Journal of Food Engineering 240 (2019) 49–55

Contents lists available at ScienceDirect

Journal of Food Engineering

journal homepage: www.elsevier.com/locate/jfoodeng

Extraction of high stearic high oleic sunflower oil (HSHO): Effect of dehulling and hydrothermal pretreatment

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ARTICLE INFO

Keywords:
High stearic high oleic sunflower
Hydrothermal pretreatment
Dehulling

ABSTRACT

The effect of a hydrothermal pretreatment on the solvent extraction of oil and minor compounds (tocopherols) from high stearic high oleic sunflower seeds (HSHO), partially dehulled and undehulled, was analyzed. Samples of sunflower seeds of different origins and oil contents (high oil, 43.0% d.b., and low oil 35.1% d.b.) were used. The oil yield increased significantly due to the hydrothermal pretreatment ($p \leq 0.05$) for HSHO samples with high oil content, both partially dehulled and undehulled. The pretreated HSHO samples with low oil content did not show significant differences in oil yield compared to the untreated samples. The effective diffusion coefficients for the hydrothermally pretreated seeds ($2.12.10^{-11}\,m^2\,s^{-1}$) and the untreated samples ($7.07.10^{-12}\,m^2\,s^{-1}$) were determined based on the oil extraction from the partially dehulled high oil HSHO samples. For both types of partially dehulled HSHO seeds, a significant increase in tocopherol content in the oils extracted from the pretreated samples was observed.

1. Introduction

Oilseed biotechnology has made it possible to produce new lines of mutant sunflowers with altered fatty acid compositions by combining breeding techniques and mutagenesis (a non-genetically modified ingredient, Fernández-Moya et al., 2005). Along these lines, a sunflower type has been developed (high stearic high oleic, HSHO) whose oil is of special interest due to its high stearic acid content (between 17 and 25% of total fatty acids) and its oleic acid levels which are about three times higher than that of the conventional sunflower oil, representing 60–70% of total fatty acids (Bootello et al., 2012). HSHO sunflower oils can be fractionated to produce fats with high levels of solids and different fusion profiles that could be used in a wide range of food formulations, thus becoming a promising, healthy and sustainable alternative source of solid fat (Salas et al., 2011; Bootello et al., 2012; Rincón-Cardona et al., 2015). The fatty acid composition of the oils affects both their quality and also human health. HSHO sunflower seeds provide commercial fats and oils with the required oxidative stability for cooking and a behavior that allows the industry to eliminate the use of oils high in palmitic and trans fatty acids, which are considered unhealthy (Hunter et al., 2000; Marset et al., 2009; Garcés et al., 2017).

In order to achieve high process efficiency in the oil extraction from oilseeds using solvents, a thorough preparation of the feed material is required, which involves cleaning, drying, grinding, dehulling, conditioning and pre-pressing steps. The mass transfer during extraction can be favored for example by grinding and dehulling operations prior to extraction. An adequate reduction in particle size along with a partial dehulling of the seeds to obtain an adequate porosity for the extraction bed are important stages because they directly affect the efficiency of the extraction process (Eggers and Jaeger, 2003).

In oilseeds, the oil is inside small, discrete intracellular organelles (oil bodies) whose walls are essentially non-permeable. These oil bodies vary in size from 0.1 to 2.5 μm, and they consist of a neutral lipid core (94–98% p/p) surrounded by a monolayer of phospholipids (0.5–2% p/p) and a strongly amphiphilic layer of proteins, oleosin and caleosin (0.5–3.5% p/p; Fisk et al., 2006). Tocopherols, which are natural antioxidants that increase the stability of fat-containing foods and also perform important biological functions, are intrinsically associated with the oil bodies (Fisk and Gray, 2011; Nantiyakul et al., 2012). The pretreatments applied to oilseeds in general modify or break the cell structure, thus favoring the release of the oil and making it more accessible to the solvent, and at the same time also facilitating the extraction of bioactive compounds. Therefore, pretreatments can affect the amount and type of minor compounds obtained in the extraction.

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https://doi.org/10.1016/j.jfoodeng.2018.07.015
Received 13 March 2018; Received in revised form 29 June 2018; Accepted 12 July 2018

Several authors have analyzed the effect of hydrothermal pretreatments on the dehulling process of various seeds: canola (Mohamadzadeh et al., 2009), black gram (Tiwari et al., 2008) and pigeon pea (Tiwari et al., 2010). Burnet et al. (2012) also studied the effect of this pretreatment on the yield and quality of canola and sunflower oils extracted from undehulled seeds. However, no study about the effect of hydrothermal treatments on the oil yield and content of minor compounds of partially dehulled sunflower seeds has been reported. It is also not known whether seeds from the same hybrid but with different oil content would show the same effect under this treatment as a consequence of their structural characteristics (Gonzáles Belo, 2017).

In the industry, it is common to find mixtures of seeds with different oil content being used for the oil extraction process. In order to improve equipment handling, optimize yields and the quality of the final product, it would be necessary to know their differential behavior during processing.

The aim of the present work was to investigate the effects of applying a hydrothermal pretreatment to the solvent extraction of oil and minor compounds (tocopherols) from high stearic high oleic sunflower seeds (HSHO) of different oil content, partially dehulled and undehulled.

2. Materials and methods

2.1. Sample characterization

Two samples of HSHO sunflower of different origin and with different oil content were used in the assays. The vegetable material with higher oil content (HO) was provided by Laboratorio de Fisiología Vegetal (UNMdP-CONICET), while the material with lower oil content (LO) was provided by Advanta Semillas SAIC (Mar del Plata, Argentina).

The samples were manually cleaned and kept at 5 °C until further use. The samples were characterized using standard procedures, determining moisture (ASAE S352.2 DEC, 1999), lipid (IUPAC 1.122, 1992), protein (AOCS Ai 4–91 1997), crude fiber (AOCS Ba 6–84, 1998) and ash content (AOCS Ba 5a-49, 1998). Acid-detergent fiber (ADF), neutral-detergent fiber (NDF) and lignin were determined sequentially using alpha-amylase, according to the procedure described by Van Soest et al. (1991) in a batch processor (Ankom Technology Corp., Fairpoint, NY, USA).

The hull percentage of the samples was determined by manually dehulling a sample of 10 g. Each fraction was dried in a forced-air oven (3 h, 130 °C), and the ratio was expressed as percentage on dry basis (%, d.b.).

2.2. Partial dehulling of the seeds

The seeds were partially dehulled under the optimal operating conditions determined for HSHO sunflower seeds (seed moisture content: 8.3% dry basis, d.b., and impact dehuller rotor speed: 39.1 m/s; de Figueiredo et al., 2015). The appropriate moisture content for dehulling was attained by drying the seeds in an oven at 35–40 °C, or by spraying them with a pre-calculated amount of distilled water. For the partial dehulling, a pilot dehulling equipment based on a centrifugal process was used. The rotor speed (rpm) was adjusted with a variable frequency drive. After the dehulling operation, the resulting product was passed through a 10 ASTM sieve in order to remove the fines (particles smaller than 2 mm). Then, after the manual separation of the hulls, the sample of partially dehulled seeds (consisting of dehulled seeds, partially dehulled seeds and undehulled seeds) was used for the experimental tests of solvent extraction.

2.3. Hydrothermal pretreatments

The samples of partially dehulled seeds from each dehulling test and the undehulled samples were ground (particle size between 0.420 and 1.000 mm) using a horizontal-blade grinder (Moulinex, Argentina), and then they were placed in trays with a metal mesh in the base (opening 149 μm) to allow circulation of air. An autoclave (VZ, Argentina) with a screen with holes at the bottom was used to subject the samples to steam. The hydrothermal pretreatments were carried out under the optimal conditions established for ground sunflower seeds (100–110 °C, 15 min; Burnet et al., 2010). After the exposure to steam, the samples were dried at 25 °C in a forced-air tunnel dryer to a moisture level of 6.5–7.4% d.b.

2.4. Scanning electron microscopy (SEM)

In order to determine the structural differences between the HSHO-HO and HSHO-LO sunflower seeds and to record any changes in their cell structure after the pretreatment, the samples (sunflower seeds cut lengthwise with a razor blade and the ground samples) were analyzed using variable pressure SEM imaging. The samples were placed into a SEM LEO EVO 40-XVP microscope (England) and observed under variable pressure at 15 kV. The pressure of the microscope chamber was adjusted between 40 and 70 Pa as needed.

2.5. Oil yield

The oil yield was determined using n-hexane (Soxhlet) according to IUPAC standard method 1.122 (1992). Both the hydrothermally pretreated and the untreated samples were ground prior to the oil extraction with hexane.

2.6. Quality measurements

The oil obtained from the pretreated and untreated samples was characterized for acid value (IUPAC 2.201, 1992), peroxide index (AOCS Cd8, AOCS, 1998), tocopherol and fatty acid composition. The tocopherol concentration in the oil was also quantified according to AOCS method Ce 8–89 (AOCS, 1997) using a Dionex Ultimate 3000 chromatograph (Thermo Scientific, Germany) with fluorescence detector (Agilent, 1100 Series Fluorescence Detector G1321A, Palo Alto, CA, USA) with excitation/emission wavelengths of 290/330 nm, equipped with a HicHROM Lichrosorb Si-60, 5 μm particle size, 250 × 4.6 mm i.d. column. Hexane:isopropanol (99.5:0.5 v/v) was used as mobile phase, with a column flow of 1.5 ml/min.

The fatty acid composition was determined after the methyl esters were extracted from the fatty acids and measured by gas chromatography. Briefly, 3 or 4 drops of oil with 1 ml of chloroform and 1 ml of methanolic acid (acetyl chloride in methanol) were placed in glass tubes. The tubes were capped and stirred for 1 min, and then transferred to a water bath at 70 °C for 1 h. After letting it cool, 1 ml of a 6% solution of K_2CO_3 was added and vortexed for 1 min. After phase separation, the aqueous phase (upper phase) was collected, and 2 ml of chloroform were added to the oil phase (lower phase). It was left to rest until phase separation. Then 1 ml of the lower phase containing the methyl esters was transferred to GC vials. The determinations were carried out with a Shimadzu GC − 2014 chromatograph (DB23 capillary column, 60 m × 0.25 mm) with FID detector. The temperature of the oven, injector and detector was 210, 240 and 300 °C, respectively. N_2 was used as carrier gas, with a split ratio of 160.4.

2.7. Statistical analysis

The experimental data were analyzed by means of analysis of variance (ANOVA) followed by Tukey's test (Infostat, 2004). The effect of the pretreatment for each sample of HSHO sunflower seeds (HO and