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Bio-derived oils and their derivatives as renewable-green solvents in organic synthesis

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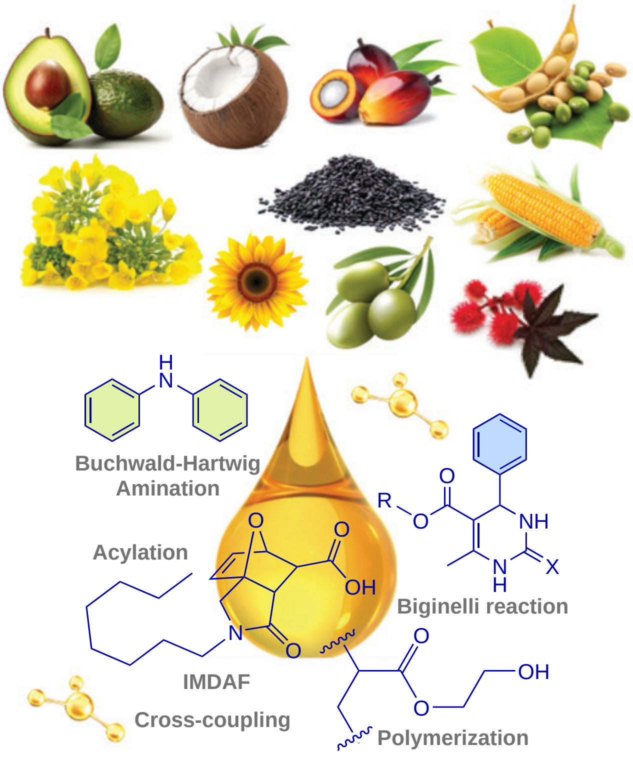
Despite numerous studies demonstrating the harmful environmental and health effects of traditional, non-renewable, toxic organic solvents derived from petroleum, these substances are still widely used in many industries for various purposes. However, there has recently been an increase in the research and development of green, renewable and biodegradable alternative substances for use in the food, pharmaceutical and chemical industries. Many substances, including ionic liquids, water, glycerol, supercritical CO_2 and various bio-solvents, have previously been widely discussed in terms of their properties as potential solvents, reactants or catalysts from the perspective of green chemistry. This review focuses exclusively on vegetable oils and their derivatives, adopting a complementary perspective. Compared to the other environmentally friendly materials listed above, their use as solvents in organic transformations remains comparatively limited. The main reason for this is that they behave differently to conventional solvents in terms of their ability to dissolve reactants and isolate products in a synthesis reaction. Nevertheless, a small number of studies have identified their potential as superior green solvents for various organic reactions. This review focuses on recent studies in which vegetable oils and their derivatives were used as effective green solvents in well-known organic synthesis reactions. It is also important to note that these materials exhibit certain deficiencies when employed as solvents in organic synthesis reactions, in comparison to traditional organic solvents. These aspects require further enhancement. It is therefore hypothesized that this review will provide organic chemists with insight to help them plan future synthetic routes in accordance with the twelve fundamental principles of green chemistry.

The bibliography includes 198 references.



Keywords: vegetable oils, renewable solvents, organic reactions, green chemistry.

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1. Introduction

A considerable number of petroleum-based solvents are used in a variety of scientific disciplines. However, it is particularly noteworthy that toxic and harmful solvents are used in enormous amounts in organic synthesis, despite the existence of environmentally friendly alternatives,[1] such as water, ionic liquids (ILs), glycerol, deep eutectic solvents (DES) and supercritical fluids (SCFs).[2–10] Terpene-based solvents derived from renewable feedstocks have the potential to replace conventional hazardous organic solvents, particularly in the fabrication of organic electronics.[8] It is recommended to utilize a terpene in conjunction with a high-boiling-point co-solvent to

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achieve an optimal morphology for high-performance organic photovoltaic devices. The investigation of various terpene solvent systems, along with the effective film formation and drying mechanisms necessary for optimal charge transport, can be facilitated by using Hansen solubility parameters. It is evident that terpenes have the potential to act as carbon-negative solvents, thus representing a distinctive and universally applicable approach to the efficient fabrication of large-scale, stable organic electronic devices. They also have the potential to be applied in a variety of areas, including pharmaceutical formulations, medical applications, cosmetics, cleaning solvents, and eutectic hydrophobic green solvent mixtures.[11–14] However, these valuable and versatile natural compounds also have negative aspects. For example, compounds in this class usually lack a suitable solvent environment for various organic transformations due to characteristics such as low thermal stability, high volatility and sensitivity to oxygen.[15–19]

Biodiesel, which is derived from vegetable oils or animal fats, serves as a sustainable alternative to diesel fuel and has recently been used in various extraction processes and polymerization reactions.[20–23] Castor oil, a non-edible vegetable oil, is a key component in the production of many functional polymeric materials.[24–26] However, we have found no studies in the literature that address the direct use of biodiesel and castor oil as solvents in organic synthesis reactions. Vegetable oils and their numerous renewable derivatives are a significant component of biomass and possess a range of physicochemical characteristics that render them promising green alternative solvents.[27,28] These characteristics include high biodegradability,[29,30] low vapor pressure and volatility,[31,32] low toxicity[32,33] and relatively low cost.[33] It is evident that vegetable oils and fats, along with their derivatives, are significant raw materials in a variety of industrial and academic applications due to their range of characteristics.

Using biologically sourced oils, such as sunflower oil, as renewable raw materials to produce bio-based chemicals, fuels and other useful materials is a promising approach. This is due to the high availability of these oils and their environmentally friendly properties. Sunflower oil is notable for its high linoleic acid content and relatively low production costs. However, the presence of unsaturated double bonds in these oils makes them susceptible to oxidation, which can have a detrimental effect on their stability, shelf life, and composition, depending on the source and processing conditions. These factors must be fully considered when evaluating the suitability of oils with a high level of unsaturation for industrial and academic applications.[34–36] Studies have shown that extracting sunflower oil with 2-methyltetrahydrofuran (2-MeTHF) increases its resistance to lipid peroxidation and that its stability can be improved by propolis.[37,38] Furthermore, research has shown that the thermal stability of different oils varies.[39] It has been demonstrated that high oleic sunflower oil and blended oils are more resistant to thermal degradation than conventional sunflower oils.[40]

2. Vegetable oils and their derivatives

Plant-based oils, also known as vegetable oils, are derived from a variety of plant sources and are widely available around the world. These oils are primarily composed of triglyceride molecules. They also contain various bioactive compounds, the quantities of which vary according to the source. These components make these oils a highly suitable alternative to many petroleum-based chemical raw materials.[41]

Vegetable oils are characterized by their renewability, environmental friendliness, ease of availability from natural sources, high functionality, and low cost. These oils are one of the main components of natural resources, which are defined as biomass, and they have enormous potential alongside animal-

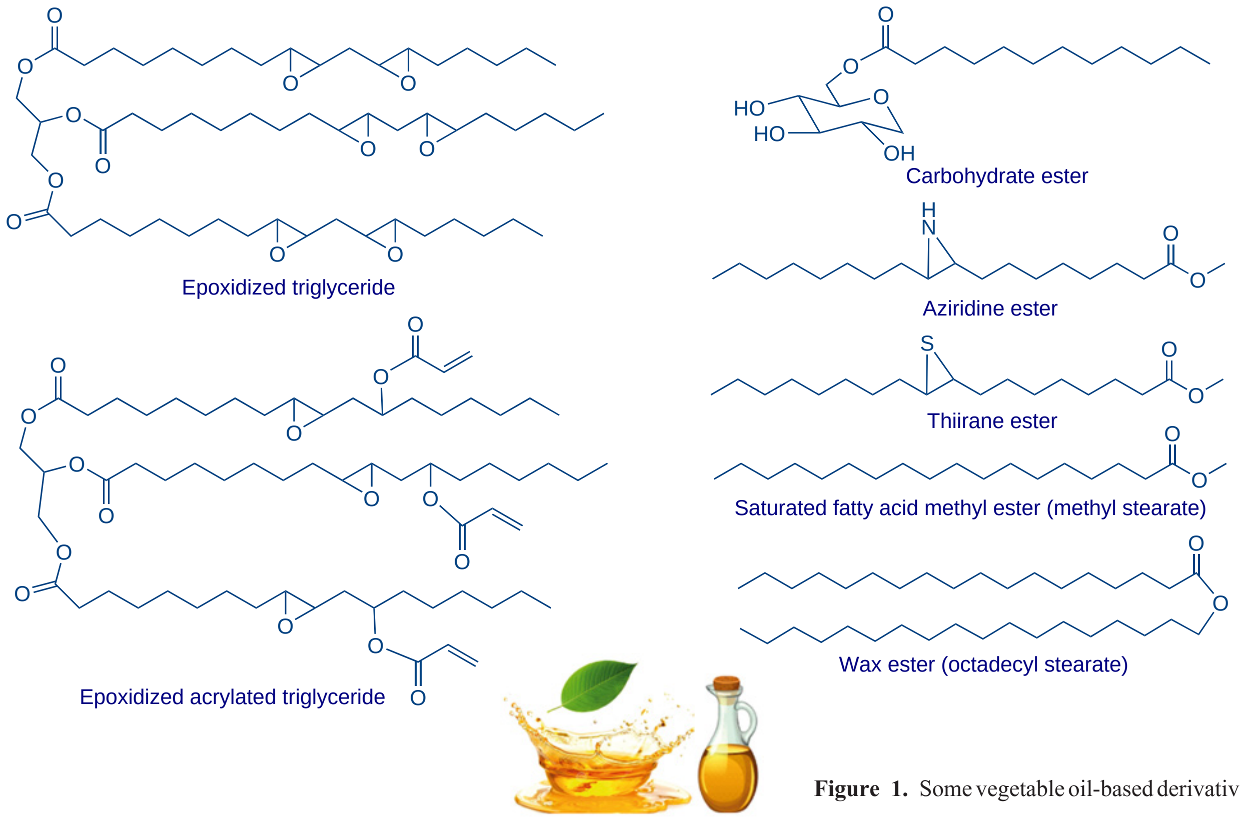


Figure 1. Some vegetable oil-based derivatives.

based oils. As crude oil reserves are set to deplete in the near future, industrial chemists and researchers are increasingly interested in exploring the potential of these resources.[42,43] Depending on their natural source, these materials can be readily converted into fatty acid methyl esters, synthetic wax esters, epoxidized triglycerides, or epoxidized fatty acid derivatives, which are known for their versatile properties when suitable functional groups, such as double bonds and/or free hydroxyl groups, are modified (Fig. 1).[44–49] Wax esters, which are defined as long-chain fatty acid esters of long-chain alcohols, represent a fascinating yet under-explored category of bio-based solvents for organic reactions. This substance is usually found in liquid or semi-solid form at ambient temperature and is characterized by its extremely low volatility and high thermal stability.[50–52]

2.1. Uses in miscellaneous applications

In the field of industrial lubrication, vegetable oils have become a topic of significant interest due to their exceptional properties. Through chemical modification, these oils have demonstrated the potential to be utilized in a wide range of applications (Fig. 2). Their superior properties make vegetable oils promising candidates for diverse industrial applications.[30] These renewable materials are attracting attention in the pharmaceutical, cosmetic, and food industries as sustainable solvents that are much safer than petroleum-based solvents for various formulations and efficient extraction processes (Fig. 3).[53–56] Odorless vegetable oils, such as sweet almond and soybean oils, have been found to be effective insect repellents for the short-term protection of various food packaging materials.[57]

The use of vegetable oils, such as palm, soybean and linseed oils, as plasticizers to replace carcinogenic, petroleum-based oils in the production of rubber composites, is becoming more widespread.[58] These materials are considered suitable for preparing lipid-based nanoparticles for use in pharmacology and cosmetics.[56] Research findings have suggested the potential efficacy of fatty acid methyl esters as a substitute for toxic, conventional solvents (*e.g.* white spirit and ligroin), which are frequently used in cultural heritage restoration applications.[51] Vegetable epoxy oil polymer resins have been reported to be suitable for use as the primary component in novel bio-derived formulations for high-temperature photopolymerization 3D printing.[59] In summary, vegetable oils and their acid derivatives have been shown to be highly reliable, renewable and environmentally friendly materials for preparing bio-based advanced materials for a variety of applications.[60]

2.2. Vegetable oils and derivatives as green solvents in organic synthesis

Vegetable oils have been identified as a class of green solvent with great potential in organic synthesis and numerous other industrial fields due to their favorable properties, such as low toxicity, biodegradability, renewability, the ability to solubilize many organic compounds, high boiling and flash points, good thermal stability, and relatively low cost compared to conventional organic solvents. The ProTox 3.0 web server was used to estimate toxicity endpoints for several organic solvents frequently used in organic reactions, as well as for triglyceride molecules present in certain vegetable oils (Fig. 4).[61] As shown in the figure, the LD50 values, which are used to measure toxicity, were found to be lower for organic solvents. It is noteworthy that chlorinated solvents may be of particular concern within the group of selected organic solvents due to their heightened toxicity relative to other compounds. The estimation results clearly show that vegetable oils can be considered solvents with reduced toxicity.

In addition, these solvents have superior properties compared to other solvents, including DESs, ionic liquids and supercritical fluids, which are regarded as green solvents. For example, using supercritical solvents often requires expensive equipment and complex operational procedures.[62] The disadvantages of DESs, a distinct class of green solvents, include potential toxicity, higher cost, increased viscosity of the reaction medium, sensitivity to water and instability at high temperatures.[63,64] In contrast, the aforementioned bio-based materials can act as cost-effective solvents in various organic transformations without causing significant issues.

Clearly, these materials have their limitations. A notable disadvantage of using them as solvents is their limited ability to dissolve specific polar organic molecules. Nevertheless, it could be argued that they exhibit superior solvation capabilities for a

Figure 2. Some applications of vegetable oils and their derivatives.

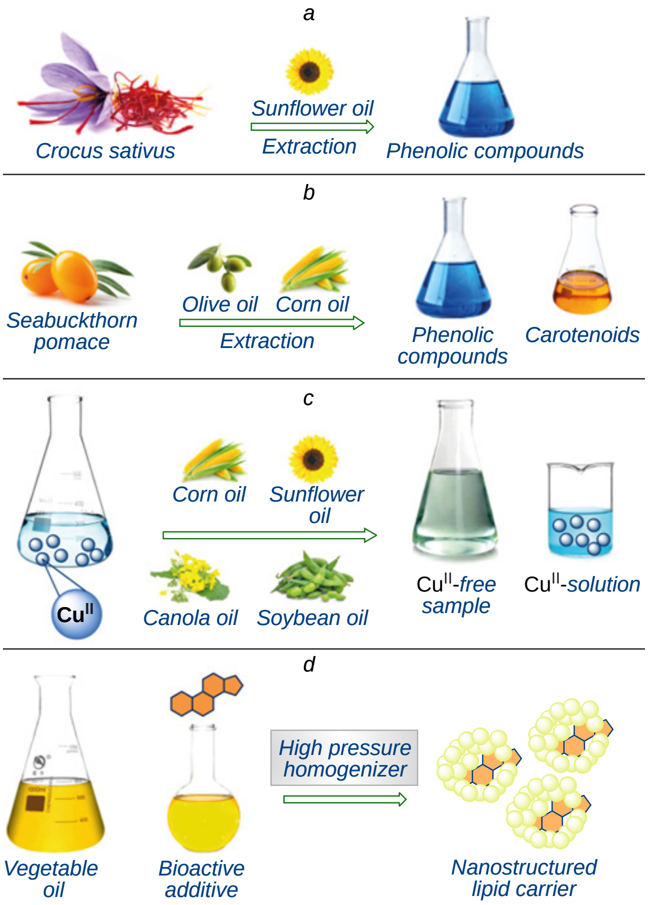


Figure 3. Applications of vegetable oils in extraction and formulation.

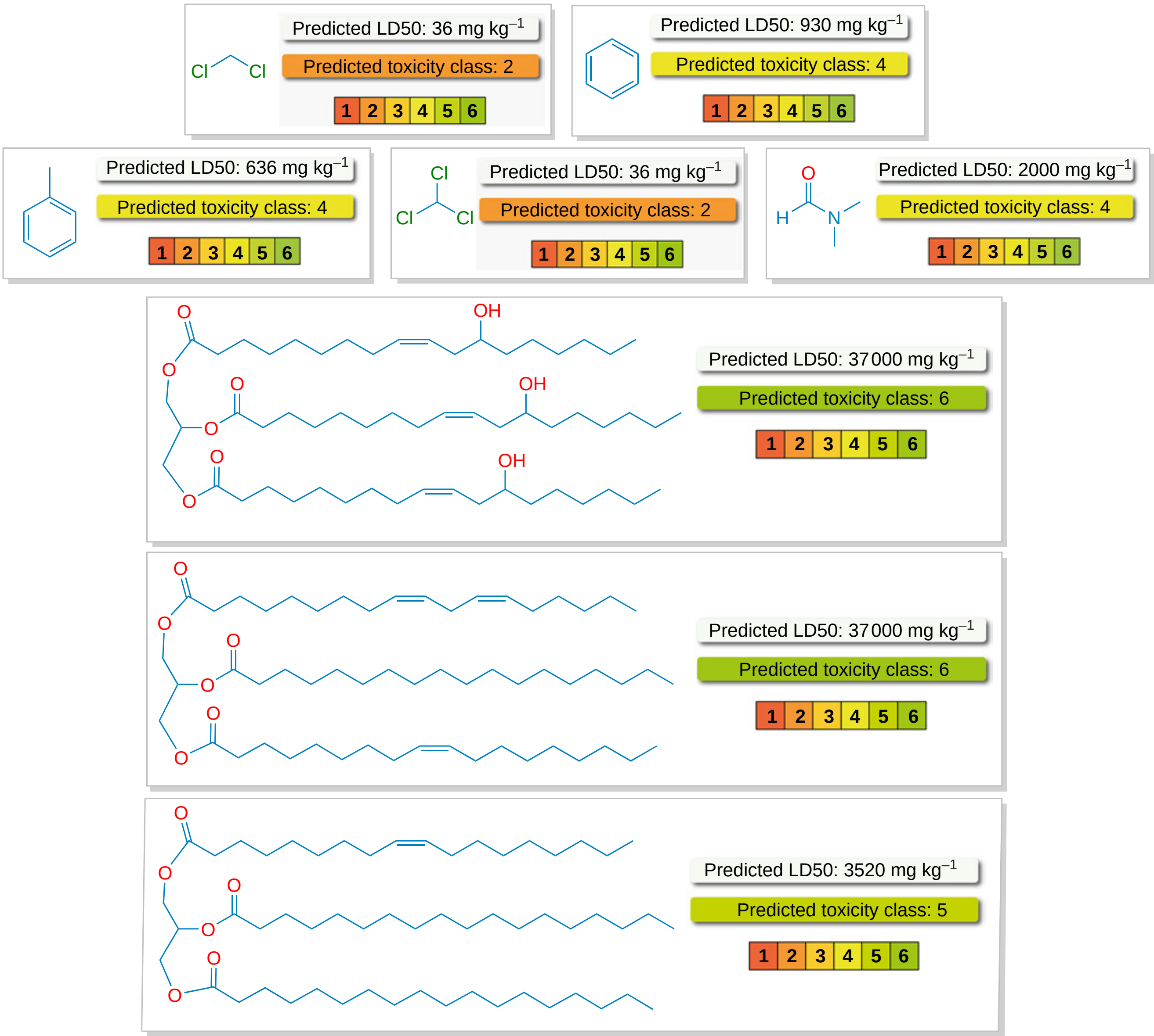


Figure 4. Predicted toxicities of common organic solvents and triglycerides using *ProTox 3.0*.

wide range of non-polar and moderately polar organic compounds, particularly at temperatures exceeding room temperature. Despite its high unsaturation content, sunflower oil has been reported to demonstrate excellent thermal stability, even at temperatures in excess of 150°C under standard atmospheric conditions.[65] In contrast, castor and coconut oils have been shown to exhibit a notable degree of thermal stability under conditions conducive to organic reactions, which often necessitate elevated temperatures of around 300°C.[66,67]

Conversely, bio-based solvents such as 2-MeTHF have been proposed as a substitute for toxic, conventional organic solvents in the isolation of vegetable oils from renewable sources.[68] The polarity of these materials can be adjusted by adding phospholipids or other amphiphilic components, thus enabling their solvation ability to be modified according to requirement.[69–71] These combinations can also increase the yield of certain reactions, which is an important consideration for those working in the field.[72] Vegetable oils can be isolated from a variety of natural sources using efficient, sustainable techniques that do not involve the use of conventional organic solvents.[73–80] The most common methods for these isolations are summarized in Table 1.

It is an established fact that vegetable oils have recently attracted interest as green solvents in the field of organic chemistry. This increased interest is due to a number of valid reasons. The chemical properties of these solvents, and the environmental benefits they offer in comparison with traditional petroleum-based solvents, make them an attractive alternative.[101] The primary advantages of these materials are outlined in Table 2.[102–104] Table 3 provides a comparative overview of green solvent metrics for potential solvent classes, including conventional petroleum-based organic solvents, vegetable oils, fatty acid methyl esters (FAMEs) and wax esters.[105–110] These metrics can guide the assessment of performance in chemical processes and their environmental and health impacts.[27,31,33,50–52,76] Consequently, recent studies (see the following sections) have indicated that these materials could serve as eco-friendly alternatives to conventional, petroleum-based organic solvents used in many well-known, versatile reactions (Fig. 5).

Table 1. Green techniques for isolating vegetable oil.

Method	Organic solvent	Oil yield (%)	Key pros	Refs
Cold pressing/mechanical extraction	–	60–80	Simple, preserves heat-sensitive compounds	81–83
Supercritical CO_2, DESs or ILs green solvent-based extraction	–	80–99	High purity, selectivity and scalability	84–89
Subcritical/superheated water extraction	–	44–94	Good extraction and bioactive retention	90, 91
Enzyme-assisted aqueous extraction	–	60–90	Valuable co-products (bioactives, enzyme residues, minerals, and nutrients for further valorization), green process	80, 92, 93
Thermo-physical methods: microwave, pulsed-electric field, *etc*.	–	80–85	Fast, good oxidative stability, solvent-free	94, 95
Bio-based solvents extraction (ethyl lactate, 2-MeTHF, limonene, *etc*.)	–	44–65	Green process, selective	96–100

Table 2. Advantages of using vegetable oils as a solvent in organic reactions.[102–104]

Features of vegetable oils	Benefits of vegetable oils
Renewable and biodegradable materials	These substances are derived from plant materials, making them sustainable. Furthermore, they are biodegradable in the natural environment
Non-toxic/low toxic and safe materials	Their low volatility and minimal health hazards make them safer to use in laboratories and industry
Materials with high boiling points	These materials are particularly well-suited to high-temperature reactions. They exhibit minimal evaporation losses, which enhances their efficacy and reduces wastage
Ability to dissolve organic compounds	These materials' unique and complex lipid composition enables them to dissolve both polar and non-polar organic compounds
No VOC (Volatile Organic Compounds) emissions	The compounds are characterized by an extremely low quantity of volatile organic compounds, resulting in reduced levels of air pollution and workplace exposure
Recyclability	A number of vegetable oils can be recovered and reused with minimal loss in performance
Enhanced selectivity	Their distinct polarity and viscosity can influence reaction pathways and product distribution, Higher viscosity has been demonstrated to influence mass transfer, phase behavior, and microenvironment, thereby modulating reaction pathways through mechanisms such as stabilizing reactive intermediates or regulating diffusion.
Green chemistry metrics	Using vegetable oils instead of conventional solvents enhances E-factors and process mass intensity (PMI)

Table 3. A comparative analysis of green metrics across different compound classes.[105–110]

Metric/Property	FAMEs	Wax esters	Vegetable oils	Petroleum solvents
Renewability	Renewable feedstocks	Renewable feedstocks (from jojoba oil, carnauba wax, beeswax, algae, or bioengineered yeast) or synthetic (from fatty acids and fatty alcohols)	Renewable feedstocks	Fossil-derived
Biodegradability	High	High	High	From low to moderate
Life Cycle Assessment (LCA)	Favorable	Favorable	Favorable	Poor
Volatility	From moderate to low	Very low	Very low	Generally high
Flash point	High (safer)	Very high	Very high	Low (flammable)
Toxicity/safety	Low	Very low	Very low	Mostly high
Thermal Stability	From moderate to high	High	Moderate	From moderate to high
Polarity	From low to moderate	Low	Low	Broad polarity range
Solubility for organics	Suitable for non-polar and some polar compounds	Suitable for non-polar and some moderate polar compounds	Suitable for non-polar and some polar compounds. May require co-solvents or emulsion techniques	Suitable for broad range of compounds
Enzyme compatibility	Excellent	Excellent	They are variable, but are generally compatible with enzymes and metal-containing catalysts including transition metal complexes used in green or bio-based chemical processes	Mostly poor
Cost and availability	Relatively cheap	Relatively expensive	Relatively cheap	Relatively cheap

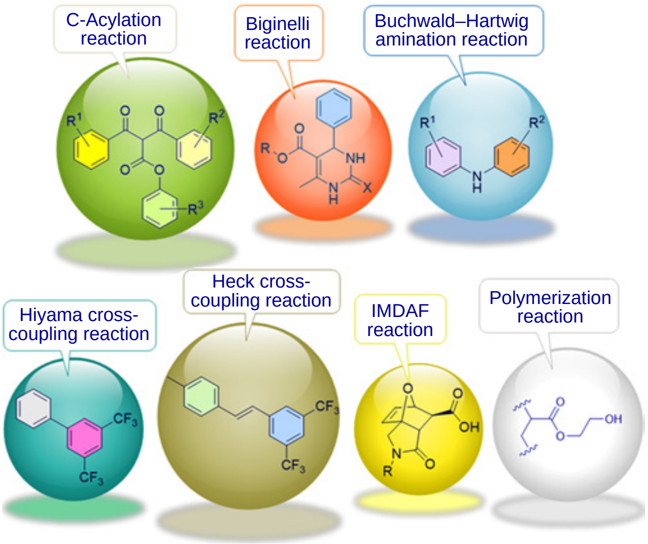


Figure 5. Bio-based oils as solvents in some organic reactions.

2.2.1. Challenges and strategies for product isolation in organic reactions using vegetable oils as solvents

The primary challenge in employing vegetable oils as solvents in chemical reactions arises from the isolation and purification of the desired products from the reaction medium in which they are utilized as solvents. However, it should be noted that such problems can also be encountered with a significant number of petroleum-based organic solvents.[101,111] Moreover, relevant chromatographic techniques may be necessary for directly isolating products from the reaction medium or for purifying products after isolation. However, as mentioned earlier in this study, if bio-based renewable alternatives can be used instead of traditional petroleum-based organic solvents in certain reactions, these should be evaluated as reaction media. In the context of vegetable oils, the main factors contributing to the challenges of product isolation are listed below:

The high boiling points of vegetable oils (typically > 300°C) can make distillation inefficient and energy-intensive. It is generally impractical to remove the oil by distillation without decomposition occurring, and vacuum distillation is also limited in feasibility due to the viscosity and thermal stability of the oil. Conversely, at elevated distillation temperatures and/or under reduced pressure, numerous organic compounds may be co-distilled with the oil, thereby rendering the separation of the product from the oil by conventional distillation processes impracticable. Furthermore, due to their high viscosity, oils become more viscous as the pressure drops, thereby preventing heat transfer and evaporation. Consequently, the processing of viscous materials under conditions of high vacuum necessitates the utilization of sophisticated and costly installations.

Comparable solubility behavior: it is evident that a significant proportion of organic compounds, particularly non-polar ones, exhibit a high degree of solubility in vegetable oils.[27] This property makes the extraction process rather challenging. Regardless of whether water or another polar solvent is applied for liquid-liquid extraction, the partitioning of the solute is more likely to favor the oil phase. It is therefore hypothesized that efficiently isolating the desired products through conventional liquid-liquid extraction techniques using vegetable oils as non-polar solvents may present significant challenges.

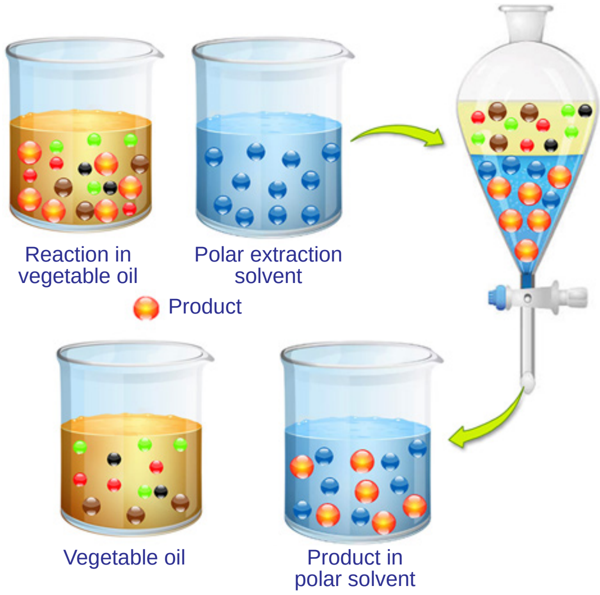


Figure 6. Principle of liquid–liquid extraction using polar solvents.

However, there are methodologies that can successfully overcome these critical challenges in the case of vegetable oils:

The process of liquid–liquid extraction using polar solvents (Fig. 6). The evaluation of polar protic or polar aprotic solvents for the extraction of highly polar products (*e.g.* methanol, dimethylformamide, *etc*.).[112,113] This method has particular importance when vegetable oils are used as solvents for organic transformations, in which case they have advantages over conventional solvents, such as providing better product yields and higher chemo- and stereoselectivity. Conversely, the necessity of employing toxic polar solvents in product isolations in a reaction where vegetable oils are considered as green solvent media appears to be a contradiction. However, the use of ethanol as an extraction solvent is advantageous due to its high affinity for certain polar compounds, especially at ambient temperature, its low toxicity, and its ease of removal under mild conditions. Furthermore, the water content in ethanol has been observed to increase the polarity of the solvent, which may have the most significant effect on the solubility of the components and their distribution between the oil and alcoholic phases. This increase in the polarity of ethanol has been found to reduce the solubility of vegetable oil in the extraction solvent while maintaining a sufficiently high extraction level of polar products. This, in turn, has been shown to result in a significant increase in the solvent selectivity between polar products and vegetable oil.

The use of the solid phase extraction (SPE) technique (Fig. 7). Selective retention of target products with specific polarity or functionality, as well as oils, can be achieved using a special SPE stationary phase product. Moreover, this technique allows for the straightforward separation of lipid classes.[114]

Precipitation or crystallization technique (Fig. 8). If the product has limited solubility in oil at low temperatures, crystallization becomes a viable isolation method. An anti-solvent (*e.g.*, cold ethanol) can be employed to induce crystallization for the product remaining dissolved in the oil medium. In the case of certain products that are solid at room temperature, the addition of a suitable solvent (*e.g.*, hexane, petroleum ether, diethyl ether or acetone) may induce precipitation.[115] In order to remove any residual oil contamination from the crystals during the filtration process, washing with cold hexane or alcohol is recommended. Despite the potential

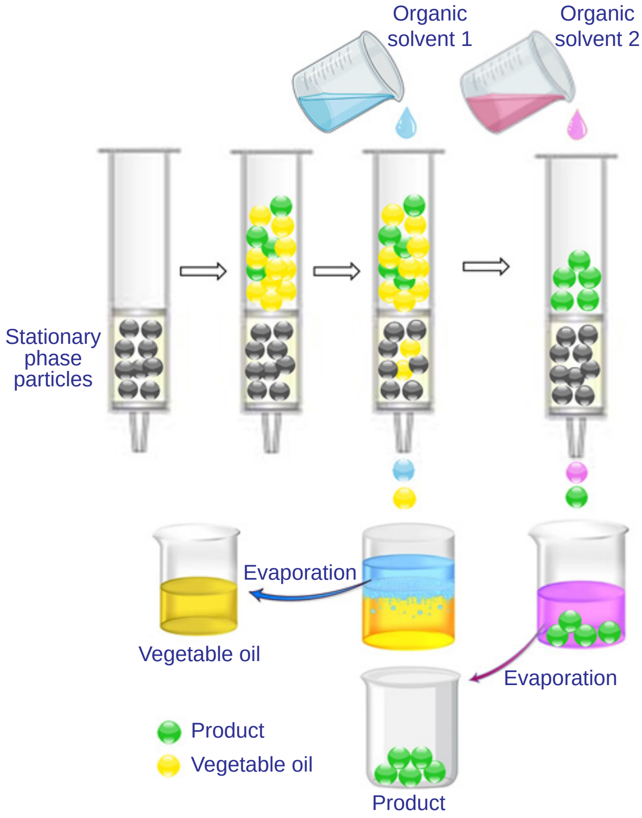


Figure 7. Principle of SPE method.

drawbacks associated with the utilization of petroleum-based organic solvents in this purification technique, whether for crystallization or washing purposes, these solvents can be readily recovered and reused repeatedly. However, from a sustainability perspective, bio-based solvents such as terpenes (less polar limonene or pinene) can also be recommended as alternatives to organic solvents such as hexane and petroleum ether to perform the same functions. It is noteworthy that terpenes typically demonstrate minimal solubility in the desired reaction product; however, they are compatible with vegetable oils. Consequently, upon the mixing of the reaction medium, terpenes have been observed to dilute the vegetable oil, reduce the solubility of the product, and cause it to precipitate.

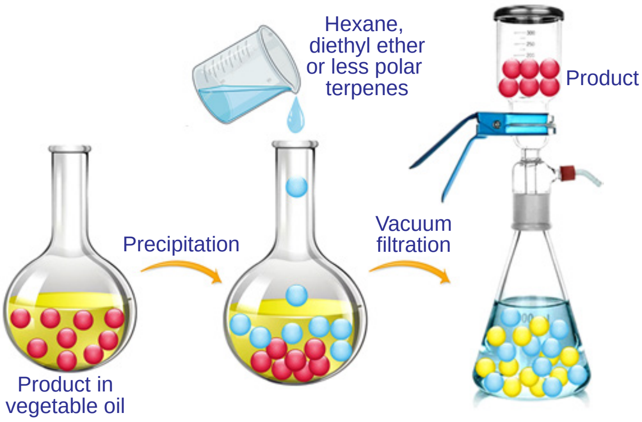


Figure 8. Principle of precipitation or crystallization technique.

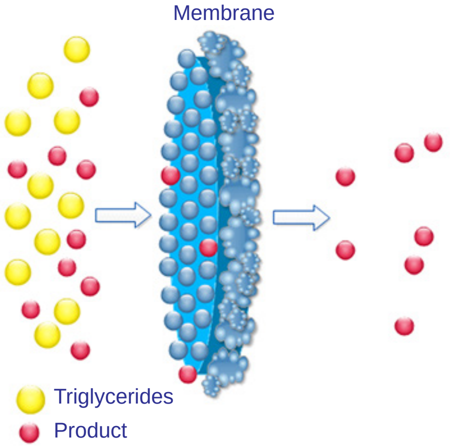


Figure 9. Principle of membrane separation technique.

Membrane separation technique (Fig. 9). The development of nanofiltration and ultrafiltration membranes has enabled the separation of low molecular weight products from high molecular weight triglycerides in oils.[116–118]

Chemical derivatization of the desired product (Fig. 10). Altering the chemical properties of the synthesized product to temporarily modify its solubility characteristics may be a viable solution for isolating the product (*e.g.* converting it to a salt

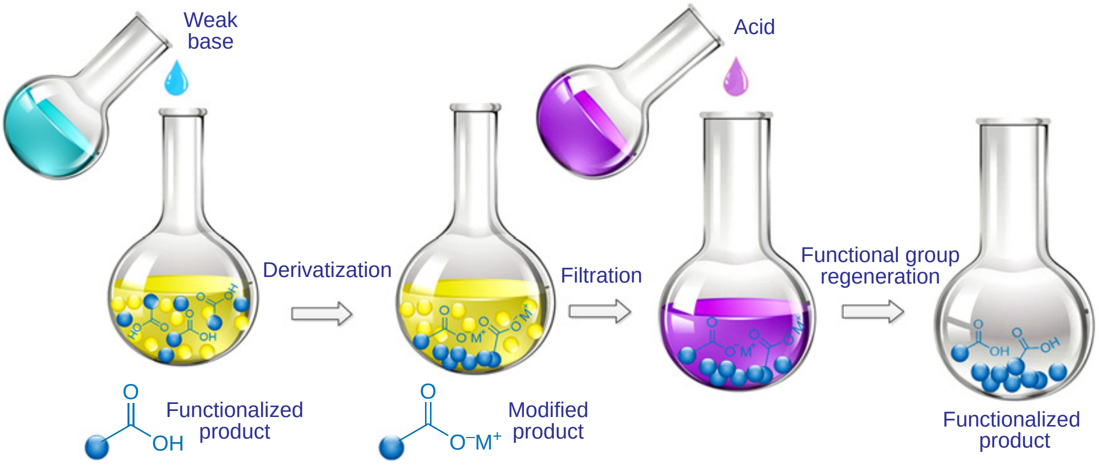


Figure 10. The principle of chemical derivatization of the desired product is illustrated using the example of salt formation.

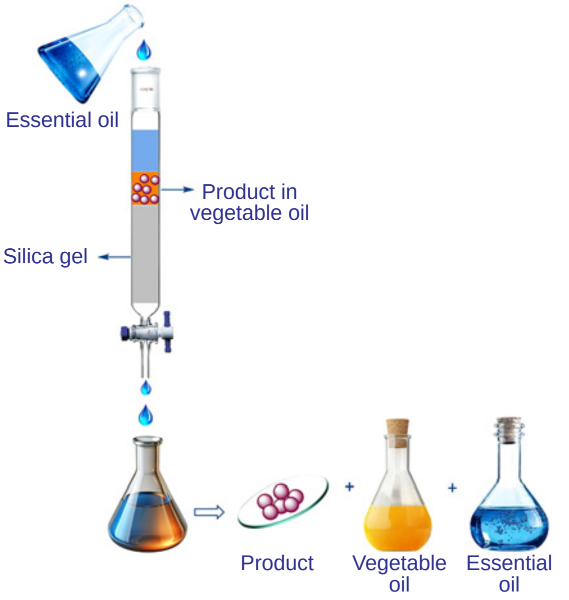


Figure 11. Scheme of the target product isolation using preparative column chromatography.

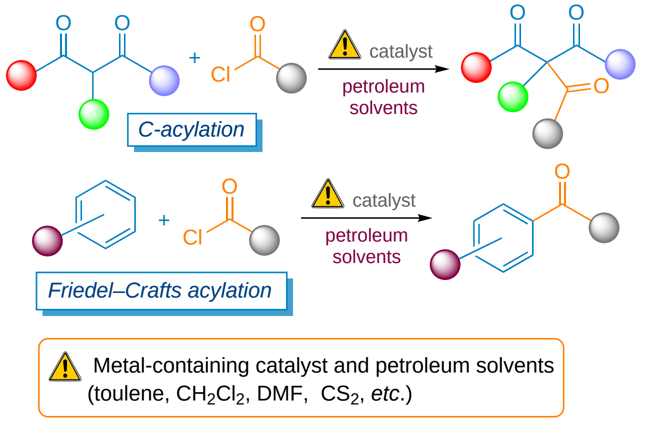
form). Following isolation, the modified product is returned to its original form.[119, 120]

Preparative column chromatography (Fig. 11). Target products in a reaction can be readily purified from vegetable oil media using column chromatography with renewable terpene-class essential oils as eluents. Consequently, following isolation and purification, the vegetable oils can be recovered for reuse.[121]

2.2.2. Common organic reactions carried out in vegetable oils

C–C bond-forming organic reactions are frequently employed by synthetic organic chemists in the preparation of complex molecules.[122] A variety of C-acylation reactions have been observed, with a particular emphasis on 1,3-dicarbonyl reactions. The solvent used in Friedel–Crafts acylation has been shown to exert a substantial influence on the reaction outcome. The C-acylations and Friedel–Crafts acylations are typically carried out in aromatic hydrocarbons, carbon disulfide and chlorinated hydrocarbons, which are toxic organic solvents [123–132] (Scheme 1). A further disadvantage associated with the use of alternative potential organic solvents in such cleavage reactions is their high volatility.[133] Recent studies have indicated that these reactions can also be conducted in solvent-free environments.[134–136] However, the development of such environmentally friendly processes has generally necessitated the use of modified solid catalysts and/or microwave support. Alternatively, it has been demonstrated that supercritical CO_2 and water can serve as effective green solvents in Friedel–Crafts acylation reactions.[137, 138] The potential for continuous catalytic Friedel–Crafts acylation exists within a two-phase environment consisting of an ionic liquid and supercritical carbon dioxide. However, it should be noted that product yields can vary significantly, ranging from low to moderate levels. Water facilitates the effortless isolation of products, contributes to reduced waste formation, enables the recycling of water-soluble reagents and catalysts, and, in certain instances, catalyzes Friedel–Crafts reactions by forming H-bonds or exhibiting weak acidity. However, the ability of water to dissolve many organic compounds that act as substrates in such reactions remains relatively weak.

Scheme 1

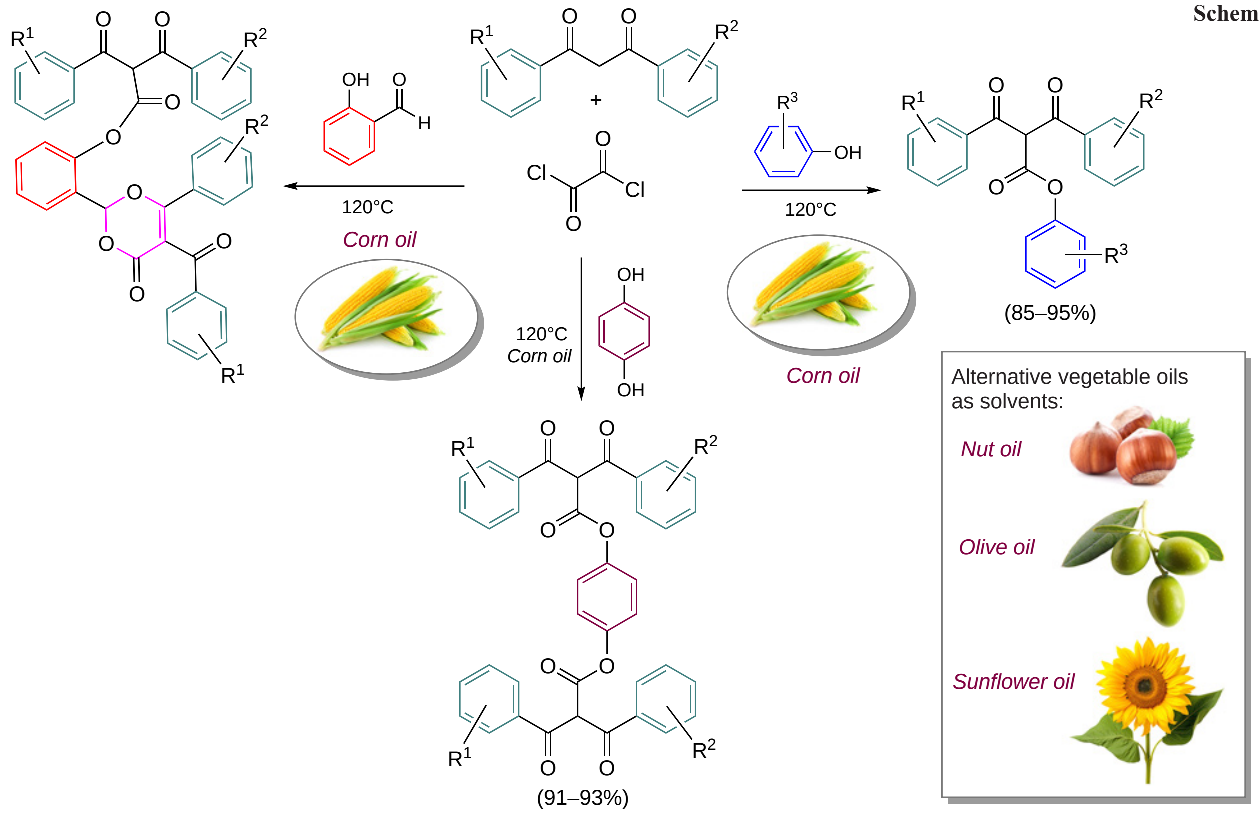


This review study clearly demonstrates the necessity for bio-based alternative solvents in such prevalent organic reactions. In this context, Menges and Sahin successfully carried out C-acylation of 1,3-diketones in a green solvent medium using vegetable oils, such as nut, olive or corn oil, in the presence of oxalyl chloride and phenol derivatives. The highest yields were obtained in the corn oil medium. This reaction has several advantages: it is very quick and does not require the addition of a base and further purification (Scheme 2).[132] Corn oil was used as the reaction medium in an acylation and cyclisation process involving phenol, dibenzoylmethane and oxalyl chloride, which was heated to 120°C. This work showed that, compared to traditional toxic aromatic solvents such as xylene, corn oil can effectively facilitate synthetic transformations due to its specific viscosity and solvation properties.

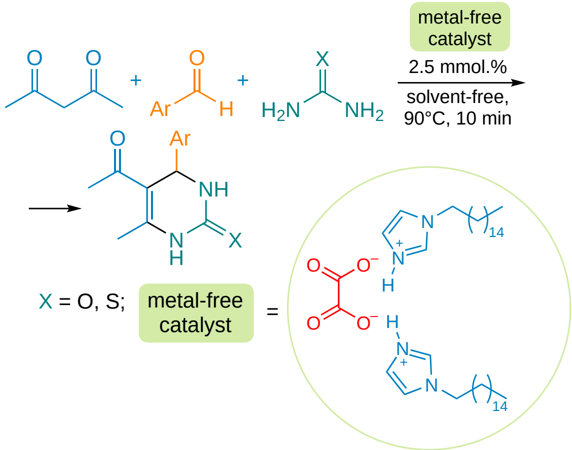
The Biginelli multicomponent reaction involves the condensation of an aromatic or aliphatic aldehyde, a β-ketoester or 1,3-diketone with a substituted or unsubstituted urea or thiourea. It is a highly effective synthetic method for producing compounds containing functionalized dihydropyrimidinone motifs, which possess a broad spectrum of biological activities.[139, 140] This reaction has attracted considerable interest due to the elegance of the process used and has historically been carried out in conventional organic solvents under hazardous catalytic conditions,[141–144] with the exception of ethanol, which is frequently used in this reaction and is considered environmentally safe and biological. However, using certain substrates, specifically aliphatic or substituted aromatic aldehydes, results in low yields of the desired dihydropyrimidinone derivative.[145, 146] Nevertheless, recent studies have proposed alternative methods to make the conditions of this reaction more environmentally friendly (Scheme 3).[147–150] In accordance with the 12 fundamental principles of green chemistry, these studies have predominantly evaluated the Biginelli reaction in relation to factors such as the solvent and solvent-free medium, and the catalyst used.

Furthermore, a recently published study has demonstrated that certain vegetable oils have the potential to serve as highly effective alternative solvents for this reaction (Scheme 4).[151] According to the researchers, who were the first to successfully perform this reaction in bio-based oil, palm oil would be a more favorable choice than cyclohexane and other bio-based solvents

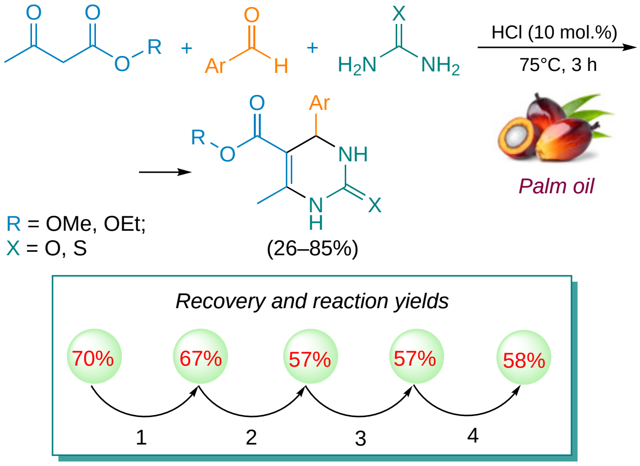
Scheme 2



Scheme 3



Scheme 4



such as p-cymene and ethyl acetate.[†] Moreover, based on the previous studies, these researchers hypothesized that non-polar solvents facilitate keto-enol tautomerization equilibrium, thereby enhancing the yield of the Biginelli product. Consequently, vegetable oils could be considered potential non-polar, environmentally friendly, bio-based solvents for this type of reaction. Vegetable oils consisting of less polar triglyceride molecules containing mainly long-chain saturated fatty acids (castor oil, for example, is a weaker solvent for this reaction) are most suitable for the Biginelli reaction. This study also investigated the recovery and reusability of vegetable oils. Palm oil was recovered and used as a solvent in four reactions, achieving a reasonable yield of the product (see Scheme 4). In this study, it was observed that although cyclohexane provides a high product yield, it cannot be recovered and recycled in this reaction because it forms a single phase that cannot be separated during aqueous washing. It was hypothesized that this phenomenon could be attributed to the increased solubility of cyclohexane in the aqueous phase, a consequence of the presence of ethanol and non-reactive starting materials. The author asserts that these compounds are capable of interacting with water through the formation of hydrogen bonds. Consequently, they disrupt the hydrogen bond network of water, thereby affording the water structure greater freedom, and this, in turn, increases the solubility of hydrocarbons such as cyclohexane in water.

The Buchwald–Hartwig amination reaction has been shown to be an invaluable process in synthesizing organic compounds.[152,153] However, it is important to note that the

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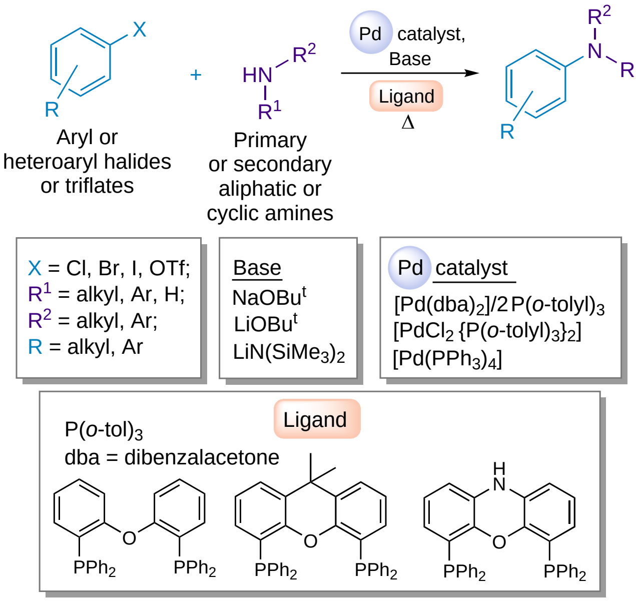
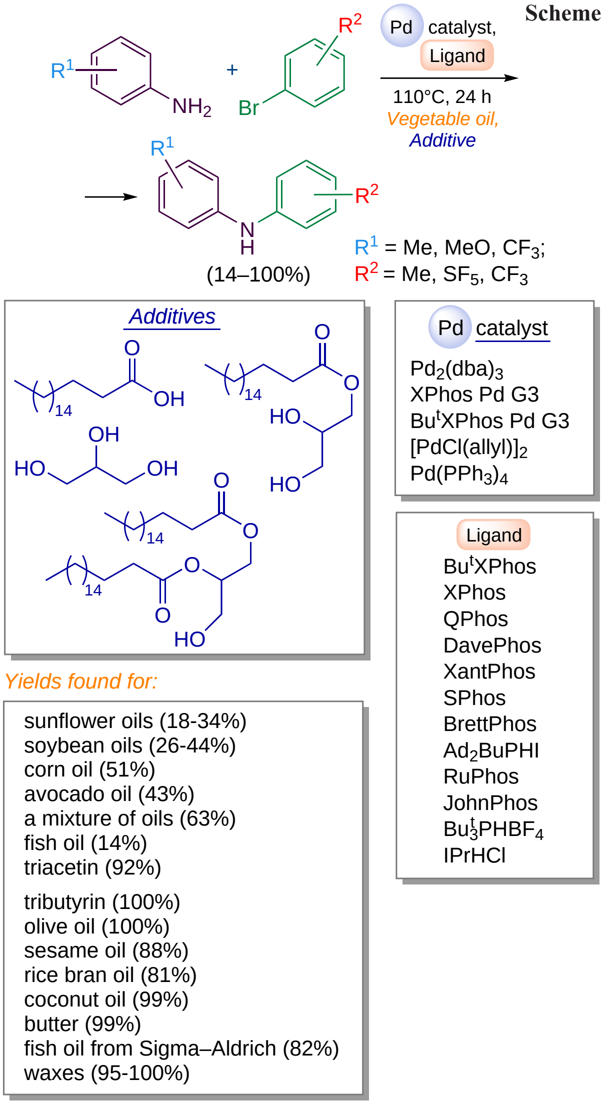
[†] In some studies, ethyl acetate is promoted as a bio-based and biodegradable solvent in terms of high synthetic efficiency and low waste generation. For example, A.Zirahi, H.S.Yamchi, A.Haddadnia, M.Zirrahi, H.Hassanzadeh, J.A.Zirahi. *AIChE J.*, **66** (2), 16828 (2019); https://doi.org/10.1002/aic.16828

nomenclature of this reaction was not established until after its initial description by Kosugi *et al.*[154] This well-known synthetic chemistry process is actually a palladium-catalyzed cross-coupling reaction.[155,156] It is widely used to form C–N bonds between primary or secondary amines and aryl or heteroaryl halides.[157,158] The coupling reaction is typically carried out in conventional organic solvents such as THF, dioxane, toluene and Bu^{t}OH, in the presence of a suitable ligand and base (Scheme 5). It has been demonstrated[159] that the nature of the metal catalyst, the base selected, the ligand structure and the solvent all have a significant impact on the outcome of this cross-coupling reaction. In a recent study, Yaseneva *et al.* developed a continuous flow Buchwald–Hartwig amination process for a pharmaceutical intermediate, demonstrating its feasibility in both laboratory-scale mini-plants and pilot-scale plants.[160]

Scheme 5



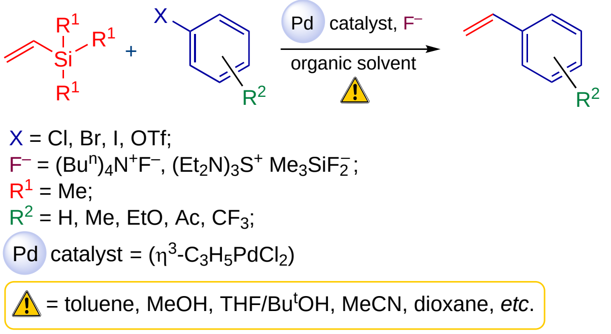
Scheme 6



In the present scientific era, efforts are being made to transform this common reaction into a more sustainable and health-friendly process, in line with the principles of green chemistry. For example, Yamada *et al.* have developed a ligand-free amination reaction catalyzed by Pd nanoparticles based on a Cu support plate under continuous microwave irradiation.[161] In addition, Shao *et al.* have developed a solvent-free mechanochemical Buchwald–Hartwig amination of aryl chlorides under high-speed ball milling conditions, eliminating the need for inert gas protection and providing the desired products in moderate to high yields.[162] Vegetable oils have been investigated[72] as potential alternatives to traditional organic solvents for use in the aforementioned reaction. Furthermore, it has been reported that amphiphiles and trace components present as additives in triglycerides substantially influence the reaction yield (Scheme 6). The presence of components such as free fatty acids, mono- and diglycerides, and glycerol in the bio-based materials used was found to affect positively Buchwald–Hartwig amination.[72] According to the researchers, these compounds possess surfactant characteristics and can act as phase transfer catalysts.

In the domain of synthetic organic chemistry, palladium-catalyzed reactions are arguably the most prevalent class of transition metal-catalyzed cross-coupling reactions.[163–166] The Hiyama coupling reaction is conceptually similar to the Suzuki reaction, but uses an organosilicon substrate instead of the organoboron compound used in the Suzuki reaction (Scheme 7).[167] This reaction involves the coupling of organosilicon substrates with organic halide compounds, catalyzed by transition metal catalysts, to produce the desired products with high yields (77–99%) and diastereoselectivity.[168–171] Liu and Tsui, for instance, developed a stereoselective C–F bond functionalization by carrying out a Hiyama coupling reaction involving tetra-substituted gem-difluoroalkenes and organosiloxanes. The reaction enables the installation of various functional groups, including challenging

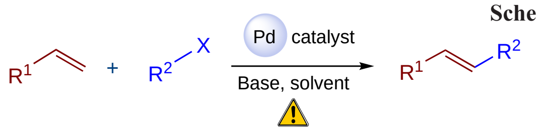
Scheme 7



alkyl groups, in the (*E*)-monofluoroalkene products.[169] In recent years, researchers have made significant progress in synthesizing a range of biologically active compounds using the Hiyama coupling reaction.[172] There are ongoing efforts to make this C–C cross-coupling reaction more sustainable. For example, Campos *et al.* carried out the Hiyama reaction using 1-phenyl-2-trimethylsilylacetylene and 7-chlorothieno[3,2-*b*]pyridine as reactants and eucalyptol as a bio-based solvent.[173] The yields obtained from this process were considered satisfactory (30–81%). The research team successfully obtained a series of cross-coupling products. For this purpose, in the study referenced above, 7-chlorothieno[3,2-*b*]pyridine or 4-chlorofuro[3,2-*c*]-pyridine was subjected to a reaction with various silylacetylenes under conditions that had been optimized for the Hiyama coupling reaction.

In contrast, the Heck coupling reaction (also known as the Mizoroki–Heck reaction) uses an alkene and an unsaturated halide or aryl halide compound (Scheme 8).[174,175] A novel Mizoroki–Heck reaction protocol with increased environmental sustainability and economic efficiency (yields, 48–91%) has recently been reported by Stini *et al.*[176] This protocol uses dihydrolevoglucosenone (Cyrene) as a green solvent and palladium (Pd/C) as a cheapest catalyst (compared to $Pd(OAc)_2$ and $PdCl_2$). The study revealed a broad scope of substrates for the coupling of aryl iodides with acrylamides, acrylates, acrylic acid, acrylonitrile, and styrene. The recyclability of Cyrene and the recovery of palladium from the final product are being investigated to boost the industrial feasibility of this protocol.

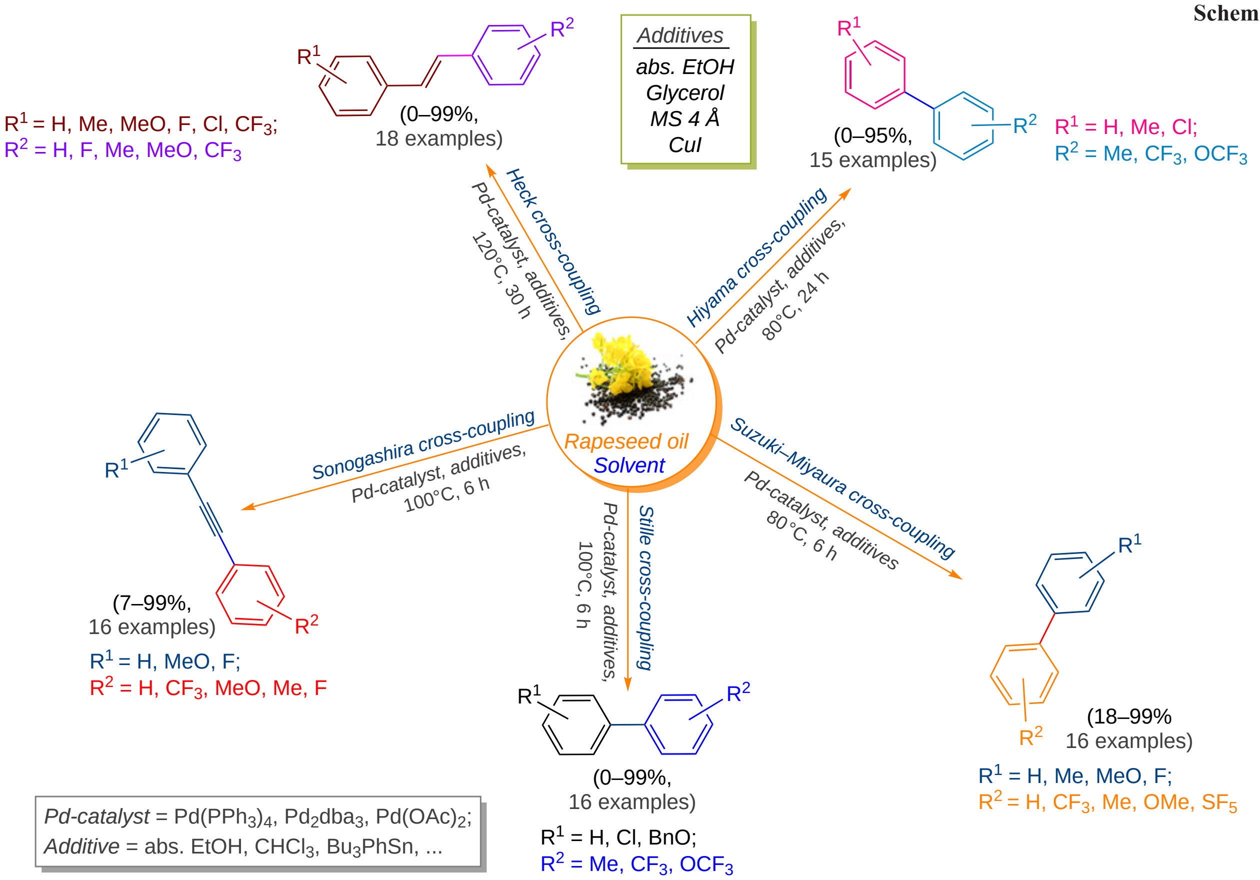
Scheme 8



X = halogen, OTf, OTs, N_2^+;
R^1 = alkenyl, heteroaryl, alkyl;
R^2 = Ar, hetero aryl, alkyl, Bn, Vi;
Pd catalyst = $Pd(PPh_3)_2Cl_2$, $Pd(PPh_3)_4$;
Base = K_2CO_3
⚠ = DMA, DMF, *etc*.

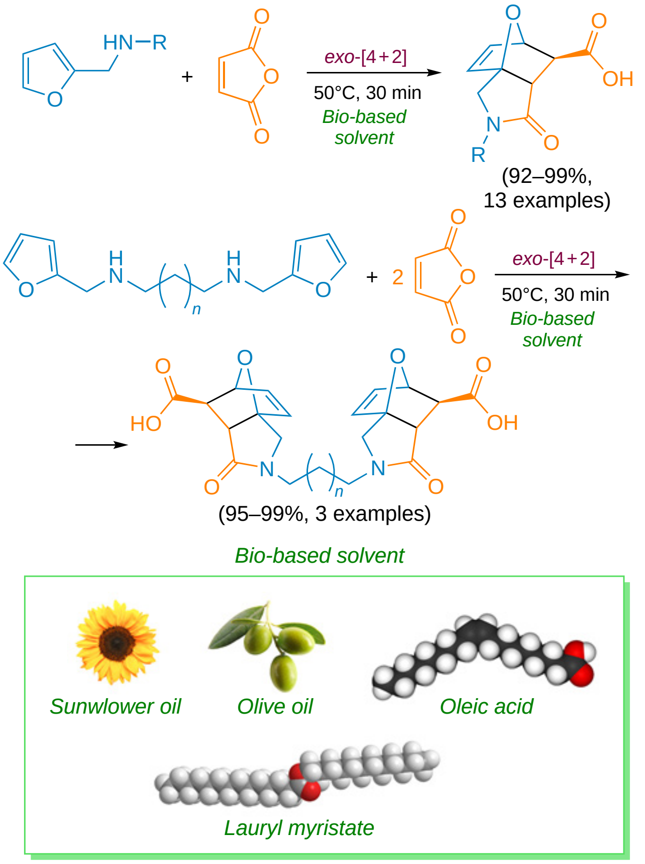
Gevorgyan *et al.* studied the use of various vegetable oils, fish oil, butter and waxes as solvents in Suzuki–Miyaura, Yamada, Stille, Sonogashira and Heck coupling reactions.[121] This study has demonstrated that vegetable oils and similar substances can be used as effective solvents in the coupling reactions, which proceed with nearly quantitative yield (Scheme 9). As outlined in an earlier section of this review, a number of isolation methods were employed to purify the products formed as a result of the coupling reactions. Examples of these methods include short-path vacuum distillation and column chromatography. However, it should be noted that the isolation steps mentioned for the reactions in this study also consisted of several steps (*e.g.*, separation with a pre-column followed by purification with normal column chromatography, or separation by distillation followed by purification with column chromatography).

Scheme 9



Intramolecular Diels–Alder reactions of furan systems, commonly referred to as IMDAF reactions, have proven to be an extremely useful method for constructing complex, oxygenated, polycyclic structures.[177,178] Reactions of this type generally occur at lower temperatures than their intermolecular Diels–Alder counterparts. Furthermore, it is important to note that these reactions typically allow non-activated alkenes to act as dienophiles, and they have a fairly wide range of substrates. This strategy is popular for forming complex polycyclic systems and natural compounds such as alkaloids,[179–182] as well as many organic intermediates and biologically active compounds.[183–185] However, these reactions are usually carried out in toxic organic solvents such as chloroform, toluene and benzene. Recent studies have demonstrated that these versatile reactions can also be successfully carried out in water and certain bio-based solvents, thereby extending their application.[115, 186, 187] Nadirova *et al*. successfully converted a series of readily available furan systems into potentially biologically active compounds, mainly possessing an exo-3a,6-epoxyisoindole skeleton, via a cascade Hinsberg/IMDAF sequential reaction involving interaction with various arylsulfonyl chlorides in water.[187] Yildirim successfully carried out the 100% atom-economic Diels–Alder reaction between a series of aminofurans and maleic anhydride in vegetable oils and waxes for the first time.[115] This reaction took less time, consumed less energy and produced higher yields (30 min, 50°C, 92–99%) compared to results obtained by Horak *et al.*[184] (360 min, 80°C, 72–82%). To achieve this, syntheses of various epoxyisoindole-7-carboxylic acids and bisepoxyisoindole-7-carboxylic acids were successfully carried out in sunflower oil, olive oil, oleic acid, and lauryl myristate under very mild reaction conditions. The corresponding products were then isolated using a practical procedure (Scheme 10). For this purpose, the mixture is cooled to room temperature at the conclusion of the reaction, whereupon the synthesized adduct is readily precipitated following trituration with hexane or diethyl ether. The precipitated product is then isolated by filtration under vacuum and washed with an ether-hexane solvent mixture. All of the cycloadducts synthesized in this study are solid organic compounds that were easily obtained in a pure form using the isolation procedure described above. However, if the products are liquid, this can be problematic as more advanced purification techniques, such as column chromatography, are required. Nevertheless, the findings of this study demonstrate that under certain conditions, it would be preferable to use vegetable oils and/or their derivatives instead of toxic organic solvents. This recommendation is based on fundamental criteria, including environmental risk factors and solvent cost. However, the ultimate decision will be made by the researchers.

Scheme 10

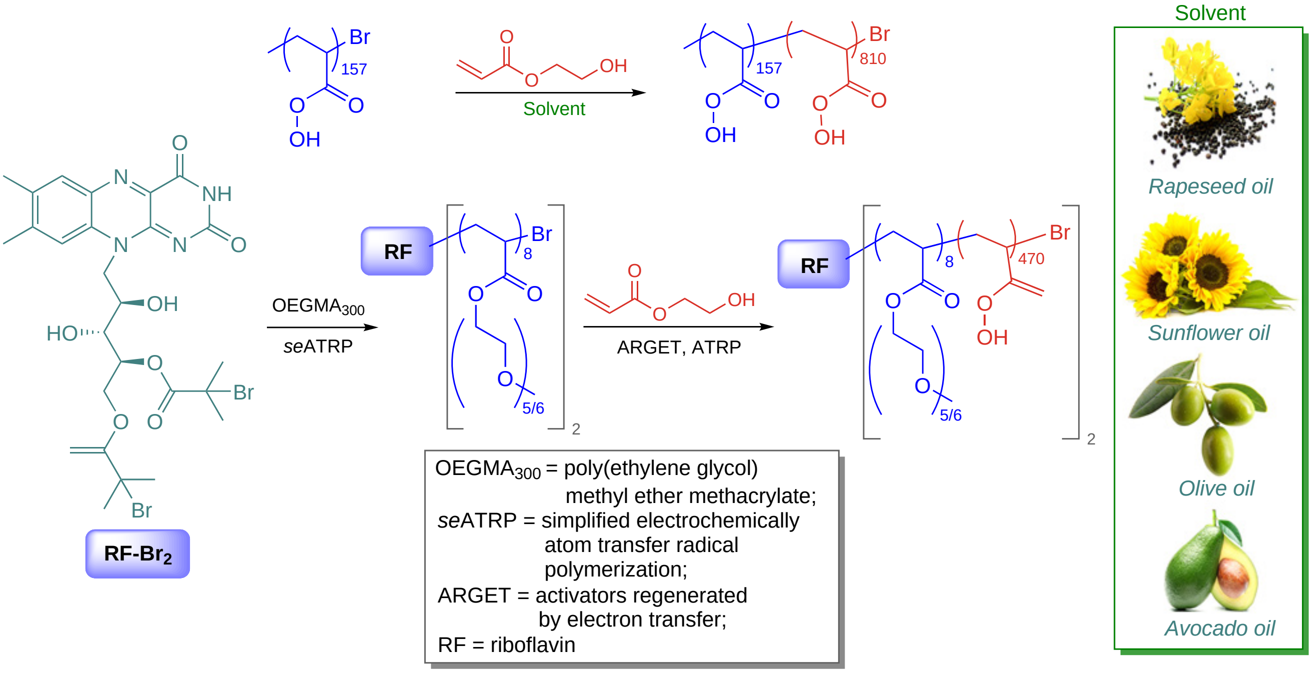


Vegetable oils can also be regarded as bio-based, renewable solvents that can be used in polymerization reactions. Controlling the polymerization of hydrophilic monomers in water is challenging due to the occurrence of various unwanted side reactions, which often result in polymers with extensive molecular weight distributions.[188] To overcome this, the polymerization of hydrophilic monomers can be carried out in a reverse dispersion system. Ascorbic acid-mediated atom transfer radical polymerization of poly(2-hydroxyethyl acrylate) can consequently be successfully carried out in a vegetable oil-based reverse emulsion to synthesize a well-controlled, high-molecular-weight, water-soluble polymer (Scheme 11).[188] In these water-in-oil emulsion polymerizations, rapeseed oil served as the continuous phase. The viscosity of the oil and its behavior at the interface affected droplet formation, emulsion stability and, accordingly, the dispersity and initiation efficiency of the polymer products. The polymerization yields of HEA using ARGET ATRP in vegetable oil-based reverse emulsions were obtained as 52–95% in rapeseed oil, 56–94% in sunflower oil, 48–63% in extra-virgin olive oil, and 36–45% in extra-virgin avocado oil.

Ishizuka *et al.*[189] developed an environmentally friendly approach that does not require the use of toxic organic solvents to produce polymeric capsule materials with a water-based core (Scheme 12). The monomer conversion was determined to be between 57–67% *via* ^{1}H NMR analysis. These materials have high potential value for a wide range of applications. Furthermore, certain vegetable oils, including castor oil, Jatropha oil and soybean oil, have been identified as biodegradable and renewable monomers in epoxidation reactions for synthesizing advanced polymeric materials suitable for various applications.[190–193]

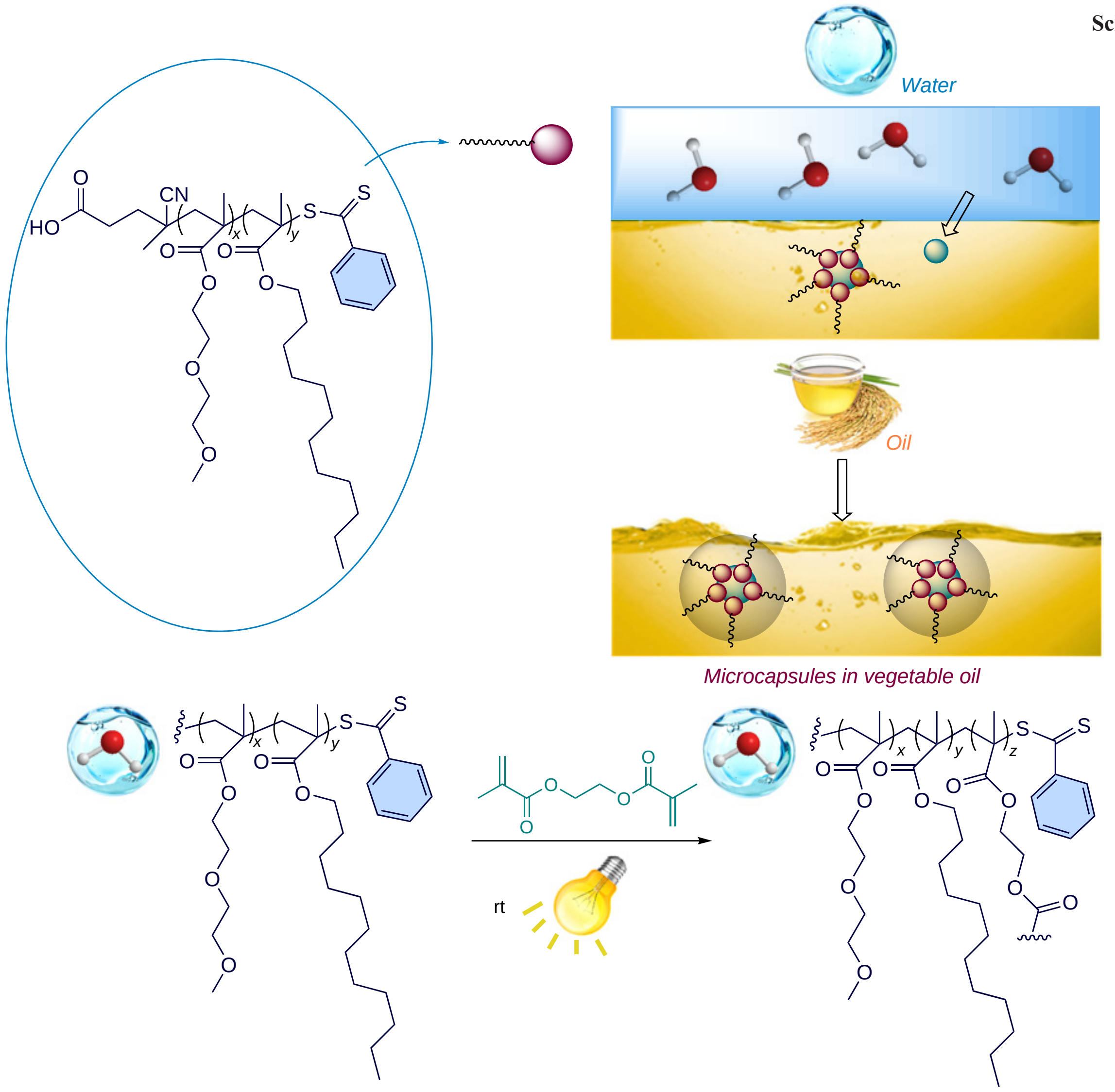
A new area of green chemistry involves using biodiesel as an environmentally friendly solvent thanks to its advantageous properties. These include low toxicity, renewability, biodegradability and dissolving properties similar to those of well-known organic solvents such as toluene and hexane. Thus, biodiesel can be considered a reliable alternative to conventional solvents used in polymerization techniques.[23, 194, 195] Biodiesel has several appealing properties. It is non-toxic or has very low toxicity; its boiling point is higher than that of many organic solvents; and its solubility properties are occasionally superior. It maintains low viscosity during polymerization reactions, averting issues associated with heat transfer and monomer mixing. Furthermore, it is readily accessible and comparatively economical. A recent study showed that biodiesel can effectively function as a solvent in the polymerization of epoxidized methyl esters. The study also demonstrated that phthalic anhydride and 2-methylimidazole can serve as effective initiators (Scheme 13). The biodiesel solvent used in this process can withstand temperatures of up to approximately 215°C. Following polymerization, biodiesel can be efficiently isolated from the polyester matrix through straightforward thermal treatment. Since biodiesel degrades at around 215°C and polyesters at around 320°C, a simple thermal treatment was introduced to separate the biodiesel solvent from the reaction products. This treatment involves heating to 250°C, followed by cooling to

Scheme 11

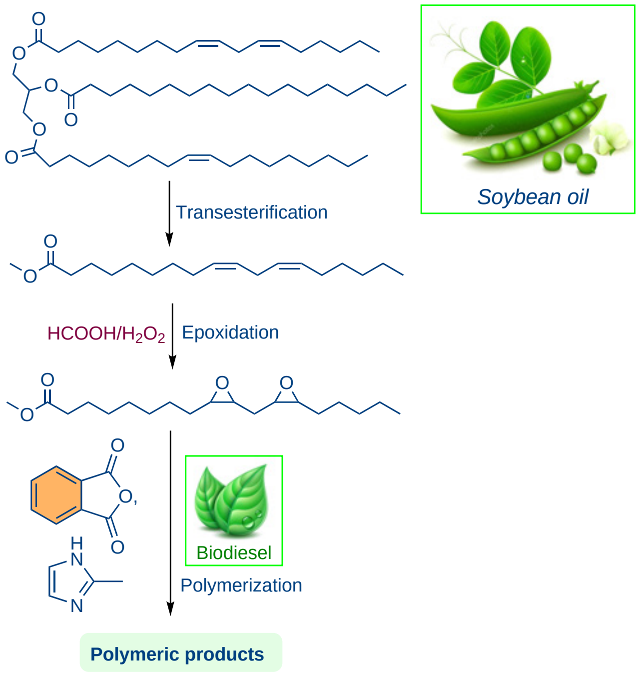


OEGMA$_{300}$ = poly(ethylene glycol) methyl ether methacrylate;
*se*ATRP = simplified electrochemically atom transfer radical polymerization;
ARGET = activators regenerated by electron transfer;
RF = riboflavin

Scheme 12



Scheme 13

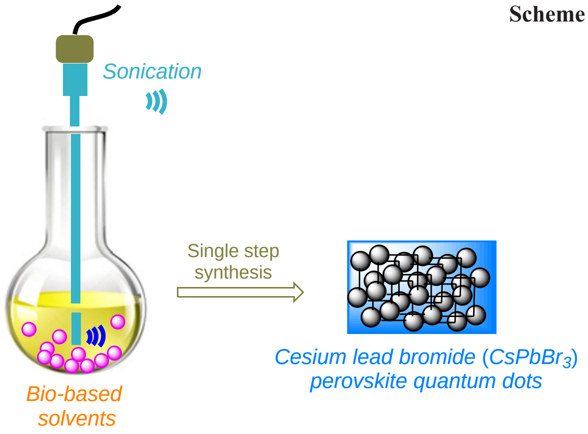


room temperature. A second heating process was then carried out, this time up to 500°C, to confirm through TGA/MS that the initial heating process (up to 250°C) had not affected the polyester matrix and was sufficient to remove the biodiesel.[22]

Despite its wide range of applications, it is challenging to reuse waste polystyrene. Volatile, petroleum-based organic solvents are usually used for this purpose. However, recent advancements have led to the development of eco-friendly methods for reusing this material. For example, one method involves dissolving polystyrene components in a fatty acid ester (methyl, ethyl or allyl esters of palmitic, stearic, oleic, linoleic or linolenic fatty acids, the most favored fatty acid ester is methyl soyate, which is a mixture of methyl esters of five main fatty acids found in soybean oil). The resulting mixture of solubilized polystyrene and a fatty acid ester can be used in various polymeric applications, including the synthesis of low-cost mixed polymers (see Scheme 14).[196]

Furthermore, vegetable oils have been shown to be effective solvents in the production of certain advanced inorganic materials. $CsPbX_3$ perovskite quantum dots (PQDs) have attracted attention due to their potential use as absorber materials in photovoltaics owing to their superior properties.[197] The colloidal preparation of these PQDs typically relies on the use of toxic, fossil-derived solvents as reaction media. However, a recent study reported an environmentally friendly, sonochemical synthesis method for $CsPbBr_3$ PQDs using vegetable oil solvents as an efficient reaction medium (Scheme 15).[198]

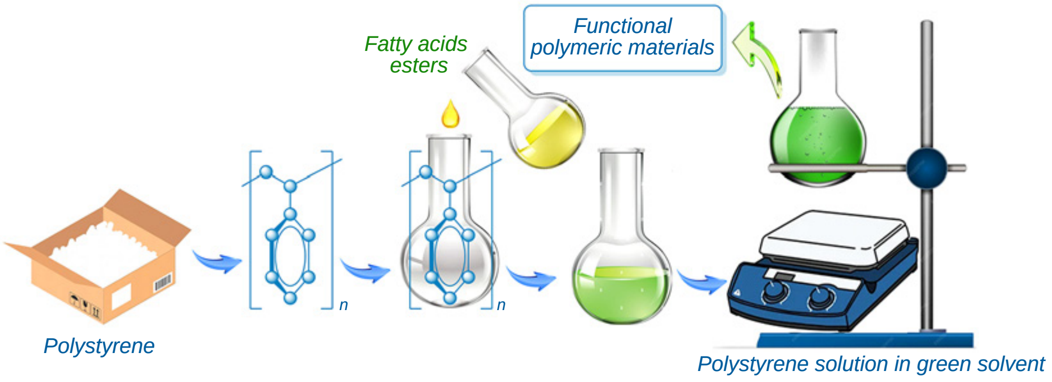
Scheme 15



3. Conclusion

A significant proportion of the petroleum-based materials used as solvents in various extraction processes and organic synthesis reactions have the potential to compromise human and environmental health. These materials are also characterized by high volatility and low thermal stability. It is crucial to explore environmentally and human-friendly alternatives to these harmful materials and cultivate them as competitors to conventional solvents, which are still widely used as reaction media in organic synthesis. Despite the recent use of vegetable oils for the isolation of certain natural compounds and in drug formulation applications, synthetic organic chemists would be well-advised to consider these materials as an alternative, and in some cases even a superior, reaction medium. Vegetable oils are non-polar, complex lipophilic systems whose composition varies depending on their origin and production method. They also belong to the class of bio-based solvents (category 1). These materials are characterized by their biodegradable nature, low toxicity and low volatility. This review reveals a lack of synthetic chemists motivated to work in this field. It can be posited that there is no conventional organic solvent that is entirely problem-free, nor any green solvent that is derived from a natural source or prepared by synthesis. Similarly, vegetable oils have their own drawbacks. Nevertheless, the existing literature suggests that these materials have high potential as green solvents for a wide variety of reactions, and could compete with conventional toxic organic solvents. Certain studies have demonstrated that

Scheme 14



vegetable oils can facilitate superior product yields in reactions compared with conventional petroleum-based solvents and a number of alternative bio-based solvents. Non-edible vegetable oils are expected to be widely used as solvents in organic synthesis reactions in the near future due to their potential for large-scale production and, consequently, their enhanced economic viability. This phenomenon is based on the inherent properties of these materials, which are characterized by their safety and harmless nature. Notably, these materials have a high propensity to function as organic solvents due to their relatively high boiling points, a property widely regarded as highly advantageous for a range of organic transformations. In summary, researchers working on organic transformations are advised to consider using these materials as solvents, as they may be a more environmentally friendly alternative to traditional solvents and would contribute to a more sustainable future.

4. List of abbreviations

ARGET — activators regenerated by electron transfer,
ATRP — atom transfer radical polymerization,
DESs — deep eutectic solvents,
FAMEs — fatty acid methyl esters,
ILs — ionic liquids,
IMDAF — intramolecular Diels-Alder furan,
LCA — Life Cycle Assessment,
OEGMA$_{300}$ — oligo(ethylene glycol) methyl ether methacrylate 300,
OTf — triflate,
OTs — tosylate,
PQDs — perovskite quantum dots,
SCFs — supercritical fluids,
*se*ATRP — simplified electrochemically atom transfer radical polymerization.

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