Chapter

Biodiesel Refining and Processing Strategies

Timothy J. Tse, Li Zhou, Farley Chicilo, Venkatesh Meda and Martin J.T. Reaney

Abstract

Biodiesel fuel is produced from triglyceride fats, and oils obtained from plant and animal sources. Typically, triglycerides are first transesterified to produce fatty acid alkyl esters (FAAE) and then refined. Traditional FAAE refining strategies are often energy-intensive, requiring large amounts of water (e.g., wet washing), adsorbents, and/or chemicals. Refining, in turn, produces substantial amounts of waste and is accompanied by the loss of biodiesel as neutral oil entrained in waste. A wide array of methods and technologies have been developed for industrial oil purification. Successful refining practices minimize waste and limit neutral oil losses. Recent studies have explored the use of adsorbents, solvent purification processes, membrane filtration, as well as novel applications of electrostatic field treatments to remove polar impurities (including free fatty acids, residues, soaps, and glycerides), and particulates from oils. This chapter will review and compare traditional current and novel strategies for refining FAAE for use as biodiesel.

Keywords: biodiesel, fuel, refining strategies, fatty acid alkyl esters

1. Introduction

An increase in global energy demand is driving a shift from traditional fossil fuels to renewable and sustainable energy, such as biofuels (e.g., bioethanol and biodiesel). Potentially, the use of renewable fuels can reduce greenhouse gas production, and pollution related to fossil fuel use [1] while also providing a sustainable source of fuel. Collectively, the biofuel markets is expected to reach $245 billion USD by 2027 [2], with biodiesel alone accounting for $73 billion USD by 2030 [3]. The top global producers of biodiesel and bioethanol and their feedstocks are listed in **Tables 1** and **2**, respectively. Currently, the United States, European Union, and Brazil are major global producers for biofuels (bioethanol and biodiesel) [4]. The COVID-19 pandemic led to restrictions to global travel and transport that resulted in a significant global decline in transport fuel [4] consumption, that in turn placed strain on the supply chain [5]. The subsequent lifting of pandemic-related restrictions in 2021–2022 led to recovery of fossil fuel and biofuel markets. The sudden increase in demand led to higher feedstock prices and bottlenecks in supply chains that have in turn increased production costs

Country	Biodiesel Production (Million L for 2019–2021*)	Feedstock
European Union	14,882	Rapeseed oil/Palm oil/Used cooking oils
United States	8905	Soybean oil/Used cooking oils
Indonesia	8476	Palm oil
Brazil	6325	Soybean oil
Argentina	1765	Soybean oil

**Refers to average estimate between 2019 to 2021.*

Table 1.
Top global producers (2021) of biodiesel and the feedstocks used [4].

Country	Ethanol Production (Million L for 2019–2021*)	Feedstock
United States	58,182	Maize
Brazil	32,748	Sugarcane/Maize
China	10,433	Maize/Cassava
European Union	6112	Sugar beet/Wheat/Maize
Thailand	1794	Molasses/Sugarcane/Cassava

**Refers to average estimate between 2019 to 2021.*

Table 2.
Top global producers (2021) of bioethanol and the feedstocks used [4].

[4, 5]. Nonetheless, the biofuel market is expected to achieve a compounded annual growth rate (CAGR) of 7.81% by 2027 [2].

Renewable biomass fuels are classified as primary and secondary biofuels. Primary biofuels are traditionally woody or cellulosic plant material and dry animal wastes that are burned for energy [6], while secondary biofuels can be further categorized into three generations dependent on the feedstock material [7]. First generation biofuels include bioethanol produced from carbohydrate rich input materials such as corn and sugar cane, and biodiesel produced from vegetable oils (such as low erucic acid rapeseed oil and soybean oil), and animal fats such as waste cooking grease [6, 8]. A portion of the inputs for production of first-generation biofuels are derived from edible resources. Second generation biofuels include those that utilize non-edible cellulosic feedstock [7] and fuels produced from non-edible oil-rich plant seeds [6, 9]. Next, third generation bioethanol production relies on algal biomass as a sustainable feedstock [7]. There is also interest in investigating the suitability of microalgae for biodiesel production [10]. Recently, fourth generation approaches for bioethanol production that use genetically engineered organisms are being investigated [11]. Similarly, a fourth generation of biodiesel is based genetically engineered organisms [12].

Second-generation biodiesel can contribute to significant reductions in carbon dioxide emissions when compared with fossil diesel sources as the feedstocks used for their production is often considered carbon neutral (e.g., plant-based) [13]. Second-generation biodiesel has also been extensively studied [14] and often utilize fully or partially refined vegetable oils (e.g., soybean oil and rapeseed oils) [15], that must be transesterified using chemical treatments (e.g., acid and/or base catalytic transesterification, with alkali-catalyzed transesterification being the most common [16]), to form fatty acid alkyl esters (FAAE) [15]. Typically, transesterification leads to the production of several contaminants in the FAAE including glycerol, glycerides, soap, alcohols, as well as accumulated moisture due to improper treatment processes or by adsorption of atmospheric moisture during storage [17]. The presence of these contaminants can have detrimental effects on the quality of the FAAE fuel, performance of vehicles being fueled, and storage properties of the fuel including, but not limited to, clogging of fuel filters [18], corrosion of storage containers [19], lower flashpoint [19], and increased deposit formation in the engine fuel system [19]. Therefore, removal of these contaminants is required to ensure quality and utility of the biodiesel product. However, conventional refining methods can require the use of many inputs, high capital cost equipment, and generate waste. For example, wet washing uses heated and softened water [20], while "dry washing" uses adsorbent materials [21] to remove polar contaminants [22]. These washing methods can result in the production of substantial waste [20] and/or require large amounts of expensive absorbent materials [1]. Furthermore, the introduction of water during wet refining processes can lead to the formation of emulsions which leads to FAAE loss [23], but also often requires specialized infrastructure to reduce moisture content in the biodiesel after refining [22].

In addition to wet and dry washing, alternative refining methods using enzymatic processes such as enzymatic degumming [24], physical refining methods [25], and membrane filtration [26] have been explored. Although these methods have been successful in removing contaminants and reducing chemical consumption and byproduct waste, implementation at an industrial scale can be prohibitive due to the cost of expensive reagents and specialized infrastructure. In addition, there are few solvent-stable membranes, and substitution of the solvent during seed oil extraction may be difficult [26]. For example, alcohols exhibit higher latent heat and lower solvent power, compared to hexane thus, requiring a larger amount of solvent to extract seed oil. More recently, newer refining methods (e.g., electrostatic field refinement) [27, 28] have been investigated as approaches to minimize the ecological footprint compared to conventional methods. The purpose of this review of biodiesel production, is to compare approaches of FAAE refining, and explore strategies for refining FAAE for biodiesel production.

2. Production of biodiesel

Most commercial biodiesel is produced by the transesterification of triglycerides with short-chain alcohols (e.g., methanol, ethanol), in the presence of a catalyst [29, 30], yielding glycerin and FAAE [31, 32]. Catalysts can include alkali catalysis (most common approach), acid catalysis, a sequence of acid catalysis followed by alkali catalyst, or a lipase catalysis [33–37] (**Table 3**). After transesterification, the FAAE phase is separated from glycerol and residual catalyst via settling or centrifugation [1].

Free fatty acids (FFA) in an oil or fat feed material can neutralize alkali catalysts and limit the transesterification reaction required for producing FAAE [15] and ultimately affect the yield and quality of biodiesel [38]. Reactions of the FFA in oils with alkaline catalysts forms soaps (e.g., carboxylic acid salts) and water. The presence of soap increases FAAE viscosity, and forms emulsions that can make separation

Catalyst type	Advantages	Disadvantages
Alkali	More effective, less corrosive	Extra alkali required when processing feedstocks with high content of free fatty acids Increased production of soap
Acid	Appropriate for low quality oil feedstocks	Slow process and results in incomplete reactions
Acid-alkali	Less depletion of catalyst, enhanced FAAE yield, generates less contaminants	Can require additional infrastructure and increase processing time
Lipase	Continuous production	Expensive and can be unstable

Table 3.
Advantages and disadvantages of FAAE production methods.

of glycerol from biodiesel difficult [39]. To prevent this, feedstocks containing FFA should be treated prior to the transesterification process. Frequently used methods to decrease FFAs include acidic esterification, enzymatic esterification, and contacting the FAAE phase with alkaline water. Common acid treatments involve the use of sulfuric acid, in the presence of an alcohol such as methanol or ethanol, to reduce FFA content in the feedstock oil [40–45]. Hydrochloric acid and phosphoric acid can also reduce FFA content in cooking oil destined for biodiesel production [46]. Treatments of high FFA oils with lipase as an enzyme catalysts and glycerol can convert FFAs to glycerides, including monoglycerides, diglycerides, and triglycerides giving an overall reduction of FFA content [47]. The decrease in FFAs helps to improve conditions for alkali transesterification reactions [48].

This transesterification of lipids produces FAAE with beneficial physicochemical properties, including lower viscosity, ignition and flash points, making it an suitable biofuel [49]. However, incomplete transesterification can produce unrefined biodiesel with a variety of contaminants including glycerol, mono–/di-glyceride, soap,

Contaminants	Contaminant limits in standardized guidelines	Potential hazards
Moisture	0.05% (by volume; ASTM D6751)	Fuel filter plugging due to microbial growth; corrosion of storage container
Methanol	1. % (by weight; EN 14110)	Lower flashpoint of biodiesel
FFA	0.5 mg/g KOH	Corrosion, low oxidation stability
Metals	5 mg/kg of Class 1 metals (Na + K; EN 14214)	Deposits in the injector, filter blockage, engine weakening
Soap	66 mg/kg when KOH is used as the catalyst (ASTM D6751)	Conferring biodiesel a degree of electric conductivity
Free glycerol	<0.02% (by weight; ASTM D6751)	Forming gum-like deposit around injector tips and valve heads
Total glyceride	<0.24% (by weight; ASTM D6751)	Increased levels of oxidative deposits

Table 4.
Common contaminants in FAAE and the potential hazards [19, 50–53].

alcohol, moisture, and catalyst [29]. The presence of these contaminants in FAAE could significantly affect engine performance, damage fuel storage equipment, and lead to engine failure (**Table 4**). To ensure that biodiesel fuel is suitable for use as a fuel the crude FAAE must be refined to meet standardized guidelines, such as the American Standard for Testing Materials (ASTM D6751) and the European Standard (EN 14214) [54]. Commercial biodiesel refining includes either wet or "dry" washing, however, these processes have significant drawbacks, such as requiring the use of expensive infrastructure and the production of waste products (e.g., wastewater). More recently, sustainable purification alternatives have been explored for biodiesel to maximize outputs, reduce the generation of waste products, and minimize the need for expensive infrastructure.

3. Conventional FAAE refining strategies

After transesterification, conventional crude FAAE refining strategies include the separation of the FAAE phase from glycerol based on differing densities (e.g., gravitational settling, sedimentation, centrifugation, decantation) [15, 29], followed by wet and/or dry washing (**Figure 1**). Wet washing removes polar contaminants (e.g., glycerol, soap, alcohol, and catalyst residue), from crude FAAE using a combination of heat and softened water utilizing the affinity of polar contaminants with water [20, 55]. The washing step is typically repeated until the biodiesel layer becomes clear and water layer becomes transparent [1]. Since this washing process must be repeated

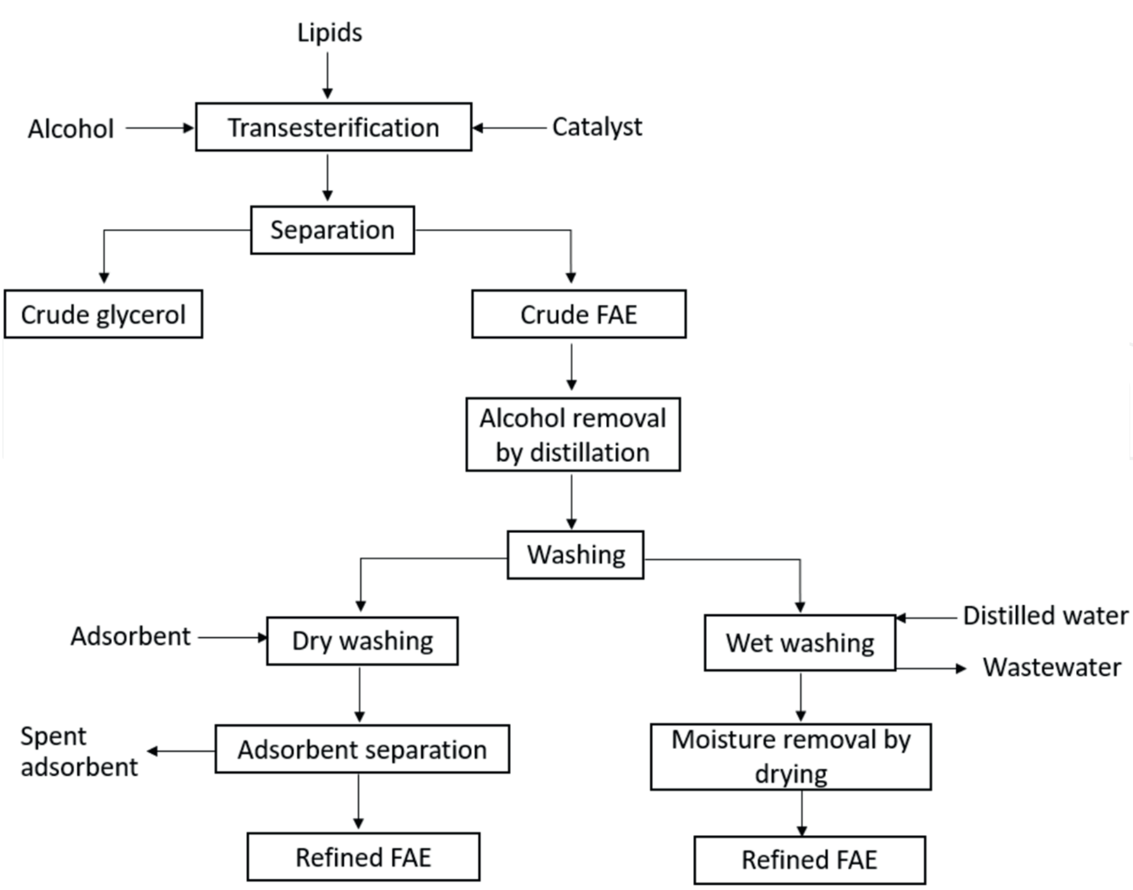

Figure 1.
Typical fatty acid alkyl esters production and purification process.

Refining method	Advantages	Disadvantages
Wet washing	> 99% FAAE purity is achievable	Requires large amounts of water
	Efficient removal of glycerol and methanol	Generates wastewater
	Removes FAAE-soluble impurities	Drying process can be energy intensive
Dry washing	Easier operation and less effort than wet washing	Lower removal rate of contaminants
	Less operation time	Re-generation of adsorbents can be challenging
	No introducing moisture into FAAE	Requires expensive machinery (e.g., pump, column)

Table 5.
Advantages and disadvantages of wet and dry washings in FAAE refining [59].

multiple times, these methods typically require large amounts of water and generate substantial waste in the form of contaminated water [56]. Typically, refining 1 L of crude FAAE can generate between 3 and 10 L of wastewater [29] and an additional drying step must then be included to remove residual moisture and prevent triglyceride hydrolysis [15]. Drying biodiesel can be costly and can result in the formation of emulsions [57].

In comparison, "dry" washing involves the removal of contaminants from crude FAAE through use of adsorbents or ion exchange resins. Suitable adsorbents include silicates (Magnesol™ or TriSyl™), ion exchange resins (Amberlite™ or Purolite™), cellulosic materials, activated clay, activated carbon, activated fiber, and others [29]. "Dry" washing is based on the affinity of adsorbents to common polar contaminants [58]. Although this method does not rely on contacting biodiesel with water, dry washing consumes a considerable amount of adsorbent material. However, this consumption can be mitigated if the adsorbent can be reused [59].

Both wet and dry washing techniques are commonly used for FAAE refining, and the advantages and disadvantages of both methods are summarized in **Table 5**. These conventional washing techniques increase production cost and further contribute to FAAE loss. The use of wet and dry washing to refine the FAAE phase generates substantial waste including wastewater and spent adsorbent. Waste is an important consideration for commercial manufacturers. As a result, there is significant interest in the development of refining technologies that are both efficient and environmentally sustainable as further elaborated below.

4. Novel fatty acid alkyl esters refining methods

4.1 Membrane filtration

Conventional FAAE refining processes have significant drawbacks including high energy usage, oil losses, use of harmful chemicals, and the production of substantial waste or effluent products [26]. Fortunately, membrane technologies offer advantages including customizable features in the process design [60] that is both efficient and environmentally sustainable for the purification and separation of many biofuels, including biodiesel [61]. Some advantages include reducing thermal damage to the end-product, improved solvent recyclability, decreased emissions and energy consumption, minimized oil losses, and reduced bleaching requirements [26]. Membrane processes have been developed for refining biodiesel and such refining can generate high quality FAAE with increased yields when compared with conventional processes [62, 63]. Specifically, membranes with suitable properties (tolerance of mechanical, chemical, and thermal stress, high surface area, enhanced selectivity can remove particles from biodiesel [62]. Micro-filtration (MFM; 0.1–10 mm pore size) and ultra-filtration membranes (UFM; 1–100 nm pore size) have been tested for the removal of particles from crude FAAE [53]. These membranes can be classified as either organic membrane (e.g., cellulose membranes) and inorganic membrane (e.g., ceramic membranes), with the latter having the advantage of increased durability, thermostability, and stability in acid, alkali, and high-pressure environments [64]. Common types of membranes used in FAAE purification are summarized in **Table 6**; the two distinct properties that influence the efficacy of the membranes are pore size and perm-selectivity [61].

Membrane filtration has been employed as a refining step for processing FAAE after transesterification [67]. Membrane based refining can reduce or remove common contaminants [67, 68], as well as residual catalysts [69]. For example, filtration through ceramic [70, 71] membranes reduce the glycerol content of FAAE [68]. Once isolated the glycerol byproduct might then be further purified and converted to value-added chemicals (e.g., solketal and glycerol carbonate) [72]. However, the use

Membranes	Pore size (μm)	Temperature (°C)	Permeate Flux ($L\ m^{-2}\ h^{-1}$)	Pressure	Results	Ref.
Ceramic membrane	0.1	60	300	1.5 bar	Low metal and free glycerol content	[65]
Ceramic membrane	0.05	40	22.17	2 bar	97.5% and 96.6% removal rate of free glycerol and soap content	[62]
Mixed cellulose ester membrane	0.22	Ambient temperature	~130	2 bar	Effect of soap and free glycerin removal	[66]
Poly (ether-sulfone) ultrafiltration membrane	10 kDa	Ambient temperature	~80	4 bar	Effect of soap and free glycerin removal	[66]

Table 6.
Application of membranes in fatty acid alkyl esters refining.

of absorbents is typically required to improve the efficiency of ceramic membranes [71]. In addition, filtration processes have been evaluated for the separation of crude biodiesel from unreacted vegetable oil [73], catalyst [65], soap [65], and methanol [1]. Ceramic or carbon microporous membranes are usually preferred due to resistance to corrosion and degradation in the presence of a base or acid catalyst during transesterification [67, 73, 74]. Filtration through ceramic membranes has been shown to effectively refine biodiesel to produce a product that meets standard specifications [65, 70, 71, 74, 75]. As glycerol and FAAE are immiscible due to their polarity differences, the lipid/oil droplets can be filtered through ceramic membranes owing to their large droplet size compared to the biodiesel [61]. These membranes have also been successful removing other common contaminants such as soap, to reach the quality defined in standard specifications [76]. However, it is often necessary to clean the membranes after each purification process either physically, chemically (hydrogen peroxide, chlorine, etc.), or hydraulically [77], to extend the reusability, efficiency, and repeatability of the membrane [76], otherwise, fouling and clogging can occur [74]. Furthermore, the membrane performance can be affected by physical operating parameters, including temperature requirements. Depending on membrane tolerance of operating conditions scale-up strategy and process design can be limited [76]. Computational modeling has also shown promising results in utilizing reverse osmosis filtration to remove impurities present in crude biodiesel [78]. Finally, the use of coagulation and flocculation processes [79], and membrane filtration methods, including nanofiltration, have been explored to treat biodiesel wastewater [80, 81], accumulated during wet washing processes [80].

Implementation of membrane filtration for biodiesel refining provides an environmentally valuable alternative to conventional biodiesel refining strategies [82]. Membrane filtration has demonstrated success in refining biodiesel from different feedstocks and produced products that meet the ASTM D6751 standard [68]. The combination of electrostatic strategies (electrostatic field, electrodialysis, etc.), with membrane filtration technologies, have also been explored and demonstrated high efficiency and success in refining biodiesel [83], and is further elaborated below.

4.2 Application of electrostatic field and nano-adsorbents in FAAE refining

The utilizing of static electric fields as a method for removing contaminants from FAAE has recently been reported [84, 85]. This method uses the behavior of particles in static electric fields [86] to remove them from FAAE. The process separates particles using electrochemical properties that separate them from bulk solution by electrophoresis (EP) and dielectrophoresis (DEP) [87]. Using EP, particles respond to a uniform direct current voltage that attracts particles, while DEP manipulates particles in a non-uniform electric field which can be tailored depending on the medium and contaminant properties [88]. Based on a range of dielectric properties of targeted molecules/particles/contaminants of interest DEP is commonly used in medical applications, diagnostics, environmental research, polymer research, and particle filtration [89]. These principles can be similarly applied for particle removal enabling partial FAAE refining.

Currently, processes involving electric fields have been applied in oil processing as a means for demulsification and dehydration [90] and removing contaminants [91]. These techniques can be employed to effectively remove polar contaminants (e.g., soap, and glycerides) [92–95] from non-polar fluids with dielectric constants below 2.8 [99] (**Table 7**). The application of electrostatic fields can remove free glycerol from crude FAAE [100–102].

Source	Dielectric constant (k)	Temperature (°C) and frequency (MHz)	Reference
Soybean oil based	3–3.24	25–70, 1 kHz- 100 kHz	[96]
Soybean, sunflower, corn, and rapeseed based	3–3.4	25–75, 20–20 MHz	[97]
Soybean oil based	2.08–2.26	25 100–2000 Hz	[98]

Table 7.
Dielectric constant of vegetable oil based FAAE.

More recently, electrostatic field treatments have been successfully employed in removing polar contaminants present in crude canola oil [27]. Reductions of 74.9%, 53.2%, and 47.0% in phospholipid, free fatty acid, and peroxide contents, respectively, were achieved using a commercial electrostatic "oil cleaner" with a fixed flow rate and equipped with pleated cellulose collector (designed to collect oil contaminants)[27]. Neutral oil loss was minimal (0.37 wt %), and pigment content (e.g., chlorophyll and carotenoid content) remained unaffected [27]. However, it should be noted that the limitations of this process included a requirement to reduce FAME moisture content prior to treatment.

To remove more polar compounds Zhou et al., [27] coupled the use of nano adsorbents (e.g., Al_2O_3, SiO_2, and TiO_2) with electrostatic separation. This implementation of nano-adsorbents coupled with electrostatic field treatment effected a significant reduction in contaminants (e.g., soap, total glycerides) from crude FAME. These reductions were observed after only 1 minute of adsorption and at low agitation speeds [28]. Although removal of spent nano-adsorbents can be challenging, electrostatic field application was successful in removing these particulates from the crude FAME solution without altering FAME chemistry [28]. Unfortunately, the application of some of these nano-adsorbents were limited by their reusability. Only Al_2O_3 was repeatedly regenerated after multiple cycles [28].

E-field and nano-adsorbent treatments offer novel approaches that can be rapidly implemented to process and refine vegetable oil and FAAE for biodiesel production [27, 28]. The ability to reuse spent nano-adsorbents, and potentially recycle solvents [26], make these approaches an attractive alternative to conventional refining. Furthermore, implementation of E-field technologies can offer a greener approach to biodiesel refining as well as economic and environmental advantages including reducing the need for expensive infrastructure, equipment, or harmful chemicals and reducing waste. Further studies should focus on the standardization and optimization of these processes at an industrial scale.

5. Conclusion

The transition from fossil fuels to a new fuel source is becoming increasingly important, and the environmental impact from the production of a new generation of fuel must be carefully considered as the market grows. The use of biofuels is unique in that it uses what can be classified as carbon neutral sources and implements feedstock which can come from primary or secondary sources. The use of these biofuels, however, requires refining which can be costly, environmentally harmful, and energy intensive. Conventional techniques for the removal of contaminants, such as dry and wet washing, produce large amounts of waste. In addition, enzymatic processes, physical refining strategies (temperature and pressure treatments), and membrane filtration can be limited in their capacity due to expensive reagents and infrastructure requirements, or solvent compatibilities. Fortunately, new refinement methods, such electrostatic field treatment and nanoparticle adsorbents have been developed to improve efficiency, minimize chemical usage, and reduce the generation of waste byproducts. These methods have successfully demonstrated the ability to remove contaminants including soap, methanol, and glycerides from vegetable oils and FAAE. Compared to conventional methods, these new techniques offer an economical and environmentally sustainable approach to current conventional methods. These techniques could be considered at an industrial scale, although further optimization is needed. As the global economy's dependence on fossil fuels continues to decrease, biofuels will play an important role in the development of environmentally friendlier materials to ensure that supply chain needs are met.

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Conflict of interest

Dr. Martin J.T. Reaney is the founder of, and has an equity interest in, Prairie Tide Diversified Inc. (PTD, Saskatoon, SK, Canada: previous company name is Prairie Tide Chemicals Inc.).

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