and Guyomard, 1997; Lacaze-Dufaure et al., 1999a,b; Amalia Kartika et al., 2005a,b), including when the twin-screw extrusion technology is used to conduct the direct extraction of oil from whole sunflower seeds according to an aqueous extraction process (Evon et al., 2007).

Co-penetrating and co-rotating twin-screw extruders are most common (Dziezak, 1989). A very wide choice of screw elements is available. The screw elements affect different functions such as conveying, heating, cooling, shearing, crushing, mixing, chemical reaction, liquid/solid extraction, liquid/solid separation, and drying (Rigal, 1996).

The screw profile (or screw configuration) is defined by the arrangement of different characteristics of screw elements (pitch, stagger angle, and length) in different positions and spacings. It is the main factor influencing performance (product transformation, residence time distribution, and mechanical energy input) during extrusion processing (Gogoi et al., 1996; Choudhury et al., 1998; Gautam and Choudhury, 1999a,b).

The forward pitch screws mainly ensure conveying action. The monolobe paddles exert a radial compression and shearing action on the matter but have limited mixing ability. In combination with forward pitch screws, the bilobe paddles exert significant mixing, shearing, conveying, and axial compression actions on the matter. The bilobe paddles are favourable to intimate mixing required in the liquid/solid extraction of soluble constituents in the cell structure. Finally, the reversed pitch screws carry out intensive shearing and considerable mixing on the matter, and exert a strong axial compression in combination with forward pitch screws (Rigal, 1996). The reversed pitch screws are frequently used to place pressure on the matter, which is essential for the separation of liquid and solid phases by filtration.

When oleic sunflower oil is expressed, a longer reversed pitch screw improves oil yield, which can rise 80% in the optimized operating conditions (Lacaze-Dufaure et al., 1999a). When two reversed pitch screws are used, the oil yield increases with the distance between them and with the decrease of the screw pitch (Amalia Kartika et al., 2005a). The best oil yield obtained with such screw configuration is close to 70% but the introduction of a second filtration zone improves it to 85% (Amalia Kartika et al., 2005a). The corresponding press cake contains less than 13% residual oil and the quality of the oil produced is similar to that of classic extraction.

Twin-screw extruders can also be used as liquid/solid extractors. The injection of a solvent promotes oil extraction by solubilizing the triglycerides. An oil yield of 80% was obtained with 2-ethylhexanol (Lacaze-Dufaure et al., 1999b) and 85% with a mixture of sunflower methyl esters (Amalia Kartika, 2005).

However, pressing efficiency can be altered by the consistency of the mixture. Liquid/solid separation is more difficult than when the seeds are expressed without injection of a solvent. Thus, the best oil yield is only 55% when the injected solvent is water (Evon et al., 2007). The oil is then extracted in the form of an oil-in-water emulsion. This hydrophobic phase is stabilized by phospholipids and proteins at the interface, which are natural surface-active agents co-extracted during the process. The aqueous extraction also produces a hydrophilic phase. This largest fraction constitutes an aqueous extract of the soluble constituents from the seeds. Its dry matter contains a high proportion of water-soluble proteins, usable for their tensioactive properties after concentration.

Besides the direct aqueous extraction of the oil from sunflower seeds (Evon et al., 2007), this study aimed to show that a co-rotating twin-screw extruder could also be used in an aqueous extraction of the residual oil from press cakes produced after pressing of the whole seeds to improve the total oil yield. Consequently, water would replace hexane used traditionally in the second step of the conventional oil extraction processes.

2. Materials and methods

2.1. Materials

All trials were carried out using a single batch of sunflower seeds (La Toulousaine de Céréales, France). The moisture content of the seeds was $7.49 \pm 0.03\%$ and the lipid content was 50% (Table 1). The lipid extract contained 1.2 g of phospholipids per 100 g of oil. The cellulose, hemicelluloses, and lignins were mainly located in the hulls. The lignocellulosic residue used was wheat straw. Its moisture content was $6.20 \pm 0.02\%$. The wheat straw was cut into small pieces using a hammer mill (Electra VS 1, France) fitted with a 6 mm screen. All solvents and chemicals were analytical grade and were obtained from Sigma-Aldrich, Fluka, Prolabo and ICS (France).

2.2. Twin-screw extruder

Experiments were conducted with a Clextral BC 45 (France) co-penetrating and co-rotating twin-screw extruder. The extruder had seven modular barrels, each 200 mm in length, and different twin-screws which had segmental screw elements each 50 and 100 mm in length. Material (whole seeds or press cakes) was fed into the extruder inlet port by a volumetric screw feeder (Clextral 40, France). Some of the modules were heated by thermal induction and cooled by water circulation. Their number varied from two to four according to adopted shape. One or two filter sections consisting of six hemispherical dishes with perforations 1 mm in diameter were outfitted along the barrel to enable the filtrates containing the extracted oil to be collected. Screw rotation speed (S_s), the seed feed rate (Q_s) or the press cake feed rate (Q_{C1}), and

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