

# Solvent solutions: comparing extraction methods for edible oils and proteins in a changing regulatory landscape. General introduction<sup>☆</sup>

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**Abstract** – Hexane is currently the dominant solvent in the oil extraction industry, valued for its efficiency, low cost, and ability to preserve the quality of oils and proteins. However, this solvent poses health and environmental risks due to its toxicity. Recently, EFSA recommended a reevaluation of hexane as a processing aid due to gaps in toxicity studies and population exposure data. Consequently, a reduction in tolerated residue limits or usage restrictions can be anticipated, potentially leading industrialists to consider alternative solutions. The series of articles introduced here aims to examine the potential consequences of replacing hexane with alternative solvents listed in Directive 2009/32/EC. This comprehensive dossier, comprising eight articles, thoroughly investigates various critical aspects of oilseed extraction. The series explores physicochemical properties (Part 1), safety considerations (Part 2), processing impacts (Part 3), energy consumption (Part 4), oil quality (Part 5), protein meal quality (Part 6), technological comparisons (Part 7), and alternative extraction technologies (Part 8). This in-depth exploration provides a holistic analysis of the challenges and opportunities associated with transitioning from hexane to alternative solvents in the oilseed extraction industry.

**Keywords:** Hexane / solvent extraction / EFSA re-evaluation / properties / alternative technologies

**Résumé** – **Comparaison des méthodes d'extraction pour les huiles alimentaires et les protéines dans un contexte réglementaire en évolution. Introduction générale.** L'hexane est actuellement le solvant dominant dans l'industrie de l'extraction d'huile, apprécié pour son efficacité, son faible coût et sa capacité à préserver la qualité des huiles et des protéines. Au revers de ces avantages, ce solvant présente des risques pour la santé et l'environnement en raison de sa toxicité. Récemment, l'EFSA a recommandé une réévaluation de l'hexane en tant qu'auxiliaire technologique en raison de lacunes dans les études de toxicité et les données d'exposition des populations. On peut donc s'attendre à une réduction des limites de résidus tolérés ou des restrictions d'utilisation pouvant amener les industriels à considérer des solutions de substitution. La série d'articles introduite ici vise à examiner les conséquences potentielles du remplacement de l'hexane par des solvants alternatifs listés dans la directive 2009/32/CE. Cette série de 8 articles, constitue un dossier complet, examinant en profondeur divers aspects critiques de l'extraction des graines oléagineuses. Ainsi seront examinés : les propriétés physico-chimiques (Partie 1), la sécurité (Partie 2), les impacts sur le traitement (Partie 3), la consommation d'énergie (Partie 4), la qualité de l'huile (Partie 5), la qualité des tourteaux protéiques (Partie 6), les comparaisons technologiques (Partie 7) et les technologies alternatives d'extraction (Partie 8).

**Mots clés :** Hexane / Extraction par solvant / Réévaluation par l'EFSA / Propriétés / Technologies alternatives

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### Highlights

- Hexane is the dominant solvent in the oil extraction industry due to its efficiency, cost-effectiveness, and ability to preserve oil and protein quality, despite its toxicity and risks to workers.
- The European Food Safety Authority (EFSA) has recommended a reevaluation of hexane's safety as a processing aid, considering new toxicity data, which may lead to stricter regulations on its use.
- This has prompted comparisons between hexane and alternative solvents, evaluating factors such as safety, energy consumption, oil quality, and processing costs to assess potential shifts in industry practices.
- The series of eight publications this article is introducing thoroughly examines various critical aspects of oil extraction, divided into eight sections covering topics such as physical and chemical properties, safety, processing impacts, energy consumption, oil and protein meal quality, technological comparisons, and alternative extraction technologies.

## 1 Hexane's industrial dominance: navigating potential regulatory shifts in the EU extraction solvent landscape

Hexane is virtually the only solvent used in the crushing industry today, due to the many advantages it offers in terms of extraction efficiency, energy savings, ease of recycling and preservation of the quality of the oil and proteins extracted. However, it owes these advantages to its great hydrophobicity, whereas living organisms, with all their biochemistry, are poorly adapted to handling these hydrophobic substances, which gives these a certain toxicity, particularly for workers exposed to their vapours. Though, given the ability of refining processes to reduce solvent residues in edible oil to below 1 mg/kg, health authorities in Europe consider that the level of risk caused by this toxicity is acceptable for consumers. In the case of proteins derived from hexane defatting, a threshold of 10 mg/kg has also been adopted. In the USA, the FDA allows the use of hexane in food processing but does not specify maximum residue limits (MRLs) for hexane in oils and proteins. The FDA considers hexane as a "processing aid" rather than an ingredient, which means it is not required to be listed on food labels. The only established threshold for that solvent is 50 ppm in fish proteins isolates (US FDA, 2024). Canada legislation has determined MRLs of 10 and 25 ppm for respectively edible oils and fats and spices extracts (Canada, 1978). In Australia and New-Zealand, the hexane residues are limited to 0.3 mg/kg (Food Standards Australia New Zealand, 2023). We have not been able to find MRLs in other world parts with the exception of countries such as Turkey, whose legislation is modelled on that of the European Union.

Recently, following a request from the European Commission, the European Food Safety Authority (EFSA) was consulted on the need for a re-evaluation of hexane as a

processing aid for this use because of doubts about the hazards it could present. The panel on contact materials, enzymes and processing aids (CEP) has a working group on solvents which has met several times since 13 November 2023 (question EFSA-Q-2023-00719, EFSA, 2024a).

The technical report resulting of this work has been released on September 16<sup>th</sup> 2024 (EFSA, 2024b). Its main conclusion is that there is a need for a re-evaluation of the safety of technical hexane used as an extraction solvent in food production. The key implications can be summarized as follows:

- about the composition of technical hexane: It is recommended to obtain analytical data on the solvent composition, including the expected range of n-hexane and other constituents, as well as toxicologically relevant impurities (such as benzene, mineral oil hydrocarbons and polycyclic aromatic hydrocarbons). Information should be gathered on the solvent extraction process for foodstuffs and food ingredients, particularly regarding the amounts of technical hexane used and its recovery after extraction, as well as current applications of technical hexane as an extraction solvent. Specifications should be established based on compositional data, including actual impurity levels.
- population exposure levels: A refined exposure assessment can only be performed after collecting information on: (i) measured data on the composition of technical hexane used for extraction purposes, (ii) its range of applications, (iii) knowledge of technical hexane residues in food and extracted food commodities, and (iv) consumption of specific products, *e.g.* hexane-extracted soy proteins.
- Toxicity: The study on which the current maximum residue limit (MRL) assessment is based is not being challenged but no longer meets current standards. Recent publications report new information on the effects of n-hexane and its metabolites, such as kidney and liver toxicity, immunotoxicity, endocrine disrupting activity, male and female reproductive toxicity, ability to cross the placental barrier, and developmental toxicity, which were not examined in the original TNO-CIVO study or considered in the SCF opinions. These publications still need to be evaluated. Initially, no new toxicity studies are requested, but an analysis of recent publications needs to be conducted.

The report did not address the issue of possible transmission of solvent residues in the food chain via oilseed meals.

## 2 Assessing the ripple effects: safety, processing, energy, and quality. implications of extraction solvents choice

Considering the conclusion of this EFSA report on technical hexane and the potential for more restricted conditions of use, it seems timely to conduct a comparison between hexane and other solvents that oilseed crushers would likely use under current regulations. This comparison will focus specifically on solvents listed in the annex to Directive 2009/32/EC that are comparable to hexane. The list of solvents

was limited to those that could be immediately adopted by the oil mill industry if more stringent restrictions were imposed on hexane emissions or residue levels in extracted products. This comparison will consider the main aspects of the operation of extraction facilities and measure the impacts induced by the physicochemical properties of these solvents on the different processing stages, weighing up the consequences of these differences on the multiple parameters that industry must manage.

Hexane is employed in the crushing industry because it makes it possible to extract almost all the oil contained in the seeds at a limited cost (Unger, 2011) contrary to mechanical extraction which has physical limitations. In another article (Carré, 2024) we explain why mechanical extraction is economically less profitable than conventional processing including a solvent extraction step. In this articles series we will examine the ability of the available solvents to replace hexane in existing facilities, *i.e.*, the solvents which boil in a similar range of temperatures.

To make this comparison, it will be necessary to study the physical and chemical properties of the solvents, especially their affinity for the oils, their minor components and their impurities. Because safety and hazard management are paramount in modern industry, we will compare the toxicological aspects as referred to in harmonised classification and labelling data from the European Chemical Agency. Explosivity, flammability and leaks prevention will follow with consideration to the prevention of these hazards. Another important aspect is the impact of the different solvents on the processing in regard to their specific requirement for change in preparation of oil-bearing materials, management of the water and solvent recovery. Obviously, these effects have consequences on the energy consumption, which will be the subject of particular attention. We will also examine how solvents affect oil quality, focusing on their capacity to dissolve desirable or undesirable substances and their interaction with the enzymatic activity of the vegetable material. These enzymatic activities could also affect the quality of the meal, as well as the volatility of the solvent which can strongly affect the protein digestibility via the intensity of the thermal transformation of the meal related to the desolventization operation. The ensemble of these parameters will impact the processing cost and market value of the products derived from the oilseeds processing.

### 3 Oil mill industry today

The European territory has about 50 oil mills falling in the scope of the European regulation about industrial activities which are presenting risks for the environment and which emissions are subject to legal limits (see Sect. 1, paragraph about the emissions). About 10 of these oil mills are processing only soybean, 35 are crushing only rapeseed or sunflower (soft seeds) and 5 are able to switch from one kind of seed to the other (Santonja *et al.*, 2019). The processing of oilseeds is generally presented as a two step process with a preliminary preparation of the oilseed and a solvent extraction. The preparation must transform the seeds to make them suitable for extraction. This means giving the solvent access to the oil droplets enclosed in the seed cells, by combining cell rupture

(flaking, screw pressing, extrusion) with other operations such as granulation and expansion. The aim of the latter is to give the material a particle size enabling rapid diffusion of the solvent into the aggregates, and a porosity of the material beds in the extractor enabling good percolation of the solvent. In the case of soft seeds, this first step includes a mechanical extraction of the oil removing about two thirds of the seed oil and leaving 18–22% of oil in the pre-press cake. With soya, the beans are cracked, heated and flaked. The flakes like the press-cake are sometimes passed through expanders or pelleting presses to improve the percolation.

The extraction step is carried out in counter-flow extractors of different technologies for minimising the mass of solvent required. They are operated under atmospheric pressure at temperatures between 50 and 65 °C. The miscella (mixture of oil and solvent) exiting the extractor contains 15–30% of oil while the marc (defatted meal solvent-laden) retains 20–35% of solvent. In both cases, the solvent is evaporated by heating and then recovered by condensation. For the oil, the operation is made under vacuum because the boiling point of the oil-solvent mixture rises sharply over 100°C when the concentration of the residues decreases below 2–3%. The stripping of the hexane residues is made in a multistage column with the aid of steam allowing to decrease the residue level below 1000 ppm. The final elimination of these residues is made during the oil refining process thanks to the deodorisation operation which involves heating the oils at temperatures from 180 to 260°C under very low pressure (~1–5 mbar) and with the aid of water steam serving as an entrainer. The meal is processed in a desolventizer-toaster where, by heat transfer and by counterflow steam entrainment the solvent is removed. The main difference with the miscella is that the desolventizer is operated at atmospheric pressure. The affinity of the solvent for the oil is much greater than for the meal therefore, it is possible to reach acceptable residues levels without the necessity to raise the temperature far beyond 100 °C. The heat of vapours exiting the desolventizer is transferred to the miscella for minimising the energy consumption (see Fig. 1 in Article 3 about the processing for a flowsheet diagram of the process).

Because direct steam is employed for stripping the oil and the meal of their solvent residues, some water is condensed with the solvent. Water and hexane are not miscible; therefore, it is easy to separate the two liquids in a static decanter. The water going out of the system can retain a weak content of hexane, in consequence it passes across a boiling step where the water is heated above the solvent boiling point for the latter's recovery. Since there is a small stream of air entering in the extractor with the oil-bearing material, this air has to be evacuated after a passage in an absorption column in which the solvent residues are absorbed in cold mineral oil. This mineral oil is then stripped of its solvent by heating.

### 4 What is a good solvent for extracting vegetable oils?

To satisfy the oil-mill operator, the solvent of choice has to meet a large number of criteria. The first one concerns the affinity of the solvent for the oils. Oils are hydrophobic and require solvent with similar behaviour. The solvent must be specific to lipids and should avoid extracting undesirable

substances. It also needs to be easy to recover as well from the oil as from the protein meals because it is a processing aid that must not be present in the final product. It must be safe to use, present no danger to workers exposed to it, not react with products, not pollute the environment through leaks or at the end of their life, and its residues that cannot be eliminated at the end of processing must not be toxic to humans or farm animals who consume them. On the economic point of view, it must be cheap, its cycle from extraction to recovery by evaporation has to require little energy consumption, the pressure at which it is used is preferably atmospheric, its flammability as low as possible, and it must be easy to contain and store. All these aspects will be discussed for the selection of solvents as well as the miscibility of the solvent with water which has several important consequences.

## 5 An article divided in several parts

This series of articles offers an in-depth exploration of a number of critical aspects of the subject at hand. Due to the breadth and complexity of the topics covered, the article is thoughtfully divided into eight comprehensive sections. Each section delves into a specific area of interest, allowing for a thorough examination and understanding. The sections are as follows:

- 1 Physical and chemical properties.
- 2 Safety and hazard management.
- 3 Processing impacts.
- 4 Energy consumption.
- 5 Oil quality.
- 6 Protein meal quality.
- 7 Overall comparison and technological levels of readiness.
- 8 Alternative technologies

The eighth part will address techniques which are out of the scope of the seven precedent ones. It will consider supercritical CO<sub>2</sub> extraction, aqueous extraction with enzymatic preparation, use of solvents out of the 2009/32 directive annex, deep eutectic solvents and other possible technologies. These techniques will be assessed for their economic feasibility in the current industrial context.

## Author contribution statement

**Patrick Carré**: conceptualization, administration, original draft, review & editing. **Sebastian Berthold**: conceptualization, administration. **Thomas Piofczyk**: conceptualization, validation, review & editing. **Sarah Bothe**: validation, review & editing. **Sara Hadjiali**: validation, review & editing.

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