

Review

A Comprehensive Review on Oil Extraction and Biodiesel Production Technologies

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Abstract: Dependence on fossil fuels for meeting the growing energy demand is damaging the world's environment. There is a dire need to look for alternative fuels that are less potent to greenhouse gas emissions. Biofuels offer several advantages with less harmful effects on the environment. Biodiesel is synthesized from the organic wastes produced extensively like edible, non-edible, microbial, and waste oils. This study reviews the feasibility of the state-of-the-art feedstocks for sustainable biodiesel synthesis such as availability, and capacity to cover a significant proportion of fossil fuels. Biodiesel synthesized from oil crops, vegetable oils, and animal fats are the potential renewable carbon-neutral substitute to petroleum fuels. This study concludes that waste oils with higher oil content including waste cooking oil, waste palm oil, and algal oil are the most favorable feedstocks. The comparison of biodiesel production and parametric analysis is done critically, which is necessary to come up with the most appropriate feedstock for biodiesel synthesis. Since the critical comparison of feedstocks along with oil extraction and biodiesel production technologies has never been done before, this will help to direct future researchers to use more sustainable feedstocks for biodiesel synthesis. This study concluded that the use of third-generation feedstocks (wastes) is the most appropriate way for sustainable biodiesel production. The use of innovative costless oil extraction technologies including supercritical and microwave-assisted transesterification method is recommended for oil extraction.

Keywords: renewable energy; potential feedstocks; oil extraction technologies; biodiesel synthesis; feasibility study



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1. Introduction

Biodiesel is a biodegradable, energy competitive, and renewable resource with the ability to meet the energy need of the world [1,2]. It is the feasible solution replacing fossil fuels causing ozone depletion and environmental degradation. It is a renewable fuel that emits fewer emissions on burning and it can be implemented to existing petroleum diesel engines without retrofitting. It is also referred to as Eco-Diesel (particularly when derived from Pig Pancreatic Lipase) [3]. Biodiesel is usually known by its primary feedstocks including vegetable oils and animal fats [4]. Since the carbon present in oils is generally originated from carbon dioxide present in the air (uptake by a plant in photosynthesis), biodiesel's contribution to global warming is much lesser than fossil fuels. Use of biodiesel as a potential energy source offers the advantage of sustainability since the CO₂ released on combustion will be used again by nature for feedstock preparation, an effect called 'Closed

Carbon Cycle' or 'Carbon Neutral Cycle'. Figure 1 shows the carbon dioxide closed cycle for biofuels. This represents the process that when renewable plant source is converted into biodiesel, emission with zero carbon dioxide takes place.

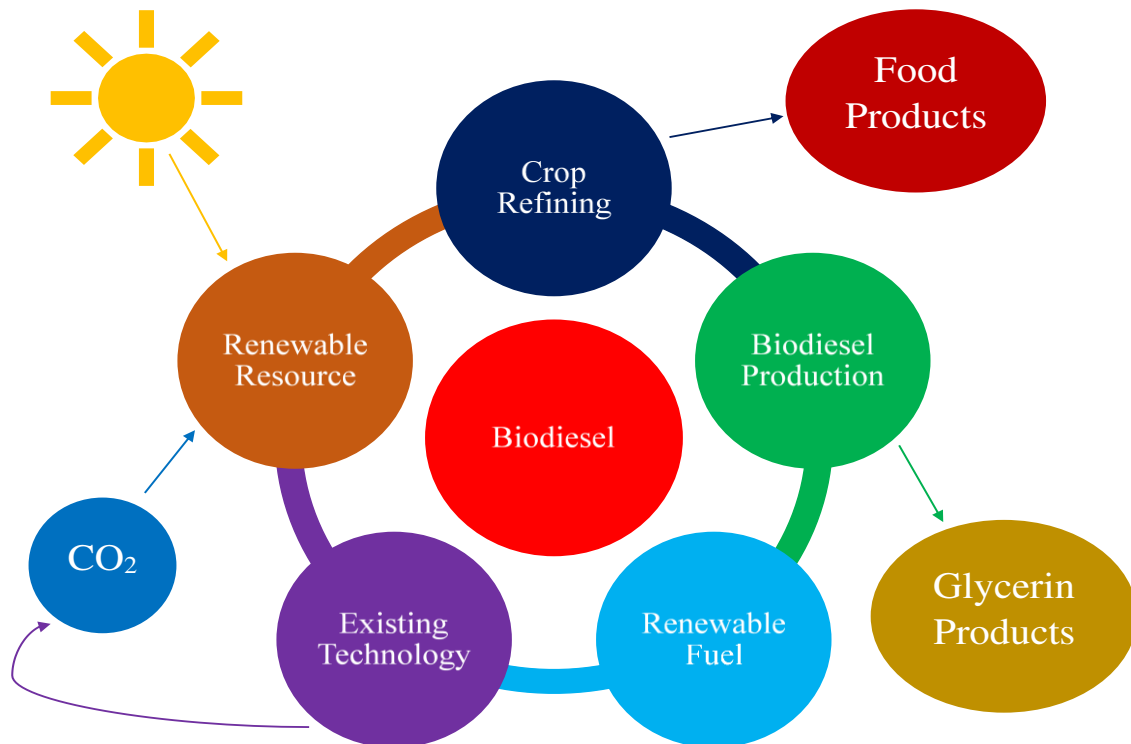


Figure 1. Carbon dioxide closed loop cycle for biofuels [3].

Like petro-diesel fuel, it can be easily stored anywhere as it has good storage properties. The chemical hazards and risks associated with handling, transportation, and storage of the biodiesel are less than the conventional diesel fuel. Biodiesel can be handled safely because of its biodegradability and greater flash point than petro-diesel [5]. Biodiesel is produced through transesterification using diverse feedstocks resources like edible, non-edible, and waste oils. The synthesis of biodiesel using waste cooking oil and animal fats offers a potential market to use waste oils [6]. The selection of feedstock has a notable effect on biodiesel synthesis as 70% of the product cost depends on the feedstock. This review highlights the advantages and disadvantages of commonly used feedstocks for sustainable biodiesel production. However, this study highlights the best available technologies for oil extraction so that the use of non-edibles can be made possible for oil extraction to implement for sustainable biodiesel production. The comparison of biodiesel production and parametric analysis is done critically, which is necessary to come up with the most appropriate feedstock for biodiesel synthesis. Since the comparison of feedstocks based on availability and cost along with oil extraction and biodiesel production technologies has never been done before, this will help to direct future researchers to use cheaper and sustainable feedstocks for biodiesel synthesis.

2. Feedstocks for Biodiesel Production

At present, more than 350 oil-producing crops have been considered globally as a potential source of triglycerides to produce biodiesel. The selection of suitable feedstock is the critical factor to produce biodiesel [7]. Preferably, suitable feedstock must fulfil two demands, which are industrial-scale production with the minimum associated cost.

Type and Availability

The cheaper and easier availability of raw material for biodiesel synthesis is influenced by the weather conditions of the region, local conditions of soil, topographical location, and farming procedures adopted by the country. The feedstocks for biodiesel synthesis are majorly classified into four main categories [8–10]. The four classifications of feedstocks for biodiesel synthesis are shown in Figure 2. The parameters like oil content and overall cultivation yield of agricultural land are the key parameters to consider any new feedstock of biodiesel synthesis. Figure 2 indicates the oil content present in various feedstocks.

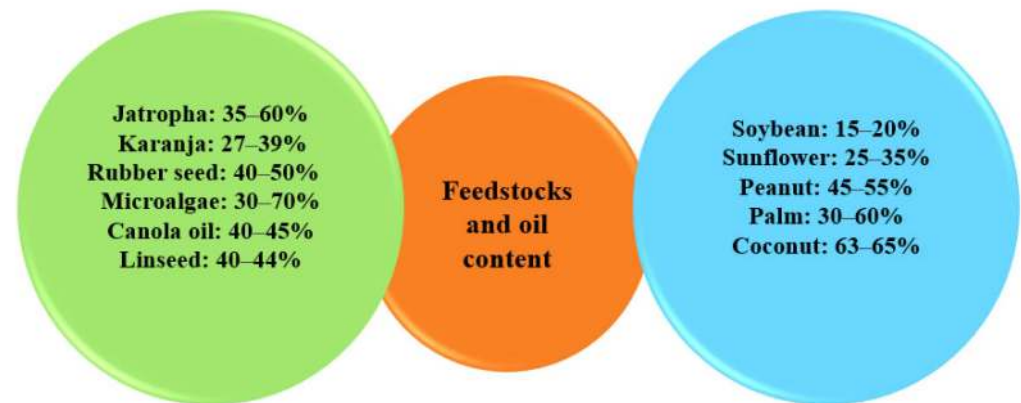


Figure 2. The oil content of feedstocks for biodiesel synthesis [11,12].

The first-generation feedstocks are considered as readily available sources to produce biodiesel. The edible oils include palm [13,14], coconut [15], olive, mustard, soybean [16], rapeseed [17], and sunflower oil [18]. The application of edible oils over the years has affected the supply, since this practice affects the food market globally, destabilizing the availability and prices. Therefore, edible oils are too costly to be used for fuel production since they have a high demand for food [19]. Hence, the world may face the ‘food vs. fuel’ soon if the use of first-generation feedstocks for biodiesel synthesis continues. Their applicability to synthesize biodiesel can cause an increase in the cost of both biodiesel and oils [14]. The process has negative effects on the environment due to its demands for a larger area of land for feedstock cultivation. This causes deforestation especially in tropical regions including Indonesia and Malaysia, supplying greater than 75% of palm oil supply to the world. Continuation of this practice for biodiesel synthesis would damage flora and fauna, with influences culminating in weather changes. The investigation of alternative feedstocks for biodiesel synthesis was aimed to decrease the dependence on first-generation feedstocks [20]. The application of non-edible oils to synthesize biodiesel is considered beneficial in comparison to edible oils to avoid the food crisis and make the biodiesel as a cost-effective process. The oil extraction and processing for biodiesel synthesis is a complex process that increases the overall production cost [21]. The second-generation feedstocks discussed above are available in large amounts in nature because of the lack of competition with food [22]. The growth rate and yield of seeds of non-edible oil plant are much lesser. The non-edible oils-producing plants have an excellent vegetative growing capacity, but the quantity of seeds obtained from a single plant is very minimal [23]. The classification of various feedstocks based on the composition of fatty acids is given in Table 1.

Table 1. Fatty acid composition of different feedstocks for biodiesel production.

Classification	Type of Feedstock	$C_{16}H_{32}O_2$	$C_{18}H_{36}O_2$	$C_{18}H_{34}O_2$	$C_{18}H_{32}O_2$	$C_{18}H_{30}O_2$
		16:0 Saturated	18:0 Saturated	18:1 Mono Saturated	18:2 Di Unsaturated	18:3 Poly-Unsaturated
First-generation feedstocks	Soybean	10.4–24.8	2.6–4.7	16.5–24.8	51.8–53.0	6.5–7.0
	Palm	37.80–43.79	2.7–4.76	39.90–42.6	9.59–12.20	0.17–0.53
	Olive	9.7	1.74	82.3	-	-
	Rapeseed	3.49–4.0	0.55–2.3	62–77.8	1.8–8.23	1.8–8.23
	sunflower	10.58	4.76	22.52	8.19	8.19
Second-generation feedstocks	Tallow	29.0	24.5	44.5	-	-
	Jatropha. C oil	14.2	7.0	44.7	32.8	-
	P. pinnata	10.2	7.0	51.8	17.7	0.2
	M. indica	24.5	22.7	37.0	14.3	3.6
	Neem oil	13.8	18.2	52.6	13.6	-
	Rubber seed oil	9.1	5.6	24.0	46.2	14.2
	Linseed oil	5.61	4.04	19.34	17.15	48.79
	Castor oil	0.92	0.16	3.53	4.21	0.91
	Mustard oil	2.80	1.09	24.98	11.64	8.61
Third-generation feedstocks	Crude castor oil	1.06	1.15	3.71	5.41	0.58
	WCO	4.1–26.5	1.4–10.9	38.6–44.7	32.8–36.0	0.2
	Chicken fat	19.82	-	37.62	-	1.45
	Yellow grease	23.24	-	44.32	2.43	0.80
	Waste frying oil	6.90	2.35	61.58	20.01	4.74
	Waste animal fat	22.31	17.02	43.26	9.76	1.71
	Crude neem oil	18.1	18.1	44.5	18.3	0.2

Extraction of oils from non-edible oils sources is a complex process that leads to the increase of cost of non-edible oil biodiesel [24]. Moreover, oils yield from seeds is low and the process of oil extraction is complex, making them an inappropriate choice. These drawbacks are considered as a hurdle to use them for industrial biodiesel synthesis [25,26]. The comparison of the cost of plantation and oil yield for various edible and non-edible oils is presented in Table 2. Among edible oils, palm oil feedstock has the highest oil yield with a slightly higher cost. Similarly, the cost of cultivation is also higher among others giving a higher oil yield.

Table 2. Comparison of cost of plantation and oil yield of edible and non-edible oils.

Feedstock	Oil Yield (kg Oil/ha)	Cultivation Cost (USD/ton)
Edible oil		
Palm	5000	950
Soybean	375	615
Rapeseed	1000	336
Non-edible oil		
Castor	1188	160
Rubber seed	120	N/A
Jatropha	1590	620

There are several other reasons to promote these oils for biodiesel production due to their sustainability: (1) These oils are feasible and sustained by the positive public opinion of biodiesel production and (2) they avoid the fuel vs. food controversy [27].

3. Oil Extraction Processes

The extraction of oil is the most important step in biodiesel synthesis. In oil extraction, physical, chemical, or enzymatic treatments are applied to the plant to recover the oil. The major products include crude oil and cakes of the already-used plant. The cake produced in this step is not used in further processing. The state-of-the-art existing oil extraction technologies are discussed in detail on the basis of applicability, cost, efficiency, and hazards on the environment.

3.1. Mechanical Extraction

In mechanical extraction of oil, manual ram pressing, or engine driven screw pressing is used. Engine-driven screw has an efficiency of 68–80% of the oil content, while manual pressing can give the efficiency of 60–65%. After mechanical pressing, filtration and degumming are used to recover the oil. The challenge of using mechanical pressing is the only application for specific seeds [28]. However, pre-treatment procedures enhanced the recovery of oil for screw presses, giving 89% efficiency in a single pass and 91% for a double pass [29].

3.2. Steam Distillation

Steam distillation is a removal process that is applicable for the extraction of oil from temperature-sensitive plants containing aromatic compounds [30]. Without applying the maceration, plant material containing oil is introduced in the setup, exposed to steam. The steam passes through the charged feed, as presented in Figure 3.

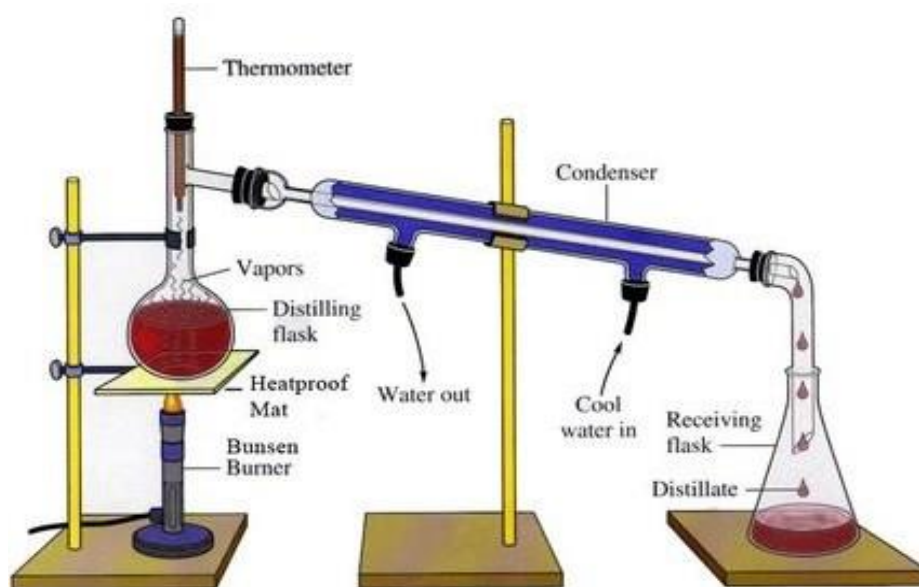


Figure 3. Apparatus for performing steam distillation.

The steam distillation system produces a product consisting of vapor and crude oil, which can be taken off after removal of vapors [31]. This method can also be followed under pressure variance based on the nature of plant material and the problem of recovering oil at the room temperature [31]. The process has the advantages of low thermal deterioration since the operating temperature does not exceed 100 °C. However, this process has some limitations i.e., raw material required for the extraction must be evenly distributed for greater recovery of oil. A different predicament related to this process is that cold feed is

charged. Until the temperature of the whole feed charged approaches the steam temperature being introduced, the wetting process continues. This process requires higher capital investment and when low market value products are produced, and the investment costs may require several years for a full recovery.

3.3. Solvent Extraction

3.3.1. Soxhlet Extraction

A Soxhlet extractor is a laboratory-scale unit [32] developed in 1879 by Franz von Soxhlet [33] to extract a component from a mixture, as shown in Figure 4. The process requires a small amount of solvent to extract a higher amount of the targeted compound. It promotes solid and liquid to recover the desired compounds from the solid matrix using a suspension of a matrix into the refluxing solvent.

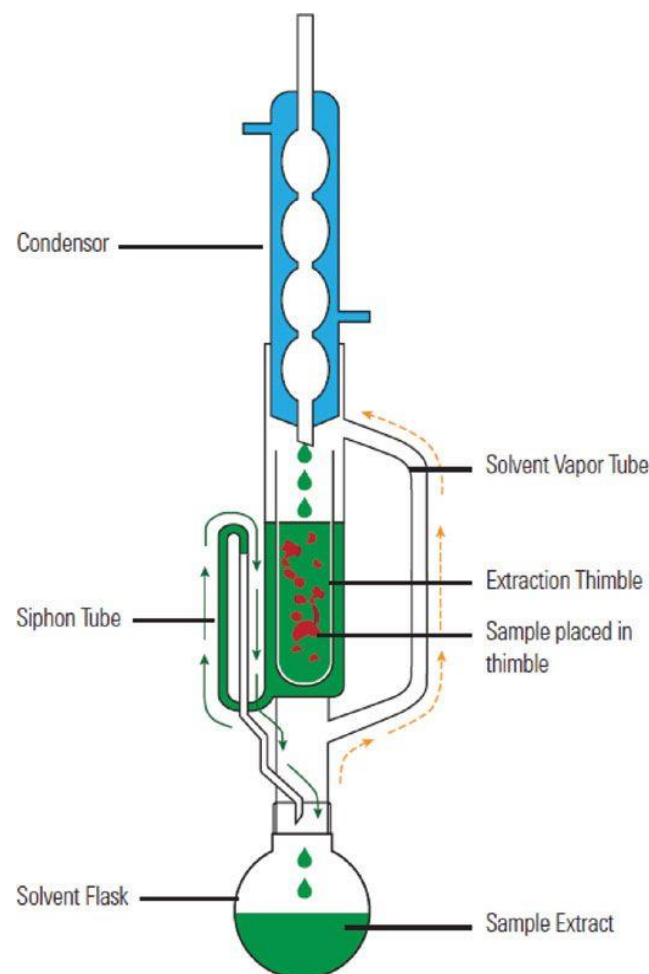


Figure 4. Soxhlet apparatus for oil recovery.

The solid matrix is kept in a space that continuously receives the solvent by the condensation of its vapors moving through the distillation arm. After achieving a certain value, the liquid is taken from the cavity by a siphon moving back into the distillation chamber. Al-Hamamre et al. [34] used a Soxhlet apparatus to extract the coffee oil from spent coffee leaves, taken from a pressurized bean-to-cup coffee machine. The extraction of oil was done using n-hexane to check the efficiency of the solvents giving maximum oil extraction yield. Hexane gave the highest yield of 15.28% in 30 min. Soxhlet extraction has various benefits such as the same solvent can be used after recycling until it is completely saturated with the analyte. Even if a certain sample contains a quantity of solvent that is not recycled, it can be utilized to extract the oil using fresh samples of the solvent. The

extraction process has some limitations such as the increase in time required, such as several hours or days. The enhanced temperature of the system for a long time causes the thermal degradation of the material of construction of distillation flask. The procedure is not in favor of the environment due to possible pollution problems occurring due to the use of flammable and toxic solvents. In addition, the process can be used for the recovery of tiny and finely divided solid samples [35].

3.3.2. Chemical Leaching

This is the process of recovery of a component from a solid feedstock using a solvent. Several factors affect the chemical leaching process such as the size of a particle, type of the liquid used, temperature, and mixing speed of the system. Small particle size is used to allow the interfacial area between the feedstock and the solvent. The viscosity of the solvent must be lower to promote free circulation. Temperature is also the significant parameter influencing the rate of extraction, the solubility of the oil rises with temperature increase. Rate of mixing is also one of the factors affecting the process, since it promotes the rate of diffusion, consequently enhancing the material transfer from the surface of the particle.

3.3.3. Enzymatic Oil Extraction

Enzymatic oil extraction is a feasible process used for oil recovery. The method utilizes suitable enzymes to extract the oil from seeds. Although the process is proving its potential, the application of this technology is still facing limitations such as higher cost of enzymes, higher incubation time, and the need for de-emulsification during downstream processing [28,36]. The implementation of techniques such as affinity chromatography and perfusion chromatography makes the downstream processing easier, while the immobilization of enzymes minimizes the enzyme losses and overall cost [37]. However, enzymes immobilization causes the reduction of reaction rates due to steric hindrance. Moreover, the application of solvents such as n-hexane increases the production of wastewater and release of volatile organic matter, besides n-hexane flammability and toxicity [38]. This requires the use of a substitute extraction process such as an aqueous enzymatic oil system along with ultrasonication pre-treatment [28,29].

3.3.4. Supercritical Fluid Extraction

The application of supercritical fluids is extensive to extract metal cations and oils. The use of carbon dioxide is most common for supercritical extraction of essential oils due to its low critical temperature and pressure conditions. Moreover, CO₂ is non-toxic and non-flammable and is available at low cost in highly pure form. It can be completely removed from the extract in a much easier way [39]. The non-polar nature of CO₂ limits its applicability for the extraction of non-polar analytes [40]. Supercritical extraction uses CO₂ as the supercritical solvent but the application of co-solvents like methanol or ethanol is recommended to add according to the nature of extracted analytes. Fadel et al. [41] found that extracts containing analytes using supercritical fluids exhibited higher antioxidation activity giving the higher yield and lower viscosity in comparison with the extracts recovered implementing other technologies. This process allows the flexible extraction of the targeted compound due to the characteristics of the supercritical fluid can be changed by varying the operating conditions. Polyphenols can be separated using pure supercritical CO₂ at elevated pressures after that unsaturated fatty acids can be recovered by adding the co-solvent (ethanol) [42]. The process is faster since the extraction process takes place on the phenomena of diffusion, which concludes that high diffusivities at supercritical conditions lead to favoring the rate of extraction. However, the need for high pressures limits the application of supercritical fluid extraction in comparison with conventional technologies.

3.3.5. Microwave-Assisted Extraction

The extraction of oil using microwave-assisted method is an innovative hydro-distillation process that uses microwave heating. It has been effectively used for the extraction of constituents from various plants and presented in previous studies. Microwave heating is done with direct contact of plant materials and is fluctuated by two phenomena: Conduction through ion movement (known as 'ionic conduction') and dipole rotation, both occurring simultaneously [43]. Therefore, the microwave heating is mainly produced due to electric field applied showing a huge dependency on the electric field. The thermal energy is produced from electrical energy in the microwave heating and its rate of conversion is presented as in Equation (1).

$$P = Kf\epsilon E^2 (\tan \delta) \quad (1)$$

where $\tan \delta$ is the dielectric loss tangent, E is the electric field strength, ϵ indicates the dielectric constant of substance, f is the applied frequency, K is a constant, and P denotes the microwave power dispersion per unit volume of the liquid. Conventional heating requires more time. The advantages of microwave-assisted extraction include a high reaction rate and extraction yield with the requirement of the solvent and controlled heating process [44]. This nullifies the CO_2 release [45] and the energy consumption is the only fraction of energy required in conventional heating [46]. However, the extraction process using microwave heating is influenced by whether the solvent or desired compounds are non-polar or volatile in nature.

3.3.6. Ultrasound-Assisted Extraction

This is one of the promising techniques used for oil extraction. The oil feedstock collected from cultivated trees is dissolved in a polar compound (such as water) or non-polar compound (alcohol) and then subjected to the ultrasonic vibrations [47]. The frequency range of ultrasound waves produced from an ultrasound source consisting of sound waves within the frequency range of 18 kHz to 100 MHz. When ultrasonic irradiation is introduced in the bulk of liquid, it causes the development of an acoustic pressure (P_a), and this developed pressure sums up in the hydrostatic pressure (P_h) of the liquid. The produced frequency of ultrasound irradiation is highly influenced by acoustic and hydrostatic pressure making it necessary to express their relationship. Acoustic depends upon the exposure time to the ultrasonic waves that follow Equation (2).

$$P_a = P_A \sin 2\pi ft \quad (2)$$

where P_A indicates the maximum amplitude of pressure of the sound wave, f is to the frequency for time t . There are some points along the wave path where the vertical lines are in proximity to each other, showing higher pressure than normal. On the other hand, some points have been found to exist and the lines seem to be pulled apart from each other, and there the pressure is lowered than the normal. The former points are known as a 'compression region' with the latter points being termed as the 'rarefaction region'. The intensity of ultrasound waves produced is influenced by the density of medium and speed of sound. The wave penetration through the bulk of the liquid is the energy dispersion per unit time per unit area that is linked with P_A as in Equation (3).

$$I = P_A^2 (\rho c)^{-1} \quad (3)$$

where ρ is the density of the medium while c indicates the speed of sound passing through the medium. For ultrasonic waves, the decrease of intensity is in inverse relation through the penetration. The sound waves penetrating through the medium produce molecular vibrations, which decreases the intensity of waves and acoustic energy is transformed into thermal energy. The heating of the liquid occurs at the points where compression takes

place, while cooling occurs in the rarefaction region. Thermal energy is produced when the acoustic energy dissipates due to the absorption that is in accordance with Equation (4).

$$I = I_0 e^{-(2\alpha d)} \quad (4)$$

where intensity (I) is the function of the distance (d) from the source producing ultrasound waves and α indicates the absorption coefficient. Ultrasound generation in the liquid medium is the root source for the generation of pressure waves resulting in the vibration of molecules from their mean location. Due to significant negative pressure (P_c) on the medium, since P_c (pressure under rarefaction) = $P_a - P_h$, the inter-molecular gap competes for the critical distance (denoted by R), which promotes the generation of cavitation bubbles, as the intermolecular arrangement of liquid is disturbed. A deteriorating bubble requires less time as compared to the time taken by acoustic waves, consequently a steady state for P_m during the bubble collapse.

$$P_m = P_h + P_a \quad (5)$$

The time is calculated using Equation (6).

$$t = 0.915 \left(\frac{\rho}{P_m} \right)^{\frac{1}{2}} \quad (6)$$

where R_m indicates the radius of the cavity formed. The rate of extraction is strongly influenced by the density of medium and acoustic pressure. Equation (6) gives the idea to select the appropriate medium to enhance the extraction rate. This process requires low capital investment and has a faster rate of extraction along with higher efficiency. However, the use of ultrasound waves can deteriorate the structure of the compounds available in the oil [48]. The overall summary of oil extraction technologies is presented in Table 3.

Table 3. Overall summary of oil extraction technologies.

Technology	Advantage	Disadvantage
Mechanical extraction	<ul style="list-style-type: none"> It has enhanced oil extraction efficiency (68–80%) as compared to manual pressing The pre-heating in mechanical extraction can increase the efficiency up to 91%. 	<ul style="list-style-type: none"> This technique is applicable for the oil extraction from limited type of seeds.
Steam distillation	<ul style="list-style-type: none"> This method can be used for the oil extraction from the plants that are not temperature sensitive Low thermal degradation is observed. 	<ul style="list-style-type: none"> The process requires equal distribution of extraction It requires high capital investment and low value product is obtained. Continuous wetting is needed to supply the cold temperature feed into the system.
Solvent extraction	<ul style="list-style-type: none"> Small amount of solvent can extract a large quantity of oil Agitation during the solvent extraction promotes the removal of targeted compound This technique is cheaper and easier to handle 	<ul style="list-style-type: none"> The use of this technology is not in favor of sustainable environment due to the use of toxic and flammable solvents.
Enzymatic extraction	<ul style="list-style-type: none"> It is one of the sustainable processes used for oil extraction due to no negative effect on the environment. 	<ul style="list-style-type: none"> It requires high cost of enzyme and incubation time and requires de-emulsification during down-stream operations.

Table 3. Cont.

Technology	Advantage	Disadvantage
Supercritical fluids extraction	<ul style="list-style-type: none"> The higher extraction takes place at supercritical conditions due to the enhanced solubility with solvent. The use of CO₂ as a solvent makes it cheaper process due to easier availability and non-flammability of CO₂. 	<ul style="list-style-type: none"> High temperature and pressure required in supercritical technique increases the overall cost.
Microwave-assisted extraction	<ul style="list-style-type: none"> The use of microwave heating for oil extraction nullifies the release of CO₂. Only fraction of energy is required as compared to conventional heating. 	<ul style="list-style-type: none"> This technique is not applicable when the solvent or desired compound is non-polar or volatile in nature.
Ultrasound-assisted extraction	<ul style="list-style-type: none"> This technology requires low capital investment, higher extraction rate and has higher extraction efficiency. 	<ul style="list-style-type: none"> The use of ultrasonic waves deteriorate the internal structure of compounds present in the oil.

3.4. Oil Refining

Oil refining used to remove the undesired components like free fatty acids, phosphatides, and colorants. The presence of these components affects the further processing steps as well as storage properties of the oil. Physicochemical characteristics of oil and nature of feedstock also affect the efficiency of oil refining. Degumming is done in the first stage of purification to remove the phosphatides. The removal of phosphatides is important because it causes a muddy appearance in the oil and also favors the formation of H₂O [49]. Acid and water degumming are the two techniques used to eliminate the Phosphatides. In acid degumming, acidic constituents are dissolved vigorously with oil to remove the insoluble phosphatides. Water degumming is applicable to remove the dissolved phosphatides. In water degumming, water is added in the oil at temperature 60–90 °C and then oil and water phases are separated using centrifugation [50,51].

De-acidification is the next processing step of oil refining. It is the most important stage for food source oils; it does not involve the synthesis of rancid flavors of free fatty acids (FFA). The removal of such components is not necessary for first-generation feedstocks. However, the removal of these components is suggested due to their direct effect on biodiesel synthesis and storage characteristics of fuel. The phenomena of de-acidification involve the unit operations such as distillation, elimination of pigments and odors using the solvents such as propane, ethanol, and furfural, and neutralization with alkali. Colorants are removed using the bleaching process. The materials like activated carbon, bleaching earth, and silica gel are used in this step for bleaching purpose. Deodorization is performed to remove odorous compounds of aldehydes and ketones are eliminated. Dehydration is the final step in the refining process in which moisture content is removed using distillation at low pressure [49].

4. Biodiesel Synthesis Processes

4.1. Direct Blending

In direct blending, crude oil is blended with petro-diesel in a specified amount as a fuel, but limitations such as higher viscosity, acid value, FFA content, and gum formation occurs making their use as a direct engine fuel inappropriate [52].

4.2. Microemulsions

In the microemulsion process, the oil is mixed with appropriate emulsifying agents such as alcohol. The most commonly used alcohols are methanol, ethanol, propanol, or butanol to produce emulsions. The main disadvantage of using micro-emulsion include the accumulation of carbon in the engine and improper burning [52].

4.3. Catalytic Cracking

This is the breakdown of the non-edible oils or animal fats via heating in the absence of air or oxygen using a catalyst, producing products with combustion characteristics similar to petroleum-diesel. The pyrolyzed material contains higher amounts of sulfur, moisture, and sediments but insufferable clinker and carbon residues [53,54].

4.4. Transesterification

The transesterification process for biodiesel synthesis involves the reaction between oil feedstock with short-chain alcohol, most likely methanol, in the availability of catalytic medium. Methanol is the commonly used solvent for biodiesel synthesis. Some researchers have suggested the use of ethanol for biodiesel synthesis [55,56]. Due to minimized cost with easier availability, NaOH and KOH catalysts are enormously used. The reaction can be proceeded with the use of homogeneous or heterogeneous catalyst; the choice of the appropriate method depends upon the FFA content of the oil. The analysis of alkyl esters is usually done with gas and liquid chromatography to determine the yield [57]. The yield of biodiesel produced can be calculated using the formula shown in Equation (7). The comparison of various transesterification techniques is shown in Table 4.

$$\text{Process yield (\%)} = \frac{\text{Pure biodiesel (g)}}{\text{Oil used (g)}} \times 100 \quad (7)$$

Table 4. Comparison of transesterification processes [58,59].

Transesterification	Advantages	Disadvantages
Homogeneous Acid-catalyzed (HCl, H ₂ SO ₄)	There is no soap formation, and it can catalyze the esterification and transesterification simultaneously.	Acid presence causes corrosion, high temperature and slower reaction rate. The acid catalysts have low catalytic activity.
Basic-catalyzed (KOH, NaOH) Heterogeneous	Higher activity of catalysts and reaction rate, low cost, and easier availability.	Soap formation and difficult separation of product.
Acid-catalyzed	No formation of soap and catalyst recyclable. Esterification and transesterification can take place simultaneously.	High cost, low activity, and diffusional problems.
Basic-catalyzed (CaO, CaCO ₃ , Al ₂ O ₃)	Non-corrosive, recyclable catalysts for a long time and higher selectivity.	Higher cost, high energy requirement, sensitive to the presence of water, low diffusion, and hence lower yield of biodiesel.
Enzymatic-catalyzed	No side reactions taking place, easier separation, and environmentally feasible process.	Slow reaction rate, degradation and higher cost of enzymes used.
Supercritical	Very high reaction rate, no catalyst requirement, easier separation of products.	Higher operation costs due to reaction taking place at high temperature and pressure.

4.4.1. Edible Oil Use for Biodiesel Production

Although biodiesel production from first-generation feedstocks affects the availability and availability in the market, research has been done to produce biodiesel using edible oils. Vujicic et al. [60] used sunflower oil via basic catalyst (calcium oxide) at 100 °C, 1 wt.% catalyst, a molar ratio of methanol and oil of 6:1, and achieved a FAME yield of 91%. Zhu et al. [61] described the temperature effect on the transesterification of soybean oil, keeping all other parameters constant and suggested that at 70 °C the highest yield of FAME was obtained. Ines Reyero et al. [62] used sunflower oil in transesterification with sodium hydroxide (NaOH) as a catalyst at 50 °C, 24:1 of ethanol to oil, 0.3 wt.% catalyst, and attained a conversion of 99% of soybean oil to biodiesel. S Nasreen et al. [63] produced biodiesel by using soybean oil with calcined (600 °C for 1.5 hr.), lanthanum (La) doped manganese (Mn) with a ratio of 2:1 using following reaction parameters; molar ratio of oil

to MeOH. 1:12, at 180 °C for 1 h using 3 wt.% of catalyst dosage to oil and attained 99% triglyceride (TG) conversion. S.K. Mohan et al. [64] compared the yield of cottonseed oil and neem oil using optimized reaction parameters. For cottonseed oil, the reaction was carried out as follows; oil to methanol molar ratio 6:1 at 60 °C for 1.5 h with 2 wt.% catalyst (NaOH) and a continuous mixing at 600 rpm. Biodiesel yield of 80% was attained.

Xia Gui et al. [65] utilized cottonseed oil in the process while Dominic et al. [66] used refined cottonseed oil in transesterification and achieved a biodiesel yield of 96% under the optimized reaction parameters; alcohol to oil molar ratio of 6:1 at 55 °C was allowed to react for 1 h reaction time and 0.6% catalyst loading. Ting Qiu et al. [67] produced biodiesel from traditional coconut oil by utilizing an ionic liquid with Bronsted acidic sites as a catalyst and obtained a yield of 98.7%. Abdurrahman et al. [68] produced biodiesel from edible oils (Hazelnut, sunflower, and a mixture of these two oils) and compared their yields. Three different yields of 97.3, 97.5, and 97.9% were obtained from hazelnut, sunflower, and their mixture, respectively, at 60 °C for 120 min with a MeOH: oil 6:1 and 0.7 wt.% of catalyst. The summary of research done on biodiesel synthesis using edible oil is presented in Table 5.

Table 5. Use of edible oil for biodiesel production.

Feedstock	Catalyst	Temperature (°C)	Time (min)	Oil to Alcohol Ratio	Yield (%)	Reference
Sunflower oil	Cs/Al/Fe ₃ O ₄	58	120	14:1	94.8	[69]
Sunflower oil	MgO/MgAl ₂ O ₄	110	180	12:1	95.7	[70]
Sunflower oil	CsH ₂ PW ₁₂ O ₄₀ /FeSiO ₂	60	240	12:1	81	[71]
Soybean oil	Sr ₃ Al ₂ O ₆	-	61	25:1	96.2	[72]
Soybean oil	ZrO ₂ /C ₄ H ₄ O ₆ HK	60	120	16:1	98.03	[73]
Palm oil	ZnO	60	300	6:1	83.2	[74]
Palm oil	TiO ₂ -ZnO	60	300	6:1	92.2	[74]
Vegetable oil	Cs-Ca/SiO ₂ -TiO ₂	60	120	12:1	98	[75]
Olive oil	C. Antarctica	60	1920	88:1	93	[76]
Soybean oil	Rhizopus oryzae	45	180	15:1	99	[77]

4.4.2. Biodiesel Synthesis from Non-Edible Oil

The application of second-generation feedstocks has been done for biodiesel synthesis. Jain et al. [9] synthesized biodiesel using Jatropha oil with both acidic (H₂SO₄) and basic (NaOH) catalysts and achieved a yield of 21.2% and 90.2% after esterification and transesterification, respectively. Rathore et al. [78] synthesized biodiesel by varying the concentration of catalyst (KOH) from 2% to 12% in the reaction of Jatropha, Karanja oil and achieved a remarkable yield of 95% after 8 h of reaction time. Jar et al. [9] used microwave-assisted transesterification of jatropha oil to synthesize fatty acid methyl esters (FAME) by adding 1 wt.% of catalyst (KOH) at 65 °C for 10 s with a molar ratio of 6:1 of MeOH to oil at 200 rpm and achieved 90% of oil to biodiesel. Olutoye et al. [79] used the clay as a catalyst to produce biodiesel using methanol at 70 °C reaction temperature. Abbah et al. [80] stated a biodiesel yield of 94% by using neem seed oil in the process of transesterification at 55 °C for 1 h with molar ratio (7:1) and a catalyst dosage of 0.75 wt.% at a constant stirring speed of 350 rpm. Samsudeen et al. [81] utilized neem seed oil transesterification process to synthesize biodiesel using 15 wt.% of catalyst loading for 1.5 h with 5:1 ethanol to triglycerides molar ratio and obtained the biodiesel yield around 90.5%. Sumit H. et al. [82] produced FAME from the reaction of rubber seed oil using carbon-based

iron (Fe/C) as a catalyst under optimized conditions i.e., catalyst concentration 4.5 wt.% to oil at a reaction temperature of 60 °C with MeOH: oil 9:1 at continuous stirring of 1250 rpm and achieved a remarkable FAME yield of 97.5%. Wuttichai Roschat et al. [83] stated that the 97% yield of fatty acid methyl esters was attained using optimized reaction conditions; 9 wt.% catalyst loading (sodium metasilicate), oil to methanol molar ratio 9:1 at 65 °C for 40 min and a constant stirring of 200 rpm.

Awais Bokhari et al. [84] claimed yield of more than 96.5% by using rubber seed oil at 55 °C with 6:1 methanol/oil, 1 wt.% catalyst concentration for 18 min and a pressure of 3 bar. Rupesh L. Patel et al. [85] obtained a biodiesel yield of 97% by using Karanja oil with 1 wt.% of catalyst (KOH) at 65 °C for 120 min and oil to MeOH molar ratio of 6:1. Shalini et al. [86] produced 98.4% biodiesel from Karanja oil under the optimum reaction parameters; 1.2 wt.% to oil catalyst dosage, Oil: MeOH 1:19 at 65 °C for 1.4 h and a continuous agitation speed of 600 rpm. Veen Singh et al. [87] claimed 98.79% yield of biodiesel by using Karnal oil with a catalyst dosage of 1 wt.% (barium zirconate), 27:1 methanol/oil at 65 °C for 180 min. M. Hashemzadeh et al. [88] produced biodiesel from the transesterification of linseed oil by using a co-solvent (diethyl ether and methanol). The fatty acid methyl ester yield of 98.08% was achieved by keeping the molar ratio of diethyl ether to MeOH (1.19:1) and MeOH to oil (9.48:1) at 30 °C at a flow rate of 1.37 mL/min.

Baskar et al. [89] produced the biodiesel using castor oil in the presence of zinc oxide (ZnO) doped by nickel (Ni) as a catalyst and achieved 95.20% yield of FAME using optimal reaction parameters; MeOH: Oil 8:1 at 55 °C for 1 h with 11 wt.% of catalyst. In another work, they used doped ZnO with Fe to produce FAME from castor oil and attained 91% yield of FAME at 55 °C for 50 min with a MeOH to oil molar ratio of 12:1 using a catalyst dosage of 14 wt.% to oil [54]. S.T. Keera et al. [90] obtained a FAME yield of 95% from the reaction of castor oil by using 1 wt.% of catalyst (KOH) at 60 °C and 9:1 of MeOH to oil after a duration of half an hour. A Deep et al. [91] achieved 96.6% FAME yield from castor oil after 30 min of reaction at 30 °C with a MeOH to oil molar ratio of 9:1 using a catalyst (KOH) dosage of 1.5 wt.%. Sergei V et al. [8] described that a FAME yield of 97.45% was obtained from the transesterification of mustard oil by using aluminum oxide (Al₂O₃) doped with zinc oxide (ZnO) as a catalyst at 350 °C, 300 bar, ethanol to oil molar ratio at 12:1 and a catalyst loading of 5 wt.%. Abdelrahman et al. [92] utilized wild mustard seed oil to produce biodiesel and claimed higher than 97.88% of yield using optimized reaction parameters; a molar ratio of MeOH to feedstock 6:1, methanol/hexane molar ratio of 1:1 with 0.80 wt.% of catalyst (potassium hydroxide) at 50 °C for a time duration of 45 min. The summary of biodiesel production from non-edible feedstocks is presented in Table 6.

Table 6. Use of second-generation feedstocks for biodiesel synthesis.

Feedstock	Catalyst	Temperature (°C)	Time (min)	Oil to Alcohol Ratio	Yield (%)	Reference
Mahua oil	KOH	-	-	6:1	98	[93]
Mahua oil	KOH	60	30	4:1	-	[94]
Karanja oil	NaOH	50	70	-	84	[95]
Jatropha oil	KOH	55	60	9:1	90–95	[96]
Jatropha oil	H ₂ SO ₄	60	120	9:1	80	[96]
Canola oil	KOH	50	60	9:1	90–95	[96]
Rubberseed oil	NaOH	-	-	9:1	80	[97]
Honne oil	KOH	45–65	30–150	4:1	89	[98]

4.4.3. Biodiesel Synthesis from Waste/Crude Oils

Biodiesel synthesis from waste oil is advantageous due to the cheaper and easier availability of feedstocks. Sahar et al. [99] achieved 94% yield of biodiesel by the reaction

of waste cooking oil at 60 °C with a molar ratio of alcohol to oil 1:3 and a catalyst loading of 1 wt.% to oil. Alex tangy et al. [100] synthesized biodiesel using WCO using 41 wt.% of catalyst (Strontium oxide) and claimed 99.2% yield after 8.2 min of reaction under microwave-assisted radiation system. Ngoya Tshizanga et al. [101] used waste vegetable oil and achieved a yield of 91% with alcohol to oil molar ratio 22.5:1 at 65 °C for 330 min with 3.5 wt.% loading of catalyst. Saowakon Suwanno et al. [102] stated a FAME yield of 92.07% by using recovered oil from POME and crude lipase as a catalyst under optimum reaction conditions; MeOH: Oil 6:1 at 35 °C for 36 h with continuous stirring of 200 rpm. Leticia Karen dos Santos et al. [103] investigated the FAME yield using crude palm oil by two steps i.e., hydrolysis and esterification. The optimum parameters for the hydrolysis process were examined i.e., a reaction temperature of 250 °C for a duration of 120 min using alcohol to oil molar ratio of 100:1 and a continuous mixing at 700 rpm. They claimed 86% and 99.1% yield of biodiesel after hydrolysis and esterification, respectively. Celian Roman-Figueroa et al. [104] stated 96.5% yield of FAME after esterifying crude castor oil by using the supercritical method at 300 °C for 1.5 h at a pressure of 21 MPa.

Rabu et al. [105] obtained FAME from WCO at 60 °C for 120 min with alcohol to feedstock molar ration of 12:1 using 1 wt.% of catalyst (NaOH) dosage and achieved 95% yield. M. Pilar Dorado et al. [106] synthesized biodiesel from used olive oil using 1.26 wt.% of the catalyst with a ratio of 12:1 alcohol to oil at an ambient temperature for a time duration of 90 min and claimed 94% ester yield. Praful Nair et al. [107] utilized waste frying oil (WFO) to obtain fatty acid methyl esters using optimal reaction parameters and obtained more than 89% yield of biodiesel. Adewale et al. [108] produced biodiesel from waste animal fat using ultrasonic radiations. They stated that a yield of 96.8% was achieved using 4:1 of alcohol: Oil and a catalyst loading of 6 wt.%. Lubomir Sanek et al. [109] compared the yields of FAME synthesized from WFO and waste animal fat, which was more than 98% from each feedstock with a molar ratio of alcohol to feedstock 6:1 at 65 °C for 2 h reaction time and a catalyst (tetramethylammonium hydroxide) dosage of 1.5 wt.% to feedstock. Aransiola et al. [110] produced biodiesel meeting the ASTM standards from crude neem oil and explained the emission from a conventional diesel engine using this fuel. Indika Thushari et al. [111] produced biodiesel by the transesterification of waste palm oil (WPO) using a green catalyst and claimed 92.7% yield of fatty acid methyl esters. V. Kavitha et al. [60] utilized dairy waste effluent to produce biodiesel and obtained 96% yield using 2.4 wt.% of catalyst at 65 °C with methanol to feedstock ratio of 6:1 after 180 min of reaction time. From the literature study, it can be concluded that palm oil mill waste can prove to be the best feedstock in terms of waste palm oil biodiesel properties.

5. Critical Reaction Parameters Influencing the Biodiesel Synthesis

FAME is usually synthesized from the process of transesterification. During the transesterification reaction, 1 mole of oil reacts with 3 moles of solvent (alcohol). The transesterification reaction is expressed as:

In the generalized transesterification reaction, oil reacts with an alcohol producing in the methyl ester and glycerol as shown in Figure 5. The formation of glycerol does not take place when methyl tertiary butyl ether is used a solvent. The yield of transesterification reaction is strongly influenced by temperature, oil to alcohol yield, agitation speed, water and FFA content, and reaction time. This section of the review describes the effect of these parameters on biodiesel synthesis.

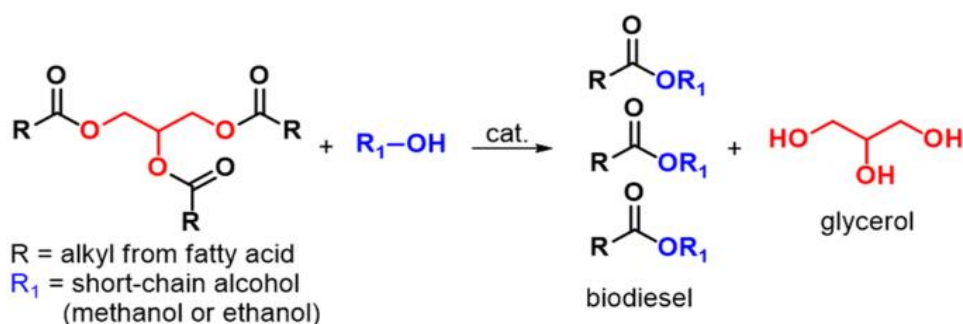


Figure 5. Generalized transesterification reaction mechanism.

5.1. Reaction Temperature

The reaction temperature is considered as one of the most significant parameters that affect the FAME yield in the transesterification reaction. The temperature increase also enhances the rate of reaction and shortens the time for the reaction completion by decreasing the viscosity [112]. The increase in reaction temperature is favorable for endothermic reactions. The energy of molecules of the reactants is directly proportional to the temperature. Thus, the rise in temperature increases the relative miscibility of the polar alcoholic media to a non-polar oil phase which makes the rate of reaction faster [113]. The increase of temperature after peak value cause to decrease the biodiesel yield. The side-reactions taking place at higher temperatures cause the reaction yield to decrease significantly. Nouredini [61] reported the production of soybean biodiesel using methanol and temperature range of 30–70 °C was studied while fixing the other parameters constant and behavior of the rate of reaction was observed. It was concluded that 70 °C was the best temperature among various temperatures, giving maximum biodiesel yield and highest reaction rate. Table 7 describes the literature findings of temperature effect biodiesel synthesis.

Table 7. Effect of biodiesel synthesis using various feedstocks.

Feedstock	Temperature (°C)	Main Outcomes	Reference
Palm oil	50–65	Temperature effect on biodiesel yield was negligible. Biodiesel yield varied between 70 to 90%.	[114]
Canola oil	25–45	A second-order reaction kinetics was observed in the range of 25–45 °C with an activation energy of 66 kJ/mol.	[115]
Palm oil	70–110	Initially, the reaction was slower. The increase of temperature increased the reaction rate and reached the equilibrium at 80 °C. The biodiesel yield was maximum at 80 °C.	[116]
Pongamia oil	30–60	Initially, a slower reaction rate was observed. The increase in temperature increased the triglycerides conversion at 45 °C and 55 °C. The highest biodiesel yield of 98.8% was obtained at 55 °C.	[117]
Sunflower oil	60–120	The biodiesel yield was maximum at 80 °C.	[118]
Linseed oil	40–60	The biodiesel yield remained between 88–96%, which raised due to increase in temperature.	

Evera et al. [119] observed the effect of temperature and assumed that temperature increases the rate of reaction up to an optimal level. The temperature increase beyond its optimized level decreases the biodiesel production due to the thermal decomposition of fatty acid methyl esters [120]. Usually, transesterification reaction is favorable below the boiling temperature to avoid the loss of solvent through boiling. The normal temperature of 50 °C is kept of conventional biodiesel production [121]. Figure 6 expresses the temperature effect FAME yield in the presence of different amounts of co-solvents.

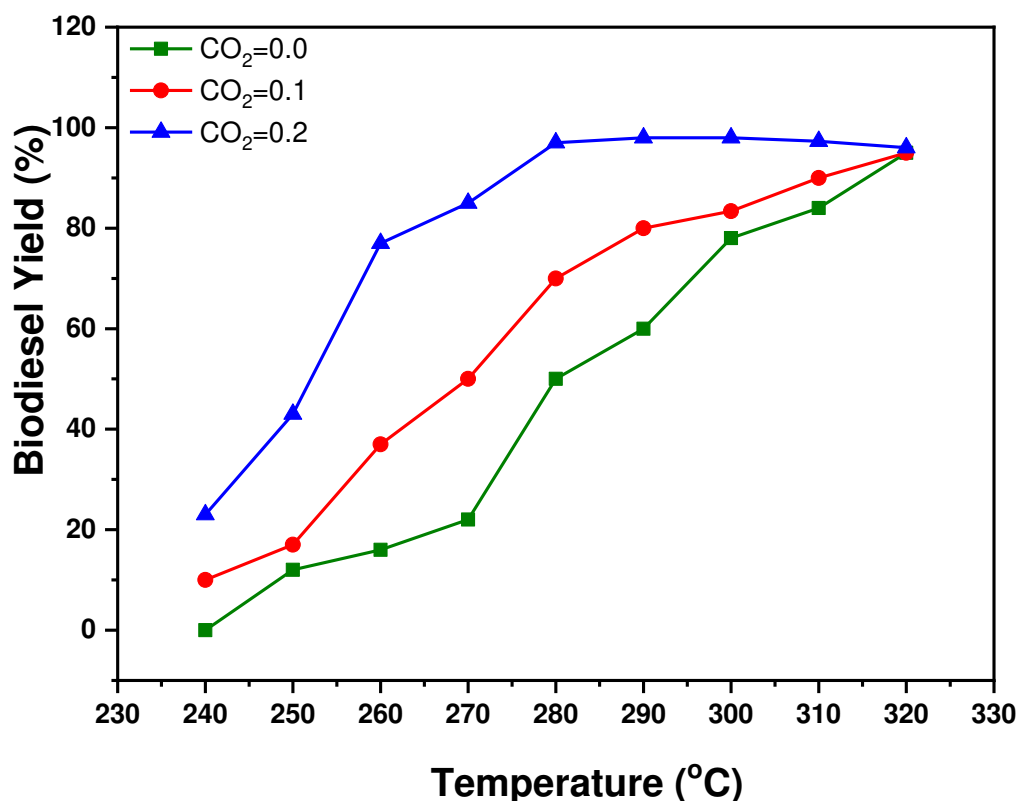


Figure 6. CO₂ and temperature effect on biodiesel yield [122].

Figure 6 states the influence of temperature on FAMES composition in the presence of co-solvent (CO₂). The increase in temperature increased the energy of reacting molecules. Secondly, the transesterification reaction is endothermic in nature. Therefore, transesterification is favorable at elevated temperatures. This graph also concludes that the optimum range of transesterification reaction is 250 to 350 °C. Further increase in temperature will lead to the thermal decomposition of the product.

5.2. Reaction Time

There is a rising trend in the concentration of fatty acid methyl ester when the reaction is carried out for a long time period [123]. The rate of reaction is relatively slower at the start of the reaction due to a little agitation and dispersion of solvent and oil. The reaction is occurring at the outer surface of oil and triglycerides only. Over time, the reaction becomes faster e.g., supercritical reaction is so fast that it is completed in 6 to 10 min [124]. Freedman et al. [123] explained the effect of reaction time and observed the increase in conversion upon higher reaction time. However, the rise in yield occurs below 90 min reaction time [125]. The longer reaction time causes the product loss i.e., biodiesel/fatty acid methyl esters because of irreversible reaction as well as soap formation [121]. Figure 7 expresses how reaction time influences the biodiesel yield in the presence of supercritical methanol as a solvent.

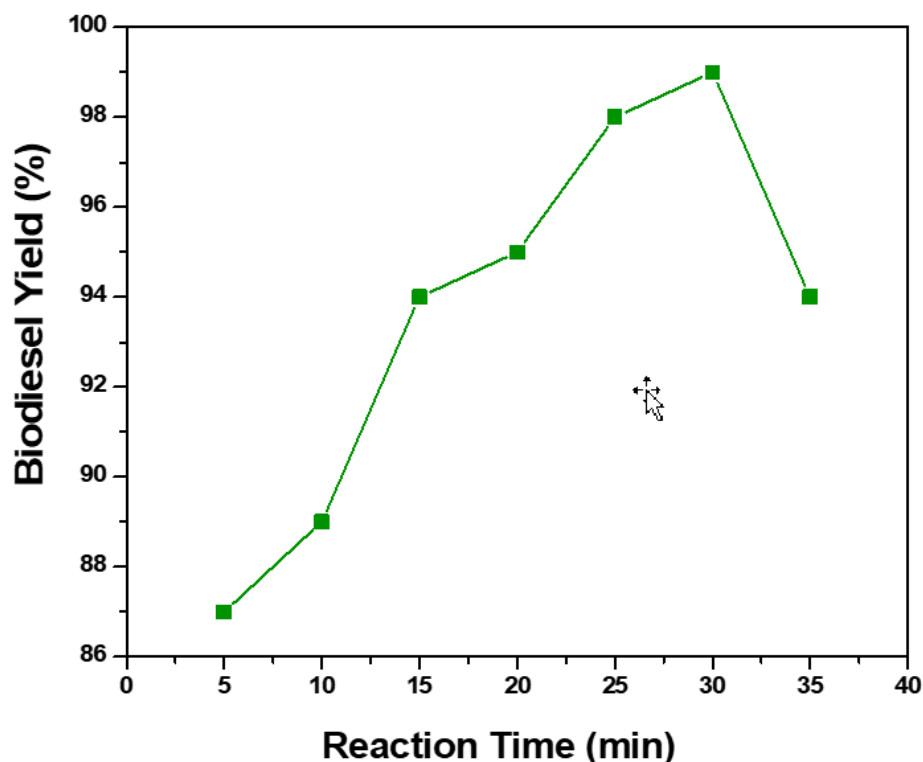


Figure 7. Influence of reaction time on biodiesel production [119].

Figure 7 describes the effect of temperature on the concentration of fatty acid alkyl esters by keeping all other reaction parameters at a constant value. It can be concluded from the graph that elevated reaction time results in the increase of fatty acid alkyl esters concentration in the product. However, this effect can be observed up to 30 min of reaction time. After 30 min, the yield of reaction became independent of reaction time. This may be because equilibrium was already achieved at 30 min [126]. It is recommended to perform supercritical transesterification between 6 to 12 min reaction time and conventional transesterification between 30 to 60 min.

5.3. Alcohol to Oil Ratio

Oil to alcohol ratio is considered as one of the most critical factors that significantly affect the biodiesel yield. The decision of oil to alcohol ratio is associated with the catalyst and process used i.e., when alkali catalyst is used, oil to alcohol ratio is kept at 6:1 [127]. It must be noted that reversible oil to alcohol nature of transesterification reaction requires a large amount of to keep the reaction towards the product side [128]. Jain and Sharma [129] reported that when the amount of methanol was enhanced from 10 to 30% (*v/v*), the highest yield of 90.6% was achieved. The increase of the molar ratio of alcohol favors the forward reaction by avoiding the reverse reaction. It has also been observed that fatty acid ethyl esters (FAEEs) have better combustion characteristics in comparison with methyl esters, but separation and purification of transesterification using ethanol is difficult. Further increase in alcohol molar ratio does not influence the reaction performance but it makes the process expensive because of the difficult separation of alcohol [130]. Similarly, when there is a high FFA content present in the oil sample, alcohol to oil ratio is kept higher than alkali catalysts. This is because of neutralization is required. In such cases, the acid catalyst is used to tolerate these conditions in the reaction. Figure 8 shows the effect of oil to alcohol ratio on biodiesel yield.

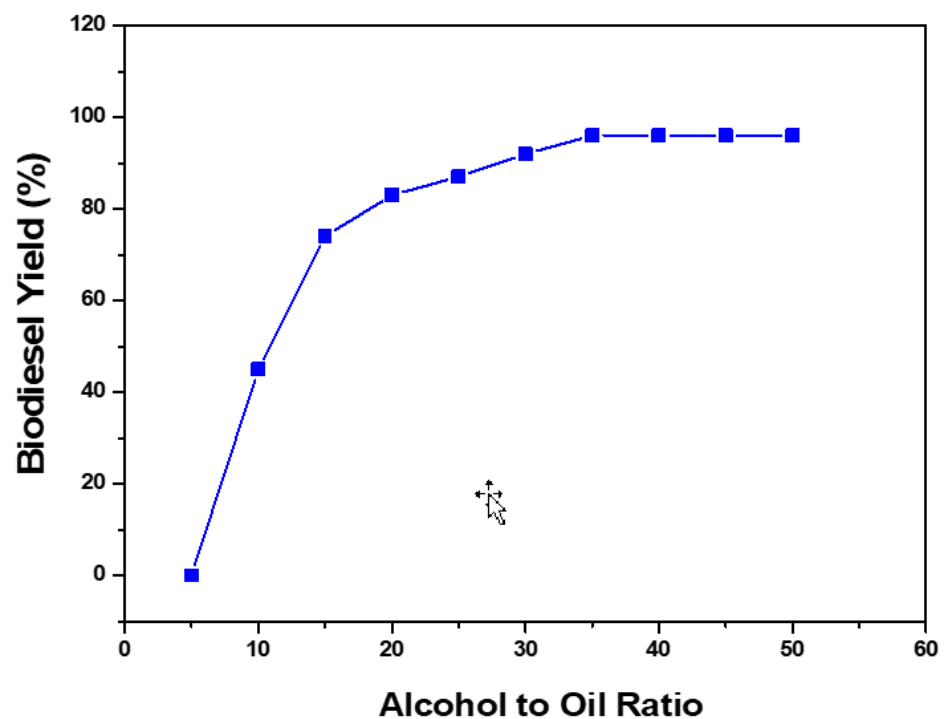


Figure 8. Oil to alcohol ratio effect on biodiesel yield [122].

Figure 8 shows the effect of oil to alcohol ratio on supercritical transesterification using methanol and carbon dioxide as solvent and co-solvent, respectively. The temperature for this study was maintained at 280 °C. However, this rising trend in biodiesel yield can be observed up to approximately 23 min. Because a higher amount of alcohol causes contamination in the product and increases the cost of the overall process, decreasing the overall biodiesel yield.

5.4. Catalyst Amount and Type

FAME yield is strongly dependent upon the type and concentration of catalyst used in the transesterification reaction. The most frequently used catalysts in biodiesel synthesis are sodium hydroxide (NaOH) and potassium hydroxide (KOH) [131]. However, Freedman et al. stated that sodium methoxide is better than NaOH for transesterification reaction because the appropriate mixing of methanol and sodium hydroxide produces water that causes the loss of product (biodiesel) due to hydrolysis of triglycerides. The amount of fatty acid methyl esters increases by increasing the amount of catalyst. This is due to the presence of more availability of active sites in the reaction mixture by the mixing of a large quantity of catalyst [132]. However, a very high amount of catalyst addition in transesterification reaction is not profitable because of the increase in the cost of the catalyst itself. Moreover, excessive usage of catalyst is found to cause emulsions reflecting on higher viscosity causing the biodiesel recovery difficult [132]. Rathore et al. [78] concluded that when KOH conversion was enhanced from 2 to 12%, biodiesel yield increased from 20 to 95%. Akhiehiero et al. [133] reported the highest yield of jatropha biodiesel with 1 wt.% catalyst. The concentration of the catalyst effect on biodiesel yield is shown in Figure 9. It shows the effect of amount for NaOH, KOH, and CaO used. It is observed that biodiesel yield initially increases with the increase of concentration of alkali catalysts (NaOH and KOH), then decreases after reaching a certain peak value.

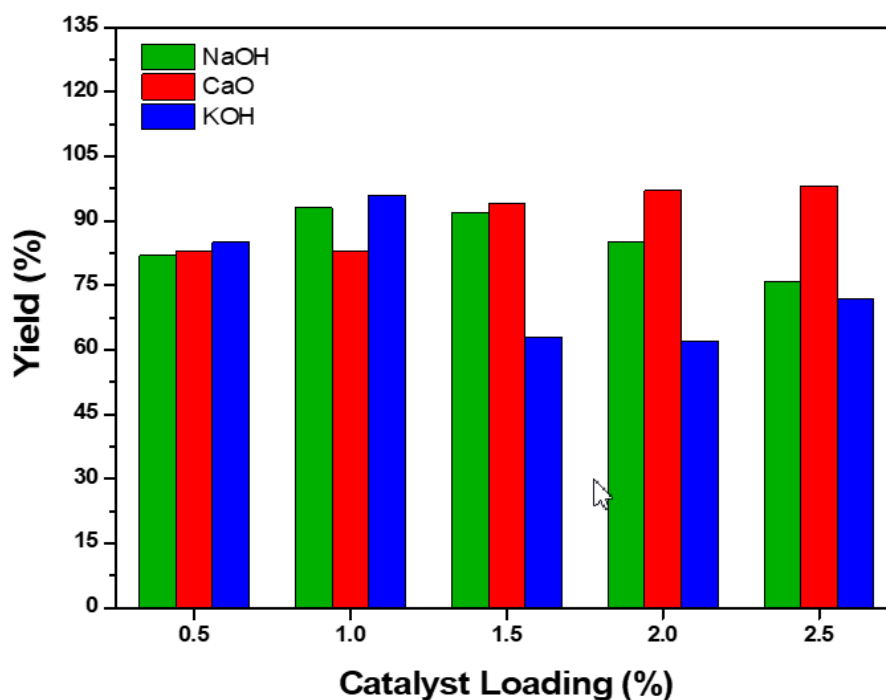


Figure 9. Influence of catalyst concentration on biodiesel yield [125,134].

Figure 9 shows the effect of amount for NaOH, KOH, and CaO used. It is observed that biodiesel yield initially increases with the increase of concentration of alkali catalysts (NaOH and KOH), then decreases after reaching a certain peak value. On the other hand, biodiesel yield increases with the addition of catalyst for heterogeneous catalysts.

5.5. Effect of Water and FFA Content

This is the most critical parameter of concern while choosing the biodiesel production process. There is a flexible tolerance limit of water and acid content in biodiesel production reaction. For example, base-catalyzed transesterification can be applied to water-free and low acid (<1) feedstocks. If the value of FFA content in the feedstock is higher than one, more amount of alkali catalyst is needed to neutralize than that of an acid content present that increases the cost of the overall process. The presence of water causes a greater negative impact as compared to FFA present because water causes soap formation and frothing increases the viscosity [135]. In the presence of water and FFA content, the catalyst is used in soap formation, which reduces the concentration of biodiesel [136]. Transesterification under supercritical conditions does not depend on water and FFA content present in the raw material [137].

5.6. Mixing Intensity

Triglycerides and alcohols are not completely soluble in each other; therefore, the reaction occurs only in the interfaces present. Thus, the process of transesterification is generally slow at the start of the reaction. Mixing intensity is an important parameter that helps to complete the reaction resulting in the formation of product (methyl esters or biodiesel yield). Since the mixing of oil and solvent increases the reaction in the inner surfaces of oil and solvent, for transesterification reaction, continuous mixing at 200 rpm, 400 rpm, and 600 rpm has been studied in the literature. The moderate stirring speed of 400 rpm was the best agitation speed giving maximum ester yield [138]. However, moderate or lower agitation speed lowers the formation of the product. On the other hand, higher agitation speed supports the soap formation irreversible behavior of transesterification reaction at higher rpm. Table 8 shows the effect of various parameters on biodiesel synthesis. The summary overall effect of each parameter is presented in Table 9.

Table 8. Effect of different parameters on biodiesel synthesis.

Feedstock	Molar Ratio	Time (min)	Catalyst Loading	Temperature (°C)	Agitation Speed (rpm)	Type of Transesterification	Yield (%)	Reference			
Palm oil	6:1 (methanol)	60	1% KOH	60	600	Homogeneous base	88	[139]			
	6:1	60	1% NaOH	60	600		93	[139]			
	9:1	480	8.5% KOH	65–75	-		96.2	[140]			
	10:1	-	0.4% KOH	70–110	-		98	[116]			
Jatropha oil Waste frying oil	10:1	480	9% KOH	60–80	-	Homogeneous base	96.8	[78]			
	4.83:1 to 9.65:1	300–480	1–4%	50–65	-		87.3	[141]			
Soybean oil	12:1	60	6% CaFeAl	60	270	Heterogeneous transesterification	90	[142]			
Jatropha oil	3:7	180	1% H ₂ SO ₄	65	400	Homogeneous acid and base	21.2	[129]			
	3:7		1% NaOH	50	400		90.2				
Waste cooking oil	3:7	180	1% H ₂ SO ₄	65	400	Homogeneous acid and base	21.2	[143]			
	3:7	180	1% NaOH	50	400		90.6				
Canola oil	3:1 to 8:1	25–75	0.2–1.2% KOH	30–70	100–600	Homogeneous base	-	[144]			
Mustard oil	-	30	KOH	40–60	450	Homogeneous base	-	[145]			
Sunflower oil	6:1	90–330	1% CaO	23–60	-	Heterogeneous	91	[60]			
	6:1		0.06–0.34	23–60	400		Homogeneous base		99	[62]	
	12:1	-				-		-			-
24:1	-	-				-		-			-
Peanut oil	30:1	30–360	-	250–310	500	Supercritical transesterification	>90	[146]			
Waste lard	6:1	20	4–6 wt.% enzyme	50	-	Ultrasound-assisted transesterification	96.8	[108]			
Silybum Marianum seed oil	6:1	75	4–6% sulfonated solid acid catalyst	60	600	Carbon acid esterification and homogeneous base transesterification	96.9	[147]			
Canola oil	6:1	-	0.5% KOH	45	-	Homogeneous base	95	[115]			
Used frying oil	6.03:1	120	0.55% KOH	60–100	-	Homogeneous base	-	[148]			
Rapeseed oil	3.5:1 to 42:1	120	-	200–500	-	Supercritical transesterification	95	[149]			
Neem oil	10:1	60	10% CZO	55	-	Heterogeneous transesterification	97.1	[150]			

Table 9. Effect of reaction parameters on transesterification.

Parameter	Effect on Transesterification
Temperature	The temperature increase also enhances the rate of reaction and shortens the time for the reaction completion by decreasing the viscosity. The temperature of 50 °C is recommended for conventional transesterification.
Time	The increase in reaction time increases the fatty acid alkyl esters concentration in the product up to optimum value. The reaction time during conventional transesterification is kept between 30 to 60 min.
Alcohol/oil molar ratio	The increase of the molar ratio of alcohol favors the forward reaction by avoiding the reverse reaction.
Catalyst amount and type	The most frequently used catalysts in biodiesel synthesis are sodium hydroxide (NaOH) and potassium hydroxide (KOH). The amount of fatty acid methyl esters increases by increasing the amount of catalyst.
Agitation speed	Mixing intensity is an important parameter that helps to complete the reaction resulting in the formation of product (methyl esters or biodiesel yield). The moderate stirring speed of 400 rpm is recommended to catalyze the biodiesel production reaction.

5.7. Kinetics of Biodiesel Synthesis

Kinetic study of biodiesel synthesis is done to study the rate of reaction and activation energy needed to complete the reaction. Some researchers proposed the kinetics of transesterification considering three steps [62,116,144]. The three-step transesterification is complex, time consuming, and requires analytical units. Therefore, it has been considered as one step kinetics considering overall transesterification reaction kinetics for triglycerides and methyl esters [26,151–157].

The rate equation for the reaction ($\text{Triglycerides} + 3 \text{CH}_3\text{OH} \rightarrow 3 \text{FAME} + \text{Glycerol}$) is represented as follows (Equation (8)).

$$-r_A = -\frac{dC_A}{dt} = -\frac{1}{3} \cdot \frac{dC_B}{dt} = k \cdot C_A^n \cdot C_B^m \quad (8)$$

Equation (8) represents the rate expression based on the overall transesterification reaction. A and B represents triglycerides and alcohol, respectively. The n and m are the reaction orders and k are the rate constant of the transesterification reaction. The rate of reaction can be taken as independent of alcohol concentration since an excess amount of alcohol present. Therefore, a simplified expression is shown in Equations (9) and (10).

$$-r_A = -\frac{dC_A}{dt} = k \cdot C_A^n \quad (9)$$

$$C_A = C_{A0} (1 - X) \quad (10)$$

Substituting Equation (3) in Equation (2) resulted in more simplified forms as follows.

$$-r_A = -\frac{dC_A}{dt} = k \cdot [C_{A0} (1 - X_A)]^n \quad (11)$$

$$-\frac{d[C_{A0} (1 - X_A)]}{dt} = k \cdot [C_{A0} (1 - X_A)]^n \quad (12)$$

Differentiating Equation (5) with time (t) gives the following.

$$\frac{C_{A0} dX_A}{dt} = k \cdot [C_{A0} (1 - X_A)]^n \quad (13)$$

$$\frac{dX_A}{dt} = k \cdot \frac{[C_{A0} (1 - X_A)]^n}{C_{A0}} \quad (14)$$

The conversion of triglycerides (X_A) can be represented with X to obtain a more simplified expression.

$$\ln \frac{dX}{dt} = n \ln \left[k \frac{C_{A0}(1 - X)}{C_{A0}} \right] \quad (15)$$

$$\ln \frac{dX}{dt} = n \cdot \ln C_{A0}(1 - X) + \ln k' \quad (16)$$

where

$$k' = \frac{k}{C_{A0}}$$

The linear graph between $\ln dX/dt$ and $\ln C_{A0}(1 - X)$ could be plotted to determine the value of order (n) and rate constant (k) of the reaction. The order and rate constant values are to find the activation energy (E_a) and pre-exponential factor (A). The Arrhenius equation is used to calculate these parameters.

$$k = A e^{\frac{-E}{RT}} \quad (17)$$

The simplified linear form of expression can be represented as in Equation (18).

$$\ln k = \frac{-E_a}{RT} + \ln A \quad (18)$$

The linearized plot between $\ln k$ and $1/T$ gives the activation energy and pre-exponential factor value. Jain et al. [143] did the kinetic study for biodiesel synthesis from waste cooking oil and reported the k and E_a as 0.0078 min^{-1} and 88.764 kJ/mol .

6. Conclusions and Future Recommendations

Plenty of feedstocks for biodiesel production are discussed in this study. The potential feedstocks for biodiesel synthesis are categorized into three classifications including edible oils, non-edible oils, and waste oils, animal fats, and microbial oils. Edible oils include soybean oil, sunflower oil, palm oil, and mahua oil. According to the study, edible oils are easily available and contain higher oil content, but they cannot be used for biodiesel synthesis because of competition with the food market. Similarly, non-edible oils can be used for biodiesel synthesis due to higher oil content and no requirement as food sources. The literature comparison of this study shows that they contain higher oil content, but it is not easy to obtain higher conversion in the reaction. This is due to the complexity of the process that requires a higher catalyst amount and time to complete the reaction. The most feasible and easily available feedstocks with higher oil content and less cost include waste oils and microbial oils. Waste oils include palm oil mill effluent (waste palm oil) and waste cooking oil. More study is required to maintain the balance between the availability of raw materials and their usage for biodiesel production. The microbial oil-based biodiesel has excellent properties, so they can meet the fuel demand solely. Nevertheless, it is difficult to grow the bacteria because they are temperature sensitive and cannot be sustained in hot weather conditions. To overcome the energy shortage by the implementation of biodiesel, the waste oils and non-edible oils should be used for biodiesel production. In the past, only edible and non-edible oils were focused on regarding use for biodiesel production causing a huge shortage of food sources. Therefore, two solutions are highlighted in the study to either increase the yield of existing biodiesel production processes or explore the new feedstocks for biodiesel. The novel oil extraction technologies are also highlighted to achieve the maximum oil extraction yield as its lower value is the main problem at the moment. The existing state-of-the-art extraction technologies include mechanical extraction, solvent extraction, supercritical technology, microwave, and ultrasound-assisted oil extraction. These oil extraction technologies are discussed and

evaluated based on their range of applicability and extraction cost. It is concluded that supercritical and microwave-assisted technologies are the most appropriate among all existing technologies due to their high oil recovery efficiency, lower cost, and the fact that they do not affect the sustainability of the environment. The transesterification reaction rate depends upon reaction temperature, pressure, oil to alcohol ratio, water and FFA content, and agitation speed. The critical parameters of transesterification reaction highlighted in this study are temperature, oil to alcohol ratio, water and FFA content, agitation speed, and reaction time. Because of the increase in temperature and molar ratios, the reaction performance is affected (i.e., an increase of temperature and pressure increases the product yield). Based on the feedstock study of biodiesel production, waste oils and algal oil use to synthesize biodiesel is an unexplored area. Therefore, more detailed study is required to explore the waste oil feedstocks that can produce high-quality biodiesel with a minimum associated cost. Similarly, in previous work, researchers have worked on edible and non-edible oils for biodiesel. The exploration of kinetics for the transesterification reaction is needed to understand the effect of every parameter influencing biodiesel yield. This will help to understand the temperature and pressure effects on biodiesel yield in a better way for practical applications. Moreover, the biodiesel properties are directly associated with chemical composition and other parameters are also necessary to be considered to address the technical difficulties involved.

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