



Extraction of sunflower oil using ethanol as solvent



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ABSTRACT

The ethanolic extraction of oil from sunflower collets was studied and compared with previous data where hexane was used as extraction solvent. First, the extractive power of ethanol was determined by Soxhlet. It gave a higher yield of extracted material, whose content of soluble hexane components (oil phase) was similar to that obtained with n-hexane. When ethanol was used as solvent, 70% less crystallizable waxes and at least 38% more tocopherols and phospholipids were extracted. In addition, ethanol showed great ability to extract sugar, mainly raffinose and sucrose, extracting over 75% of the initial sugar content.

Then, the kinetics of ethanolic extraction was studied at 50 and 60 °C in a batch reactor. At equilibrium conditions, it was observed that extraction could be limited by the solubility of the extractable material. Oil effective diffusivities were $9.94 \cdot 10^{-10}$ at 50 °C and $3.11 \cdot 10^{-9}$ m²/s at 60 °C. From the point of view of the quality of the obtained products, this work demonstrated the feasibility of using ethanol as an alternative solvent to hexane in the oil extraction from sunflower collets.

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1. Introduction

The industrial oil extraction process has not changed much for the last few decades, although there have been some technical developments in optimization and better control. The conventional procedure for sunflower oil extraction involves seed preparation, mechanical extraction (in which a press cake or oilcake containing 16–24% of oil is obtained after continuous pressing), and a final stage which is the solvent extraction from ground oilcakes or expanded material, known as “collets” (porous cylinders obtained from pressed sunflower cakes by expanding). This last stage is commonly achieved by counter-current flow extraction using n-hexane as solvent (Li et al., 2014). The success of the industrial extraction process depends on how fast the compound to be

extracted is dissolved, and on the balance achieved in the solvent phase. Solvent extraction is considered a mass transfer process between a solid phase (insoluble solid matrix), a liquid phase occluded inside the pores within the particle, and the miscella that moves through the spaces between the particles (Cacace and Mazza, 2003). Among the particles, the extracted material diffuses from the occluded liquid phase to the bulk liquid phase.

In the past, some alcohols were considered as possible solvents to extract oils from collets. But eventually, oil yields and some economic aspects favored the selection of hexane, an inflammable petroleum-derived solvent (Lusas et al., 1994). Nowadays it is known that this solvent can be emitted during extraction, being necessary its recovery because it has been identified as an air pollutant since it can react with other pollutants to form ozone and photochemical oxidants (Wan et al., 1995b; Hanmoungjai et al., 2000). Safety, environmental and health concerns have prompted increased interest in alternative solvents to hexane to reduce the emissions of volatile organic compounds to the atmosphere as well as potential traces of hexane in edible oils after refining. In light of this new trend leading to greater environmental protection and the development of a green chemistry, hexane should be gradually substituted by alternative solvents that are recognized as economically viable and environmentally safer (Li et al., 2014).

Several studies have been carried out, both on laboratory and pilot scale, aimed to replace hexane with other hydrocarbons

Abbreviations: C_L , concentration in bulk phase (extracted material kg/miscella kg); C_s , concentration in solid-occluded miscella phase (extractable material kg/inert solid kg); d.b., dry basis; D_e , effective diffusivity (m²/s); e.m, extracted material; R , collet average radius (m); K , equilibrium constant (C_s/C_L); M_t/M_{inf} , relation between mass extracted at time t and the one at infinite time; PA, phosphatidic acid; PC, phosphatidylcholine; PE, phosphatidylethanolamine; PI, phosphatidylinositol; t , time (s); λ_n , roots of $J_0(R \lambda_n) = 0$; J_0 , Bessel function of the first kind of order zero.

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(Conkerton et al., 1995; Wan et al., 1995a, 1995b) or alcohols (Abraham et al., 1988; Rittner, 1992; Hron et al., 1994; Sineiro et al., 1998) as solvents for oil extraction. Isopropanol and ethanol are the most promising solvents for the oil extraction from cottonseed (Abraham et al., 1988; Hron et al., 1994), sunflower seed (Sineiro et al., 1998) and soybean (Baker and Sullivan, 1983; Rittner, 1992). Ethanol is a worthy candidate to be studied as alternative solvent because it is cheap and it can be produced by fermentation from a large variety of biological materials using simple technology, and therefore labeled as “natural” or “biorenewable”. In addition, although flammable, this alcohol is recognized as non-toxic and has less handling risks than hexane (Rittner, 1992). The use of ethanol as an extraction solvent also avoids eventual toxicity problems of meals for animal feedstuff (Ferreira-Dias et al., 2003), but it has been reported that the solubility of lipids in ethanol is drastically affected by the moisture content of the solvent and the extraction temperature (Rao and Arnold, 1956, 1957).

Due to the lower selectivity of ethanol towards triglycerides, during the extraction process other compounds such as phosphatides, polyphenols, pigments and soluble sugars are extracted jointly (Hron et al., 1982, 1994; Sineiro et al., 1996). After the ethanol extraction, and when the miscella cools down, it can be expected that part of the extracted material becomes insoluble (in ethanol) and separates as an emulsion. At about 38 °C, the gums obtained (mucilaginous material consisting of emulsified oil, phospholipids, pigments and sugars) lose some of their entrained miscella, agglomerate and sink to the bottom of the oil phase (Hron and Koltun, 1984). The aim of this work was to determine the performance of ethanol (extractive power, quantity and quality of extracted oil, and oil extraction kinetics) when used in the oil extraction from sunflower collets.

2. Materials and methods

2.1. Sample characterization

All experimental determinations were made with sunflower expanded material, known as “collets”, which were kindly provided by a local factory. Collets were stored in polyethylene containers with screw caps in the dark at 4 °C until used in the extraction experiments. A sample of 20 collets was randomly selected to determine the average size of the collets. Collet dimensions, namely length (L) and radius (R), were measured using a gage with an accuracy of 0.01 mm. The initial moisture and oil content were determined according to IUPAC method 1.121 and 1.122 (IUPAC, 1992), respectively. The oil content of the solid was determined by an exhaustive extraction with analytical-reagent grade n-hexane (90%, bp 68–72 °C) in a Soxhlet apparatus. Similarly, the maximal ethanolic extraction yield was determined using analytical-reagent grade ethanol (95%, bp 78 °C). Solvents were removed from the miscellas firstly by rotary evaporation at 50 °C and low pressure, and then by nitrogen displacement until constant weights were reached.

The extracted material obtained using ethanol as solvent was fractionated in hexane-soluble material and other compounds (hexane-insoluble fraction) by phase separation with n-hexane. The hexane-soluble material constituted the lipid material that was used to study oil extraction kinetics.

2.2. Analyses of minor components

For the extracted material obtained using ethanol, contents of minor components (tocopherols, phospholipids, waxes and sugars) are expressed on a total extracted material basis and then referred to as moisture-free sunflower collets mass, in order to allow

comparison with the results obtained using n-hexane as extraction solvent.

2.2.1. Tocopherol analysis

Tocopherol content was determined using AOCS method Ce 8–89 (AOCS, 2009) with a Waters e2695 HPLC (Waters Associates, Milford, MA, USA) equipped with a Nucleosil Si-100A column (250 mm length, 4.6 mm i.d., 5 µm particle size, Phenomenex, USA) and a fluorescence detector (Waters 2475) with the excitation wavelength set at 290 nm and the emission wavelength set at 330 nm. The mobile phase used was n-hexane:isopropanol (99.5:0.5 v/v, HPLC solvent, J.T. Baker, Phillipsburg, USA) at a flow rate of 1.0 mL/min. Tocopherol content was determined by the external standard method. Areas were converted to concentrations using the standard curve of α -tocopherol (Sigma Chemical Co., 95%) in n-hexane in the relevant concentration range for the sample concentrations ($R^2 = 0.99$). Determinations were performed in triplicate.

2.2.2. Phospholipid analysis

Quantitative determination of phospholipids (PL) was carried out by enrichment using a diol solid-phase extraction cartridge (J.T. Backer Inc., Phillipsburg, NJ, USA) and subsequent analysis by HPLC-UV (Carelli et al., 1997). A Waters 600E HPLC system (Waters Associates, Milford, MA, USA) and a Lichrosorb SI-60 column (250 × 4 mm, 5 µm particle size, Merck, Darmstadt, Germany) were used. Identification of PL was carried out by comparing the retention times with pure standards. The following PL reference standards with purities greater than 98% were supplied by Sigma Chemical Co. (St. Louis, MO, USA): L- α phosphatidylethanolamine (PE), L- α phosphatidylcholine (PC), L- α phosphatidylinositol (PI) and phosphatidic acid (PA). Calibration curves were obtained using standard solutions prepared by dissolving the standard PL in HPLC mobile phase at different concentrations. Determinations were performed in duplicate.

2.2.3. Wax determination

Wax composition was determined by separation on a silica gel chromatographic column and analysis by GLC (Carelli et al., 2012). Briefly, column chromatography was performed using a glass column (i.d. = 15 mm, length = 400 mm) with a double phase of silver nitrate-impregnated silica gel (3 g) placed at the bottom of the column and hydrated silica gel 2% (12 g) placed on top as a solid stationary phase. The waxes were eluted with n-hexane/ethyl ether (99:1 v/v) at a flow rate of 3 mL/min using Sudan I dye to control the end of the elution (Carelli et al., 2012). The eluted wax fraction was evaporated to dryness and diluted with n-heptane, and analyzed by capillary GLC with an on-column injection system. A Perkin Elmer AutoSystem XL gas chromatograph equipped with an FID detector, a temperature programmable on-column injector and a TotalCrom Workstation data processor version 6.3.1 (Perkin Elmer, MA, USA) was used. The capillary column was an HP-5 (5% diphenyl and 95% dimethyl-polysiloxane), fused-silica 15 m length × 0.32 mm i.d., 0.25 µm film thickness (Hewlett–Packard, Palo Alto, CA). C32 to C44 wax standards of 99% purity were obtained from Sigma Chemical Co. (St. Louis, MO, USA). The C32 standard was used as internal standard, and the purified sunflower wax recovered from a filter cake following the method proposed by Carelli et al. (2012) was used as standard for the identification of waxes with more than 44 carbon atoms. Waxes were categorized as follows: with less than 40 carbon atoms they were considered the oil soluble fraction, waxes with 40 and 42 carbon atoms were the partially soluble fraction, and waxes with more than 44 carbon atoms constituted the crystallized fraction. Determinations were performed in duplicate.

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