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Parameters affecting enzyme-assisted aqueous extraction of extruded sunflower meal

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Microscopic observation of sunflower meal before and after extraction indicated that extensive cellular disruption was achieved by extrusion, but that unextracted oil remained sequestered as coalesced oil within the void spaces of disrupted cotyledon cells. A full factorial design experiment was defined to develop aqueous extraction processing (AEP) with and without enzymes to improve vegetable oil extraction yields of extruded sunflower meal. This experimental design studied the influence of four parameters, agitation, liquid/solid (L/S) ratio, and cellulase and protease addition, on extraction yield of lipid and protein. Agitation and addition of cellulases increased oil extraction yield, indicating that emulsification of oil and alteration of the geometry of the confining cellular matrix were important mechanisms for improving yields. Protease and liquid-solid ratio of the extraction mixture did not have significant effects, indicating key differences with previously established soy oil extraction mechanisms. Maximum yields attained for oil and protein extraction were 39% and 90%, respectively, with the aid of a surfactant.

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1. Introduction

Pressing, with single screw extruders, is usually the first step of oil production. For seeds with high oil content such as sunflower, extraction yields of 70–85% can be achieved (Evon & [Dissertation] Toulouse Université de Toulouse, 2008; Kartika, Pontalier, & Rigal, 2006). However, to maximize yields, residual oil in the extruded meal is extracted with an organic solvent, most commonly hexane. An important part of the Green Chemistry (Anastas & Warner, 1998) movement is to develop technologies that are environmentally friendly and reduce the use of petroleum-derived materials. Aqueous extraction processing (AEP) and enzyme-assisted aqueous extraction processing (EAEP) are safe water-based extraction processes that, with the use of enzymes, have succeeded in achieving free oil yields as high as 88% in soybean oil extraction (Moura & Johnson, 2009; Moura et al., 2008).

In an immiscible oil-water system, the ability to extract oil is dependent on its mobility within the solid matrix confining the unextracted portion (Campbell & Glatz, 2009). Therefore, one important factor in AEP/EAEP is the geometry of the confining matrix as determined by the nature of the oilseed itself, as well as the mode of comminution used to disrupt cells. In soy, grinding and extruding produced substrates with very different physical geometries from which the oil must escape (Campbell & Glatz, 2009). In the case of extrusion, oil was released from a matrix of insoluble denatured protein, while in flour from flakes; oil was released from partially disrupted cells.

Cellulases increase the extraction yield of oil from ground sunflower in EAEP by cellular disruption (Dominguez, Nunez, & Lema, 1995; Sineiro, Dominguez, Nunez, & Lema, 1998) but could also act by modifying the geometry of cells previously disrupted, thus facilitating oil transfer out of the remaining matrix. Furthermore, Campbell and Glatz have established that emulsification is a key parameter in the extraction mechanism for EAEP of soybean flour (Campbell & Glatz, 2009). In an aqueous environment, where the extract (oil) is immiscible with the solvent (water), extraction is increased when coalesced oil entrapped within ruptured cells can be emulsified into smaller, more mobile droplets by turbulent forces in the extraction medium.

Another important factor for soy oil extraction is the nature of the oil-water interface. Campbell and Glatz proposed that the mechanism, by which protease increases oil yields in soy flour extraction, is by disruption of a viscoelastic interfacial protein film

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at the oil-water interface, facilitating emulsification. Badr and Sitohy demonstrated that at pH 5 proteases can also increase the yields of sunflower oil from dehulled chopped seeds, which they attributed to a disruption of lipid-protein complexes (Bair & Snyder, 1980).

The objectives of this work were to identify the conditions to increase the oil recovery yield from extruded meal, using aqueous extraction or enzyme-assisted aqueous extraction instead of the classical hexane extraction procedure.

2. Materials and methods

2.1. Preparation of extruded sunflower meal

Common variety sunflower kernels (with hulls) obtained from Toulgrain, Inc. (Toulouse, France) were extruded in an Omega 20 single screw bench top press-extruder (Eurl Laplace Co., Pau, France), equipped with a heated collar around the die housing. Steady-state exit temperature of the extruded cake was measured to be around 100 °C (±5 °C) with an infrared thermometer. Expressed oil was collected, weighed, and centrifuged. The resulting precipitate was rinsed three times with cylcohexane, dried, and weighed to determine the fraction of foots in the expressed oil. The resulting cake was cooled and then ground in a Pulverisette 19 (Fritsch Ltd. Idar-Obersteen, Germany) knife mill with a 2 mm outlet screen. Extruded meal was stored at −20 °C until use.

2.2. Extraction

The appropriate quantity of extruded meal was added to 1 L of DI water in a 2 L jacketed reactor with an agitator, maintained at 50 °C with a water bath and at constant pH 6.5 using a 716 DMS Titrino autotitrator (Metrohm Ltd., Herisau, Switzerland) with 1 N NaOH. Samples were collected by siphon into a 500 mL bottle, weighed, and centrifuged (Sigma 6–16 k) at 3000*g* for 15 min at 20 °C. The supernatant was discarded and the remaining residual solid was weighed, freeze dried, and weighed again for moisture determination. Freeze-dried precipitate was ground in a coffee grinder for approximately 30 s and then stored in a dessicator until oil and protein content determination. Yield was calculated as one minus the fraction of total material remaining in the residual fraction. Protein dissolution was defined as the protein extraction yield plus the fraction of dissolved protein entrained in the solid-fraction, estimated by multiplying the liquid fraction protein concentration by the mass of water in the solid fraction. The liquid fraction protein content was determined by mass balance based on the protein content of the residual fraction.

For microscopy experiments, extraction was carried out by placing extruded meal in 500 mL centrifuge bottles with DI water for a solid-liquid ratio of 1:10. Bottles were placed on a stir plate in a water bath maintained at 50 °C, and agitated with a magnetic stir bar at 1000 rpm. Centrifugation (3000*g* 15 min at 20 °C) resulted in two distinct layers in the centrifuge bottles. Therefore, samples for microscopy were from the bulk mixture before centrifugation and from each of the two layers after centrifugation.

2.3. Full factorial design experiment

To elucidate the effects of enzyme, solid-liquid ratio, and agitation, a randomized full factorial design experiment was conducted using two continuous two-level parameters: solid-liquid ratio (0.05 and 0.10) and agitation rate (160 and 350 rpm), plus two discrete parameters: with and without protease Protex 7L and with and without cellulase Multifect CX 13L, kindly provided by Genencor (Rochester, NY), both 2% w/w solid, giving a total of 16 possible experimental conditions. Cellulase Multifect CX 13L, with a specific activity of 3900 CMC/g, exhibits significant activity towards cellulose, hemicelluloses, β-glucans and arabinoxylans. The Protex 7L (also named Multifect Neutral) has an activity of 1600 AU (Azo Unit)/g define by hydrolysis of Azo-casein substrate at pH 7.5 for 5 min at 30 °C. The active pH ranges of these enzymes overlap in the pH 6–7 region, and so pH 6.5 was selected for all of these experiments. Measured responses were oil extraction yield, protein dissolution, and non-lipid material dissolution. Trials for the full factorial design experiment were not replicated, while all other trials reported were made in triplicate. Error estimation for analysis of variance (carried out using JMP 7 software from SAS Institute, Inc. Cary, NC) was based on the assumption of interactions of an order higher than two, being nonsignificant.

2.4. Analytical methods

Oil was extracted from residual samples four times for 10 min, at 105 °C and 95 bar with cyclohexane using an ASE 200 Accelerated Solvent Extractor (Dionex Corp, Sunnyvale, CA). Extract was transferred from vials to preweighed glass beakers (dried 1 h at 103 °C, cooled to room temperature on the bench top), rinsing twice with cyclohexane. Cyclohexane was evaporated by placing beakers in a boiling water bath and then drying them for 1 h in a 103 °C oven. Beakers were cooled to room temperature on the bench top, and weighed again to determine mass of oil. Protein content was determined by the Kjeldahl total nitrogen method using a nitrogen to protein conversion factor of 6.25 g protein per g nitrogen. Residual moisture content was determined by loss of mass upon freeze-drying. Moisture gained during sample storage was analyzed simultaneously with oil content determination, by measuring the loss of mass upon drying samples at 103 °C for 24 h. This was used to correct the oil content determination.

2.5. Particle size distribution of extruded meal

Particle size distribution of extruded meal was determined by sieving. 250 g of extruded meal was placed in a sieve-shaker equipped with four different sieve sizes: 1.25 mm, 0.80 mm, 0.50 mm, and 0.25 mm. Material was fractionated for 15 min at a frequency of 50 s^{-1}, and then weighed from each screen. As the entire meal was used for the experiments, specific extrusions were done for particle size distribution determination.

2.6. Differential Scanning Calorimetry (DSC)

The extent of protein denaturation was determined by measuring the heat absorbed by 12 mg samples of dry material, heated at a rate of 10 °C per minute from room temperature to 190 °C using a Pyris 1 differential scanning calorimeter (Perkin Elmer, Waltham, MA).

2.7. Microscopy

Samples were fixed and embedded following Bair and Snyder (Boy & Snyder, 1980) with minor modifications, at the Centre de Microscopie Electronique Appliquée in Toulouse, France. Sections were made at the Iowa State University NanoImaging Facility using a Reichert Ultracut S ultramicrotome (Leeds Precision Instruments, Minneapolis, MN). Thick sections were contrast stained using 1% toluidine blue. Light microscopy images were made using a Zeiss Axioplan 2 light microscope (Carl Zeiss MicroImaging, Inc., Thornwood, NY).

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