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Surfactant-Based Oil Extraction of Corn Germ

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Abstract An aqueous surfactant-based extraction system was developed for the extraction of corn oil from corn germ with anionic extended surfactants. The surfactants used in this study were sodium linear-alkyl polypropoxylated polyethoxylated sulfates ($C_{12,14}$–P_{10}–E_2–SO_4Na and C_{10}–P_{18}–E_2–SO_4Na). Interfacial tension, critical microemulsion concentration (CμC), and optimum salinity values of the extended surfactants with corn oil were determined. In the extraction process, the ground corn germ was shaken with predetermined surfactant and salt concentrations at room temperature for 45 min. About 83%, the sum of total free oil and total oil-in-water emulsion, of the corn oil was extracted from the corn germ using a formulation of 0.4% $C_{12,14}$–P_{10}–E_2–SO_4Na and 1% NaCl. A solid/liquid ratio of 1/10 performed best for efficient oil recovery. The chemical compositions of the extracted corn oils were found to be similar to that of hexane extracted corn oil.

Keywords Surfactant · Corn germ · Extraction · Microemulsion · Interfacial tension · Corn oil · Extended surfactant

Introduction

Currently, commercial corn oil is obtained from corn germ by either hexane extraction [1, 2], or a process that combines pressing and hexane extraction [3]. To date, hexane extraction is still much less expensive than alternative vegetable oil extraction methods. However, the US Environmental Protection Agency (USEPA) has identified hexane as a hazardous air pollutant and issued stricter rules for hexane emissions, providing incentives to develop alternative methods of oil extraction [4].

Alternative solvents have been evaluated to replace hexane including ethanol [5, 6], vegetable oil itself [7], or carbon dioxide–ethyl alcohol mixture [8]. Aqueous [9, 10], aqueous enzymatic [10–13], and enzyme-assisted solvent extraction [14] methods have also been evaluated for corn germ oil extraction.

The use of non-toxic surfactants to extract oil from plant seeds is an alternative approach which is considered to be a clean technology since the surfactants used in oil extraction are not hazardous. Surfactants have been used in numerous applications, including cleaning technologies, cosmetics, drug delivery, biodiesel applications [15], and environmental remediation [16]. However, surfactant-based extraction of oil from oil seeds and/or plants is quite a new application. In recent studies, surfactants have been used for the extraction of oil from cruciferous [17] and palm kernel oil seeds by selecting optimum surfactant and salt concentrations based on interfacial tension (IFT) and phase behavior data [18].

The role of surfactants in oil seed extraction is to reduce the IFT between the aqueous extracting phase and oil in crushed seed. IFT reduction promotes the snap-off, roll-up, and oil liberation mechanisms and hence promotes the oil extraction [19]. While conventional surfactants are not able to produce low interfacial tension (<0.1 mN/m) with vegetable oils at ambient conditions without alcohol or co-oil addition, it has been reported that ultralow IFT can be attained with vegetable oils using extended surfactants [19, 20]. An IFT value of 0.01 mN/m was reported for canola

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oil [equivalent alkane carbon numbers (EACN) = 16.9] which has EACN identical to corn oil (EACN = 16.9) [20].

Extended surfactants are surfactants in which groups of intermediate polarity, such as polypropylene oxides or block copolymers of propylene oxides and ethylene oxide, are inserted between the hydrocarbon tail and hydrophilic head group. Due to the resulting unique molecular structure, the surfactant is stretched out further into both the oil and water phases, providing a smoother transition between the hydrophilic and hydrophobic regions of the interface, which provides a more suitable environment for solubilizing hydrophilic and lipophilic molecules [19]. Additionally, the Gibbs adsorption equation indicates that with an increase in adsorption at the interface, a reduction of interfacial tension is expected [21]. Despite their relatively large molecular size, these surfactants are water-soluble and can be formulated at a relatively high electrolyte concentration while avoiding surfactant precipitation.

In this study, surfactant-based extraction using anionic extended surfactants was evaluated as an alternative to hexane for corn oil extraction from corn germ. Fundamental surfactant-oil interaction properties are reported as well as oil seed extraction efficiencies and the impact of selected operating parameters.

Materials and Methods

Materials

The anionic extended surfactants used in this study are sodium linear-alkyl polypropoxylated polyethoxylated sulfate (R–$(PO)_y$–$(EO)_z$–SO_4Na) surfactants and were synthesized and donated by Huntsman Petrochemical Corporation (Houston, TX). Surfactant $C_{12,14}$–P_{10}–E_2–SO_4Na has an alkyl group consisting of a branched hydrocarbon chain with an equal mixture of 12 and 14 carbons, with 10 propylene oxide units (denoted as "P" in abbreviation) and 2 ethylene oxide units (denoted as "E" in abbreviation). This surfactant solution is 22.3 wt% active (mass concentration of surfactant) with 1–2% Na_2SO_4 as received from the manufacturer. The other surfactant used was C_{10}–P_{18}–E_2–SO_4Na and has an alkyl group consisting of branched hydrocarbon chain with 10 carbons, with 18 propylene oxide units and 2 ethylene oxide units. C_{10}–P_{18}–E_2–SO_4Na is 22.55 wt% active (mass concentration of surfactant) with 1–2% Na_2SO_4. The extended surfactants were used as received from the manufacturer.

Dry milled corn germ samples having 17–41% oil, 13–21% protein, 6–21% starch and 4–5% moisture content were donated by the United States Department of Agriculture (USDA) and stored at 4°C upon receipt. For IFT measurements analytical grade corn oil (Sigma-Aldrich, USA) was used.

Methods

Preparation of Corn Germ Samples

Based on the US standards of vegetable oil seed size [18], grain particles can be classified in three groups: coarse size (>0.425 mm), fine size (between 0.212 and 0.425 mm) and a very fine size (<0.212 mm). Samples (20–30 g) of dry-milled corn germ were ground for 20 s with a coffee mill (Krups, Model 203B). Samples of ground corn germ were sieved to a fine particle size (between 0.212 and 0.425 mm) with a standard test sieve (ASTM).

Hexane Extraction

For hexane extraction, corn germ (4 g) was weighed into a 50-ml glass screw-top tube and 40 ml of hexane was added. The mixture was shaken horizontally at 250 rpm for 30 min at room temperature. The slurry was then centrifuged for 20 min at 3,500 rpm. The hexane phase was removed with a pipette and put into a pre-weighed 50 ml glass tube. Hexane was completely evaporated at 70 °C and the remaining oil was weighed. Two sets of corn germ were received over the course of the study. Because of the batch-to-batch variation in the oil content of the corn germ, a sample of the germ used in each set of experiment was also extracted with hexane and this control value was used to calculate the 'relative oil yield'. Based on the assumption of 100% oil yield from corn germ with hexane extraction, total oil content was calculated as 17 ± 2.0 wt%.

Aqueous Surfactant-Based Extraction

The procedure for surfactant-based corn oil extraction is summarized in Scheme 1. Since a small amount of corn germ was used, a small amount of free oil was obtained in each experiment. To remove the free oil phase, a freezing/scraping method (Scheme 1) was preferred, since it was found to be more reliable as compared to removal of oil with a syringe. It should be noted that this step, common in the literature [22], is for analytical quantification only and is not intended as part of the overall oil removal process. Free oil refers to the amount of oil floating on top of the surfactant solution at the end of the surfactant extraction method which is clear in appearance (Scheme 1, Step 2). Oil-in-emulsion refers to the oil extracted from interface emulsion (Scheme 1, Step 2). Residual oil refers to the oil remaining in the corn germ samples after surfactant extraction (Scheme 1, Step 2B). Combination of free oil and oil in-emulsion was reported as oil extracted by surfactant extraction method. To confirm the oil yield by the surfactant extraction method, a mass balance was

Scheme 1 Method for the surfactant oil extraction of corn oil from corn germ (adapted from [30])

STEP 1

1. Put predetermined amounts of surfactant solution + salt solution + water into 50 ml. glass screw-top tube and stirred for 5 min. by magnetic stirrer (Cole-Parmer, Model 4802, Chicago,IL).
2. Weigh 4 g samples of corn germ and add into the prepared surfactant mixture.
3. Shake the mixture horizontally for 45 min. at room temperature in a shaker (Cole-Parmer Ping-Pong™ #51504-00) at a frequency of 200 oscillations/min.
4. Centrifuge (Thermo Scientific IEC CL10) the slurry for 20 min. at 3500 rpm
5. Remove the **aqueous solution (A)** from precipitated **solid part (Corn germ particles) (B)** with a pipet.

Aqueous Part (A)

STEP 2: Detection of oil extracted with surfactant

A1. Centrifuge the solution for 20 min. at 3500 rpm. At the end of the centrifugation free oil phase appears on top. Directly below the free oil phase was white interface-emulsion.
A2. Put the solution into a freezer at -10°C for 2 hr.
A3. Scrap off the top free oil layer.
A4. Thaw the oil paste and weigh to calculate the amount of free oil extracted.
A5. Remove the white interface emulsion with pipet and transfer to 40ml. glass tube.
A6. Add 20 ml hexane and shake for 15 min.
A7. Centrifuge for 10 min. at 3500 rpm.
A8. Remove the hexane solution with pipet
A9. Evaporate the hexane and weigh the remaining oil .

Solid Part (Corn germ particles) (B)

STEP 3: Detection of residual oil remaining in the corn germ

Residual Oil in washing solution

B1. Wash the remaining corn germ particles with 10 ml. of water.
B2. Centrifuge the slurry for 10 min. at 3500 rpm.
B3. Remove water with pipet
B4. Repeat steps B1-B3 once more.
B5. Add 10ml. hexane to water solution to extract the remaining oil in washing solution
B6. Shake the water-hexane mixture for 15 min.
B7. Evaporate the hexane at 70°C
B8. Weigh the remaining oil.

Residual Oil in corn germ

1. Evaporate the residual corn germ for dryness at 100°C for 2 hours.
2.Add 20 ml. hexane
3.Shake for 15 min.
4.Centrifuge for 10min. at 3500 rpm.
5. Remove the hexane phase with pipet.
6. Evaporate the hexane at 70°C
7. Weigh the remaining oil.

conducted. The amount of residual oil was subtracted from total oil content and reported as oil yield. Mass balance on the oil extraction showed mass recovery within ±2% error margin.

Interfacial Tension Measurements

The IFT between the aqueous surfactant solution and the oil phase was measured using glass capillary tubes and a spinning drop tensiometer (Model 500, University of Texas). The capillary tube was 2 mm in diameter and had a volume of 300 µl. An amount of 1–3 µl of corn oil was injected into the tube filled with the surfactant solution. All the measurements were done in duplicate at 25 ± 1°C. The IFT measurements were commenced immediately after injecting 1–3 µl of the corn oil (Sigma-Aldrich, USA) into a capillary tube filled with the surfactant formulation. Since IFT values were observed to reach equilibrium within 15 min, IFT values are reported throughout this work at a 15 min reading. It was demonstrated that the IFT values reached equilibrium within 15 min [20].

Statistics

All extraction experiments were replicated with triplicate samples. Error bars on the charts are calculated from treatment replications. For interfacial tension experiments each sample was conducted in triplicate.

Nonpolar Lipid Analysis

Analysis of the corn oil (free oil phase obtained in Scheme 1-Step 2) was made with a normal phase HPLC method with ELSD. The column was a LiChrosorb 7-µm DIOL column (3–100 mm, packed by Chrompack, Raritan, NJ). The binary gradient had a constant flow rate of 0.5 ml/min, with solvent A as hexane/acetic acid (1,000:1) and solvent B as hexane/isopropanol (100:1). Details of the method are described in Moreau's study [23].

Results and Discussion

Interfacial tension measurements were conducted as a function of surfactant and salt concentrations in order to estimate the point at which the surfactant system attained ultralow interfacial tension with the corn oil.

Interfacial Tension Measurements: *Effect of Salt Concentration*

Figure 1 presents IFT values as a function of the electrolyte concentrations for the systems corn oil–$C_{12,14}$–P_{10}–E_2–SO_4Na–brine and corn oil–C_{10}–P_{18}–E_2–SO_4Na–brine. It is encouraging to note that both surfactant systems were able to produce ultra-low IFT (<0.001 mN/m) with the corn oil. The optimum salinity (S^*) of the system denotes the electrolyte concentration where the surfactant system

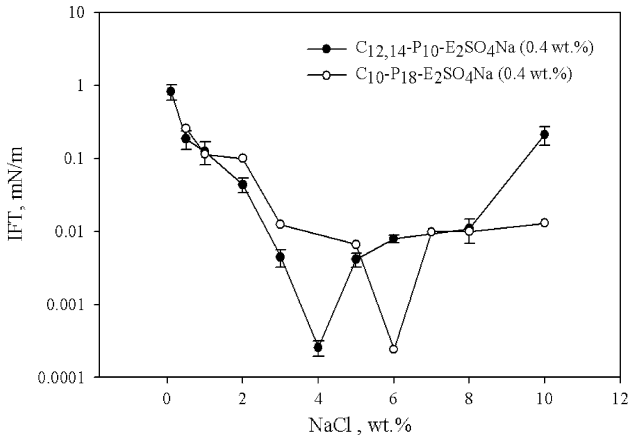

Fig. 1 IFT as a function of electrolyte (NaCl) concentration (salinity scan) for the systems corn oil–$C_{12,14}$–P_{10}–E_2–SO_4Na (0.4 wt%)–brine and corn oil–C_{10}–P_{18}–E_2–SO_4Na(0.4 wt%)–brine. Measurements were conducted at 25 ± 1 °C

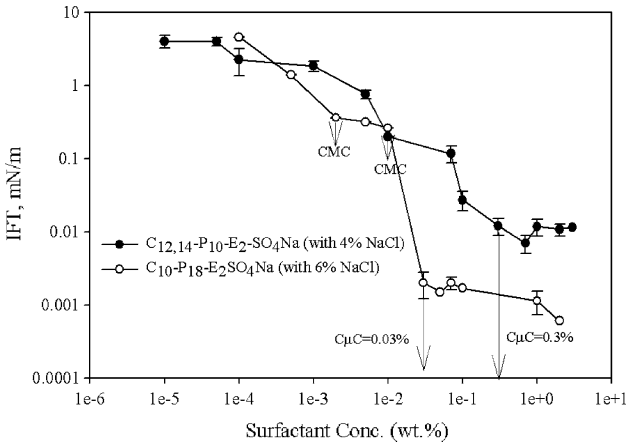

Fig. 2 IFT versus surfactant concentration at optimum electrolyte concentration for the systems corn oil–$C_{12,14}$–P_{10}–E_2–SO_4Na–4% NaCl (*closed circles*) and corn oil–C_{10}–P_{18}–E_2–SO_4Na–6% NaCl (*open circles*). CMC (critical micelle concentration) and CμC (critical microemulsion concentration) are *highlighted*. Measurements were conducted at 25 ± 1 °C

attained minimum IFT. For $C_{12,14}$–P_{10}–E_2–SO_4Na ultralow IFT (0.0004 mN/m) was attained with *S** of 4% NaCl and for C_{10}–P_{18}–E_2–SO_4Na ultralow IFT (0.0002 mN/m) was attained with *S**of 6% NaCl.

Interfacial Tension Measurements: *Effect of Surfactant Concentration*

In this set of experiments, the electrolyte concentration was fixed at the optimum salinity, and the corn oil-surfactant IFT was measured as a function of surfactant concentration. Figure 2 shows the IFT measurements for the systems corn oil–$C_{12,14}$–P_{10}–E_2–SO_4Na–brine and corn oil–C_{10}–P_{18}–E_2–SO_4Na–brine. The observed decrease in IFT with the addition of surfactant follows the trend previously observed in systems where equilibrium IFT values of conventional surfactants were measured [24]. This decrease in IFT follows two stages. The first stage corresponds to the adsorption of the surfactant at the oil–water interface, which occurs at concentrations less than the CMC. Above the critical micelle concentration (CMC), surfactant monomers aggregate to form micelles [25]. Many system properties remain unchanged above the CMC since additional surfactant forms micelles rather than increasing the surfactant aqueous activity [26].

The second stage corresponds to the change in curvature of the micelles which ends at the point where the first droplet of microemulsion forms (the CμC) [24]. CμC is referred to as the concentration at which a microemulsion first forms. Microemulsions are thermodynamically stable systems that contain water and oil domains separated by surfactant films [25]. While the IFT is commonly observed to decrease between the CMC and CμC [20], the phenomenological reason for this is not yet understood. From Fig. 2, we observe that the CμC value is 0.03 wt% for C_{10}–P_{18}–E_2–SO_4Na and 0.3 wt% for $C_{12,14}$–P_{10}–E_2–SO_4Na.

Oil Extraction Efficiency

The effects of surfactant and salt concentrations on oil recovery efficiency were evaluated. As mentioned above, the goal of oil extraction with surfactant-based microemulsion systems is to liberate the oil droplets from corn germ particles by reducing the IFT. The study of Wang et al. [27] provides a proof of concept that surfactant can displace the oil fine droplets that are attached to oil seed particles and found that the addition of surfactant significantly enhanced the oil release. In this study, we want to go one step further and analyze the effects of surfactant concentration, salt concentration and IFT on improved oil release from corn germ particles. It was presumed that maximum oil recovery would be achieved at a point where the system attains ultralow IFT. To test this hypothesis, oil extraction studies were carried out at different salt and surfactant concentrations corresponding to different levels of IFT.

From Fig. 3, we observe that a maximum of 83% of the corn oil was extracted (total of free oil and oil potentially recoverable from oil-in-water emulsion) with $C_{12,14}$–P_{10}–E_2–SO_4Na with a contact time of 45 min. Throughout the experiments it was observed that the majority of the oil was liberated as free oil with only 7–9% of the extracted oil existing in an oil-in-water emulsion. This maximum extraction efficiency occurred at 0.4 wt% surfactant, which is just above the CμC value of 0.3 wt% reported above (see Fig. 2). It may be seen that while the extraction efficiency

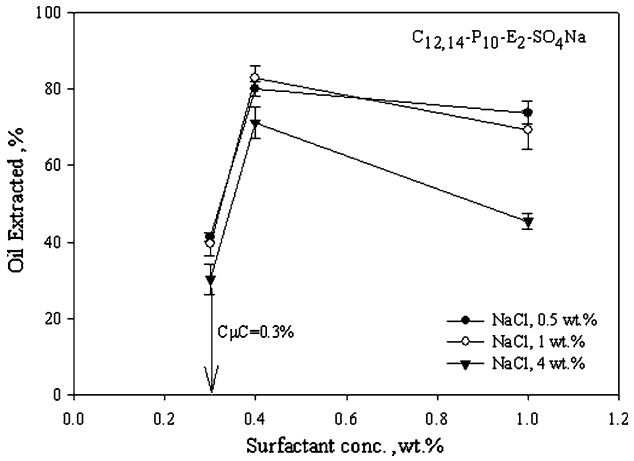

Fig. 3 Oil extraction versus $C_{12,14}$–P_{10}–E_2–SO_4Na concentration and salinity (NaCl), contact time of 45 min, solid to liquid ratio of 1/10, temperature of 25 ± 1 °C. CμC (critical microemulsion concentration)

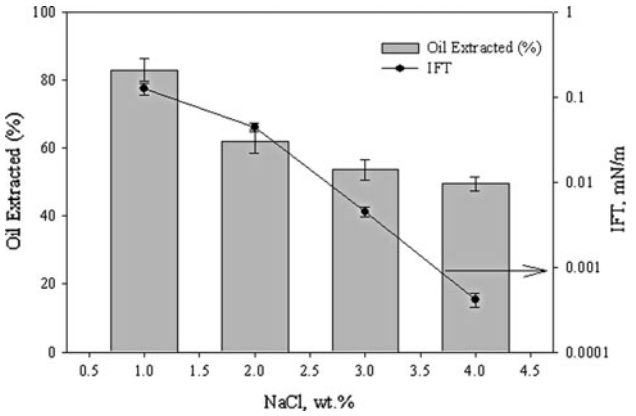

Fig. 4 Oil extraction and IFT for $C_{12,14}$–P_{10}–E_2–SO_4Na (0.4 wt%) versus NaCl concentration. Measurements were conducted at 25 ± 1 °C, contact time of 45 min, solid to liquid ratio of 1/10

reaches a maximum near the surfactant CμC where the IFT reaches a minimum, further increases in surfactant concentration do not improve the oil extraction, as the IFT remains constant. It is also noted that oil extraction ≥80% was achieved at 0.5 and 1% NaCl, where IFT values are expected to be around 0.1 mN/m (Fig. 1). It is encouraging to see that effective oil extraction can be achieved using relatively low concentrations of the $C_{12,14}$–P_{10}–E_2–SO_4Na surfactant (0.3–0.4 wt%). The oil recovery at optimum salinity (4%), where IFT values were $<10^{-3}$ mN/m, was statistically lower (~70%), as discussed below.

C_{10}–P_{18}–E_2–SO_4Na surfactant also produced low IFT values (Fig. 2). The maximum oil yield with this surfactant was around 60% (data not shown). Maximum oil extraction was also achieved just above the CμC point and further increase in surfactant concentration did not enhance the oil extraction. These results illustrate that the best oil extraction efficiency was achieved at low values of IFT, which is experienced around the CμC.

Since much higher oil yields were achieved with surfactant $C_{12,14}$–P_{10}–E_2–SO_4Na, the remaining experiments were performed with this surfactant. As can be seen from Fig. 4, IFT values of 0.1 mN/m were sufficiently low to produce effective oil extraction. Further reducing the IFT by one order of magnitude (0.01 mN/m) actually reduced the oil extraction efficiency. One explanation for this observation is that at IFT values around 0.1 mN/m, oil droplets can be detached and hence removed from the crushed corn germ. When the IFT drops down to a value around 0.01 mN/m, oil droplets may spread on the oil seed making it harder to remove the oil from the corn germ. The attached oil is partially detached by a roll-up and/or snap-off mechanism since the contact angle is reduced by the surfactant adsorption. Later, the oil detachment gradually ceases, and the spreading of the oil becomes dominant if the system has an ultralow IFT. Similar results have also been observed for detergency tests and attributed to the spreading effect at ultralow IFT [28]. This behavior may be explained by coating film hypothesis at optimum conditions (i.e., optimum salinity for ionic surfactants or optimum temperature for nonionic surfactants). The ultralow IFT causes the oil to spread on the wet fabric surface, making the oil harder to remove. At lower electrolyte levels in the solution (less than optimum salinity), the system lowers the IFT but not to ultralow values. Since the oil is still non-wetting on the surface, the roll-up mechanism can easily detach the oil from the surface because of the lower energy of cohesion within the oil which results from the reduced IFT [29].

Oil Extraction Efficiency: Effect of Solid/Liquid Ratio

Experiments were conducted with 4 g corn germ and varying amounts of surfactant solution of $C_{12,14}$–P_{10}–E_2–SO_4Na and NaCl. Solid/liquid ratios of 1/5, 1/7 and 1/10 (m/m) were selected.

The maximum oil extraction of 80% was achieved with solid/liquid ratio of 1/10 (Fig. 5). Decreases in the amount of surfactant solution (solid/liquid ratio of 1/7 and 1/5) drastically reduced the oil extraction to 25–30%. One explanation for these results is that when there is not enough surfactant solution in the medium, adsorption losses are more noticeable and it affects the detachment of oil droplets and hence the oil yield.

Composition of Corn Oil from Hexane versus Surfactant-Based Extraction

Non polar lipid analysis of corn oil extracted by surfactant microemulsion based extraction method was examined by

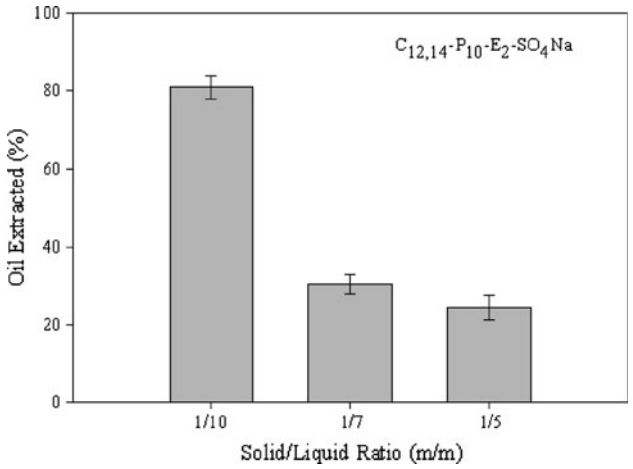

Fig. 5 Oil extraction versus solid/liquid ratio for $C_{12,14}$–P_{10}–E_2–SO_4Na–1% NaCl system. Contact time is 45 min

Table 1 Nonpolar lipid composition of corn oil obtained by different extraction methods

Lipid class	Hexane-extracted oil (wt% of oil)[a]	Aqueous enzyme-extracted oil (wt% of oil)[a]	Surfactant-extracted oil (wt% of oil)[b]
Sterol fatty acyl esters	0.61	0.48	3.43
Triacylglycerols	97.1	98.0	92.9
Palmitic acid[c]	0.30	0.10	0.191
Oleic acid[c]	0.13	0.09	0.649
Free sterols	0.61	0.24	0.344

Standard deviation of the data in Table 1 are in the range of ±3–5%

[a] Ref. [30]

[b] Analysis of the free oil extracted is as shown in Scheme 1, Step 2

[c] Free fatty acid component

HPLC and compared with the reported composition of corn oil extracted by hexane and aqueous enzyme extracted method [30] (Table 1). Basically, the surfactant-based extraction method produced crude (unrefined) corn oil samples that were quite similar to hexane-extracted corn oil. It is mostly triacylglycerols with low levels of free fatty acids (palmitic and oleic), some plant sterols, and 1,2-di-acylglycerols. The amount of sterol fatty acyl esters is much higher in surfactant extracted corn oil as compared to hexane extracted and enzyme extracted corn oil (Table 1). In corn oil, the predominant esterified fatty acid is linoleic acid and usually is also the predominant free fatty acid. However, linoleic acid was not detected in our sample.

Conclusion

surfactant-based extraction of corn oil from corn germ offers several advantages. First, hexane and/or other organic solvents were avoided in the process. Greater than 80% of corn oil can be extracted with low surfactant (0.4 wt%) and salt (1 wt%) concentrations. The surfactant-based extraction process proved to be efficient at room temperature (25 ± 1 °C) with short process time (45 min). It may be concluded that aqueous-based surfactant micro-emulsion oilseed extraction is a promising alternative approach for oil extraction.

Further studies should be performed for scale up options for industrial use. To enhance the oil recovery from the oil-in-water emulsion, demulsification processes should be adapted. Various single and combined treatments including thermal treatments and enzymatic treatments may be used to increase the free oil yield [31, 32]. Future research should further explore this extraction process.

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