View metadata, citation and similar papers at core.ac.uk

brought to you by CORE

provided by Open Archive Toulouse Archive Ouverte

Open Archive Toulouse Archive Ouverte (OATAO)

OATAO is an open access repository that collects the work of Toulouse researchers and makes it freely available over the web where possible.

This is an author-deposited version published in: http://oatao.univ-toulouse.fr/
Eprints ID: 11955

To cite this version:
Evon, Philippe and Vandenbossche, Virginie and Pontalier, Pierre-Yves and Rigal, Luc *Aqueous extraction of oil from sunflower seeds in batch reactor: reorganization of the mixing in three formulated fractions.* (2007) In: 98th AOCS Annual Meeting & Expo, 13 May 2007 - 16 May 2007 (Québec City, QC, Canada). (Unpublished))

Any correspondence concerning this service should be sent to the repository administrator:
staff-oatao@inp-toulouse.fr

Aqueous extraction of oil from sunflower seeds in batch reactor: reorganization of the mixing in three formulated fractions

Ph. Evon, V. Vandenbossche, P.Y. Pontalier & L. Rigal
Laboratoire de Chimie Agro-Industrielle – UMR 1010 INRA/INP-ENSIACET
118 Route de Narbonne – 31077 Toulouse Cedex 4, France
Tél. + 33 (0)5 62 88 57 38 – Fax + 33 (0)5 62 88 57 30
E-mail address: Philippe.Evon@ensiacet.fr (Ph. Evon, corresponding author)

Aqueous extraction process is an alternative to the solvent oil extraction process from oilseeds. It enables simultaneous recovery of oil and protein. Water extraction of sunflower oil is carried out with a mixer (model Waring Blendor, USA) as batch reactor (seeds/water: 15/85). This only apparatus carries out two essential unit operations: conditioning and grinding of sunflower seeds and liquid/solid extraction. However, lixiviation of kernels is incomplete.
After five minutes of extraction, the mixture reorganizes by centrifugation in three formulated fractions: the insoluble phase (31.1% of the mixture), the hydrophilic phase (61.4% of the mixture) and the hydrophobic phase (7.5% of the mixture). Oil extraction yield obtained is 46.6%.
Residual oil content of raffinate is 39.2% instead of 49.7% in sunflower seeds. Aqueous extraction process also results in an impoverishment of oil cake into water-soluble substances contained initially in kernels: proteins and minerals. Raffinate is on the contrary richer in insoluble proteins and fibers (26.1% for cellulose and lignins instead of 17.1% in sunflower seeds). An additional stage of extraction would allow a better oil impoverishment of the insoluble phase before its possible use for animal feeding. It would be also possible to upgrade it by thermopressing. Panels obtained have mechanical characteristics comparable with those of other experimental materials.
Hydrophilic phase is the dominating fraction. It contains proteins (31.8% of dry residue), minerals (8.2% of dry residue) and hemicelluloses. Its oil content means that centrifugation does not allow an expedient separation between hydrophilic phase and hydrophobic phase. After concentration of organic substances by ultrafiltration, it is possible to collect proteins by isoelectric precipitation. These water-soluble proteins can be used for their surface-active properties.
Hydrophobic phase is an oil-in-water emulsion. It is lighter than hydrophilic phase (0.94 for its density). Lipids represent 83.8% of its dry residue. Its stability is ensured by the presence at interface of natural surface-active agents also extracted during the process, phospholipids and proteins. Demulsification is possible by alcoholic extraction. It enables the edible oil isolation. Oil consumption is possible for human feeding but also for non food uses like biolubricants market. Hydrophobic phase can also be used without any modification for the manufacture of paintings and cosmetics or for the treatment of surfaces with hydrophilic matter.

Aqueous extraction of oil from sunflower seeds in batch reactor: reorganization of the mixing in three formulated fractions

Ph. Evon*, E. Lacroux, V. Vandenbossche, P.Y. Pontalier, L. Rigal

Laboratoire de Chimie Agro-Industrielle, UMR 1010 INRA/INP-ENSIACET
118 Route de Narbonne, 31077 Toulouse Cedex 4, France
** Tel: (33)(0)5 62 88 57 38 – Fax: (33)(0)5 62 88 57 30 – e-Mail address: Philippe.Evon@ensiacet.fr*

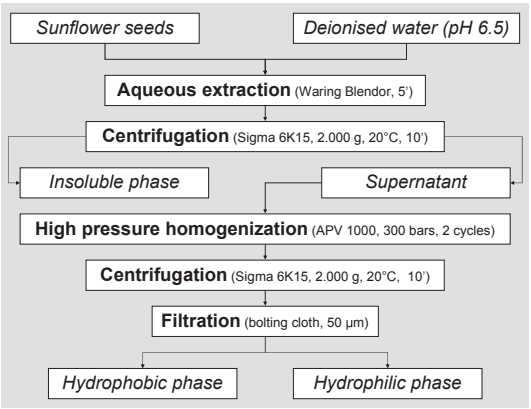
Introduction

► Industrial process for the extraction of edible oil from sunflower seeds involves a solvent extraction step which is preceded by pressing [1]. Hexane is the usual solvent in commercial operations for a long time. For such process, it is possible to achieve oil yields higher than 95% with a solvent recovery of over 95%.
► Hexane is toxic and carcinogenic. It can also contribute to the industrial emissions of volatile organic compounds (VOCs) which are greenhouse gases [1].
► Safety, environmental and health concerns have generated interest in alternative solvents. Current interest in ethanol, isopropanol and methylene chloride is high [2].
► Aqueous extraction process is another environmentally cleaner alternative technology to the solvent oil extraction process from oilseeds [1,3]. It enables the simultaneous production of oil-in-water emulsion (hydrophobic phase) and protein isolate (hydrophilic phase) in the same process.

Experimental

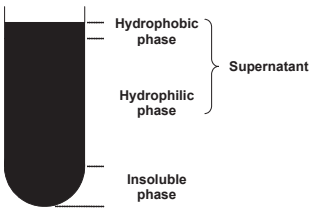
► **Sunflower seeds** (linoleic type, 2 mm homogenate) (La Toulousaine de Céréales, France):
- 7.5% of moisture content.
- Composition (% dry mass): 3.1% of ashes, 49.7% of lipids, 0.6% of phospholipids, 15.7% of proteins, 12.5% of cellulose, 6.9% of hemicelluloses, 4.7% of lignins.

► **Representation of aqueous extraction process:**



Results and discussion

► Aqueous extraction in mixer Waring Blendor produces three fractions. Hydrophobic phase is the lightest and the most valuable fraction. It is appeared as an oil-in-water emulsion (7.5% of the total mass of the medium).



Schematic distribution of the three fractions.

	Hydrophobic phase	Hydrophilic phase	Insoluble phase
Moisture (%)	55.7%	98.1%	69.8%
Ashes (% dry mass)	1.7%	8.2%	3.0%
Lipids (% dry mass)	83.8%	39.4%	39.2%
Phospholipids (% dry mass)	1.0%	-	-
Proteins (% dry mass)	10.9%	31.8%	15.0%
Cellulose (% dry mass)	-	-	19.0%
Hemicelluloses (% dry mass)	-	-	10.3%
Lignins (% dry mass)	-	-	7.1%

Chemical composition of the three fractions.

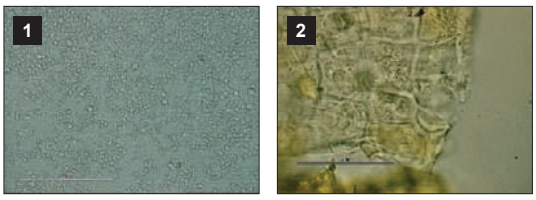
	Extraction medium (500.0 g)		Separate phases after centrifugation (500.0 g)			
	Sunflower seeds	Deionised water	Hydrophobic phase	Hydrophilic phase	Insoluble phase	Mass balance
Mass composition	75.0 g (15.0%)	425.0 g (85.0%)	37.7 g (7.5%)	307.1 g (61.4%)	155.2 g (31.0%)	
Water	5.6±0.0 g	425.0 g	21.0±0.2 g	301.2±0.0 g	108.3±0.1 g	100.0%
Lipids	34.5±0.1 g	-	14.0±0.1 g	2.3±0.1 g	18.4±0.2 g	100.6%
Proteins	10.9±0.1 g	-	1.8±0.0 g	1.9±0,0 g	7.1±0.0 g	98.4%
	Lipids extraction yields		40.6%	6.7%	46.6%	
	Proteins extraction yields		16.6%	17.0%	35.2%	

Mass balance for water, lipids and proteins. Extraction yields for lipids and proteins.

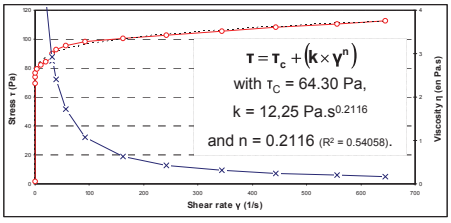
► Medium size of oil droplets in hydrophobic phase equals 1.54±0.38 µm. Good stability in time of this fraction is due to the presence of phospholipids and proteins at the interface.
► Demulsification is possible with ethanol but it is quicker with ethanol/ether mixture (3/1).
► Hydrophilic phase is the main fraction (61.4%). It constitutes an aqueous extract of the water-soluble components of the seeds, mainly proteins. The presence of lipids indicates that the separation between hydrophilic phase and hydrophobic phase is not optimal.
► Insoluble phase is relatively wet. It contains the cellular remains resulting from the destructuration process of sunflower seeds. All cotyledon cells are not lysed. This explains why the insoluble phase still contains as much oil.

	Hydrophobic phase	Water	Sunflower oil
Density (20°C)	0.943 ± 0.001	0.998 [4]	0.925 [5]
Minimal stress for flowing (25°C)	64.30 Pa	None	None
Viscosity (25°C)	142.14 Pa.s for 75 Pa, 0.64 Pa.s for 100 Pa	0.89 10^{-3} Pa.s [4] (Newtonian fluid)	53.65 10^{-3} Pa.s [5] (Newtonian fluid)
Surface tension (GBX 3S, 20°C)	84.3±6.9 mN/m	46.3±0.8 mN/m	34.3±0.4 mN/m

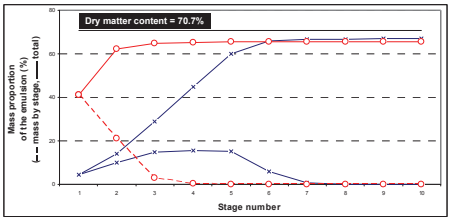
Physical characteristics of hydrophobic phase, water and sunflower oil.



Microscopic sights of hydrophobic phase (1) and insoluble phase (2) (optical microscope Nikon Eclipse E 600, objective with oil immersion, × 1,000).



Flow curve of hydrophobic phase at 25°C (rheometer CSL 500, diameter of cone: 40 mm, angle: 3°59).
○ stress τ, × viscosity η, ----- Herschel-Bulkley model.



Demulsification kinetics of an hydrophobic phase from twin-screw extruder with ethanol/ether mixture (3/1) (○) and ethanol (×) (w/v = 4/10, vortex 2').

Conclusion

► Aqueous extraction process enables simultaneous recovery of oil and protein from sunflower seeds. Water extraction of sunflower oil is made with a mixer which carries out two essential unit operations: crushing of the seeds and liquid/solid extraction. However, lixiviation of kernels is incomplete. After aqueous extraction, the mixture reorganizes in three formulated fractions: the insoluble phase, the hydrophilic phase and the hydrophobic phase.
► Aqueous extraction results in an impoverishment of the insoluble phase in oil and water-soluble proteins and in an increase in its fibers content. A reduction of the medium size of solid particles is observed (45 µm against 125 µm before extraction). One more stage of extraction would allow a better oil impoverishment of cake before its use for animal feeding.
► Hydrophilic phase contains mainly proteins. After concentration of organic substances by ultrafiltration, it is possible to collect these water-soluble proteins by isoelectric precipitation and to use them for their surface-active properties.
► Hydrophobic phase is an oil-in-water emulsion. Its viscosity and its surface tension are higher than those of water and commercial sunflower oil. Lipids represent nearly 85% of its dry residue. Its stability is ensured by the presence at the interface of surface-active agents, phospholipids and proteins.
► Hydrophobic phase is lighter than the aqueous extract. Its separation can be carried out by centrifugation but also by decantation.
► Demulsification of hydrophobic phase is possible by alcoholic extraction. It enables the edible oil isolation.
► Hydrophobic phase can also be used without any modification, for paintings and cosmetics but also for its surface-treatment properties.

References

[1] **Rosenthal A.**, **Pyle D.L.** & **Niranjan K.**, Aqueous and enzymatic processes for edible oil extraction, *Enzyme and Microbial Technology*, **19**, 402-420 (1996).
[2] **Johnson L.A.** & **Lusas E.W.**, Comparison of alternative solvents for oils extraction, *Journal of the American Oil Chemists' Society*, **60**, 229-242 (1983).
[3] **Mechling E.**, Mise au point d'un réacteur multitâches adapté à la production des composés oléophiles à partir des milieux réactionnels issus des graines oléagineuses (PhD), *Institut National Polytechnique de Toulouse*, France (2002).
[4] **IAPWS**, Revised release on the IAPS formulation 1985 for the properties of ordinary water substance, *International Association for the Properties of Water and Steam*, Erlangen, Germany, **15** (1997).
[5] **Oliomobile**, *Réseau des utilisateurs d'huile végétale carburant*, www.oliomobile.org (2006).

Laboratoire de Chimie Agro-Industrielle
Unité Mixte de Recherche INRA / INP - ENSIACET (UMR 1010)