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Review Article

A Review on the Recent Breakthrough Methods and Influential Parameters in the Biodiesel Synthesis and Purification

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Abstract. Biodiesel has recently received much attention as an energy source with numerous benefits such as high degradability, negligible toxicity, and minimal emissions of carbon monoxide gases as well as particulates. Therefore, this research aims to compare, review, and summarize the conventional and advanced methods of biodiesel production. Currently, some emerging processes that were developed for advanced biodiesel production include microwave-assisted synthesis, ultrasonic-assisted synthesis, supercritical transesterification, and liquid phase plasma discharge technology. The types of feedstocks, catalysts, and operating conditions as the influential parameters in biodiesel synthesis are also discussed. Moreover, in the purification process, the effectiveness of purification depends on the type of catalyst applied in the synthesis process. This research also reviewed and compared several commonly used purification methods such as wet and dry washing, ion exchange and precipitation, complexation, and membrane-based separation that have shown significant results along with the impacts of biodiesel production on environmental and economic sectors.

Keywords: Biodiesel synthesis, biodiesel purification, catalysts, advanced biodiesel production, biodiesel impact

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1. Introduction

Biodiesel has been widely promoted one of the most promising renewable energy sources. Basically, biodiesel is a blend of fatty acid and methyl esters (FAME) that can be used in standard diesel engines with little modification. It has mostly been created through the esterification of free fatty acids (FFAs) and/or homogenous basic or acid transesterification of triacylglycerols (TAGs) from diverse raw materials such as used cooking oils, plant, and animal oils (Athar and Zaidi, 2020). The TAGs conversion into methyl esters takes approximately one hour at ambient temperature and pressure conditions due to the high activity of conventional basic catalysts and the moderate operating condition of reaction (around 60–65°C) (Athar and Zaidi, 2020; Fayyazi *et al.,* 2021; Syafiuddin *et al.*, 2020). However, the disadvantages of these procedures include the generation of soap, a decrease in catalytic effectiveness due to catalyst depletion, a rise in viscosity, and the development of gels.

In the wet washing process, a large amount of water is required to remove the remaining catalyst and purify the biodiesel, leading to a substantial amount of wastewater that needs to be effectively handled (Fayyazi *et al.*, 2021; Šalić *et al.*, 2020; Sokač *et al.*, 2020). This extra stage in the synthesis of the biodiesel process raises the total cost of production, which makes it uncompetitive with petroleum-based diesel generation (Kumar *et al.*, 2020; Noriega and Narváez, 2020). Lastly, the cost of producing biodiesel is approximately three times higher than that of petroleum. Moreover, one of the most significant disadvantages of homogeneous catalysts is that they cannot be regenerated (Hariprasath *et al.*, 2019; Kasirajan, 2021; Shankar *et al.*, 2017).

Previous research has stated that the conventional biodiesel production methods such as catalytic transesterification and esterification have achieved production high capacity, however, it requires high expenditure, especially for the purification process (Veljković *et al.*, 2015). This becomes the main reason biodiesel has not been able to compete with diesel oil from petroleum products in the transportation market (Keera *et al.*, 2018; Lee and Saka, 2010; Marchetti *et al.*, 2007). Therefore, the need for the development of novel, clean, eco-friendly, and efficient processes is increasing in past decades to produce the standard biodiesel product with a shorter time and easier purification method. To solve this problem, numerous approaches of biodiesel production from the conventional to the advanced methods such as microwave-assisted synthesis, ultrasonic-assisted ultrasonic, supercritical transesterification, and plasma discharge were deeply reviewed, compared, and discussed. This research also reviews the influential parameters in biodiesel syntheses such as feedstocks, catalysts, and

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operating conditions. Furthermore, several commonly applied purification methods were discussed and summarized to present a holistic understanding of biodiesel production. The methods presented were wet and dry washing, ion exchange and precipitation, complexation, membrane-based separation, and simultaneous synthesis and purification techniques along with their benefits and drawbacks. Similarly, the impacts of biodiesel production on environmental and socio-economic aspects were also briefly analyzed.

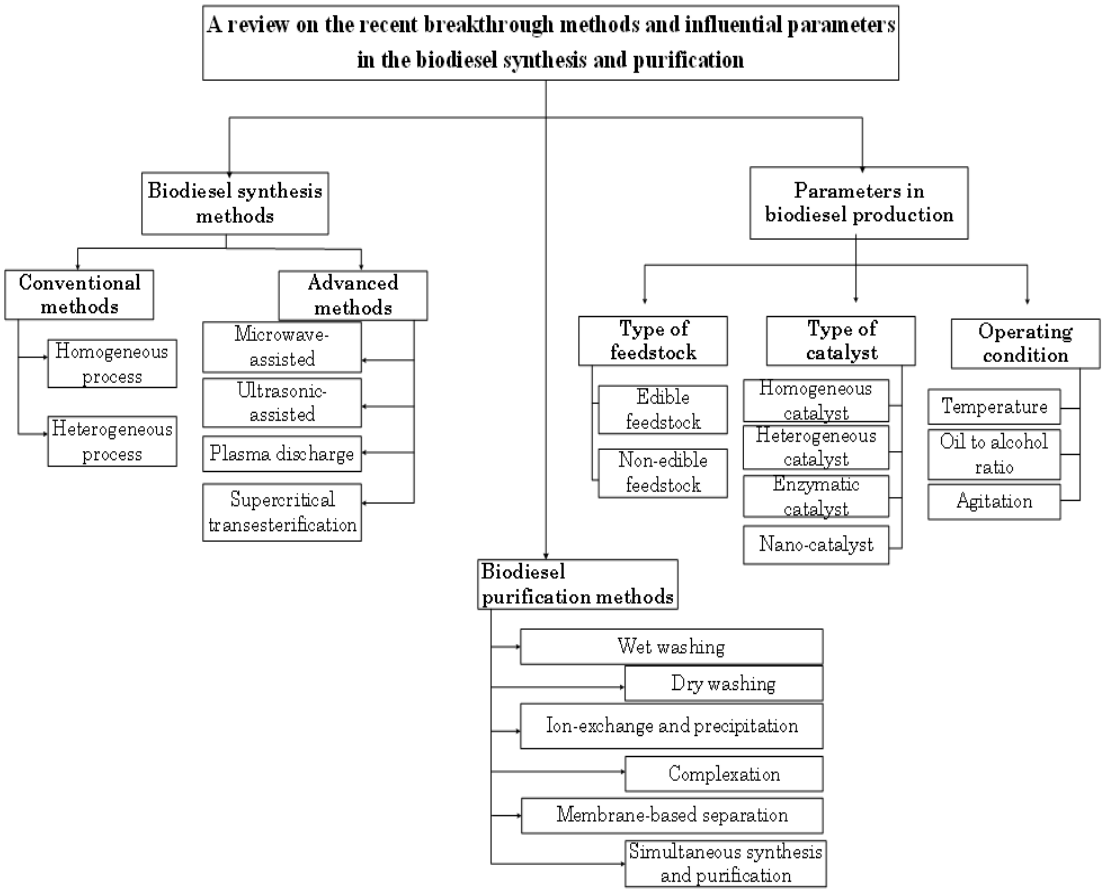

Fig. 1. Overview of the review's contents.

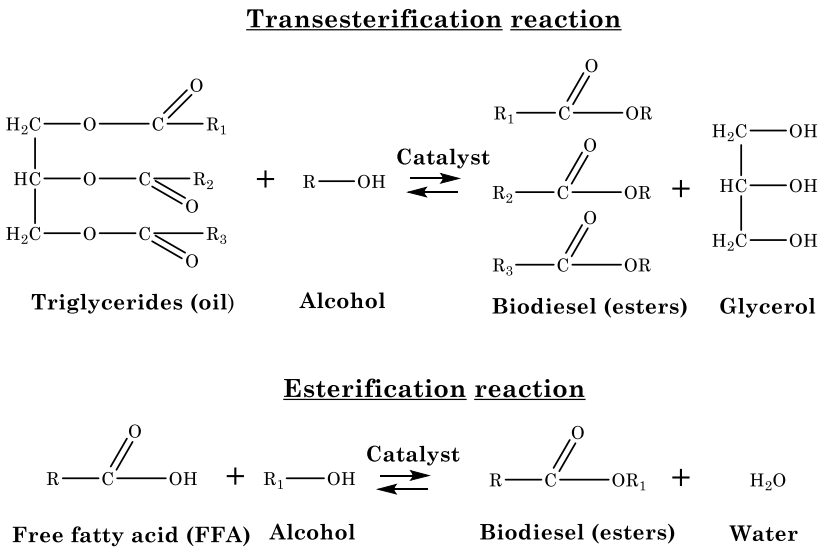

Fig. 2. The transesterification and esterification reactions in biodiesel production (Chozhavendhan *et al*., 2020).

2. Production process of biodiesel

In this section, the conventional and several advanced methods in biodiesel production are discussed, as shown in Fig. 1. Conventional methods from the literature include homogeneous and heterogeneous processes, while advanced methods such as microwave-assisted, ultrasonic-assisted, plasma discharge, and supercritical processes in biodiesel production are reviewed and compared.

2.1. Conventional method of biodiesel production

Algae, plant oil, food sources, lignocellulosic materials, and other sources can be used to produce biodiesel (Abo *et al*., 2019; Alami *et al*., 2021; Baskar *et al*., 2018; Keera *et al*., 2018; Li *et al*., 2012; Saputra Nursal *et al*., 2021). Moreover, biodiesel is commonly made through the transesterification process, which involves reacting oils with short-chain alcohol to form alkyl esters and glycerol with the use of a catalyst. The reaction can be occurred at a temperature of (50-70 ^{o}C), and it is usually heated by external thermal heater (Fayyazi *et al*., 2021). Transesterification is a three-step process that occurs in a sequential (Al-Saadi *et al*., 2020; Hariprasath *et al*., 2019; Sokač *et al*., 2020), which involves the transformation of triglyceride in the oil to diglyceride, followed by monoglyceride, and glycerol in the first stage. A significant amount of oil to alcohol (usually 1:3) molar percentage is constantly operated in the system to promote the forward reaction and a large amount of alcohol is typically applied to tip the balance to the right side (product). The reaction flow is divided into two stages, namely biodiesel product (upper) and glycerol rich phase (lower) at the end of the transesterification procedures (Fayyazi *et al*., 2021). It was also reported that the unreacted alcohol is distributed in both phases (Al-Saadi *et al*., 2020; Hariprasath *et al*., 2019; Sokač *et al*., 2020). The general reaction mechanisms for producing biodiesel (methyl esters) through the transesterification and esterification process can be seen in Fig. 2. Further details regarding homogeneous and heterogeneous catalytic process in biodiesel synthesis can be seen in part 3.2.1 and 3.2.2, respectively.

2.2. Advanced method of biodiesel production

In conventional methods, energy in form of thermal is used to heat the reactants to a specific temperature to force the chemical reaction. Meanwhile, the activation energy is fulfilled using electric, sound, and electromagnetic wave powers in the current methods of biodiesel production. In this section, the current technology of biodiesel production is discussed.

2.2.1. Microwave-assisted biodiesel synthesis

In common biodiesel production, the reactors are designed based on conventional heat transfer, which transfers some amount of energy to the material's surface and initiates the reaction. However, this method is thermodynamically inefficient and time-consuming, because it requires a large amount of energy and longer time to achieve a uniform temperature distribution (Lawan *et al*., 2020). Meanwhile, microwave technology can irradiate the electromagnetic energy directly to the molecular level for early chemical reaction (Mamo and Mekonnen, 2020). Research has shown that microwave energy transfer is faster and more efficient than conventional heating technology (Binnal *et al*., 2021; Mamo and Mekonnen, 2020). In the practical situation, it is essential to identify the dissipation factor of the material that is used in a microwave system because its efficiency of heat transfer depends on the ability of the material to absorb the electromagnetic energy and initiate the chemical reaction. Therefore, homogeneous and heterogeneous catalysts are still used in microwave-assisted biodiesel synthesis to provide higher efficiency (Tangy *et al*., 2017). In the synthesis process, the microwave energy is directly transferred to the reactants and degraded into different side products. This makes it necessary to maintain the input power of microwave irradiation to achieve a good result.

Microwave-assisted transesterification process using can be applied using homogeneous and heterogeneous catalysts. Homogeneous acid catalyst process is the best choice for the feedstock that containing high level of free fatty acid and water (Zhang *et al*., 2010; Zhang, 2003). Several homogeneous acid catalysts including sulfuric acid (H_2SO_4), hydrochloric acid (HCl), and boron trifluoride (BF_3) were found useful for the biodiesel synthesis from high acid level feedstocks (Dall'Oglio *et al*., 2015; Zhang *et al*., 2010). Other polyacids such as $H_3PW_{12}O_{40}$, $H_4SiW_{12}O_{40}$, and $Na_3PW_{12}O_{40}$ can be dissolved in alcohol, forming the homogeneous system (Zhang *et al*., 2010). The polyacids have strong Bronsted acidity than that of H_2SO_4 and other acids with uniform acid sites. A study conducted by Dall'Oglio and coworkers revealed the results of comparative study on microwave-assisted biodiesel synthesis using maize oil as feedstock and applied some acid catalysts such as HCl, H_2SO_4, and $CISO_3H$ was found to have > 85% of conversion (Dall'Oglio *et al*., 2015). The better conversion using H_2SO_4 showed the better mobility of ions and counterions in the reaction system that supported by microwaves compared to the conventional system (Dall'Oglio *et al*., 2015; Zhang *et al*., 2010). Cheng and coworkers studied the comparation of microwave-assisted biodiesel synthesis of chlorella pyrenoidosa using two different processes namely, (i) microwave-assisted transesterification followed by a conventional solvent extraction, and (ii) microwave-assisted extraction followed by a conventional transesterification (Cheng *et al*., 2014). It was found that in the first approach the FAME concentration was found to have a maximum content of 95% by using 30 min of 500 W microwave power (Cheng *et al*., 2014). Other process conditions were 6 ml/g alcohol to oil ratio, 3% H_2SO_4, and 90 ^{o}C of temperature. It was concluded that microwave was useful to boost the FAME yields (Cheng *et al*., 2014). Since the microwave does not interrupt the bonds at molecular levels, but just helping to cause the electron excitation, thus, the reaction pathways of a microwave-assisted biodiesel synthesis follow the same route as the conventional process (Dehghan *et al*., 2019; Huang *et al*., 2015). Despite acid catalysts showed improvements in microwave-assisted biodiesel production, some drawbacks were still found such as it requires higher alcohol to oil ratio, formation of water molecules, complex product separation and purification, and the spent acids in final product (Dehghan *et al*., 2019).

Due to many drawbacks of acid catalysts in the homogeneous microwave-assisted system, researchers gave more attention to the alkali catalysts (Nayak *et al*.,

2019). The main profit of using homogeneous alkali catalyst in microwave-assisted biodiesel synthesis is a higher yield of biodiesel in a shorter time compared to the conventional heating method (Qu *et al*., 2021). This is promoted by the direct absorption of electromagnetic energy of microwave by the hydroxyl (-OH) groups, that subsequently break the stricture of two-tier of oil and alcohol, which improved the solubility of these reactants (Lin and Chen, 2017; Sharma *et al*., 2016). Numerous alkali group 1 materials such as KOH, NaOH, $NaOCH_3$, which are dissolved in alcohol and can be applied for microwave-assisted biodiesel synthesis (Nayak *et al*., 2019). Under bombardment of electromagnetic power from microwaves, due to the higher dielectric characteristics, alcohol quickly heated to reach the boiling point and provide sufficient energy to initiate the transesterification reaction (Rokni *et al*., 2022). A study performed a microwave-assisted biodiesel synthesis using papaya oil found nearly 99% biodiesel conversion (Nayak and Vyas, 2019). This process was conducted by applying 0.95%-wt NaOH as catalyst, 62.3 ^oC of temperature, 9.5:1 alcohol to oil ratio, 450 W of microwave power for 3.3 min (Nayak and Vyas, 2019). An attempt was performed using microalgae as feed oil with 0.5%-wt NaOH in methanol as catalysts, 12 ml n-hexane, constant agitation of 600 rpm, 45 ^oC, with 350 W of microwave power (Chen *et al*., 2015). In addition, the main advantage of microwave-assisted biodiesel synthesis using the homogeneous alkali catalysts is the faster process compared to the acid catalysts and can be performed for wet biomass feedstocks such as microalgae by a single stage (Chen *et al*., 2015; Qu *et al*., 2021).

Microwave-assisted biodiesel synthesis using acid and alkali homogeneous catalysts reported to have an improvement in shorter reaction time, but the homogeneous catalytic process suffers from problems regarding product separation process, treatment of acid or base disposals, and catalyst reactivation process (Kamel Ariffin and Idris, 2022; Zhang *et al*., 2022). This problem can be solved by using heterogeneous catalysts (Dehghan *et al*., 2019). Various study on the microwave-assisted transesterification using heterogeneous catalysts have been reported (Alcañiz-Monge *et al*., 2018; de Aguiar *et al*., 2017). Acidic metal oxides, alkali metal oxides, cation exchange resins, clay supported catalysts, and carbon supported catalysts have been utilized for advanced microwave-assisted biodiesel synthesis (Alcañiz-Monge *et al*., 2018; de Aguiar *et al*., 2017; Guldhe *et al*., 2017; Nayak *et al*., 2019). The presence of catalysts can reduce the required microwave energy level, maintain the reaction equilibrium, and conduct the reaction at lower input energy input that provides a fast conversion rate (Nayak *et al*., 2019). The operating condition in microwave-assisted biodiesel synthesis depends on the feedstock properties, fatty acid content, reaction time, mixing speed, and catalyst dosage (Mamo and Mekonnen, 2020; Tangy *et al*., 2017). Some of these parameters have been optimized in the previous research (Thirugnanasambandham *et al*., 2017). Table 1 summarizes the most recent application of microwave in biodiesel synthesis. where it was concluded that microwave-assisted biodiesel production technology is one of the most promising new technologies with faster and high conversion. However, some obstacles such as high capital cost and complex equipment requirement decelerate the development of this method in the industry.

2.2.2. Ultrasonic assisted biodiesel synthesis

Another modern technology in biodiesel production is ultrasonic-assisted biodiesel synthesis, which is carried out to homogenize the reactants and catalysts. The higher level of homogenization between reactant and catalyst can provide a better contact area to convert reactants into biodiesel with a higher conversion rate (Salamatinia *et al*., 2012). The use of ultrasonic radiations in transesterification reaction does not disturb the chemical equilibrium and thermodynamic conditions (Sajjadi *et al*., 2015; Stavarache *et al*., 2007). Similarly, it can also enhance the mass transfer, thereby improving the overall rate of kinetic (Sajjadi *et al*., 2015). This process radiates the acoustic energy that creates the cavitation mechanism and increases the molecular kinetic energy (Gogate, 2008). To apply the ultrasonication in biodiesel production, there is a need to understand the required intensity, produce the optimized cavitation, and distribute a uniform ultrasound condition across the mixture to obtain a better result. In an ultrasonic system, the electrical energy is converted into acoustic/ sound energy through piezoelectric transformers, which are emitted into the chemical transformation (Luo *et al*., 2014). Therefore, an optimized design is needed to minimize the energy loss during the conversion from electrical into sound energy.

The physical effects of ultrasonic-assisted transesterification reaction have been reported by a huge number of investigations. Physically, the ultrasonic waves cause bubble cavitations close to the boundary layer between oil and alcohol, which generating a vast number of micro bubbles. The bubbles' collapse produces microturbulence and disrupts the phase boundary layer. The speed of collapse of the bubbles can reach up to 200 m/s, generating the immiscible mixing and starting the emulsification between the oil and alcohol. The mass transfer rate increases due to this emulsification as well as generating faster reaction kinetics. The use of an ultrasonic system in the transesterification process enhances the reaction efficiency and attributes to the less ratio of oil to alcohol (Florez Marulanda and Ortega Alegria, 2019). Meanwhile, one of the superiorities of the ultrasonic system in biodiesel production is the massive formation of oil and alcohol emulsion, which is carried out by simple mixing in the conventional method. The recent developments of ultrasonic-assisted biodiesel production are listed in Table 2. In the homogeneous catalytic process, the ultrasonic energy does not directly enhance the reaction rate; however, the ultrasonic waves intensify the mass transfer by providing massive emulsion of reactants and catalysts to achieve an 80% faster process. Moreover, it can also be seen that the operating temperatures to conduct the biodiesel synthesis using an ultrasonic system are relatively low compared to the conventional process that needs at least 60^oC to initiate the reaction (Asakura *et al*., 2008).

It is noteworthy that, even though transesterification assisted with ultrasonic wave to synthesize biodiesel is very helpful, further studies are required to overcome the technical drawbacks.

Table 1.
Comparison of several microwave-assisted biodiesel productions.

Feedstock	Type of catalytic process	Catalyst	Operating conditions	Ester yield (%)	Ref.
Waste cooking oil	Homogeneous	NaOH (1%)	P = 700 W, t = 10 min, T = 60 °C	88.9	(Eguchi., 2015)
Microalgae oil	Homogeneous	H_2SO_4 (2.5%)	P = 450 W, t = 11 min, T = 60 °C	97.1	(Binnal and Nirguna Babu, 2019)
Dairy scum oil	Homogeneous	KOH (1%)	P = 600 W, t = 5 min, T = 60 °C	93.5	(Binnal *et al.*, 2021)
Waste cooking oil	Homogeneous	KOH (1%)	P = 750 W, t = 2 min, T = 80 °C	96	(Sawangkeaw *et al.*, 2011)
Maize oil	Homogeneous	$ClSO_3H$ (3%)	P = 700 W, t = 20 min, T = 80-84°C	93.1	(Dall'Oglio *et al.*, 2015)
Chlorella vulgaris oil	Homogeneous	H_2SO_4 (1.5%)	P = 700 W, t = 40 min, T = 60 °C	91.8	(Sharma *et al.*, 2016)
Yellow horn oil	Homogeneous	H_2SO_4 (1%)	P = 500 W, t = 30 min, T = 90 °C	95.2	(Zhang *et al.*, 2010)
Yellow horn oil	Homogeneous	$H_3PW_{12}O_{40}$ (1%)	P = 500 W, t = 10 min, T = 90 °C	96.2	(Zhang *et al.*, 2010)
Algae oil	Homogeneous	NaOH (0.5%)	P = 800 W, t = 20 min, T = 50 °C	83.3	(Chee Loong and Idris, 2014)
Waste cooking oil	Homogeneous	CH_3ONa (0.75%)	P = 750 W, t = 3 min, T = 65 °C	97.9	(Chen *et al.*, 2012)
Tallow	Homogeneous	NaOH (0.6%)	P = 700 W, t = 5 min, T = 55 °C	96.3	(Murillo *et al.*, 2019)
Jathropa oil	Homogeneous	NaOH (1%)	P = 312 W, t = 7 min, T = 65 °C	98.7	(Nayak *et al.*, 2019)
Safflower oil	Homogeneous	NaOH (1%)	P = 300 W, t = 6 min, T = 60 °C	98.4	(Duz *et al.*, 2011)
Waste cooking oil	Heterogeneous	SrO/SiO_2 (41%)	P = 100 W, t = 15 min, T = 60 °C	99.2	(Tangy *et al.*, 2017)
Yellow horn oil	Heterogeneous	$Ag_3PW_{12}O_{40}$ (1%)	P = 500 W, t = 10 min, T = 60 °C	95.7	(Zhang *et al.*, 2010)
Castor oil	Heterogeneous	$Cs_{2.5}H_{0.5}PW_{12}O_{40}$ (15%)	P = 300 W, t = 4 h, T = 70 °C	90.0	(Yuan and Shu, 2013)
Acidified oil	Heterogeneous	CERP/PES (3%)	P = 360 W, t = 90 min, T = 60 °C	97.4	(Zhang *et al.*, 2012)
Waste oil	Heterogeneous	Amberlyst-15 (25%)	P = 360 W, t = 30 min, T = 75 °C	85.5	(Ayas and Yilmaz, 2015)
Rapeseed oil	Heterogeneous	K10 montmorillonite (10%)	P = 1000 W, t = 60 min, T = 170°C	10.2	(Mazzocchia *et al.*, 2004)
Oleic acid	Heterogeneous	Sulphated metakaolin (5%)	P = 400 W, t = 40 min, T = 115°C	96.5	(de Oliveira *et al.*, 2013)
Jathropha oil	Heterogeneous	$[MMBIM]HSO_4$ (20%)	P = 245 W, t = 12 min, T = 80 °C	96.2	(Soni *et al.*, 2014)
Microalgae oil	Heterogeneous	Goat bone nano-catalyst	P = 600 W, t = 3h, T = 60 °C	92	(Abomohra *et al.*, 2017)
Waste lard	Heterogeneous	CaO/zeolite (8%)	P = 595 W, t = 1.25 h, T = 70 °C	90.9	(Lawan *et al.*, 2020)

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Table 2
Comparison of several ultrasonic-assisted biodiesel productions.

Feedstock	Type of catalytic process	Catalyst	Operating conditions	Ester yield (%)	Ref.
Waste cooking oil	Homogeneous	KOH (1%)	P = 240 W, f = 20 kHz, t = 8 min, T = 60 °C	88.4	(Martinez-Guerra and Gude, 2015)
Canola oil	Homogeneous	NaOH (1.25%)	P = 150 W, f = 20 kHz, t = 2 min, T = 50 °C	96.8	(Fayyazi *et al.*, 2014)
Karanja oil	Homogeneous	NaOH (1%)	P = 500 W, f = 20 kHz, t = 45 min, T = 50 °C	98	(Parida *et al.*, 2016)
Waste cooking oil	Homogeneous	NaOH (1%)	P = 500 W, f = 20 kHz, t = 1 min, T = 55 °C	99	(Khosravi *et al.*, 2016)
Waste cooking oil	Homogeneous	KOH (1%)	P = 500 W, f = 20 kHz, t = 1 min, T = 50 °C	99	(Khosravi *et al.*, 2016)
S. triguga oil	Homogeneous	$Ba(OH)_2$ (3%)	P = 250 W, f = 20 kHz, t = 80 min, T = 50 °C	90.8	(Sarve *et al.*, 2016)
Waste cooking oil	Homogeneous	KOH (1%)	P = 400 W, f = 25 kHz, t = 10 min, T = 60 °C	96.5	(Aghbashlo *et al.*, 2018)
R. trisperma oil	Homogeneous	H_2SO_4 (2%)	P = 1000 W, f = 40 kHz, t = 150 min, T = 55 °C	95.3	(Wongwuttanasatian and Jookjantra, 2020)
Castor oil	Homogeneous	KOH (3%)	P = 560 W, f = 20 kHz, t = 6 min, T = 42 °C	97	(Florez Marulanda and Ortega Alegria, 2019)
Palm oil	Heterogeneous	CaO (8%)	P = 140 W, f = 68 kHz, t = 37 min, T = 50 °C	96.2	(Aghbashlo *et al.*, 2018)
Rapeseed oil	Heterogeneous	Li/Fe_3O_4	P = 1000 W, f = 37 kHz, t = 35 min, T = 35 °C	99.8	(Fallah Kelarijani *et al.*, 2020)
Waste cooking oil	Heterogeneous	CaO (1.5%)	P = 1000 W, f = 20 kHz, t = 60 min, T = 70 °C	98	(Aghbashlo *et al.*, 2018)
Waste cooking oil	Heterogeneous	CaDG (1%)	P = 120 W, f = 22 kHz, t = 30 min, T = 60 °C	93.5	(Gupta *et al.*, 2015)
Karabi oil	Heterogeneous	CaO (5%)	P = 50 W, f = 20-30 kHz, t = 120 min, T = 60 °C	94.1	(Yadav *et al.*, 2018)
Jatropha oil	Heterogeneous	K_3PO_4 (1%)	P = 600 W, f = 20 kHz, t = 45 min, T = 50 °C	98	(Jogi *et al.*, 2016)
Jatropha oil	Heterogeneous	SrO-CaO (6%)	P = 210 W, f = 20 kHz, t = 30 min, T = 65 °C	95.4	(Ali *et al.*, 2017)
Jatropha oil	Heterogeneous	TPA/AC (4.23%)	P = 400 W, f = 20 kHz, t = 40 min, T = 65 °C	91	(Badday *et al.*, 2013a)
Waste cooking oil	Heterogeneous	SO_3H-CD (11.5%)	P = 300 W, f = 20 kHz, t = 8.8 min, T = 117 °C	90.8	(Badday *et al.*, 2013b)
Oleic acid	Heterogeneous	PTA-MOF (30%)	P = 100 W, f = 20 kHz, t = 15 min, T = 30 °C	98	(Nikseresht *et al.*, 2017)
Kernel oil	Enzymatic	Lipase (3%)	P = 140 W, f = 25 kHz, t = 4h, T = 25 °C	55.2	(Murillo *et al.*, 2019)
Waste cooking oil	Enzymatic	Lipozyme TLIM (3%)	P = 80 W, f = 25 kHz, t = 3h, T = 25 °C	96.1	(Subhedar and Gogate, 2016)
Crambe oil	Enzymatic	Novozym® 435 (20%)	P = 80 W, f = 20 kHz, t = 6h, T = 30 °C	98.2	(Tavares *et al.*, 2017)

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Yasvanthrajan and coworkers conducted an ultrasonic assisted transesterification method to boost the process efficiency of biodiesel production from waste cottonseed oil using the immobilized lipase enzyme as the catalyst (Yasvanthrajan *et al*., 2021). This approach effectively decreased the time for reaction to 6 h with low amount of enzyme (5 %w) and relatively milder temperature condition (45°C). Moreover, the immobilized lipase remains intact during this ultrasonic assisted transesterification process and has a great reusability up to four consecutive cycles. Therefore, this emerging approach can be adapted to provide the more efficient biodiesel production process. Another study by Oliveira and co-workers reported that the removal of external heating source and mechanical agitation is beneficial for ultrasonic assisted transesterification process (Oliveira *et al*., 2021).

2.2.3. Supercritical transesterification

The supercritical transesterification process is one of the modern methods in biodiesel production, which is carried out without the presence of catalysts. This process allows direct transesterification of triglycerides and esterification of free fatty acid into biodiesel (Farobie and Matsumura, 2017). The supercritical process also improves the solubility and mass transfer properties of the reaction. In this process, the reactants containing oil and alcohol are transferred into a reactor with supercritical operating conditions. The temperature and pressure conditions are above the critical point of the alcohol, with methanol having a critical temperature and pressure of 240°C and 1140 psi, respectively (Deshpande *et al*., 2017). In this supercritical condition, the chemical and physical properties of methanol change such as density, diffusivity, polarity, and viscosity (Bernal *et al*., 2012). Previous research has shown that some influential parameters in supercritical transesterification include pressure, temperature, type of alcohol, reaction time, and oil to alcohol ratio (Deshpande *et al*., 2017; Farobie and Matsumura, 2017). According to the previous research on biodiesel production using supercritical transesterification, it was discovered that in the supercritical region, methanol disintegrated is a free monomer due to the weakening of hydrogen bonding (Lee and Saka, 2010). The free methanol monomer reacts with carbonyl groups of the triglyceride that generates methoxide transfer and form the methyl ester and diglyceride that further transform into biodiesel and glycerol.

The supercritical transesterification was firstly studied by Lee and Saka and they found that the thermal decomposition of biodiesel (ester) may occur at high temperature condition and ambient pressure, which resulting in lower biodiesel quality (Lee and Saka, 2010). However, at high temperature and pressure condition in supercritical process, the transesterification reaction is easier to occur due to the formation of free monomer of the reactants. Yin and coworkers investigated a biodiesel synthesis using soybean oil in supercritical condition in a high pressurized vessel (Yin *et al*., 2008). They found that the ester conversion was 95% at 10 min of process at a temperature of 350 °C, whereas at 260 °C, it was only 30% of ester conversion after 60 min. Another study by Tsai and coworkers reported that waste cooking oil showed better efficiency compared to the refined cooking oil due to its free fatty acid content (Tsai *et al*., 2013). The ester yield was 65% at 300 °C and 100 bar within 4 min.

Even though the supercritical process seems to generate many benefits, including, no pretreatment needed, catalysts free, no soap formation, wastewater free, and faster process. However, the high expensive operational cost is a significant limitation due to the supercritical process that requires high temperature and pressure (Han *et al*., 2005; Kusdiana and Saka, 2004; Yin *et al*., 2008). Some simulation, environmental life cycle assessment, and feasibility studies reported that the supercritical process provided high biodiesel production capacity, faster process, technical benefits, and significantly reduce wastes compared to the conventional process in biodiesel production (Han *et al*., 2005; Kusdiana and Saka, 2004; Lee and Saka, 2010; Yin *et al*., 2008b). The operational efficiency of supercritical technology can be achieved by planning an efficient heat flow plant using Pinch technology (Pleşu *et al*., 2015).

2.2.4. Liquid phase plasma discharge technology

In recent years, plasma discharge technology has been applied in biodiesel production. Moreover, plasma is the ionized gas phase that contains electrons, ions, as well as neutral particles (Mostaghimi and Boulos, 2015), and can be obtained in two forms, namely, thermal and cold plasma (Takai, 2008). The thermal plasma is generated when at a high temperature of at least 3500°C, which is the equilibrium temperature between ions and electrons (Mostaghimi and Boulos, 2015). However, it cannot directly be used for reaction due to its high temperature, which decomposes all reactants, and makes the reaction uncontrollable. Currently, liquid phase plasma discharge (LPPD) was introduced as the controllable plasma technology and has been used in several applications such as nanomaterials, catalyst, organic compounds degradation, and triglyceride cracking for biodiesel production (Du *et al*., 2007; Meeprasertsagool *et al*., 2017). The energy input of LPPD can easily be adjusted for certain purposes. The plasma introduction in biodiesel production can be conducted in the non-catalytic and catalytic systems.

The plasma can transform triglyceride into diglyceride and monoglyceride for easy ester formation through transesterification (Khani *et al*., 2015), which also increases the rate of reaction due to the massive triglyceride bond breaking (Gharibi *et al*., 2015). During the plasma discharge assisted transesterification process, electrons with high energy run through the electrode with high voltage to the other electrode in ground state and excite the bulk gas molecules. This gas molecules excitation generates the ionization of atoms and forming the metastable species such as the reactive radical molecules (Wu *et al*., 2020). The collisions between the reactive species and the metastable species with the reactant molecules result in the production of variety compounds. Specifically, during the plasma assisted transesterification of oil feedstock, the C-C bonds in the triglyceride split, with the presence of methanol as the oxygen donor in the reaction, the oxygen molecules replace the C-C double bonds, subsequently result in the saturated

molecules (Kongprawes *et al*., 2021). Istadi and coworkers conducted a study of plasma assisted transesterification process using palm oil as a feedstock. They found that using a plasma voltage of 10 kV, electrode distance of 1.5 min, and a reaction time of 2 min, they achieved ester yield of 75.65% (Istadi *et al*., 2014). Similarly, another study also reported that plasma introduction in the transesterification is a promising method for efficient biodiesel synthesis (Almarashi *et al*., 2020).

Table 3 shows the recent biodiesel production using plasma discharge technology. This showed that plasma technology provides a new approach to initiate biodiesel formation using a different form of energy compared to the conventional method. It has process flexibility that can be carried out in the catalytic and non-catalytic systems. However, further researches are needed to determine the optimum operating conditions and understand the reaction mechanisms to make the method more controllable. As a summary for an overview of advanced technology in biodiesel production, the comparison including advantages and disadvantages of the discussed advanced process is summarized in Table 4.

Table 3
Comparison of several plasma discharge-assisted biodiesel productions.

Feedstock	Type of catalytic process	Catalyst	Operating conditions	Ester yield (%)	Ref.
Palm oil	Non-catalytic	-	Coaxial plasma discharge, Voltage = 220 V, Flowrate = 0.1318 L/min	10.1	(Fan *et al*., 2018)
Mixed oil	Non-catalytic	-	Dielectric barrier plasma discharge, Voltage = 10.2 kV, t = 120 min, 40 °C	65	(Nabilla *et al*., 2019)
Palm oil	Non-catalytic	-	Dielectric barrier plasma discharge, Voltage = 10 kV, t = 120 min, Ar gas	56.25	(Nabilla *et al*., 2019)
Mixed oil	Non-catalytic	-	Dielectric barrier plasma, Voltage = 10 kV, t = 120 min, 40 °C	60.72	(Nabilla *et al*., 2019)
Used oil	Non-catalytic	-	Dielectric barrier plasma, Voltage = 10 kV, t = 120 min, 50 °C	56.25	(Zara *et al*., 2020)
Used oil	Non-catalytic	-	Dielectric barrier plasma, Voltage = 10 kV, t = 100 min, ambient condition	70	(Zara *et al*., 2020)
Soybean oil	Homogeneous	NaOH (0.8%)	LPPD plasma, V = 1.2 kV, Flowrate = 2.7 mL/s	99.5	(Wu *et al*., 2019b)
Ethyl acetate	Homogeneous	$NaOCH_3$ (1%)	Dielectric barrier plasma, Voltage = 15.6 kV, t = 90 min, ambient condition	77	(Oliveira Palm *et al*., 2022)
Ethyl acetate	Homogeneous	H_3PMo (1%)	Dielectric barrier plasma, Voltage = 15.6 kV, t = 90 min, ambient condition	90	(Oliveira Palm *et al*., 2022)
Oleic acid	Homogeneous	H_2SO_4 (1.25%)	Dielectric barrier plasma, Voltage = 2.2, t = 4 min, ambient condition	80	(Wu *et al*., 2020)
Oleic acid	Homogeneous	H_2SO_4 (1%)	Dielectric barrier plasma, Voltage = 2.12, t = 2 min, ambient condition	78	(Bashir *et al*., 2021)
Waste cooking oil	Homogeneous	NaOH (1%)	Flying jet plasma DBD, Voltage = 2.5 kV, t = 45 min	95.4	(Abdul-Majeed *et al*., 2016)
Rape straw	Heterogeneous	Zn-Ti-Zeolite (3%)	Power = 15 – 45 W	25	(Wu *et al*., 2019a)
Soybean oil	Heterogeneous	K_2O/CaO-ZnO (6%)	Dielectric barrier plasma, Voltage = 5 kV, t = 1.25 min, 65 °C	77.19	(Buchori *et al*., 2017a)
Soybean oil	Heterogeneous	Activated carbon	Dielectric barrier plasma, Voltage = 7 kV, t = 5 min, 65 °C	92.3	(Buchori *et al*., 2017b)
Soybean oil	Heterogeneous	Sulfonated ZnO	Dielectric barrier plasma, Voltage = 7 kV, t = 1.25 min, 65 °C	56.91	(Buchori *et al*., 2017b)
Sunflower oil	Heterogeneous	MgO (3%)	Glow discharge plasma, 45 min	95	(Rahmani Vahid *et al*., 2017)

Table 4
Comparison of the advanced process of biodiesel production.

Advanced method	Main properties	Pros	Cons	Ref.
Microwave-assisted method	• T = 40 – 100 °C • t = 5 - 60 min • Catalyst = 0.5-5% • Energy = 500 - 800 W • Yield = 80 - 99%	• Efficient heating • Uniform heat distribution • Faster process • High yield • Low wastes • Easier separation • Less energy lost	• Low product quality • Scale up problem • Need high voltage • Not efficient with high amount of catalyst	(Binnal *et al*., 2021; Mamo and Mekonnen, 2020)
Ultrasonic-assisted method	• T = 40 – 65 °C • t = 10 – 240 min • Catalyst = 1-6% • Frequency = 25 – 60 kHz • Energy = 1.4 kWh/m^3 • Yield = 90 - 99%	• Reduce reaction temperature • Improve emulsion formation • Faster process • Improve product yield • Reduce alcohol requirement • Low production cost	• High electrical energy consumption • Difficult equipment design • Require high frequency	(Florez Marulanda and Ortega Alegria, 2019; Stavarache *et al*., 2007)
Supercritical transesterification	• T = 280 - 350 °C • t = 20 – 30 min • Catalyst = catalyst free • P = 5 – 40 MPa • Yield = 10 - 94%	• Catalyst free • No pretreatment • Short residence time • Low wastes • Available for all types of feedstocks	• Dangerous process • Need high safety during process • High energy requirement • Need hi-tech equipment • High temperature and pressure • Costly process	(Deshpande *et al*., 2017; Lee and Saka, 2010)
Plasma discharge technology	• T = 20 - 50 °C • t = 0.02 - 2 min • Catalyst = 0.5 – 3% • Energy = 100 – 300 W • Yield = 78 - 99%	• Less loss of energy • Faster reaction • Shortest residence time • Low energy of activation • Low catalyst dosage	• Difficult to control • Expensive process • Still emerging technology • Complicated reactor design	(Mostaghimi and Boulos, 2015; Takai, 2008)

3. Parameters in biodiesel production

Several factors affecting the performance of biodiesel production have been previously reported. Meanwhile, the type of biodiesel feedstock, type of catalysts, and operating conditions are discussed in this following sections as previously described in Fig. 1.

3.1. Types of feedstocks

The initial generation of biodiesel derived from agricultural goods such as peanut, soybean, and canola have implications on food production and the environment for people's use. In the early 1940s, 5% of biodiesel with gasoline as an alternative fuel was used for vehicles. Fig. 3 shows the classification of the biodiesel feedstock generations. In this first generation of biodiesel, all raw oily feedstocks were derived from edible agricultural products such as soybean, palm oil, and peanuts, due to their ease of handling and large availability (Kumar *et al*., 2020; Noriega and Narváez, 2020). Meanwhile, the second-generation biodiesel was generated using cellulose substrates such as short-rotation trees, grassland plants, and urban trash. Jatropha is a non-edible feedstock, which necessitates a large quantity of farmland and produces low oil (Baskar *et al*., 2018; Keera *et al*., 2018; Saputra Nursal *et al*., 2021). Based on current research, the development of alternative ways to lessen food farmland rivalry was continued until the discovery of microalgae as a reliable and rich biofuel feedstock recognized as the third-generation of biodiesel. Microalgae-based biodiesel is a feasible third-generation option since it has no impact on food sources (Abo *et al*., 2019; Alami *et al*., 2021; Saputra Nursal *et al*., 2021). It can also be used for both effluent phytoremediation and fabrication of biodiesel and also to remove carbon dioxide from the air (Abo *et al*., 2019). Furthermore, algae may be cultivated on any accessible surface such as lands, lakes, oceans, and generate more triglycerides that have been identified as a possible source for the production of biodiesel.

The accessibility of oil sources is a crucial determinant of biodiesel production's economic viability because it contributes approximately 80% of the overall cost of biodiesel (Kumar *et al*., 2020; Noriega and Narváez, 2020; Hadiyanto *et al* 2020). Various attempts have been devoted to determining a low-cost feedstock that is available throughout the decade. Compared to standard fuels, FAME from algae, plant oils, and animal fats have been demonstrated to be a source of biodiesel synthesis due to enhanced combustion behavior, volatility, and viscosity. Fish oils and beef tallows are the most common animal fat sources, while vegetable oils such as rapeseed oil, castor berry, palm pulp, palm kernel oil, sunflower seeds, coconut kernel, cottonseed, peanut grain, and canola seed were used in the biodiesel manufacturing process (Athar and Zaidi, 2020; Pinzi *et al*., 2014; Yusuff *et al*., 2021). Microalgae can increase their biomass in 24 hours, with the quickest half-life of approximately 3.5 hours, which makes them an attractive sustainable source for biodiesel synthesis (Abo *et al*., 2019; Alami *et al*., 2021; Saputra Nursal *et al*., 2021). Its enormous amount and use of richer

nitrogen, as well as phosphorus in wastewater as an inexpensive source of nutrients, benefits from algae cultivation. Algal species have the disadvantage of being obligatory phototrophs, which indicated that they need light to survive. The amount of FFA and contaminants in biodiesel also affects the kind of manufacturing technique employed and the amount of fuel produced. Similarly, lipid leftovers such as waste cooking oil and non-edible beef tallow have recently attracted much attention from the biodiesel industry. However, the discovery of new additional options that do not interfere with food sources is critical.

3.2. Types of catalysts

3.2.1. Homogeneous catalysts

Acids and alkalis are included in the homogeneous catalyst, where the alkalis such as sodium hydroxide (NaOH), sodium methoxide ($NaOCH_3$), and potassium hydroxide (KOH) are usually used (Vicente *et al*., 2004). Meanwhile, in the acidic homogeneous catalyst (H_2SO_4), sulfuric acid has been commonly used (Wongwuttanasatian and Jookjantra, 2020). KOH behaved well in the transesterification reaction of Karanja oil, yielding methyl esters above 95% at one percent of catalyst dosages in an hour of reaction at 60°C with a mixing rate of 7 rps (Boey *et al*., 2011). Saponification arises due to an undesirable by-product in a homogeneous catalytic reaction mechanism, which necessitates an extra separation procedure to eliminate catalytic pollutants and increases the final operating costs. In the homogeneous alkali catalytic reactions, (i) the optimal temperature is adjacent to the boiling temperature of the alcohol employed, (ii) sufficient alcohol is required to facilitate excellent conversion, where 1:6 is regarded as the ideal oil/Me-OH molar ratio (Dias *et al*., 2008). Furthermore, various basic catalysts such as $NaOCH_3$, $KOCH_3$, NaOH, and KOH for methanolysis of sunflower oil were compared with all reactions in an agitated reaction vessel and the following purification stages in a decanter under the test condition. Methoxides are more efficient than hydroxides, with a yield of around 100% (Boey *et al*., 2011). The benefits of using a homogeneous catalyst for biodiesel synthesis include low reaction temperatures, the high catalytic performance of base catalysts, and conversion in a relatively short period. The basic type of heterogeneous catalysts is also substantially more active than acid catalysts. After the processing and treatment of wastewater, saponification generates a stable colloid, the used catalysts that cannot be regenerated, basic catalysts which are delicate to the concentration of water and FFA are the key restrictions faced by homogeneous catalysts. The comparison of used catalyst for biodiesel production is listed in Table 5.

3.2.2. Heterogeneous catalysts

Heterogeneous catalysts have the potential for the transesterification process of plant oils to make biodiesel. Meanwhile, heterogeneous catalysts without homogeneous catalysts can be regenerated, renewed, and employed in continuous operations (Ganesan *et al*., 2021; Helmi *et al*., 2021; Jayakumar *et al*., 2021). Since their employment in the transesterification process substantially simplify and optimizes the post-treatment of the products, there seems to be a recent surge in the invention of acidic or basic solid heterogeneous catalysts for FAME generation (Jayakumar *et al*., 2021; Xie and Wang, 2020). Generally, heterogeneously catalyzed biodiesel manufacturing techniques have fewer processing steps, post-treatment stages, and do not entail neutralization. The kind of catalyst (acid or base), the catalyst quantity, the flow reaction time, the extent of stirring and mixing, the oil/alcohol percentage, and the quality of the feed affect heterogeneous catalysis (Deeba *et al*., 2020; Helmi *et al*., 2021; Xie and Wang, 2020). The detailed advantages and disadvantages of heterogeneous catalysts to homogeneous catalysts can be seen in Table 6.

Furthermore, the use of a solid catalyst can lead to faster and less expensive separation procedures, minimum wastewater discharge, lower production and energy expenditures (Rezania *et al*., 2022; Siddiquee *et al*., 2011). Solid catalysts can be carried out in the packed bed reactors as the catalyst bed that simultaneously perform catalytic reaction and separation. Therefore, the extra physical separation processes and their related operational expenses can be eliminated. Heterogeneous catalysts are also less harsh and are used in fixed-bed reactors, leading to a safer, better cost-effective, and eco-friendly process. Their advantage is that they consume less energy during the transesterification process (Awogbemi *et al*., 2021; Jayakumar *et al*., 2021; Rezania *et al*., 2022; Siddiquee *et al*., 2011). In this process, 10 tons of sodium hydroxide are needed to make 1,000 tons of biodiesel, while the heterogeneous requires only 0.7 tons of magnesium oxide solid catalysts to make 12,500 tons of biodiesel (Jayed *et al*., 2009). Although the heterogeneous process has shown a more effective catalyst to biodiesel product, there are still some challenges, which includes slow reactions, partial conversions, short lifespan, and expensive prices (de Oliveira and Coelho, 2017; Eguchi *et al*., 2015; Krishnan *et al*., 2021). Therefore, the homogeneous process is widely selected for biodiesel synthesis in the current industrial processes. The major drawback of a heterogeneous catalyst, which is its slower reaction rate compared to homogeneous catalysis can be solved by increasing the methanol-to-oil proportion, temperature, and pressure of the process (Alagumalai *et al*., 2021; Hamza *et al*., 2021; Krishnan *et al*., 2021; Zailan *et al*., 2021). These treatments have been reported to attribute to the faster rate and higher biodiesel yield using the heterogeneous catalytic process (Awogbemi *et al*., 2021; R. Ganesan *et al*., 2021; Hamza *et al*., 2021; Jayakumar *et al*., 2021; Krishnan *et al*., 2021; Patiño *et al*., 2021). To protect combustion damage to the engine, several pollutants such as unreacted FFAs, TAGs, glycerol, soaps, catalyst, mono- and diglyceride, water, and other impurities need to be separated from unrefined biodiesel by post-production purification stages, such as adsorption, filtration, ion-exchange, and other methods (Catarino *et al*., 2020; Gomes *et al*., 2015; Hajra *et al*., 2015; Li *et al*., 2012; Wang *et al*., 2021).

Inorganic-synthesized catalysts such as calcium oxide (CaO), silicon dioxide (SiO_2), zircon oxide (ZrO_2), iron oxide (Fe_2O_3), and others are usually used due to their low cost, accessibility, safety, robustness, and ease of regeneration (Hadiyanto *et al* 2016). Meanwhile, current studies have focused on low-cost, environmentally friendly, and incredibly efficient heterogeneous-base

catalysts, where calcium oxide-based from waste or natural sources has shown the most potential (Baskar *et al*., 2018; Jayakumar *et al*., 2021; S. Silviana *et al*., 2021b; Xie and Wang, 2020). Calcium-compounding shells, limestone, and lime mud have been proven to have superior catalytic characteristics, which make them appropriate for biodiesel generation (Ajala *et al*., 2020). Furthermore, they make the reaction more cost-effective, ecologically beneficial, and also reduce waste. This occurs because they needed a relatively small amount of catalyst for the biodiesel synthesis, which generates smaller liquid waste compared to the homogeneous catalyst process. Strong acid such as sulfuric acid that simultaneously facilitates both transesterification and esterification processes are particularly important for biodiesel synthesis from low-cost raw stocks with high FFA concentration (Binnal and Nirguna Babu, 2019). Therefore, the invention of the novel, more powerful, and less expensive solid catalysts is expected to reduce the overall cost of producing biodiesel from the various oily feedstock. A better catalyst activity can also be accomplished through porous materials support, which increases TAG and FFA transport to reactive acid/basic sites and improves the rate of the reaction. In addition, environmentally friendly innovations such as membrane reactors, ultrasounds, and microwaves can also be used to solve operational issues such as excessive power consumption, long response times, and poor catalytic performance, which increases the costs of production.

Table 5
Biodiesel production using several types of feedstocks, catalysts, and stoichiometric ratio of methanol and oil.

Type of catalyst	Feed oil	Catalyst addition (%wt)	Oil to methanol ratio	Ester yield (%)	Ref.
Homogeneous catalysts					
Sodium hydroxide (NaOH)	Sunflower oil	1.00	1:6	97.0	(Vicente *et al*., 2004)
Sodium methoxide ($NaOCH_3$)	Sunflower oil	1.00	1:6	99.0	(Vicente *et al*., 2004)
Potassium hydroxide (KOH)	Sunflower oil	1.00	1:6	91.7	(Vicente *et al*., 2004)
Potassium methoxide ($KOCH_3$)	Sunflower oil	1.00	1:6	98.0	(Vicente *et al*., 2004)
Sulfuric acid (H_2SO_4)	Oleic acid	1.25	1:3	80	(Wu *et al*., 2020)
Sulfuric acid (H_2SO_4)	Oleic acid	1.00	1:3	78	(Bashir *et al*., 2021)
Barium hydroxide ($Ba(OH)_2$)	Microalgae	3.00	1:6	90.8	(Sarve *et al*., 2016)
Chloro-sulfonic acid ($ClSO_3H$)	Maize oil	3.00	1:6	93.1	(Dall'Oglio *et al*., 2015)
Heterogeneous catalysts					
Calcium oxide (CaO)	Sunflower oil	1.00	1:12	91.0	(Boey *et al*., 2011)
Potassium fluoride (KF)	Tallow seed	4.00	1:6	96.8	(Jayakumar *et al*., 2021)
Potassium nitrate (KNO_3)	Rape oil	1.00	1:12	98.0	(Jayakumar *et al*., 2021)
Titanium oxide (TiO_2)	Canola oil	6.00	1:30	100	(Jayakumar *et al*., 2021)
Magnesium oxide (MgO)	Mutton fat	4.00	1:22	98.0	(Foroutan *et al*., 2020)
Aluminum oxide (Al_2O_3)	Palm oil	5.97	1:12	98	(Boey *et al*., 2011)
Zinc oxide (ZnO)	Used cooking oil	1.30	1:14	90	(Yusuff *et al*., 2021)
CeO_2/Li/SBA-15	Cotton seed oil	10	1:40	98	(Malhotra and Ali, 2018)
Cu impregnated TiO_2	Palm oil	2	1:20	90.93	(De and Boxi, 2020)
$NaAlO_2/Al_2O_3$	Palm oil	10.89	1:21	97.65	(Zhang *et al*., 2020)
Ca/APB-700	Waste cooking oil	5	1:30	93.4	(Wang *et al*., 2019)
$LiFe_5O8$-$LiFeO_2$	Soybean oil	8	1:36	96.5	(Dai *et al*., 2018)
Enzymes					
Lipase from *Rhizopus oryzae*	Soybean oil	1.00	1:3	85.0	(Adewale *et al*., 2017)
Lipase from *Aspergillus niger*	Canola oil	1.00	1:3	69.0	(Andrade *et al*., 2019)
Lipase/APTES-magnetite	Rapeseed oil	20	1:6	89.4	(Gojun *et al*., 2021)
Thermomyces lanuginosus/ magnetite/ Au NPs	Waste tomato oil	20	1:6	98.5	(Sarno and Iuliano, 2019)
Novozyme 435	Fish oil	50	1:35	82.9	(Marín-Suárez *et al*., 2019)
Palatase	Soybean oil	10	1:3	71	(Pedro *et al*., 2020)
Immobilized CLEAs of Km 12 lipase	Waste cooking oil	0.3	1:3	71	(Badoei-dalfard *et al*., 2019)
NS 40116 lipase	Chicken fat	0.3	1:4.5	77	(da Silva *et al*., 2018)
NS 40116 lipase	Soybean oil	3	1:6	96	(Mibielli *et al*., 2020)
Rhizopus oryzae lipase	Palm oil	1.5	1:3	91.3	(Muanruksa and Kaewkannetra, 2020)
Lipase immobilized Fe_3O_4-poly (GMA-co-MAA)	Soybean oil	1.5	1:4	92.8	(Xie and Huang, 2020)
Nano-catalysts					
Nano-CaO	Soybean oil	1.00	1:7	96.0	(Foroutan *et al*., 2020)
Nano-MgO	Soybean oil	3.00	2:3	99.0	(Foroutan *et al*., 2020)
Nano-KF	Canola oil	3.00	1:15	82.1	(Jayakumar *et al*., 2021)
Ni doped ZnO nanocomposite	Castor oil	1.40	1:12	91	(Baskar *et al*., 2018)
Diatomite CaO@MgO	Waste cooking oil	6	1:15	96.47	(Rabie *et al*., 2019)
Nano sized waste animal bone	Honge oil	7	1:12	96	(Chingakham *et al*., 2019)

Table 6
Comparison of biodiesel production using conventional homogeneous and heterogeneous catalytic process.

Type of production	Pros	Cons
Homogeneous	• Mild operating condition • The mixture of FFA and water has little effect on acid catalysts • Hydroxides are ineffective compared to methoxides • Catalysts that are alkali are substantially more active than those that are acidic • Both esterification and transesterification can use the same acid catalyst	• After-reaction separation issues with wastewater treatment • Acid catalysts are corrosive and have a sluggish reaction rate • The existence of FFA and water makes basic catalysts sensitive • The used catalyst cannot be regenerated • Needing more operating cost than heterogeneous process
Heterogeneous	• Product separation is easier, selectivity is better, and catalyst life is longer • Eco friendly, noncorrosive, reusable, and less problematic in terms of disposal • The existence of FFA and water has little effect on acid catalysts • Relatively low-cost compared to homogeneous process • Possibly used in continuous process using fixed-bed reactors	• The multi-phase reaction system has mass transfer restrictions • In comparison to conventional homogeneous base catalysts, it is apparently less effective • Acid catalysts are more expensive than basic catalysts • The raw oil with low water and FFA content are required for basic catalysts • Temperature and pressure, as well as a high alcohol-to-oil molar ratio, may be necessary • The catalyst is possibly leached out during process.

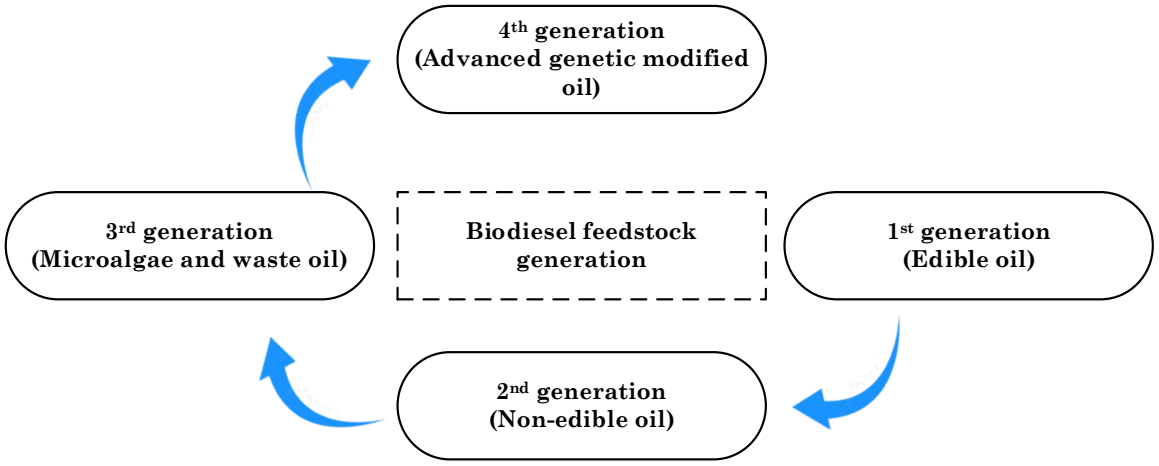

Fig. 3. Classification of biodiesel feedstock generations.

A transesterification method with base catalyst is used when an oil feed contains high water, FFA, and contaminants, such as used cooking oils. Extra consideration must be given to the production planning and catalyst selection especially more favorable on that condition with high contents of water, FFA, and contaminants (Deeba *et al*., 2020; Siddiquee *et al*., 2011). However, the design process needed an extremely high temperature, longer time of reaction, and equipped in corrosion resistance vessel. To overcome these challenges, a pre-esterification must be carried out using an acidic catalyst to convert FFAs into esters, followed by transesterification with a basic catalyst to also synthesize TGAs. Since both processes are reversible, a large amount of alcohol is frequently used to drive the process to the synthesis of methyl ester. Meanwhile, biodiesel synthesis is carried out in a single step through a bi-functional catalyst that can perform the processes simultaneously (Al-Saadi *et al*., 2020). Table 6 shows the advantage and disadvantage of homogeneous and heterogeneous biodiesel synthesis method relative to each other.

3.2.3. Enzyme-based catalysts

Lipases are currently the most used enzyme in biodiesel synthesis, grouped into the hydrolases process, which is an enzymatic process that hydrolyzes the

molecules of triglyceride into fatty acids and glycerol. The lipase enzyme can be obtained from numerous sources such as bacteria, plants, fungi, and animals. Recently, the lipase enzyme from microbes (bacteria and fungi) is commonly used as biodiesel catalysts (Adewale *et al*., 2017; Andrade *et al*., 2019; Chang *et al*., 2021). Some advantages of enzyme-based catalysts in biodiesel synthesis include an environmentally friendly type of catalysts that are derived from microbes and can carry out the transesterification reaction in the high level of free fatty acids and water. Moreover, the enzymatic catalysis reacts in the mild ambient condition that caused a lower energy requirement, which shows the sustainability of this process for biodiesel production (Marín-Suárez *et al*., 2019). Immobilized lipase differs from pure lipase because it can be easily recovered from the reaction medium, allowing for repeated usage. The efficiency of the enzymatic process is great, and the enzyme can be possibly immovable upon the solid matrix, which includes the amount of biodiesel that is retrieved (Marín-Suárez *et al*., 2019). There are several reaction pathways of the biodiesel synthesis using enzyme catalysts. The active sites on the enzymes attributing the positive or negative charge initiate the reaction by accepting or releasing protons via the Bi-model mechanism based on the enzyme types (Pedro *et al*., 2020). The lipase enzyme plays as the proton acceptor of hydroxyls (OH^-), while on the amine group-based enzymes, they release protons (Pedro *et al*., 2020; Sarno and Iuliano, 2019).

The main drawback in the use of enzymatic catalysts is the high cost of extraction from the sources which usually appears in low yield. Therefore, research was carried out on the integration of enzymes with several nanomaterials to increase the catalyst activity and the yield of biodiesel. This includes the use of the amino-coated iron oxide (Fe_3O_4) material, which was crosslinked with the Km12 lipase enzyme (Sarno and Iuliano, 2019). It was discovered that when stored for 24 days at a temperature of 4 ^oC, it gave a 60% higher initial enzymatic activity compared to the pristine enzyme. The current works of enzymatic catalyst utilization in biodiesel production with their findings are summarized in Table 5. By carefully examining the data in Table 5, this indicated that enzymatic catalysts averagely gave lower biodiesel yield compared to homogeneous and heterogeneous catalysts. Furthermore, the results showed that different types of enzymes, feedstocks, and oil to alcohol ratios gave different yields of biodiesel. Therefore, selecting an appropriate enzyme and operating parameter is essential to achieve a desirable yield of biodiesel.

3.2.4. Nano-catalysts

The heterogeneous catalysts have a different phase from the reactants in biodiesel production, which can be attributed to the acidic and alkali properties based on the chemical composition (Athar and Zaidi, 2020; Jayakumar *et al*., 2021; Kumar *et al*., 2010). Their use considerably reduces the number of separation treatments compared to the homogeneous ones. However, lower conversion carried out by the common heterogeneous catalysts initiated the scientists and engineers to develop higher efficient catalyst by synthesizing in form of nanoparticles. The heterogeneous catalysts that appeared as nanomaterials are commonly named the nano-catalyst (Naveenkumar and Baskar, 2020). Meanwhile, research has been carried out on the influence of nano-catalyst on triglycerides transesterification to FAMEs (Ashok *et al*., 2018; Dehghani and Haghighi, 2020; Foroutan *et al*., 2020; Liu *et al*., 2020), as the result can be seen in Table 5. The transesterification process of soybean oil production is improved using MgO as a catalyst (Rahmani Vahid *et al*., 2017). This process uses ferromagnetic ZnO nanocomposite as a solid catalyst for generating biodiesel from castor oil as a feedstock (Baskar *et al*., 2018). Research on X-ray diffraction (XRD) showed that the nanoparticles were in a single phase. However, after six cycles of soybean oil and four cycles of poultry fat, biodiesel made from soybean oil using CaO nanoparticles under ambient temperature exhibited a poor speed of the reaction and needed six to 24 hours to achieve a high yield (Foroutan *et al*., 2020). The use of nanomaterial is costly, but it produces more than 90% of biodiesel conversion. Nanomaterials can provide larger active site for catalytic reaction compared to the conventional heterogeneous catalysts.

3.3. Operating condition

3.3.1. Temperature

One of the most critical elements that affect the output of biodiesel synthesis is the process temperature. This is because an increase in temperature and a decrease in oil viscosity produced a faster response speed. However, increasing the temperature above the optimal level reduces biodiesel yield caused by the higher temperature, which accelerates triglyceride soap formation. To minimize the alcohol loss, the operating temperature must be lower than the alcohol's boiling temperature. Generally, the optimal temperature for biodiesel synthesis is between 50 and 60°C based on the oil quality and the catalyst (Al-Saadi *et al*., 2020; Deeba *et al*., 2020; Krishnan *et al*., 2021).

3.3.2. Alcohol and oil ratio

Alcohol and oil commonly have a 3:1 stoichiometric ratio, however, excessive use of alcohol is harmful to the advanced transesterification reaction (Sawangkeaw *et al*., 2011). The most widely known alcohols used in the transesterification process are methanol and ethanol. In a 2.5-hour reaction period at 65°C with a 10:1 methanol/oil stoichiometric ratio and 450 rpm of stirring, approximately 80% biodiesel production was achieved from soybean oil with 0.1 percent $NaOCH_3$. It was also stated that waste cooking oil converted to FAME in 69 hours at a temperature of 65°C and a stoichiometric ratio of 1:3 oil to methanol (Marchetti *et al*., 2007; Sawangkeaw *et al*., 2011).

3.3.3. Agitation

Since the mixing of the oil feed and catalyst combination promotes the process, agitation speed is significant in the synthesis of FAME (Demirbas, 2010; Elgarhi *et al*., 2020). For illustration, the mixing intensities of 200-800 rpm were selected for 1 hour, while other variables remain unchanged and the final product conversion rate was greater at 400 rpm (Demirbas, 2010). Moreover, soap production happens at greater mixing speeds, while byproduct creation occurs at slower agitation

speeds due to the transesterification reaction's reversible character.

4. Biodiesel purification method

The purification treatment aims to separate the generated soap, glycerol, water, unreacted alcohol, and catalysts from biodiesel products. The generated esters should be free of those impurities to fulfill the standard product of biodiesel. Moreover, the different production processes of biodiesel caused a variation in the product specification, as shown in Table 5. Based on the mechanisms, the purification techniques that have been used since last decades for biodiesel until now can be grouped into wet washing, dry washing, ion exchange and precipitation, complexation, membrane-based separation, adsorption, as well as simultaneous synthesis and purification.

4.1. Wet washing method

The wet washing method is operated using pure or acidified water (Díaz-Ballote *et al*., 2020; Mendow *et al*., 2012; Shirazi *et al*., 2013). Before the process, water is used at ambient temperature or as heated as possible, and the extra alcohol is occasionally removed by distillation. The use of acidified water was reported to be more effective than pure water (Iglesias *et al*., 2014). Meanwhile, several dilute acids that are commonly used include phosphoric acid, hydrochloric acid, and sulfuric acid. Although the process needs a lower amount of acidified water than pure water to treat the same amount of unrefined biodiesel, the use of acidified water can acidify the biodiesel product. In industry, this problem is usually addressed by adding the pure water wash at the end of the acidified water washing to remove the spent acid content in the biodiesel product (Gomes *et al*., 2015). Currently, the ionic liquid compounds have been used in the wet washing process such as organometallic substances, which were added into the wash water to improve the efficiency and effectiveness (Veljković *et al*., 2015). The benefits of the wet washing method include a very convenient and straightforward approach for biodiesel refining, extremely useful separation of glycerol and alcohol, as well as the ability for using aqueous acids (Veljković *et al*., 2015). Table 7 shows some reported works regarding the wet washing process. However, some drawbacks still appear such as the need for a large amount of water, product drying to separate the detectable water content, increase in the consumption of energy, greater surface area for washing unit, and generating a large amount of wastewater that contain hazardous substances, which is difficult to be treated.

4.2. Dry washing method

Dry-cleaning was created to replace biodiesel purification with no-water purifying processes that are more eco-friendly, where chemicals such as adsorbents and acidic polymers are used to remove contaminants from unrefined biodiesel (Catarino *et al*., 2020; Gomes *et al*., 2015). After mixing for 20 minutes at 55°C, the unrefined biodiesel was treated with 2% magnesol and the adsorbent was recovered by filtering (Zhu *et al*., 2006). However, to increase the separation efficiency, the refined biodiesel from dry washing process is sometimes washed with pure water in a stirred tank unit (Ilmi *et al*., 2017). The biodiesel phase is separated from the mixture by decantation and centrifugation.

Another type of adsorbent that has been applied in biodiesel purification was silica derived from geothermal solid waste, which is one of the potential silica sources (Silviana *et al*., 2021). Previous research reported that the silica adsorbent has an optimum glycerol adsorption capacity of 10.06 mg/g (Silviana *et al*., 2021a). The benefits of dry washing include no threat of water, continuous process, a reduction in overall process time, and wastewater. However, the drawbacks include adsorbent that do not eliminate alcohol, the requirement for additional apparatus, and slightly higher operating expenses.

4.3. Ion-exchange and precipitation

Precipitating agents are used to separate Ca^{2+} ions from raw biodiesel in this technique. If a precipitation chemical, like oxalic and citric acid are introduced to calcium-containing unrefined biodiesel, an insoluble substance forms in the mixture (Musiał *et al*., 2011; Vieira *et al*., 2017). Another study showed that washing the crude biodiesel in a mixture of sodium carbonate (Na_2CO_3) and methanol at boiling temperature. Calcium soap have a more non-polar property, which being less soluble in water and more soluble in biodiesel, thus increases the difficulty in separation. After exchanging calcium ions by sodium ions, the generated sodium soaps can be easier to remove using wet or dry method (Musiał *et al*., 2011; Vieira *et al*., 2017). This process was carried out to exchange the calcium ions from the crude biodiesel with the sodium ions from sodium carbonate to form the water-insoluble calcium carbonate, with reaction as follows (Veljković *et al*., 2015):

$$Ca^{2+}_{(aq)} + Na_2CO_{3(aq)} \rightarrow CaCO_{3(s)} + 2Na^{+}_{(aq)}$$

Methanol is used to prevent ester hydrolysis and as a reagent in the transesterification process. The formed calcium carbonate has water-insoluble properties that are easily separated while the remaining sodium ions can be removed using wet or dry washing methods.

Further development in ion exchange and precipitation method has been carried out as a pretreatment process, followed by subsequent acid resin, ceramic membrane, and wet washing (Hajra *et al*., 2015; Li *et al*., 2012). At the beginning of the process, the crude biodiesel was treated using methanol containing sodium carbonate and was mixed under a constantly stirred vessel at 60°C for 5h at a speed of 1,200 rpm. The calcium ions were exchanged by sodium ions to generate the calcium carbonate. The calcium carbonate and unreacted sodium carbonate were separated using conventional filtration, while methanol was separated with gravitational settling treatment. Moreover, centrifugation or filtration can be used to remove the deposited component from pure biodiesel. The microfiltration membrane and wet washing method (pore size 0.1 μm) were also used to remove the sodium ions and the results are compared (Li *et al*., 2012). Overall, the pretreatment and membrane filtration or wet washing methods were efficient in sodium and calcium removals from crude biodiesel.

Table 7
Summary of some recent works using wet washing as purification method of biodiesel.

Biodiesel production			Purification		Findings	Ref
Feed oil	**Catalyst**	**Operating conditions**	**Method**	**Operating conditions**		
Brown grease	ZnO/ZrO_2	Autopressure, 200 °C, 2h	Wet washing: pure water	Washing and product drying 24 h	The product meets the ASTM diesel fuel standard for sulfur content below than 500 ppm	(Kim *et al.,* 2011)
Palm oil	SrO_2	Stirred batch reactor, 65 °C	Wet washing: distilled water	Washing, centrifuging, and drying	Wet washing slightly reduced the biodiesel density, while increased in viscosity	(Salamatinia *et al.,* 2012)
Crude palm kernel	Sulfonated ZrO_2	Standard batch reactor. 200 °C, 50 kPa, 1 h	Wet washing: hot acidified water	Washing and drying	The product meets the ASTM standard for biodiesel	(Jitputti *et al.,* 2006)
Curcas oil	Na/SiO_2	Ultrasonic equipped batch reactor, 24 kHz, 15 min	Wet washing: hot water	Washing and drying	The product meets the ASTM standard for biodiesel	(Kumar *et al.*, 2010)
Crude palm oil (CPO)	SBA-15 functionalized ZrO_2	Up flow fixed-bed reactor, 200 °C, 70 kPa, 6h	Wet washing: two times washing	Washing and drying	The product meets the ASTM standard for biodiesel	(Iglesias *et al.,* 2014)
Waste cooking oil	KOH	Continuous centrifugal contactor separator (CCCS) 10 Hz, 35 °C	Wet washing: pure water	Washing and centrifugation	Optimized result was 96% of ester yield and 75% energy saving.	(Fayyazi *et al.,* 2021)
Sunflower oil	CaO	Continuous centrifugal contactor separator (CCCS) 60 °C, spacetime 0.05h	Wet washing: pure water	Washing and centrifugation	Optimized result was 83.2% of ester yield and productivity of 638 kg/m^3h	(Fayyazi *et al.,* 2018)
Sunflower oil	TransZyme A	Continuous centrifugal contactor separator (CCCS)	Wet washing: pure water	Washing and centrifugation	85% mol of ester yield was achieved during 9 h of process	(Ilmi *et al.*, 2017)
Waste cooking oil	KOH	Prewashing and wet washing	Wet washing: pure water	Prewashing and wet washing	Purified biodiesel meets the standards required to be a replacement for diesel. This approach reduces 60% consumption of fresh water in the purification process	(Bashir *et al.*, 2018)
Waste cooking oil	KOH	Wet washing and repeating microfiltration	Wet washing: pure water	Wet washing and repeating microfiltration	Up to 15% less water consumption after two rounds of biodiesel production operations. Considerably less wastewater generation toward zero discharge-based operation	(Jaber *et al.*, 2015)

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This showed that the benefits of the ion exchange method include a good output of pure biodiesel, a lower volume of water, and easy filtering of the residue. The drawbacks include the dependence of the effective precipitation on operational parameters; therefore, additional investigations are necessary to improve the precipitation settings.

4.4. Complexation

Complexation is a method for biodiesel purification, causing the incorporation of chemical groups, atoms, or ions to form a larger molecule. This method is currently used to separate Ca^{2+} ions using ethylenediaminetetraacetic acid (EDTA) as the initiator to form the complexation with a molar ratio of 1:1 of EDTA to calcium ions. When the EDTA solution is added to the raw biodiesel product, the reaction takes place in a stirring condition and complex substances between EDTA and Ca^{2+} are formed (Zhu *et al*., 2006). The Ca-EDTA residue can be removed from the mixture through centrifugation, where supernatant is biodiesel and the residue is the Ca-EDTA (Zhu *et al*., 2006). Decalcification can achieve 84% of efficiency with 92% yield in biodiesel. Some benefits of using this method include a high amount of calcium ions that can be separated from the raw biodiesel and soluble in water. However, the disadvantages include the difficult preparation of EDTA and are not fully eco-friendly.

4.5. Membrane-based separation

Membrane filtration is a well-known modern technology that is usually applied in water science, protein separation, and purification purposes. The technology is commercially used in aqueous mixture separations, however, the purification of the non-aqueous mixture by the membrane is currently thriving. The membrane can be in form of organic, inorganic, or a combination of both. Since they are thermally and chemically stable, the former type especially for the inorganic ceramic membrane has been applied in a cross-flow system to separate triglycerides from FAMEs mixture (Shirazi *et al*., 2013; Sokač *et al*., 2020; Bansod *et al*., 2021; Goswami and Pugazhenthi, 2021). The pore diameter for the oil emulsion was set to be about 40 microns. Ultrafiltration and tubular ceramic membrane are the most efficient for biodiesel purification that attributed the environmentally friendly technology compared to the other type of membranes. Several benefits of the membrane-based technology in biodiesel purification include high rejection of unreacted alcohol and generated soap, simple and easy to operate, low energy consumption, easy to control and scale-up, and eco-friendly. However, some of its drawbacks include the membrane that needs to be cleaned up periodically and flux reduction gradually over time. The collective information regarding previous studies results about biodiesel purification can be seen in Table 8.

4.6. Simultaneous synthesis and purification of biodiesel

Research has shown that the simultaneous synthesis of biodiesel formation and purification enhances the overall manufacturing process. Such coupled mechanism has been studied so far (Gojun *et al*., 2021; Song *et al*., 2021). It can possibly overcome several drawbacks of current purification methods for biodiesel as shown in Table 8. During the ester formation, they introduced ion exchange preceded by precipitation. It has also developed a bench-scale biodiesel synthesis and purification simultaneously using a reactor equipped with packed anion- and cation-exchange resins at a temperature of 50°C. The cation-resin was firstly introduced to the reactor to accelerate the esterification of FFA, while the anion-resin was loaded after that for transesterification of TAG and also as the adsorbent for the purification treatment of the generated biodiesel. Subsequently, the biodiesel was generated at a flowrate of 0.23 l/h having a TAG content lower than the standard (Veljković *et al*., 2015). The glycerol, water, alcohol, and unreacted FFA as well as TAG were adsorbed onto the anion exchange resin. Therefore, this process is called simultaneous biodiesel formation and purification because they take place at the same time. Several advantages of this method include shorter time for the processing of the oily feedstock to biodiesel, does not require additional equipment, and is relatively cheaper than the conventional method. However, it has some drawbacks such as the resins needed to be regenerated periodically and properly designed to adsorb various types of impurities with different chemical properties.

5. Impacts of biodiesel

In previous years, a substantial body of knowledge about the socioeconomic and environmental implications of biodiesel has been accumulated. The petroleum-based fuel reserve will be drained in the next few years approximately at 2030 due to the rapid increase of people's needs, which will generate the greatest issue of the 21st century (Edwan Kardena, 2015). Based on the Organization of Petroleum Exporting Countries (OPEC), global fuel oil consumption is forecast to reach approximately 100 million barrels a day by 2040, with 5.0 million barrels of diesel fuel consumption a day. Furthermore, the unpredictable petroleum-based fuel prices are jeopardizing energy supplies and degrading the payment balance by increasing the cost of imported energy. This has led to a resurgence of curiosity in the manufacturing and use of energies derived from organic waste and plantations. Therefore, biodiesel is an environmentally sustainable fuel source and energy with several potentials that can be used as an alternative source identical to petroleum. The use of biodiesel instead of coal and petroleum has the potential to provide several advantages. This is because biodiesel can be generated locally, reducing the demand for petroleum importation, lowering the cost, and ensuring the country's economic stability. The negative effects of supply interruptions can be minimized when biodiesel demand and use to minimize the application of petroleum fuels. Moreover, biodiesel manufacturing provides countries without oil reserves energy sovereignty. The financial effects of biofuel are not restricted to the biodiesel industry and agriculture because of the interdependencies across producing areas that influenced the national economy (Anwar, 2021; Naveenkumar and Baskar, 2020)

Table 8
Comparison of the commonly used methods for biodiesel purification

Purification technique	Sample work	Pros	Cons
Water (wet) washing	• Feedstock: curcas oil • Purification: wet washing • Ester yield: 98% • Ref: (Shirazi *et al*., 2013)	• Efficient removals of alcohol and glycerol • Simple and straight forward process • Can be used to efficiently separate biodiesel from catalysts such as ZnO, SiO_2, ZrO_2, and SrO	• Require high volume of water for washing • Long time needed for the process • Require further drying for the product after washing • Large amount of wastewater is generated during the washing process • Non-eco-friendly method
Dry washing (Ion-exchange and adsorption)	• Feedstock: Waste cooking oil • Purification: ion-exchange • Ester yield: 95.8% • Ref: (Hajra *et al*., 2015)	• Relatively faster process • Needing smaller space for the process • Less amount of waste is generated • Might be run in continuous system • The purifying agents i.e., resins, adsorbents, zeolites, etc., can be regenerated and reused • Eco-friendly method	• Might be increase the acidity of biodiesel due to the used of high acidity adsorbents or resins • Not efficient in decalcification • Can be reduce the acylglycerol from the product • Cannot completely remove methanol or glycerol in one stage of process
EDTA complexation	• Feedstock: curcas oil • Purification: complexation • Ester yield: 98% • Ref: (Zhu *et al*., 2006)	• Calcium removal ability is remarkable • Ca-EDTA complexes is water soluble • The Ca-EDTA complexes can be removed using centrifugation	• Needing further wet washing and drying • The complexation agent i.e., EDTA has a high toxicity • A very time requiring process • Generate wastewater similarly to the wet washing method
Precipitation	• Feedstock: curcas oil • Purification: citric acid precipitation • Ester yield: 98% • Ref: (Vieira *et al*., 2017)	• Citric acid can be utilized to precipitate the calcium ions in the crude biodiesel (yield >95%) • High product yield • The residue/precipitate can be simply separate using a conventional filtration • The precipitate Ca-citrate can be regenerate back into citric acid by the addition of strong acid	• The purified biodiesel has a higher value of viscosity, making it unfitted the biodiesel standard • Require long time with maintained operating temperature, pressure, and agitation. • Priorly, the calcium content in the crude biodiesel should be measured • Additional purification is required to remove soap, alcohol, and glycerol
Membrane filtration	• Feedstock: Waste cooking oil • Purification: membrane filtration • Ester yield: 5.6% • Ref: (Bansod *et al*., 2021)	• Simple and flexible in operation • Require low energy • High product flux and selectivity • Can be engineered for the special uses • Relatively easier to separate higher size such as, soap and glycerol. • Ease to scale-up	• Less effective in ion removal • The membrane should be periodically checked, cleaned, or changed • Gradual flux reduction over time due to natural fouling on the membrane

The use of biodiesel is also considered environmentally beneficial since the greenhouse gasses emission is remarkably reduced. In the process of biodiesel formation, the use of heterogeneous catalysts is environmentally safer than the homogeneous catalyst process due to the minimized generated waste. The purification step in the heterogeneous catalyst process is easier to conduct, however, the process still generated some wastes (Alagumalai *et al*., 2021; de Mello *et al*., 2017; Jayed *et al*., 2009). This includes wastes from the product separation such as used catalyst and adsorbent, fouled membranes during the application, and complexation product of Ca-EDTA. The second type of waste is from the reactivation of regeneration of catalyst or ion-exchange resins such as solvents, used alcohol, and wastewater. Therefore, there is a need to develop the stability of catalyst properties through its use over several consecutive cycles and the handling catalyst. This approach is useful for minimizing the generated wastes upon the application of the heterogeneous method in biodiesel production, which will create more environmental benefits.

6. Conclusion

Emerging processes have been developed in recent years fo such as microwave-assisted, ultrasonic-assisted, supercritical transesterification, and liquid plasma discharged-assisted biodiesel synthesis processes. These methods were reported to have a variety number of conversion yields from 75 to 99%, especially the supercritical transesterification that is carried out in a catalyst-free process. However, the main problem to use

these advanced processes is the high operating cost and a higher risk compared to the conventional process. Furthermore, the types of catalysts play an essential part in biodiesel synthesis because catalysts with different sources, phases, chemicals, and sizes have been applied to achieve better biodiesel quality and yield. Homogeneous and heterogeneous catalysts can achieve at least 80% to 95% biodiesel conversion. Nano-catalysts are the nanoparticle form of heterogeneous catalyst that showed a better catalytic performance due to their large active surface area. The enzymatic catalysts are eco-friendly with a variety of conversions from 70 to 99%. Moreover, the effectiveness of the separation process in biodiesel purification depends on the type of catalyst applied in the synthesis process. In certain cases, a simple wet washing can lead to the biodiesel product meeting the specification standard. Some efforts using several dry methods such as adsorption, cation- and anion-exchange resins, precipitation, complexation, and membrane-based separation have shown a great performance in calcium ions removal. However, some drawbacks of those methods include the complex process from upstream until downstream that needed to be simplified. Therefore, further research on this topic is necessary, especially on the reusability of the catalysts and purifying agents. This development will not only contribute to the intensification of biodiesel production and purification but also maximize the economic impact and minimize the environmental hazard of biodiesel.

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Silviana: Conceptualization, methodology, writing—original draft., Didi Dwi Anggoro; Validation, resources. Hadiyanto: Writing—review and editing, supervision, Cantika Aulia. Salsabila: Writing—review and editing, and validation., Kevin Aprilio: Formal analysis, and resources, Anisa Widia Utami: Formal analysis, and project administration, Afriza Ni'matus Sa'adah: Formal analysis, and resources., Febio Dalanta: Visualization, Literature review, Validation, and writing—original draft - review and editing. All authors have read and agreed to the published version of the manuscript.

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