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Solvent Solutions: Comparing Extraction Methods for Edible Oils and Proteins in a Changing Regulatory Landscape.
Part 7: Overall comparison between solvent solutions[☆]

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Abstract – Oilseeds extraction of oils requires the use of a processing aid : a solvent. This seventh article on the comparison of solvents potentially capable of replacing hexane presents a multi-criteria analysis, systematically revisiting aspects from previous investigations. The methodology addresses the problem's complexity by aggregating different dimensions representative of each criterion, resulting in a nuanced ranking approach. The comprehensive study examined critical dimensions including solvent extractive efficacy, operational safety, explosion risk, toxicity to workers, environmental impact, process modifications, energy consumption, effects on products quality, residual toxicity, technology readiness level, and economic feasibility. From a technical standpoint, hexane today remains the most efficient solvent for oilseed crushers. Dichloromethane may be a viable alternative due to its non-explosive nature, though its toxicity concerns are significant. 2-methyloxolane is the subject of ongoing research and development investments, which may ultimately establish it as a credible alternative solution. Different stakeholders present varied perspectives on solvent replacement. For worker safety, isopropanol and ethanol are preferable options. Environmental advocates may favor ethyl acetate because of its lower energy requirements during processing. Regarding food safety, both ethanol and ethyl acetate are generally regarded as acceptable solvents. Public authorities, for their part, must balance what is desirable with what is feasible, in a multi-criteria framework of balancing safety, toxicity, environmental impact, availability, and cost on a global scale.

Keywords: Solvents / Extraction / Safety / Quality / Environment

Résumé – Solvants d'extraction : Comparaison des méthodes d'extraction pour les huiles alimentaires et les protéines dans un contexte réglementaire en évolution. Partie 7 : Comparaison globale des solvants. L'extraction d'huile à partir de graines nécessite aujourd'hui le recours à un auxiliaire technologique: un solvant. Ce septième article sur la comparaison des solvants potentiellement capables de remplacer l'hexane présente une analyse multicritères, revisitant systématiquement les aspects des précédentes investigations. La méthodologie aborde la complexité du problème en agrégeant différentes dimensions représentatives de chaque critère, créant une approche de classement nuancée. L'étude exhaustive a examiné des dimensions critiques incluant l'efficacité extractive du solvant, la sécurité opérationnelle, la le risque d'explosion, la toxicité pour les travailleurs, l'impact environnemental, les modifications du process, la consommation d'énergie, la qualité des produits, la toxicité résiduelle, le niveau de maturité technologique et la faisabilité économique. D'un point de vue technique, I'hexane demeure aujourd'hui le solvant le plus performant pour les triturateurs. Le dichlorométhane pourrait constituer un substitut pertinent en raison de sa nature non explosive, mais sa toxicité est problématique. Le 2-méthyloxolane fait l'objet d'investissements continus en recherche et développement, susceptibles d'en faire à terme une solution alternative crédible. Pour la sécurité des travailleurs, l'isopropanol et l'éthanol sont des choix préférables. Sur le plan environnemental, l'acétate d'éthyle pourrait être favorisé en raison de sa moindre consommation d'énergie; l'éthanol serait plus acceptable du point de vue de la sécurité sanitaire

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[☆] Contribution to the Topical Issue: "Extraction solvents / Solvants d'extraction".
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des aliments. Les pouvoirs publics doivent arbitrer entre le possible et le souhaitable et penser en termes de coûts et bénéfices multicritères (sécurité, toxicité, environnement, accessibilité, etc.) à l'échelle globale.

Mots clé : Solvants / Extraction / Sécurité / Qualité / Environnement

Highlights:

The desirability of solvents depends on one's perspective, and each viewpoint is legitimate,

- Hexane: Currently used but raises health and sustainability concerns,
- Ethanol/isopropanol: Consumer-friendly but challenging for manufacturers and energy efficiency,
- Ketones: Low TRL (Technical readyness level),
- Dichloromethane: Reduces explosion risks but highly toxic,
- Ethyl acetate: Sustainable but lacks industrial references,
- 2-methyloxolane: Better toxicological and environmental profile, advanced TRL whilst not industrially proven, and less economically competitive.

1 Introduction

In the preceding sections, we have endeavoured to compile the existing knowledge on the properties of solvents potentially used for vegetable oil extraction. We have observed that their physicochemical properties lead to substantial differences in the operation of extraction units, ranging from the quantities of solvent to be implemented, to the quality of oil and meal, through raw material preparation, explosion prevention, energy consumption, solvent recycling methods, treatment of used air and water We have also noted that the level of existing knowledge is rather heterogeneous when moving from one solvent to another. This knowledge gap extends even to hexane, despite its decades of use. The latest EFSA opinion regarding hexane dates back to 1994 and is based on a toxicological study from 1989. This 90-day study aimed to determine a no-observed-effect level (NOEL), which does not allow for the evaluation of potential effects from long-term exposure to very low doses. Similarly, knowledge about the residual solvent quantities in refined oils is rather limited. In a recent article, Cravotto *et al.* (2022) reviewed the available studies on this subject. Of the 9 studies identified, only 6 focused on oils intended for human consumption. Collectively, these studies evaluated residues in 105 oils samples and identified ten cases where concentrations exceeded of 1 mg/kg. Among these 10 out-of-limit analyses, 6 came from a single study (Ramezani *et al.*, 2018) examining 10 oils randomly sampled from markets in Iran. This study contradict those of Yousefi and Hosseini (2017), who found no samples with concentrations above 42.6 µg/kg in 40 oils sampled in the same country. It is worth noting that the values observed by Ramezani *et al.* reach levels of around 10 mg/kg, which seems atypical for normally refined oils. Of the 4 other out-of-limit samples, 2 concerned olive pomace oils sold mixed with virgin oils (Peña *et al.*, 2003), which may suggest atypical deodorisation, as the taste would be masked by that of the added oil. Another out-of-norm oil is described as Chinese pepper oil, which is also an considered atypical. The last out-of-norm oil is a soybean oil sampled in Korea (Oh *et al.*, 2005). Cravotto *et al.* did not mention the study by Downes and Rossel (1984), who examined hexane content in 23 samples of refined oils in Great Britain and found found no values above the detection limit" (50 µg/kg). Hirayama and Imai (1977) studied the evolution of hexane content in oil during refining and showed that levels were no longer detectable after deodorisation (quantification limit 1 mg/kg).

From a theoretical perspective, Van Duijn (2016) estimates that the majority of hexane is eliminated in the early stages of refining (drying and bleaching) due to its high volatility, thereby creating a risk of explosive atmospheres in gaseous effluents. In conclusion, it can be considered that, in the absence of anomalies, refining ensures compliance with the maximum set by Directive 2009/32/EC (EP&CoEU, 2009), with residues generally below 50 µg/kg and mostly below 5 µg/kg. It is important to note that these values primarily originate from the findings of Youssefi and Hosseini (2017), who employed a gas chromatography method having a quantification limit of approximately 100 µg/kg (ITERG, personal communication). Consequently, caution should be exercised when interpreting these values.

It is also interesting to mention the study by Flecker *et al.* (2019) which reported the presence of various hydrocarbons in virgin oils caused by the thermal decomposition of organic substances. This study demonstrated that virgin pumpkin seed oils can contain up to 1.6 mg/kg of pentane and that hexane forms when the seeds are subjected to a temperature of 120°C. Liu *et al.* (2023) detected in rapeseed and soybean oils obtained by mechanical extraction only the presence of n-hexane up to 0.36 and 0.58 mg/kg respectively, which the authors attribute to the contamination of these oils by lubricants. The possibility of contamination from laboratory air should also be considered since hexane is ubiquitous in laboratories analyzing fats.

Regarding the oilseed meals used in the animal feed sector, residue levels are seldom measured. However, the IO7 database has 11 results for rapeseed meal ranging from 0 to 41 mg/kg (average 6.4 mg/kg) and 12 results for soybean meal, all below the quantification threshold. These values are much lower than the residue levels found in meals at the exit of extraction plants because hexane continues to desorb during handling and transport operations, especially in the case of soybean meals which are generally produced in Brazil or Argentina before reaching feed mills in Europe. Although relatively reassuring, these data do not cover a sufficient number of samples to establish reliable statistics. Despite our best efforts, we were unable to find studies addressing the potential risk of hexane residues in farm animals or the possibility of these residues passing into animal products. This lack of available research suggests that the issue has not yet attracted significant attention among scientists in the field of animal science.

Ultimately, the data on the level of presence of hexane residues in the food chain remains insufficient, as highlighted in the recent EFSA technical report (EFSA, 2024). The report emphasises that too few reliable studies have been conducted using sufficiently precise methods on samples representative of the market. Furthermore, EFSA notes a significant knowledge gap regarding the general population's exposure to solvent residues, calling for further in-depth investigation in this area.

In order to produce a general comparison, given the multiplicity of criteria that we have examined one after the other, we need to apply a degree of simplification, taking care not to over-simplify. Overly simplistic approaches such as absolute prioritisation of safety are not effective because they tend not to take into account the complexity of issues and can lead to undesirable effects economically, environmentally, and in some cases, on safety through indirect effects. Thus, the simplest way to eliminate the proven and potential dangers of solvents would be to do without them entirely and return to a purely mechanical extraction accepting the associated economic burden. However, in doing so, we would introduce additional risks, such as the risk of fire in expeller cake warehouses by residual oil autoignition, restricting the use of European meals in feed formulations for compound feeds, while increasing their importation, reduce local vegetable oil production, inducing the need to produce more oilcrops with a deforestation effect damaging to climate and biodiversity, or to give up energy uses of oils which would be compensated by increased use of fossil fuels and, in the medium term, an increased risk for populations due to the accentuation of GHG emissions.

Recognising this complexity of phenomena should not be a cause of paralysis, but rather an invitation to develop a thought capable of accounts for uncertainty, contradictions and interdependencies inherent in our reality (Morin, 1987). It is therefore necessary at this stage to proceed with a simplification by grouping the criteria examined while trying not to erase the most important dimensions relating to the broader analysis. This simplification, which leads us, for instance, to posit the industrialist as the bearer of economic constraints, is deliberately reductive. We must remain cognizant that the industrialist in question is an abstraction that should not be conflated with the real industrialist–a human being who is multidimensional, unique, and concerned with values beyond the purely economic.

For this, we have retained seven criteria: 1/ Extraction efficiency and oil yield, 2/ Safety and hazard management, 3/ Environmental impact, 4/ process compatibility and retrofitting, 5/ energy consumption, 6/ product quality and compliance, 7/ Technical and economic feasibility. These choices were driven by the necessity of having objective data to enable the intended comparisons, which led us to exclude other criteria, such as solvent losses in water, air, and meal, for which published results are virtually non-existent. This does not imply that the criteria not mentioned are unimportant or that they should not be the subject of further studies before any decision is made to proceed with the replacement of hexane by another solvent.

For each criterion, we have retained a small number of parameters for which the solvent desirability was assessed. A score of 5 was assigned to the most desirable outcome and 1 to the least desirable.

2 Extraction Efficiency – oil yields

2.1 Neutral oil solubility

The criterion taken into account was the Hansen Solubility Parameter Ra distance between the rapeseed oil and the different solvents. The values given in Table 3 of the first article of this series were used for this scoring (Carré et al, 2024c). A score of 5 is assigned for Ra distance below 6, score 4 for Ra distance between 6 and 8; score 3 for Ra distances between 8 and 12, score 2 for distance between 12 and 20, and score 1 for Ra distance beyond 20.

2.2 Phosphatidylcholine solubility

Phosphatidylcholine is the most important phospholipid in vegetables. It has been chosen as a marker of miscibility in polar lipids. The scoring assumes that a good extraction of phospholipid is a desirable achievement which is not always desirable. The scoring scale is the same as for rapeseed oil, relying on the Ra distance between the PC and the solvents. The values given in Table 4 of the first article of this series were used for this scoring (Carré et al, 2024d). The ability to efficiently extract polar lipids is considered beneficial to reduce their presence the extraction of total lipids and reducing their presence in protein meals. It is also justified since residual polar lipids in protein meals can lead to organoleptic issues when these meals are utilised for human consumption (Vioque *et al.*, 2000).

A score of 1 was given to the ketones although the RA HSP distance is predicting much better affinities. adjustments were made for the known poor miscibility of phospholipids in acetone and by extension in ketones. Methanol is also a mediocre solvent for PC and receives a score of 2. Hexanes and ethanol share the score of 3 while isopropanol receives a score of 4 while EA, DCM and 2-MeOx are rated 5.

2.3 Non-lipids solubility

Two families of compounds have been assessed: phenolic compounds (isoflavone, kaempferol and sinapic acid) and chlorophyll using the Ra distance with the solvent using Table 4 in the first part of this work (Carré *et al.*, 2024d).

Contrarily to previous scales, the highest distances were considered as more desirable because although beneficial antioxidants, phenolic compounds may impart bitterness and chlorophyll an undesirable dark colour. These compounds are removed during the refining process. In consequence the highest grade was given to Ra distances above 20, score 4 to distances between 15 and 20, score 3 for distance between 12.5 and 15, score 2 between 11.25 and 12.5, and score 1 for Ra distance under 11.25.

For phenolics, no solvent receives a score of 1, DCM is the only solvent rated 2, Ethanol, IPA, Acetone, MEK, EA and 2-MeOx fall into category 3, methanol obtains a score of 4, and hexanes get a score of 5.

For chlorophyll, it is ethanol which gets the lowest score followed by methanol which is rated 2. IPA and DCM are in medium position with a score of 3. Acetone, MEK, EA et

Table 1. Solvent selectivity and fitness for lipids

Criteria	nHx	Ihx	CHx	MeOH	EtOH	IPA	Acetone	MEK	EA	DCM	2-MeOx
Global selectivity	**4.2**	**4.2**	**4.2**	**1.4**	**2.1**	**2.3**	**2.3**	**2.9**	**4.3**	**3.6**	**4.3**
Neutral lipid score	5	5	5	1	2	2	3	4	5	4	5
Polar lipids score	3	3	3	2	3	4	1	1	5	5	5
Phenolics score	5	5	5	4	3	3	3	3	3	2	3
Chlorophyll score	5	5	5	2	1	3	4	4	4	3	4

2-MeOx have lower chlorophyll affinity and receive a score of 4 while hexanes are the solvents which are the least prone to extract this substance.

2.4 Global solvent selectivity

Global selectivity has been assessed with a ponderation considering that a good affinity for neutral lipids is much more attractive that the affinity for polar lipids; the score for neutral lipids was pondered with a coefficient 3. Non-lipids miscibility being represented by two criteria, each criterion is pondered with a coefficient 0.5.

The global selectivity gradation (Table 1) gives the best score to hexanes, ethyl acetate and 2-methyloxolane with a small advantage for the non-hexane solvents explained by a better affinity for polar lipids. This ranking should be interpreted with nuance, as the actual Ra distance for rapeseed oil, as well as for other major edible oils, is significantly more favourable for 2-MeOx than for EA. Moreover, a substantial body of published results supports this assessment. In consequence, the 2-methyloxolane could be considered as the best choice for this criterion.

On the other extremity of the scale, methanol is definitely the worst choice for extracting vegetable oils. Ethanol and isopropanol and acetone present limited selectivity for lipids but this characteristic may allow separation from oil by of neutral lipids as a means to separate the solvents from oil by cooling the miscellas and avoiding the distillation steps. MEK and DCM have intermediate scores for the selectivity which make them less attractive. In the case where phospholipids are not recovered for the valuation as lecithin, the poor miscibility of phospholipids in ketones may change the perception of these solvents because they also have a relatively interesting selectivity for the chlorophyll. DCM is the solvent which is likely to bring the highest level of impurities in the oils.

3 Safety management

3.1 Method

This criterion will encompass most of the aspects concerning the safety related to the use of the solvent at industrial level. It will gather the questions related to fire and explosion hazard and occupational safety. As for other parameters, the solvents will be graded in 5 levels of desirability, from the least to the most desirable using a non-linear scale based on parametric indicators. The parameters for the flammability are the flash point, the potential to form explosive atmospheres at ambient temperature, the potential to accumulate static electricity. For the health of workers, the occupational exposure limits (OEL), and a composite composite occupational safety score (COSS) were considered. OEL has been separated from COSS as they impose regulatory constraints on the processor, necessitating the implementation of detection and ventilation systems, which have implications for costs and process control. For further details and origin of the information refer to Carré *et al.* (2024b).

3.2 Flash point (FP)

With FP below −20°C, hexanes clearly could be considered as the most flammable solvents and receive the score 1. The second group, scored 2, includes solvents with flash points between −10 to −4.4°C (EA, MEK, acetone and 2-MeOx). The third group (score 3) presents FP from 12 to 16°C and is constituted of alcohols (MeOH, EtOH, IPA). DCM, which is not flammable, is rated 5.

3.3 Potential to present explosive atmospheres at ambient temperatures in air-tight vessels

A solvent like hexane does not generate ATEX (explosive atmospheres) in enclosures where it is present at ambient temperature, significantly reduces the frequency at which installations present ATEX conditions. This is not always the case with less volatile solvents, for which ATEX classification rules may need to be revised, potentially necessitating changes in equipment specifications or implementation procedures. Our classification assigns a score of 5 to DCM, which does not form ATEX; 3 to hexane and acetone, whose volatility precludes ATEX formation at ambient temperature; 2 to 2-MeOx, which slightly exceeds the LEL (Lower Explosive Limit) at 20°C; and 1 to all other solvents that form ATEX at this temperature.

3.4 Static electricity accumulation potential

The ease with which a solvent can accumulate static electricity is a key safety concern, as it may serve as a triggering factor for ignition in the presence of an ATEX. Solvents were classified into 5 desirability levels based on their electrical conductivity. Hexane is rated 1 due to its very low conductivity and the very low solubility of water in this solvent. DCM is rated 2 for its significantly higher conductivity coupled with low water solubility. 2-MeOx and ethyl acetate are rated 3 as they exhibit comparable conductivity and the ability to dissolve a limited, easily saturated amount of water. All remaining solvents are scored 5 as they present a lower probability of existing in a biphasic state in the presence of water and exhibit higher electrical conductivities.

3.5 Occupational exposure limit values

With an occupational exposure limit (OEL) of 20 ppm in France, **hexane** imposes significant constraints on the working environment and is therefore rated 1 for this criterion. DCM, with an OEL of 50 ppm, is rated 2. Most other solvents have OELs of 200 ppm and are rated 3. **Acetone** and **isohexane**, which are permitted up to 500 ppm, are rated **4**. Ethanol shows a substantial difference between its German and French OELs; consequently, it is also rated 4. 2-MeOx does not yet have an established OEL. It receives a temporary grade of 3 for the sake of this comparison.

3.6 Harm severity index and composite occupational safety score

The severity index was constructed based on the ECHA' harmonised classification & labelling by assigning a severity score according to the harmfulness criteria presented in Article 2 of this series. A score of 10 was assigned to criteria H304 (fatal if swallowed or inhaled) and H351 (suspected carcinogen). A score of 5 was assigned to hazards H301 (moderate acute toxicity), H318 (severe eye damage), H341 (suspected of causing genetic defects), H351 (when notified by a few suppliers to ECHA), H370 (specific target organ toxicity – single exposure, higher severity), and H372 (specific target organ toxicity – repeated exposure, higher severity). A score of 3 was applied only to ethanol for the H370 risk, which is outside the harmonised classification and whose risk associated with excessive ingestion is poorly suited to the context of industrial use. A score of 1 was assigned to all other indicators of lower harmfulness. Table 2 presents the detailed noxiousness indicators and corresponding severity index which is the sum of the score for each solvent. In Table 3 of the second article of this series focused on risk management, a solvent may appear at two levels of the same risk category, being either officially recognised by ECHA, identified by a majority of suppliers, or by a minority thereof. The risk considered is the one validated by ECHA or, in absence of harmonised C&L, by a majority of suppliers.

With a score of 23, *n*-hexane receives the highest severity rating, followed by dichloromethane (DCM) and methanol, which aligns with expectations. The isomers of *n*-hexane display significantly lower scores but remain higher than most than other solvents, primarily due to their H304 classification. 2-MeOx presents elevated risks due to its classification for ocular damage (although not harmonised within the ECHA C&L inventory). Ethanol follows, owing to the intoxication risks it may cause, which could be debatable in an industrial context of workplace hazards. IPA, ketones, and ethyl acetate are the solvents that can be considered the safest.

The severity index was converted into an Occupational Safety Score using a 5-point scale, where 1 corresponds to scores ≥ 20, incrementing by 1 for each 5-point decrease in hazard score, up to 5 for scores < 5.

3.7 Global safety appreciation

Table 3 summarises our safety criteria and provides a comprehensive evaluation of solvent safety in the workplace, incorporating criteria related to flammability and health hazards. The composite criterion COSS, which synthesises a significant number of aspects, was given double weight in calculation of the overall score. This overall score is the weighted average across all criteria.

No solvent achieves the maximum score of 5, as the only non-flammable option, DCM, exhibits significant toxicity concerns. The lowest-rated solvent is *n*-hexane, which has a score of 1.3. Its isomers score one point higher but still rank among among the least favourable solvents, placing them close to methanol and DCM. 2-MeOx and ethyl acetate occupy an intermediate position, closely aligned, compared to ketones, ethanol, and IPA. Acetone ranks highest, followed by IPA, while ethanol and MEK are tied at 3.5 points.

Technical hexane would currently be considered marginally less harmful than *n*-hexane according to the harmonised ECHA classification in terms of neurotoxicity (STOT RE 2 vs. STOT RE 1). However, considering the recent reclassification of *n*-hexane to the maximum severity level for this risk, it is reasonable to disregard this distinction, given the shared H361f classification (reprotoxic) applied to technical hexane if it contains more than 5% n-hexane. Out of caution, the occupational risk score would decrease from 24 to 19, its COSS score would increase by one point, and the safety desirability index would shift from 1.3 to 1.7, without impacting the current ranking.

4 Environmental impact

4.1 Method

The criteria considered for evaluating environmental impacts encompass the ozone-formation potential of solvent vapours and the classification provided by the pharmaceutical industry, which synthesises a broad range of criteria. The criterion concerning ozone is the Photochemical Ozone Creation Potential (POCP), which we have adopted from Anderson-Sköld *et al.* (1992) and Derwent *et al.* (1996). The POCP is a relative index that compares the ozone formation under European summer conditions for a given substance to that produced by ethene. These values vary over time, which is why in each study, the values over the longest periods were retained. Neither of these studies mentions the POCP of 2-MeOx, and we have not identified any document citing a value for this solvent. However, Mapelli *et al.* (2022) published values for TMO (*2,2,5,5*-tetramethyloxolane), a tetramethylated variant of tetrahydrofuran comparable to 2-MeOx. As 2-MeTHF is also an oxolane derivative, it is likely to have a POCP value in a comparable range to TMO, though possibly higher due to having fewer methyl substituents. Solvents were classified using desirability scores from 1 to 5. A score of 5 was assigned for POCP values below 15, decreasing by one point for each 15-unit interval up to 65.

Table 2. Harm severity index and occupational safety score

Criteria		Hexane	Iso-Hx	Cyc Hex	MeOH	EtOH	IPA	Acetone	MEK	EA	DCM	2-MeOx
Occupational safety		**1**	**3**	**3**	**2**	**4**	**5**	**5**	**5**	**5**	**1**	**2**
Acute Tox. 3	H301	0	0	0	5	0	0	0	0	0	0	0
Acute Tox. 4	H302	0	0	0	0	1	0	0	0	0	1	1
Asp. Tox. 1	H304	10	10	10	0	0	0	0	0	0	0	0
Acute Tox. 3	H311	0	0	0	1	0	0	0	0	0	0	0
Skin Irritant 2	H315	1	1	1	0	1	0	0	0	0	1	1
Eye dam. 1	H318	0	0	0	0	0	0	0	0	0	0	5
Eye Irritant 2	H319	1	1	0	0	1	1	1	1	1	1	
Acute Tox. 3	H331	0	0	0	1	0	0	0	0	0	0	0
STOT SE 3	H336	1	1	1	0	0	1	1	1	1	1	1
Mutagen 2	H341	0	0	0	0	0	0	0	0	0	5	0
Carcinogen. 2	H351	0	0	0	5	0	0	0	0	0	10	0
Reprotoxic 2	H361f	5	0	0	0	0	0	0	0	0	0	0
STOT SE 1	H370	0	0	0	5	3	0	0	0	0	0	0
STOT SE 2	H371	0	0	0	0		0	1	0	0	0	0
STOT RE 1	H372	5	0	0	0	0	0	0	0	0	0	0
STOT RE 2	H373		0	0	0	0	0	0	0	0	1	0
Severity index		**23**	**13**	**12**	**17**	**6**	**2**	**3**	**2**	**2**	**20**	**8**

Abbreviations meaning: Acute Tox. 3/4: Acute Toxicity Category 3/4; Asp. Tox. 1: Aspiration Toxicity Category 1; H311: Toxic in contact with skin; Skin Irritant 2: Skin Irritation Category 2; Eye Dam. 1: Serious Eye Damage Category 1; Eye Irritant 2: Eye Irritation Category 2; STOT SE 1: Specific Target Organ Toxicity – Single Exposure Category 1; STOT SE 2: Specific Target Organ Toxicity – Single Exposure Category 2; STOT SE 3: Specific Target Organ Toxicity – Single Exposure Category 3; STOT RE 1: Specific Target Organ Toxicity – Repeated Exposure Category 1; STOT RE 2: Specific Target Organ Toxicity – Repeated Exposure Category 2; Mutagen 2: Germ Cell Mutagenicity Category 2; Carcinogen. 2: Carcinogenicity Category 2; Reprotoxic 2: Reproductive Toxicity Category 2. The categories generally indicate the severity of the hazard, with lower numbers typically representing more severe effects.

Table 3. Overall safety assessment

Criteria	*n*Hx	*hx*	*C*Hx	MeOH	EtOH	IPA	Acetone	MEK	EA	DCM	2-MeOx
Global safety	**1.3**	**2.5**	**2.3**	**2.7**	**3.5**	**3.7**	**4.0**	**3.5**	**3.2**	**2.7**	**3.0**
Flash point	1	1	1	3	3	3	2	2	2	5	2
ATEX potential	3	3	3	1	1	1	3	1	1	5	2
Electrostatic risk	1	1	1	5	5	5	5	5	3	2	3
OEL	1	4	3	3	4	3	4	3	3	2	3
coss	1	3	3	2	4	5	5	5	5	1	4

Table 4. Environmental impacts rating

	nHx	CHx	MeOH	EtOH	IPA	Acetone	MEK	EA	DCM	2MeOx
POCP/Andserson-Sköld et al (1992)	45.2	34.9	21.3	22.5	20.3	12.4	17.8	20.9	2.3	
POCP/Derwent et al. (1996)	64.8	59.5	20.5	44.6	21.6	18.2	51.1	32.8	3.1	*18**
POCP score	1.5	2.5	4	3.5	4	4.5	3	3.5	5	4
Prat et al (2014) score	1	2	4	5	5	4	4	5	2	3
Environmental impact rating	**1.2**	**2.2**	**4.0**	**4.5**	**4.7**	**4.2**	**3.7**	**4.5**	**3.0**	**3.3**

* Estimated POCP for 2-MeOx based on TMO analogue (Mapelli et al., 2022).

Table 5. Overall desirability assessment of each solvent for process compatibility and retrofitting

	Hexane	MeOH	EtOH	IPA	Acetone ND	Acetone	MEK	EA	DCM	2MeOx
Process Compatibility and Retrofitting	**5.0**	**1.8**	**2.6**	**2.8**	**3.8**	**3.1**	**2.4**	**3.4**	**4.7**	**3.2**
Evolution of DTDC heat exchange capacity	5	1	1	1	3	3	2	3	4	3
Number of additional operations	5	3	1	1	2	4	4	4	5	4
Heat exchange enlargement for distillation	5	1	5	5	5	2	1	4	5	4
Boiler and cooler duty	5	1	2	3	5	3	2	3	5	3
Equipment sealing	5	4	5	5	4	4	4	3	4	1

When values differed between sources, the average score was used.

Regarding the pharmaceutical ranking, we kept the one of Prat *et al.* (2014) which divided the solvent between 5 categories as in this work. The grade 1 was given to “hazardous” solvents, 2 to the “problematic to hazardous” solvents, 3 to the “problematic” class, 4 for the solvent between “recommended” and “problematic” and 5 for the “recommended” ones. The final rating assigned double weight to the pharmaceutical industry classification compared to our POCP-based evaluation.

4.2 Results

Table 4 recapitulates our findings. With 4.7, IPA is close to the ideal score of 5. This rating being relative to the context of this study, this conclusion does not mean that this solvent has very low environmental impacts but that it represents the most favourable option. Ethanol and ethyl acetate are tied with a score of 4.5, placing them close to the top ranked IPA.

Acetone receives a score of 4.2, which may underestimate its environmental performance given its “recommended” status in Prat *et al.* (2016) classification. Methanol is similarly classified, though it has a slightly higher POCP than acetone. MEK’s score is negatively impacted by its POCP value from Derwent *et al.* (1996). In contrast, 2-MeOx was clearly classified as problematic by pharmaceutical experts, resulting in its lower ranking among other solvents, ranking it just 0.3 points above DCM. Between *n*-hexane and cyclohexane, there is a notable improvement, yet it is not sufficient to consider cyclohexane an environmentally friendly option.

5 Process compatibility and retrofit compatibility

5.1 Method

Variations in thermal load requirements necessitate a reassessment of heat exchanger sizing. Additionally, the increased complexity of operations designed to reduce energy consumption and compensating for water-solvent miscibility effects increases the number of unit operations, consequently increasing process capital costs. Table 5 presents a desirability scale for evaluating these costs. To conduct this evaluation, we considered the four most impactful parameters: changes in DTDC size, the number of additional unit operations, alterations in heat exchange capacity for miscella distillation, and variations in heating and cooling requirements (Carré *et al.*, 2024c).

5.2 Evolution of DTDC heat exchange capacity

We established comparison thresholds relative to hexane for assessment purposes. A score of 5 is assigned for capacity variation limited to 12.5%, 4 for variation between 12.5% and 25%, 3 for variation between 25% and 50%, 2 for variation between 50% and 100%, and 1 for variation greater than 100%.

As described in Article 3 of this series (Carré *et al.*, 2024c), multiple factors influence the heat transfer requirements during the desolventisation stage. The primary determinants include the solvent’s latent heat of evaporation, the presence and nature of any water-solvent azeotrope (including its composition and boiling point), and the solvent retention of the marc. These factors collectively dictate the thermal energy necessary for effective desolventisation. In addition, the water content of the meal exiting the desolventiser must be considered, as it determines the drying requirement, i.e., the size of the heat exchanger that will heat the air used to remove excess water.

Table 2 in the fourth article of this series (Carré *et al.*, 2024d) demonstrated that methanol requires a particularly high amount of heat, primarily due to its latent heat of vaporisation, which explains its classification in the least desirable category. Ethanol and IPA also exhibit substantial heat exchange requirements, attributed to higher solvent retention in the marc, relatively high latent heats of vaporisation (especially in the case of ethanol), and a water-rich azeotropic composition for IPA. A distinctive characteristic of these solvents is that the incoming product must undergo pre-drying before extraction. The direct steam condensed for heat transfer during desolventisation encounters a meal with low water content. Consequently, the moisture uptake remains limited, and the meal does not require subsequent drying. However, this effect is insufficient to compensate for the significant heat transfer needs, which represent more than double the desolventisation requirements of hexane-extracted meals.

For acetone, we observe a variation in desolventization heat requirement of just over 25%, placing this solvent in the middle range for this criterion. This improvement compared to alcohols is justified by a smaller increase in retention in the marc, a slightly lower latent heat of vaporisation, and the absence of an azeotrope limiting the amount of water vapor at the vapor outlet of the desolventiser. In contrast, MEK, which has a lower latent heat of vaporisation, is nevertheless classified as 2 because its azeotrope with 14% water requires the presence of 52 kg of water in the DT vapour, which is almost as much as IPA. This classification could be debated since the lower latent heat of vaporisation and relatively modest retention in the marc 288 kg vs. 248 kg t^{-1} of flakes for hexane, which seems low compared to 359 kg t^{-1} for IPA) should limit the need for exchange surface, while the proportion of direct steam could be increased without necessarily increasing the size of the trays. The score of 2 was maintained due to significant uncertainty in evaluating retention in the marc, and prudence leads us to adopt a conservative classification.

Ethyl acetate is estimated to introduce approximately the same amount of solvent to the desolventiser as MEK, and its latent heat of vaporisation is similar. However, it received a score of 3. The distinction is based on small differences. Due to the lower miscibility of water in the solvent, this distinction will be maintained. Aside from assumptions about the determinism of the marc's solvent retention capacity, we can assume that MEK is likely to transfer more water into the marc than ethyl acetate. This would result in an increased need for heat during desolventisation. With DCM, the heat transfer requirement would be solely related to a small increase in the marc's retention capacity. 2-methyloxolane was classified at the same level as ethyl acetate despite having a more favorable energy consumption profile according to our estimates. This decision is justified by the findings published by Bartier *et al.* (2024), which indicate a higher solvent retention capacity in the meal than what we had initially accounted for and highlight the uncertainty associated with these estimations.

5.3 Number of additional unit operations

The evaluation method employed here is straightforward: we subtracted one point from the maximum score of 5 for each additional operation compared to the hexane process. For methanol, we estimated that because of its high water miscibility, a stripping operation would be necessary to treat wastewater, and a distillation or pervaporation step would be required to limit the solvent's water content. Ethanol and isopropanol, using a non-distillation scheme, require pre-drying, low-temperature decantation of miscella, a pervaporation step to limit water in the solvent, and a separate distillation step for a fraction of lean miscella to produce a polar lipid-rich concentrate. This concentrate would likely require an additional operation to recover neutral lipids (as discussed in Article 3 of this series [Carré et al, 2024c], §3.1 on non-distillation). Acetone, when used with the non-distillation scheme, would have essentially the similar additional operations, except for the need for pervaporation if predrying can be considered sufficient to remove process water. This hypothesis is quite credible since acetone does not form an azeotrope with water, and the system receives little water due to the requirement to surpass the azeotropic concentration during meal desolventisation. For the same solvent in a conventional distillation process, we retained a pre-drying operation to ensure maintenance of an acceptable water concentration. MEK, ethyl acetate, and 2-MeOx have a solubility in water that necessitates adding a stripping operation for wastewater. DCM, on the other hand, does not require additional unit operations.

5.4 Heat exchange enlargement required by the distillation of the miscella

The classification of solvents for this criterion follows the same rule as for assessing differences in DT size. A score of 5 is assigned for capacity variation limited to 12.5%, 4 for variation between 12.5% and 25%, 3 for variation between 25% and 50%, 2 for variation between 50% and 100%, and 1 for variation exceeding 100%. Unsurprisingly, methanol is classified as 1 due to its high latent heat of vaporisation and the required solvent mass. Ethanol, isopropanol, and acetone in non-distillation (ND) processes are classified in category 5, as they do not require distillation. Acetone used with distillation requires 92% more heat than hexane and is classified as 2. Methyl ethyl ketone exceeds 100% and falls into category 1. Ethyl acetate and 2-methyloxolane require only 24.7% and 19.7% more heat than hexane, respectively, and can be classified as 4. Dichloromethane allows for limited heat transfer requirements and would be classified as 5.

5.5 Variation of boiler and cooling tower duty

This consideration concerns the need to enlarge the utilities like the boiler producing steam in the oil-mill and the cooling towers used to cool the water circulating in the condensers. These variations in capacity are directly related to the overall change in energy requirement (last row of Table 2 in the fourth part of this series of articles [Carré et al, 2024d]).

The classification was made using the same scale as for previous parameters related to change in energy demand (5 when change <12.5%, 4 for 12.5 to 25%, 3 for 25 to 50%, 2 for 50 to 100% and 1 for >100%).

5.6 Equipment sealing and elastomer compatibility

A critical consideration in solvent substitution is preserving seal integrity in the installation. Solvents incompatible with elastomers typically used with hexane will necessitate a significant replacement of gaskets. Our classification system assigns a score of 5 to solvents compatible with Viton and NBR, which can be used directly without changing gaskets (compatibility 1 to 2 in Table 2 of the second article of this series [Carré *et al.*, 2024b]). Only ethanol and isopropyl alcohol demonstrate this level of compatibility. Methanol, while incompatible with Viton but compatible with all other elastomers, receives a score of 4. Dichloromethane, incompatible with NBR but showing adequate compatibility with Viton, is also classified as 4. Ketones, incompatible with both these elastomers but for which alternatives exist, are classified as 3. Ethyl acetate, also incompatible with Viton and NBR, has several alternatives, though only FKM achieves rating 1. It is nonetheless classified as level 3, considering that a compatibility rating of 2 is deemed acceptable. 2-methyloxolane, which is only compatible with FFKM, presents more significant challenges due to cost, poor elasticity, and fluorinated composition of this material. Consequently, it is scored 1.

Table 6. Energy efficiency ranking of solvents in oilseed extraction

Criteria	nHx	Ihx	CHx	MeOH	EtOH	IPA	Ac. ND	Acetone	MEK	EA	DCM	2-MeOx
Energy	**5.0**	**5.0**	**4.3**	**1.0**	**1.3**	**3.0**	**4.8**	**3.3**	**2.5**	**3.3**	**4.8**	**3.3**
Heat	5	5	4	1	1	3	5	3	2	3	5	3
Electricity	5	5	5	1	2	3	4	4	4	4	4	4

5.7 Aggregation of the criteria and final ranking

Table 5 summarises the criteria considered to evaluate the significance of the retrofit process to be considered. The issue of seals, being less substantial than other points, has been given reduced importance through the application of a weighting factor of 0.5.

DCM is the solvent that could be implemented in existing extraction plants with the minimal capital costs although some modifications, such as upgraded pumps may have been insufficiently considered here. Acetone with a non-distillation scheme would rank above ethyl acetate and 2-methyloxolane; however, this assessment may be attributed more to the method's limited precision than to any inherent superiority. The implications of a nondistillation scheme are not well understood, so this appearance of superiority should not be taken literally. EA and 2-MeOx having relatively low water miscibility are both incompatible with elastomers frequently used in contact with hexane, such as Viton or NBR, which could necessitate costly replacement work for all seals. Their major impact would be at the level of the desolventiser capacity enlargement.

Acetone used with conventional distillation would generate less capital costs than alcohols which seems credible. Between ethanol and isopropanol, the similarity of the scores does not account for the relatively significant differences that distinguish these solvents. For example, both require predrying of the material, but this drying is much less extensive for IPA. Similarly, because 95% ethanol has a more limited capacity to dissolve lipids compared to 88% IPA, larger equipment is required.

6 Energy consumption

6.1 Method

Thermal energy costs constitute a significant portion of oilseed crushing costs. Laisney (1983) estimated energy costs to be between 15% and 20% of total crushing costs, with an energy consumption of 210 kWh t^{-1} for extraction. Dada (1984) reported energy costs representing 40% to 50% of operating expenditure, with a consumption of 250 kWh t^{-1} in facilities lacking an economiser. According to Dada, the addition of this device can reduce heat requirements to between 175-210 kWh/t. More recently, Bockisch's Handbook (1998) reported steam requirements for sunflower seed processing at 240 to 260 kg per tonne, equivalent to 145 to 160 kWh/t, which aligns with the assessment presented in Table 2 of the fourth article in this series.

Electrical energy consumption is estimated at approximately 40 kWh per tonne according to Dada, compared to only about 10 kWh per tonne in Bockisch's assessment. However, this electrical energy is more expensive than thermal energy. According to SDES (2024) figures, the gas used to produce steam had an average value of 78 €/MWh in 2023 in France for industrial applications, while electricity cost 205 €/MWh. Based on these figures, assuming an electrical energy requirement of 20 kWh/t, the cost of electricity represents approximately 25% of the total energy cost.

The comparative assessment of solvents was conducted using two key criteria, both referenced to hexane as the baseline: the relative change in thermal energy costs and the change in electricity demand. These evaluations were based on data presented in Article 4 of this series, providing a standardised framework for comparison across different solvent options. The thermal energy criterion was assessed using a 5-point scale: a score of 5 was assigned to solvents showing negative or no variation, 4 for variations between 0% and 25%, 3 for 25% to 50%, 2 for 50% to 75%, and 1 for variations exceeding 75%. Electricity requirements were estimated based on the mass of solvent to be handled per tonne of flakes, calculated as the sum of rows α and β in Table 2 of Article 4. These variations are attributed to the limited solubility of oil in specific solvents, such as ethanol, which necessitates the utilisation of increased solvent volumes. Furthermore, higher retention in the marc mandates the transfer of a greater quantity of solvent to the extractor. This criterion was also evaluated on a 5-point scale: 5 for masses $\leq$ 930 kg, 4 for 930–1000 kg, 3 for 1000-2000 kg, 2 for 2000-3000 kg, and 1 for masses $>$ 3000 kg. The final ranking was determined by calculating a weighted average of these two scores, with the thermal energy score weighted three times more heavily than the electricity requirement score.

Results

Table 6 presents a comprehensive summary of the energy efficiency comparison results. Acetone, utilised in a non-distillation scheme, appears as the least energy-intensive option, alongside dichloromethane. However, as previously noted, there is considerable uncertainty regarding the actual performance of acetone in this configuration. Further verification under industrially representative conditions is required to validate this assessment. If corroborated, it could offer strong incentive to processors who do not valorise lecithin. The scoring system may unfairly penalise cyclohexane, as its performance differential relative to hexane is marginal. Subsequent alternatives–acetone, ethyl acetate, and 2-methyloxolane–exhibit a one-point reduction in score, which could entail substantial economic costs on industrial operators if implemented. Isopropyl alcohol, with a score of 3.0, remains comparatively competitive compared to the aforementioned

Table 7. Evaluation of the solvent effect on oil quality

	Criteria	MeOH	EtOH	IPA	Acetone	EA	DCM/Chlor of	2-MeOx
	Denaturant	Yes	Yes	Yes	Yes	No	No	No
	FFA	+	9/5	5/10	4/1	4/0	2/1	2/0
	PV	–	3/6	3/2	1/3	3/0	1/2	3/0
	PL	?	4/2	2/6		1/0	1/1	3/0
Results count	USM	+	3/1	0/1	1/0	0/5		1/0
	Phenolics	++	5/0	5/0	5/0	4/1	3/0	11/0
	Tocopherols	+	8/4	5/3	3/2	2/2	3/0	4/1
	Sterols	++	4/1	2/2	2/2	1/1	2/0	0/3
	nb of studies	1	19	19	8.0	13	7	14
Score	Denaturant	1	1	1	1	-1	-1	-1
	FFA	-1	-1	1	-1	-1	-1	-1
	PV	1	1	-1	1	-1	1	-1
	PL		1	1		-1	0	-1
	USM	1	1			-1		
	Phenolics	1	1	1	1	1	1	1
	Tocopherols	1	1	1	1	0	1	1
	Sterols	1	1	0	0	0	1	-1
Mean		0.71	0.75	0.57	0.50	-0.50	0.29	-0.43
Score		**5**	**5**	**4**	**4**	**2**	**4**	**2**

three solvents. In contrast, methyl ethyl ketone, scoring 2.5, demonstrates inferior performance. Ethanol and methanol are are not competitive in this context.

7 Product quality and market acceptance

7.1 Method

To assess the impact of the various solvents in our study on product quality and consumer acceptability, we will review the observations made in Articles 5 and 6 of the series, which address oil quality and meal quality, respectively. We will also provide an assessment of the oral toxicity of residual solvents and regulatory constraints to which the solvents are subject.

7.2 Oil quality

To evaluate the effect of solvents on oil quality, we have utilised the comparison tables from Article 5 of this series (Carré *et al.*, in press-a). Consequently, we have used hexane as a reference and assessed other solvents in relation to it. For instance, regarding the acidity data, this methodology entails that we considered the differential between the acidity of the oil extracted using hexane and that extracted with the solvent in question. We considered the number of positive and negative studies for the following criteria: free fatty acids, peroxide value, phospholipid content, unsaponifiable compounds, phenolic compounds, tocopherols, and sterols. We also took into account the solvent's ability to inactivate enzymes, which is considered beneficial in this context as it can inhibit the activity of lipases and phospholipases that may reduce oil quality. When the number of studies showing a positive effect was predominant, we assigned a value of +1 for the parameter. Conversely, we assigned −1 if the number of negative results was greater, and 0 in case of a tie. When the number of studies for a particular criterion was significantly lower than other criteria, we excluded that criterion from our analysis. Table 7 summarises these evaluations.

Due to the limited availability of data, the results for methanol are uncertain, as we had access to only a single study. Similarly, it was not possible to evaluate methyl ethyl ketone owing to lack of data on the quality of oils extracted with this solvent. For dichloromethane, we supplemented the data by incorporating results from studies that used chloroform, due to similarities between these solvents. An increase in free fatty acid content or peroxide value was considered to have a negative impact on oil quality. Similarly, an increase in phospholipid content was viewed negatively, as most extraction facilities perform water degumming immediately after extraction and return the gums to the oilcake, resulting in increased neutral oil losses. This approach contrasts with the approach in the section on solvent selectivity, where the extraction of phospholipids was considered positively. This choice was made to take into account the range of perspectives, specifically the perspective of industrialists who value lecithin and those who return it to the oilcake and consequently suffer losses in neutral oil.

In this section, we have also adopted an alternative perspective regarding phenolic compounds relative to our approach in the selectivity section. While previously viewed negatively, here we consider these compounds in a positive light due to their antioxidant properties.

In the case of solvents that would be used in a non-distillation scheme, we considered that phospholipids would be retained in the lean miscella phase, resulting in oils with low

Table 8. Effect of the solvents on the proteins damaging during desolventisation

Criteria	Hx	i-Hx	c-Hx	MeOH	EtOH	IPA	Acetone	MEK	EA	DCM	2-MeOx
Enthalpy of the vapours (MJ/t of flakes)	118	115	148	864	354	365	162	242	201	118	185
Variation		-2.5%	25%	632%	200%	209%	37%	105%	70%	0.0%	57%
Score	5	5	4	1	2	2	4	3	3	5	3

phospholipid content, and thereby producing a positive effect for these solvents. As previously indicated, the assessment of methanol remains uncertain, as it is predicated on a single result. However, given the structural similarity of this solvent to ethanol, it seems reasonable to accept its evaluation. The quantity of data for acetone and dichloromethane is relatively limited, yet their findings are largely consistent. For instance, in the case of acetone, four studies demonstrate an increase in acidity, while only one indicates a decrease. Regarding the unsaponifiable matter for the same solvent, where only one positive study was identified, we have chosen to exclude the result due to insufficient corroborating evidence. The mean was calculated relative to the number of available observations. The final score is derived directly from this mean, using a tiered scoring system from 5 to 1. A score of 5 was assigned to means between 1 and 0.6, 4 to means between 0.6 and 0.2, 3 for 0.2 to −0.2,2 for −0.2 to −0.6.

Ethanol and methanol received the highest scores in this evaluation due to their minimal negative effects. The increase in acidity could potentially have been excluded for ethanol, considering that in a non-distillation scheme, free fatty acids would be retained in the lean miscella. Isopropanol is assigned a less favourable score owing to a negative rating for the peroxide value and a lack of effect on sterols, which is considered justified. In the case of acetone, its use in a non-distillation configuration could similarly warrant a reassessment of its impact on acidity; however, we lack adequate experimental evidence to substantiate this. Dichloromethane's primary drawbacks include the absence of enzymatic inactivation and increased acidity. 2-methyloxolane shows beneficial effects only on the extraction of phenolic compounds and tocopherols. The impact of this solvent on the oxidative stability of oils is likely one of its most significant disadvantages, given the solvent's tendency to form hydroperoxides. Ethyl acetate, for its part, shows beneficial effects only on the phenolic compound content. This advantage is somewhat mitigated by the fact that the majority of these compounds are eliminated during the refining process.

7.3 Potential impact on meal quality

As discussed in Article 6 of this series (Carré *et al.*, in press-b), which focused on the effect of solvents on meal quality, the main expected impact is related to the duration of meal exposure to desolventisation temperatures. Consequently, to classify solvents on the same graded scale as other criteria, we concentrated on the amount of energy that must be transferred to the meal to evaporate the marc. This data is derived from Table 2 of our fourth article (Carré et al, 2024d), which addressed energy impacts. Specifically, we utilised rows A and B : the enthalpy of vaporisation of water contained in the vapours and the enthalpy of solvent vapours, respectively. The effect of sensible heat was not considered, as this heat is transferred before the meal reaches the temperature at which Maillard reactions occur.

Scores presented in Table 8 were assigned based on the differential compared to normal hexane and its acyclic isomers. Dichloromethane, which requires the same amount of heat for desolventisation, is rated 5. It is followed by cyclohexane and acetone, with variation rates below 50%. 2-methyloxolane and ethyl acetate received a score of 3 due to variations between 50% and 100%. Methyl ethyl ketone was also classified as 3 because it is near the threshold and clearly distinct from alcohols. Ethanol and isopropyl alcohol are classified as 2, and methanol receives the lowest score of 1.

7.4 Oral toxicity of the solvent

The quality of oils and oilseed meals can also be evaluated in terms of food safety through the toxicity of solvent residues present in these products. To assess this, we employed two toxicity indicators: the median lethal dose (LD$_{50}$), which corresponds to the dose at which 50% mortality is observed in rats to which the product is given, and the No Observed Adverse Effect Level (NOAEL), expressed in mg per kg body weight per day. The LD$_{50}$ values were obtained from the ECHA database, while the NOAEL values were sourced from published literature. Table 9 presents the compiled results. For iso-hexane, methanol, and MEK, we were unable to find NOAEL values; consequently, Lowest Observed Adverse Effect Level (LOAEL) indications were reported instead.

The oral toxicity scores incorporate the two toxicity indicators. For solvents lacking NOAEL values, LOAEL values divided by 2 were considered as a surrogate.

Figure 1 illustrates the distribution of various solvents on a plane defined by these two axes and defines the boundaries used for score assignment, namely 1, 5, 10, and 15 g kg^{-1} for LD$_{50}$ values, and 200, 1000, and 3000 mg kg^{-1} d^{-1} for NOAEL values. Lines connecting these threshold values were drawn, and scores from 1 to 5 were assigned based on the position of solvents within these limits. In absence of NOAEL and LOAEL, and due to its low LD$_{50}$, isohexane has been ranked at the same level as *n*-hexane. This classification of isohexane is conservative, given that its neurotoxicity is notably lower than that of *n*-hexane. However, LD$_{50}$ values for both solvents are similarly low, which justifies this classification.

7.5 Regulation compliance (Directive 2009/32/EC)

Solvents are subject to differing treatment under the regulations under Directive 2009/32/EC. Some solvents are authorised without usage limitations or maximum residue

Table 9. Oral toxicity indicators and toxicity scores

Criteria	NOEAL (mg/kg bw/day)	LOEAL (mg/kg bw/day)	Source	LD_{50} (rats, g/kg)	LD 50 (Midpoint)	Score
n-Hexane	23.0		CECSCF, 1988	0.024-0.049	0.0365	1
Iso-hexane				0.0158	0.0158	1
Cyclohexane	1721.0		Malley et al., 2000	5	5	4
Methanol		500	ABC., 1986	1.19-2.77	1.98	2
Ethanol	2400.0		ECHA, 2018	1.18 - 15.0	8.09	5
Isopropanol	400.0		OECD, 2007	5.84	5.84	3
Acetone	200.0		Dietz et al., 1991; NTP, 1991	5.8	5.8	3
MEK		1771	Krasavage et al., 1982	2-6	4	3
EA	900.0		OECD, 2006	10.19	10.19	4
DCM	6.0		OECD, 2011	2	2	2
2-MeOx	250.0		Antonucci et al., 2017	0.3	0.3	2

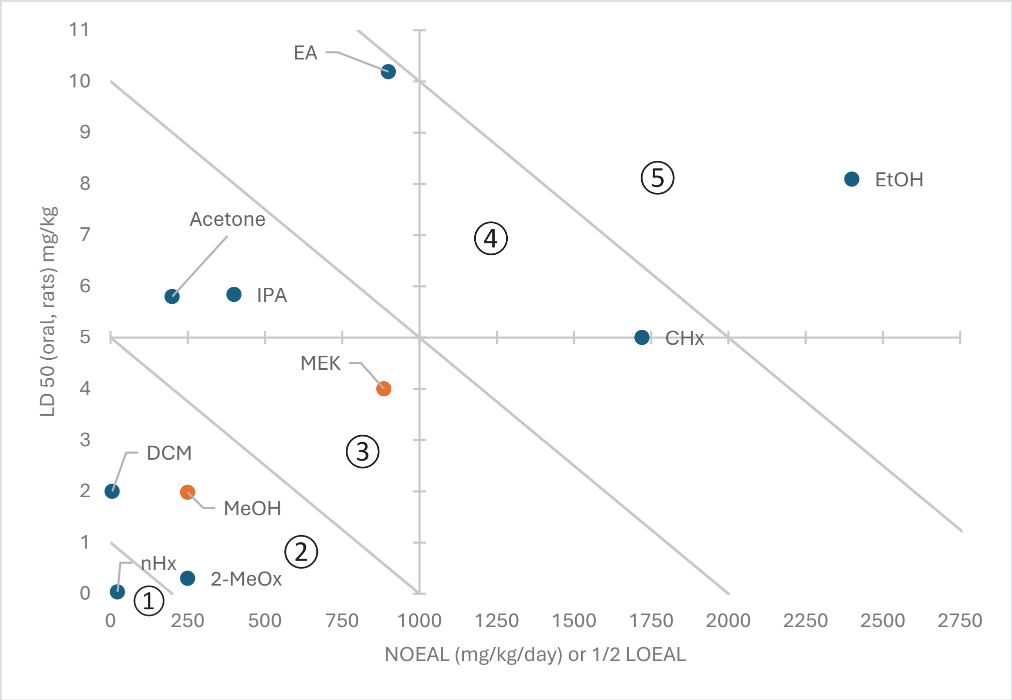

Figure 1. Oral Toxicity Scoring of Solvents (NOEAL: No Observed Effect Level; LOEAL: Lowest Observed Effect Level; LD 50: Lethal Dose 50)

Table 10. Regulatory compliance difficulty

Criteria	Hexane	MeOH	EtOH	IPA	Acetone	MEK	EA	DCM	2-MeOx
LMR oil (ppm)	1	10	NL	10	NL	5	NL		1
LMR (others)	10	10	NL	10	NL		NL	2	10
All uses	-1	+1	+1	+1	+1	-1	+1	-1	-1
Extraction	+1	+1	+1	+1	+1	-1	+1	-1	+1
Score	**3**	**4**	**5**	**4**	**5**	**2**	**5**	**1**	**3**

NL: no residual limits given in the annex of the Directive 2009/32/EC

Table 11. Summary of explored criteria related to products quality

Criteria	Hx	i-Hx	c-Hx	MeOH	EtOH	IPA	Acetone	MEK	EA	DCM	2-MeOx
MEAN	**3.0**	**3.0**	**3.3**	**3.0**	**4.3**	**3.3**	**4.0**	**2.8**	**3.5**	**3.0**	**2.5**
Oil quality	3	3	3	5	5	4	4	3	2	4	2
Meal quality	5	5	4	1	2	2	4	3	3	5	3
Toxicity	1	1	4	2	5	3	3	3	4	2	2
Regulation compliance	3	3	2	4	5	4	5	2	5	1	3

Table 12. Scoring of technical readiness of solvents solutions.

Solvents	TRL	Comment	Source	Score 1	Score 2	TRL score
n-hexane	9	Current technology		5	5	5.0
Iso-hexane	6	Tested on bench-scale	Wan et al, 1995	3	5	4.0
Cyclo-hex.	6	Idem	Idem	3	5	4.0
Methanol	4	Tested in direct interestrification at lab scale	Makareviciene et al, 2020	2	5	3.5
Ethanol	7	Currently used in other industries	Hron et al, 1982	3	3	3.0
Isopropan ol	7	Extensive experiment in TAMU	Lusas et al, 1997	4	3	3.5
Acetone	8	The Vaccarino process operated at industrial scale	Vaccarino et Vaccarino, 1961	4	2	3.0
MEK	2	Tested with cotton flakes in a 55 kg extractor	Eaves et al. 1952	2	2	2.0
EA	4	Lab-scale extraction	Lohani et al., 2015	2	4	3.0
DCM	6	Pilot-plant comparison.	Johnson et al, 1986)	3	4	3.5
2-MeOx	7	Industrial demo and ongoing qualifications	Claux et al, 2021	4	4	4.0

limits, such as ethyl acetate, acetone, or ethanol. Others have residue limits for oils and protein-rich products, while some are not listed for oil extraction among their authorised uses. Table 10 indicates the Maximum Residue Limits (MRLs) for oils and proteins, or specifically for coffee in the case of dichloromethane. It also specifies whether solvents have all-purpose authorisations and if they are authorized for oil extraction. A score of 5 was assigned to solvents authorised for all uses without MRLs. A score of 1 was given to solvents that lack authorisation for oil extraction and have no MRLs for oils, which is the case for DCM. Methyl ethyl ketone, while not authorised for extraction, nonetheless has an MRL for oil and was thus classified as 2. Hexane and 2-methyloxolane, which have extraction authorisation and MRLs of 1 ppm, received a score of 3. Methanol and isopropyl alcohol, with MRLs of 10 ppm, were classified as 4.

7.6 Aggregation of criteria related to product quality

Table 11 shows that when aggregating all criteria related to product quality, ethanol emerges as the most favourable solvent with an average score of 4.3 out of 5. This score is somewhat diminished due to the negative impact on meal quality associated with the intensity of thermal treatments during desolventisation. Acetone follows with a score of 4. Its strengths lie in the absence of regulatory constraints and a lesser impact on meal quality; however, the reliability of the data supporting its rating is slightly less robust than for ethanol. Ethyl acetate ranks third due to a slightly negative effect on oil quality and thermal damage to proteins. With a score of 3.3, isopropanol and cyclohexane are relatively close to ethyl acetate. Cyclohexane is mainly penalised as it does not meet the regulatory definition of hexane, and its industrial use would need to overcome this barrier to be accepted. Otherwise, it possesses advantages in terms of toxicity profile and meal quality. Isopropanol, although having a profile similar to ethanol, lags behind because it appears to have a marginally more negative impact on quality across most criteria. Additionally, from a regulatory standpoint, it is subject to maximum residue limits, which is not the case for ethanol.

Methanol and DCM are equivalent to normal hexane and iso-hexane for different reasons. DCM has a profile similar to hexane, as it is slightly less toxic but potentially more effective in extracting compounds of interest for oil quality. Methanol, on the other hand, markedly reduces protein quality but would be more favourable from a regulatory perspective. Interestingly, 2-MeOx, despite being marketed as a green solvent, performs poorly in this comparison, scoring lower than hexane. Available comparisons seem to attribute marginally less favourable effects to 2-MeOx compared to hexane in terms of oil and meal quality, while its toxicity profile distinguishes it only slightly from hexane in the frame of this scoring method.

8 Technical and economic feasibility

8.1 Technical level of readiness

The technical readiness levels (TRL) must be considered when evaluating potential solutions, as unproven options remain impractical at the industrial level. Combining economic and technical feasibility may be problematic due to their differing evaluation bases. Therefore, this comparison examines each aspect independently. TRLs are standardised

Table 13. Economic feasibility scores

	n-hexane	Isohexane	CycloHexane	MeOH	EtOH	IPA	Acetone ND	Acetone	MEK	EA	DCM	2-MeOx
Capital	5	5	4.5	1.8	2.6	2.8	3.8	3.1	2.4	3.4	4.7	3.2
Energy	5.0	5.0	4.3	1.0	1.3	3.0	4.8	3.3	2.5	3.3	4.8	3.3
Solvent cost	3	3	3	5	4	2	3	3	4	3	4	1
Score	**4.7**	**4.7**	**4.1**	**1.8**	**2.0**	**2.8**	**4.2**	**3.2**	**2.7**	**3.3**	**4.6**	**2.9**

classifications established by the European Union, ranging from 1 to 9, encompassing basic principles to proven operational systems. For this study, we have mapped the TRL scale to a 5-point desirability scale. The first level comprises TRLs 1–3, representing experimental proof of concept. The second level comprises TRLs 4 and 5, indicating technology validated in a relevant environment. The third level corresponds to TRL 6, demonstrating technology in a relevant environment. The fourth level includes TRLs 7–8, which involve prototype demonstration and validation in an operational environment. Finally, the fifth level represents the existing industrial reference (TRL 9), specifically *n*-hexane mixtures.

Table 12 presents the classification of solvent systems according to Technical Readiness Levels. Wan *et al.* (1995) tested hexane isomers in a laboratory-scale pilot plant. The current regulations in the United States do not allow for circumventing emission thresholds of polluting substances by replacing *n*-hexane with its isomers. This is because the regulations apply to hexane isomers in general, without distinguishing their specific types (EPA, 2020). Regarding methanol, only the direct interesterification process is reported, as no practical extraction experiments have been conducted for oil extraction with this solvent. In this configuration, methanol is assigned a score of 2, reflecting experiments conducted at the laboratory level with no scale-up reported. Isopropanol was assigned a score of 4 due to extensive experimentation. In the 1990s, Texas A&M University conducted numerous studies on isopropanol, supported by multiple published reports. For ethanol, a substantial body of literature exists on its oil extraction performance but its use in non-distillation schemes was less documented than IPA and it was therefore assigned a lower score of 3.

Acetone receives a score of 4 due to its historical use in the Vaccarino process for cottonseed extraction and gossypol removal. Johnson and Lusas (1983) documented its use in an operational oil mill, where oil recovery involved adding water to the miscella, followed by water removal via rectification. Butanone (MEK) has received limited attention in the literature. Eaves *et al.* (1952) compared this solvent with hexane and acetone in a batch extractor using approximately 55 kg of cotton seed flakes. However, these conditions were not representative of continuous extraction, with an extraction period of 19 hours and the use of dry MEK. As a result, we assigned a score of 2 to this solvent on our scale of 1 to 5. Ethyl acetate has showed promising results at the laboratory scale but lacks evidence of pilot-scale trials. This places it at a TRL of 4, corresponding to a score of 2 in this comparison. Dichloromethane has undergone more advanced testing, with pilot plant experiments conducted by Johnson *et al.* (1986). This places DCM at TRL 6, resulting in a score of 3 in this comparison.

2-methyloxolane is currently undergoing qualification work by the EcoXtract company, which is expected to advance it from TRL 6 to 7. Consequently, it is assigned a score of 4 in this comparison.

Another approach to assessing Technology Readiness Level involves examining the existence of similar applications in other sectors, such as caffeine removal or the production of food ingredients like colorants or aromas. This second assessment is the score 2 of Table 12. Hexane isomers would not require significant changes in existing processes allowing a high score (5). Ethyl acetate and dichloromethane are established solvents for coffee decaffeination, used in both direct and indirect extraction (Franca, 2016) and indirect methods. In the latter, beans are soaked in hot water to create an extract from which caffeine is removed using ethyl acetate through liquid-liquid extraction (Durango Coffee Company, 2019). This existing know-how in engineering companies reduces the gap to market for oilseed extraction applications, allowing for a relatively high TRL score (4) for these solvents. Methanol is widely used in the transesterification of vegetable oils, suggesting that its recovery and handling processes are well-established by oilseed processing companies. For butanone, industrial examples in food ingredients, aromas, and colorants are scarce, potentially leading to greater caution among equipment suppliers due to unfamiliarity. Consequently, it receives a lower score (2). Ethanol, commonly used in various extraction processes, wouldn't pose significant challenges for equipment suppliers when used with distillation. However, the adaptation of non-distillation systems may require further demonstration, resulting in a score of 3. Isopropanol, being similar to ethanol, receives the same score. Acetone in non-distillation schemes has less experimental data available, warranting a score of 2. According to its supplier, the 2-MeOx is expected to soon undergo the completion of extensive industrial condition testing, which would advance its TRL to a level of 7 or 8. However, due to the uncertainty surrounding the achievement of these results, its score under this secondary assessment remains unchanged. This second TRL scoring approach combined with the first one provides a more nuanced evaluation of solvent readiness for oilseed extraction applications, considering both direct industry experience and potential for adaptation from related sectors.

8.2 Economic feasibility

To assess the economic feasibility of our various solutions, we have utilised the impact ratings on the process previously inventoried in Chapter 5 for investment costs and in Chapter 6 for energy costs, which constitute a significant portion of operational expenses. The evaluation of these parameters was conducted using our desirability scales ranging from 1 to 5. Additionally, we considered the costs of different solvents, which were also rated on a scale of 1 to 5. A score of 5 was assigned to solvents with a price below 500 €/t, 4 for those between 500 and 1000 €/t, 3 for 1000 to 1500 €/t, 2 for 1500 to 3000 €/t, and 1 for those exceeding 3000 €/t. We applied weighting factors of 2,1 and 0.5 to these three cost factors, corresponding to energy costs, capital costs, and solvent costs, respectively, to compute the final weighted score.

Table 13 provides a comprehensive summary of scores and presents the overall ranking of solvent alternatives. Hexane isomers appear as the most cost-effective option, requiring minimal process modifications and capital expenditure. While cyclo-hexane incurs higher costs due to a higher boiling point increasing energy requirements, it maintains competitiveness against other options. Dichloromethane presents an economically viable option, as it does not demand significant retrofitting, and its energy consumption is comparable to that of hexane. However, its toxicological profile precludes its consideration as a credible substitute for hexane from a safety perspective. In a non-distillation scheme, acetone demonstrates potential competitiveness owing to its reduced energy consumption. Nevertheless, this assessment requires caution due to the limited understanding of this solution's actual performance. Ethyl acetate and acetone, when employed conventionally, offer substantial improvements in safety and technical feasibility. However, these advantages are accompanied by a notable increase in costs compared to hexane. 2-methyloxolane ranks marginally lower in economic feasibility and seems less desirable from a safety point of view but exhibits superior technical readiness. Isopropanol shares similar economic characteristics with 2-methyloxolane; however, its implementation poses greater technical hurdles. Ethanol and methanol rank lower than the other alternatives in terms of economic viability.

9 Conclusion

As announced in the introductory section, it would be inappropriate to conclude this article with final aggregate score encompassing all the proposed classifications. These classifications span various domains, including extraction efficiency, safety management and environmental impacts, the magnitude of required process modifications, energy costs, implications for product quality and residues toxicity, and the economic ramifications of the choices to be made. Such an aggregation exercise would necessitate weighting each selection criterion according to preferences that are beyond our purview to determine. The objective of this study was to present an impartial assessment of the present state of knowledge regarding the potential implications of a solvent change in the vegetable oil extraction industry. Concurrently, this work provides insights into the factors contributing to hexane's dominance in this sector and exposes the challenges impeding a straightforward substitution with an alternative solvent. It is important to recall that the solvent predominantly used by the industry is technical hexane, which contains roughly 50% *n*-hexane. While this distinction does not diminish the overall harmfulness of the actual solvent, it does call for a more nuanced evaluation.

In the context of solvent selection for vegetable oil extraction, several key factors emerge as key determinants of technical and economic feasibility. The solvent hydrophobicity is a key parameter in influencing their selectivity, recyclability, and associated thermal energy requirements. However, it is noteworthy that hydrophobicity does not directly correlate with health and environmental hazards, as evidenced by the significant disparity observed between *n*-hexane and cyclo-hexane, despite their comparable hydrophobic properties. This study also highlights the heterogeneous nature of knowledge and technical readiness levels across various solvent alternatives. Consequently, certain potentially attractive solutions, such as acetone in non-distillation schemes, have not been sufficiently investigated to conclusively establish their viability. In this particular case, the role of water in solvent recycling economics remains not well understood. It is noteworthy that this analysis did not account for the potential necessity of solvent rectification in scenarios where repeated recycling may result in increased water content. Although the concept of Technology Readiness Level is introduced towards the conclusion of this work, its significance cannot be overstated, particularly in the context of potential regulatory changes. Any solution that has not been rigorously validated is effectively unavailable for industrial application.

Switching from technical-grade hexane, which contains approximately 50% *n*-hexane, to a more refined composition of isomers presents challenges. This shift would alter the solvent's boiling point, potentially placing it outside the regulatory definition characterised by a range of 64 to 70°C (Directive 2009/32/CE). For instance:

- 2-methylpentane boils at 60°C
- 3-methylpentane boils at 63°C
- Dimethylbutanes have boiling points below 60°C
- Methylcyclopentane boils at 72°C
- Cyclohexane boils at 80°C

To maintain a boiling point within the regulatory range, it would be necessary to formulate a mixture of aliphatic and cyclic hexanes. This process would require a different distillation cut, thereby increasing solvent cost, although it is technically feasible. The desirability of this solution is questionable. While it may achieve the goal of eliminating *n*-hexane from the process, the toxicological data for 2-methylpentane appears unfavourable based on LD_{50} toxicity data. Furthermore, the environmental evaluations of the alkane group are generally negative.

In the context of solvent alternatives for vegetable oil extraction, 2-methyloxolane stands out as the only substance undergoing a comprehensive qualification process (Sicaire *et al.*, 2015; Sicaire *et al.*, 2016; Rapinel *et al.*, 2020; Claux *et al.* 2021; Bartier *et al.*, 2024). The studies conducted on isopropanol in the United States during the 1990s (Lusas, 1997) are now considered dated, and their applicability to oilseeds such as rapeseed and sunflower remains uncertain. Based on the current state of research and development, it can be inferred that if the qualification studies for 2-methyloxolane were to be completed and their results made public, this solvent could potentially emerge as a viable alternative from the perspective of industrial stakeholders.

As we conclude this article, the future direction of regulatory changes remains uncertain. The European Food Safety Authority (EFSA) opinion from September 2024 suggests that the specification of technical-grade hexane will likely require more precise specification. In case of reduction in Maximum Residue Limits for refined oils, concerns may arise regarding laboratory capability to measure low concentrations of hexane isomers in these matrices. The current Limit of Quantification (LOQ) is approximately 0.1 mg kg^{-1} (ITERG, pers. comm.).

Abbreviations concerning solvents names

- nHx: normal hexane (CAS 110-54-3);
- Ihx: Isohexane (CAS 107-83-5)
- CHx: Cyclohexane (CAS 110-82-7)
- MeOH: methanol
- EtOH: ethanol
- IPA: isopropanol
- MEK: Methyl ethyl ketone (butanone)
- EA: ethyl acetate
- DCM: dichloromethane (methylene chloride)
- 2-MeOx: 2 methyloxolane (2-methyltetrahydrofuran).

Conflicts of interest

The authors have no conflicts of interest to disclose.

Author contribution statement

Patrick Carré: conceptualisation, administration, original draft, review & editing. Thomas Piofczyk: conceptualisation, validation, review & editing. Sarah Bothe : validation, review & editing.

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