



## Review article

# Recent advances and challenges in the utilization of nanomaterials in transesterification for biodiesel production



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## ABSTRACT

Due to diminishing fossil fuel supplies and rising energy needs, there has been an ever-increasing demand for renewable energy sources. The available renewable energy resources, such as solar, wind, hydropower, and biofuels, provide a new way of supplying the world's energy needs. Biofuels stand out among them because they are sustainable and have the potential to bring the idea of a global bioeconomy to life. As a result of their production of biofuels like biomethane, biohydrogen, and biodiesel, atmospheric CO<sub>2</sub> is being fixed, eventually lowering the world's carbon footprint. Current developments in the production of bioenergy have concentrated on producing biodiesel among other biofuels. Biodiesel is being produced from a variety of feedstocks using a number of processes, including transesterification, micro-emulsion, direct mixing, and pyrolysis. The most popular method among these is transesterification, which makes use of a variety of catalysts. As a result of the development of nanotechnology, nanocatalysts with desirable properties, such as increased catalytic activity, increased surface area, and superior thermal stability, have been made and modified. In this review, various nanocatalyst types and manufacturing processes are examined in relation to transesterification. It explores how crucial nanocatalysts are in boosting biodiesel production, highlights potential barriers, and makes recommendations for their widespread use in the future.

## 1. Introduction

A vast majority of global energy demand is met by fossil fuels, which poses threats to the environment. Continuous globalization and industrialization have increased the use of fossil fuel products such as petroleum and coal, rendering their reserves in an

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### Abbreviations

TAG	Triacylglyceride
GHG	Greenhouse gas
FFA	Free fatty acid
FAME	Fatty-acid-methyl-ester
CNT	Carbon nanotube
MWNT	Multi-walled carbon nanotube
s-MWCNT	Sulfonated multiwalled carbon nanotube
BCL	Burkholderia cepacia lipase
MNP	Magnetic nanoparticle
ZSM-5	Zeolite Socony Mobil-5
NBC	Nanobiocatalyst

endangered state [1]. The annual worldwide energy requirement is approximately 580 TJ, a whopping 80% of which is met by burning conventional fossil fuels. Increasing population and accessibility necessitate greater energy consumption, further accelerating the depletion of fossil fuel resources [2]. The shortage of energy, limited deposits, rapid increment in petroleum oil prices, and adverse effects of greenhouse gas emissions have necessitated a shift of focus to alternative energy sources. The pursuit of environmentally friendly technology is propelling research attempts to discover viable energy sources that are carbon-neutral, biodegradable, renewable, and non-toxic [3]. With technological advancements, implementing many cutting-edge concepts can drive perceptions and methodologies of curating renewable energy to new horizons and offer greener solutions to address rising demands while conserving the environment [4]. The existing renewable energy sources, such as, hydro, geothermal, solar, wind, and biofuels, contribute to meeting only about 18% of global energy demand, mostly due to the non-implementation of the technologies on a large scale [5]. As a substitute for traditional fuels, biofuel production has garnered much attention from scientific circles in recent decades. Biomass-derived energy is expected to generate half of the net energy demand in industrial nations by 2050 because of its accessibility and eco-friendly characteristics, such as low carbon footprint and generating minimal concentrations of sulfur. The combustion of fossil fuels releases around 27 billion tonnes of CO<sub>2</sub> each year, with this figure expected to surge by 60% by 2030. As a result, employing biomass-derived fuels is critical to lowering the carbon footprint [6]. Fig. 1 shows the contribution of biomass-derived energy in meeting global energy demands. Biofuel production primarily focuses on biomass conversion to fluid fuels such as biogas, biohydrogen and biodiesel, which is accomplished mostly by photosynthesis. Among them, biodiesel as a possible fuel is rapidly developing, thus limiting and minimizing the utilization of carbon energy and the rest of nonrenewable resources [7].

Biodiesel is a sustainable energy source made composed of hydrocarbons having a long carbon chain and an ester group (-COOR) that is produced from many types of feedstocks, such as oils derived from plants and animals or other lipids termed triacylglycerides (TAGs) [8]. Biodiesel generated from non-food sources is a cost-efficient and reliable substitute for petroleum-derived fuels. It can be utilized directly or mixed in any ratio with conventional diesel. However, production costs, feedstock availability, and complexities in production methods are among the most important elements influencing commercial biodiesel production [9]. Biodiesel generation technologies such as pyrolysis, the supercritical fluid process, and transesterification are widely employed. Transesterification is the most extensively utilized of these processes [10]. Some recent works in the field of biodiesel production can be seen Table 1.

Transesterification is a catalytic process that requires using methanol to transform triglycerides into fatty acid chains in the presence of a homogeneous/heterogeneous catalyst. Homogeneous catalysis is where the catalyst stays in the same state as reactants throughout the process. The catalyst is heterogeneous if it is in a different phase than the reactants [19]. Contrary to those that are catalyzed heterogeneously, homogeneously catalyzed processes are often quicker and call for less loading. However, solid catalysts are very expensive and have a smaller surface area, thereby depreciating the catalyst's total effectiveness [20]. The Development of nanotechnology has led nanomaterials to execute an important role in enhancing biodiesel output and grade while shortening reaction

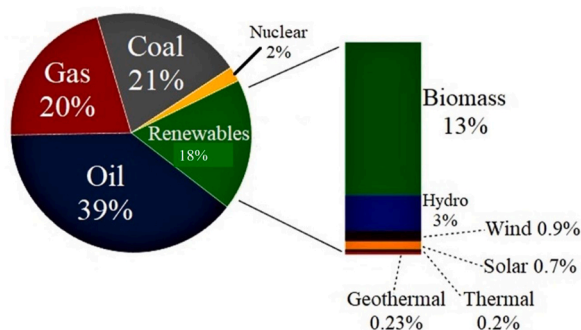


Fig. 1. Contribution of different renewable energy sources for meeting global energy demands.

**Table 1**  
Different nanocatalysts for the synthesis of biodiesel using several feedstocks.

Feedstock	Nanomaterial	Yield (%)	Reference
<i>Mesua ferrea</i>	Co doped ZnO	98.03	[11]
Waste cooking oil	MgO	93.3	[12]
Waste cooking oil	ZnFe <sub>2</sub> O <sub>4</sub>	98.6	[13]
<i>Mesua ferrea</i> linin	Egg shell derived Cao	94	[14]
FFA	MoO <sub>3</sub> /B-ZSM-5	98	[15]
Goat fat	MgO	93.12	[16]
Waste cooking oil	CaO over Zr- doped MCM-41	88.5	[17]
Rapessed oil	Potassium impregnated	96.1	[18]

time. Using a nanocatalyst decreases catalyst weight, the reaction temperature, and the oil-to-alcohol ratio [21,22]. It has already been established that nanocatalysts significantly contribute to accelerating the velocity of reaction without getting damaged. It also decreases the activation energy of the reactants substantially.

Furthermore, the quantity of nanocatalysts used is crucial in delimiting biodiesel production output [23]. It was further reported that with no catalyst loading in the specified reaction prerequisites, net biodiesel production was zero, implying that the presence and number of nanocatalysts determine the efficacy of biodiesel synthesis [24]. The esterification process mediated by nanocatalysts has various distinctive benefits, such as a faster mixing rate with the reactants, shorter reaction periods, and quick and simple purification [25].

This review explores different types of nanomaterials used in mediating the transesterification process and emphasizes the importance of nanotechnology in biodiesel production. In addition, this article evaluates their applicability and related obstacles in the transesterification process, focusing on reusability, selectivity, catalytic activity and catalyst loading [26–28]. The prime motive of the review is to gain a thorough understanding of the application of nanotechnology in bioenergy generation and serve as a roadmap for future innovations for sustainable development.

## 2. Biodiesel

Biofuels are mostly sourced from renewable resources such as wood, bagasse, feedstock, edible and non-edible commodities, and cellulosic and lignocellulosic materials. They are generated using a biological fixation process, resulting in the generation of several kinds of biofuels like bioethanol, biohydrogen, biomethane, and biodiesel [29]. Biodiesel is a fatty acid alkyl ester synthesized by transesterifying triglycerides in presence of an alcohol. Depending on its composition, the generated biodiesel mostly comprises various fatty acids. It has a low sulfur content, is non-toxic, renewable, and biodegradable, and has fewer hazardous gas emissions. Biodiesel can be used in combination with conventional diesel to scale back on the number of pollutants and environmentally hazardous gases generated by petrol, diesel and other fuels [30].

### 2.1. Feedstock for biodiesel production

A vast range of possible sources for the feedstock, including as oilseeds, algae, and animal fats, have been studied for the manufacturing of biodiesel. The choice of raw materials for biodiesel synthesis is an important area of consideration since some of the feedstock materials are expensive and utilizing them will render the production unprofitable [31]. Feedstock is mostly constituted of oils and fats sourced from organic biomass of plants and animals and can be classified into three main generations.

**Table 2**  
Advantages and Disadvantages of three generations of feedstock.

Feedstock generation	Source	Advantages	Disadvantages	Reference
First	Edible oil	1.Environmentally safe 2.Easy to handle 3.Sustainable	1.Food vs Feed problem 2.Lack of agricultural lands for high yield of feedstock and biodiesel	[37–41]
Second	Non – edible oil	1.Sustainable 2.Easy to handle 3.Economically more viable 4.No Food vs Fuel duel 5.Promotes recycling	Purification process is required which is often complex	[42–46]
Third	Yeast, algae, microbial biomass	1.Sustainable 2.High yields of biodiesel 3.Easily available 4.Economically viable, if used to full potential 5.Doesn't lead to Food vs Feed competition	1.Has not been applied in commercial set ups 2.Lack of statistical knowledge on scale-up	[47–51]

1. First generation feedstock comprising of edible oils derived from seeds of sunflower [32], canola, rapeseed, coconut, etc. [33].
2. Second generation feedstock consisting of non-edible oils like jatropha oil [34], oil derived from sewage and kitchen wastes, discarded frying oil, etc. [35].
3. Third generation feedstock which mainly include oil derived from microorganisms such as algae, yeast and other lipid-rich microbes [36].

The three generations of feedstocks come with their own benefits and drawbacks, a few of which have been listed in Table 2 and Fig. 2 [31].

The fourth generation of biofuels' raw materials are genetically altered microalgae. These microbes have undergone genetic modification to boost photosynthesis, produce more biofuel, and create an artificial carbon sink. Several algal strains, including *Chlamydomonas reinhardtii* sp., *Phaeodactylum tricoratum* sp., and *Thalassiosira pseudonana* sp., have undergone genetic modification to speed up development and improve their capacity to flourish in environments with little nutrients. The reduction of GHGs, CO<sub>2</sub> assimilation, and use of genetically modified microalgae as a wastewater treatment medium are all benefits for the environment. Although though the research is still in its early stages, the fourth generation of biofuel aims to have the least negative environmental impact when compared to prior generations. As a result, the majority of literature research concentrate on the effects of gene alteration on the environment.

### 3. Transesterification in biodiesel production

Biodiesel production from feedstock oils is achieved via three main routes: pyrolysis, emulsification and transesterification. All of the techniques have some degree of viability, as well as certain benefits and drawbacks that are detailed in Table 3 [52]. Since traditional methods like pyrolysis, blending, and emulsification technologies for synthesis are incompetent for large volumes of biodiesel output, transesterification or alcoholysis is the standard method for producing biodiesel.

Transesterification involves reacting triglycerides with alcohols (usually methanol) with the aid of a catalyst to speed up the reaction and create fatty acid esters (often, fatty-acid-methyl-esters, or FAME) [54]. Most oils derived from organic biomass are triglycerides and hence, the glycerol backbone needs to be removed to obtain fatty acid ester chains, having similar structural and combustion properties as conventional diesel. The glycerol backbone is removed using transesterification method, which is sped up using a catalyst. The reaction proceeds in stages, beginning with triacylglycerol and progressing through di- and monoacylglycerols to glycerol, generating methyl ester at each stage [55].

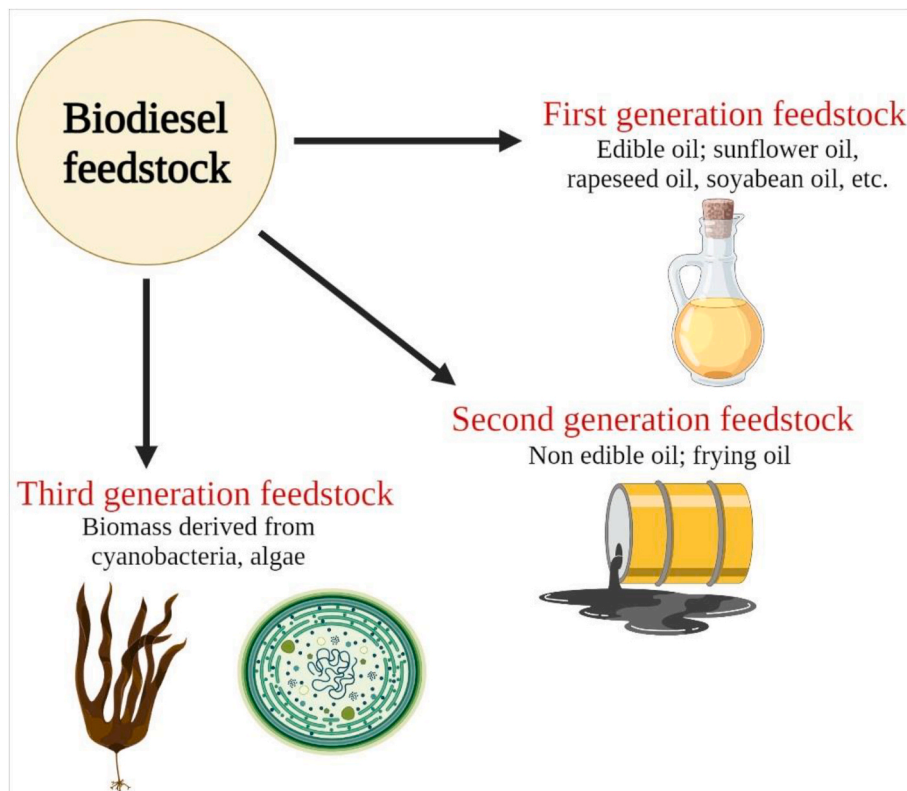


Fig. 2. The three generations of feedstock for Biodiesel production.

**Table 3**  
Techniques used for Biodiesel production (Adapted from Karmakar and Halder [53]).

Technique	Advantage	Disadvantage
<b>Direct blending</b>	No specific feedstock requirement and no need of additional preparations	Not suitable for long haul use Higher viscosity could lead to other issues and blockage
<b>Thermal cracking or pyrolysis</b>	Potent process with almost no contaminating byproducts. No requirement for additional detachment or filtration	Expensive set-up and quite costly process Low biodiesel quality because of impurities
<b>Microemulsification</b>	Straightforward cycle It is utilized for extremely sticky feedstock	Fragmented ignition and arrival of inordinate carbon stores
<b>Transesterification</b>	Gentle response conditions High conversion rate Effortless handling	Requirement of large volumes of water Development of side residues Treatment required for the unsaturated triglycerides
<b>Enzyme catalyzed transesterification</b>	Possess the capacity to transesterify both triglycerides and FFAs, and demand a low oil to alcohol molar ratio. Are able to produce biodiesel with fewer processing steps, using less energy in the process.	The creation of waste water hinders the approaches' efficiency. A low rate of reaction, especially when immobilized for enhancement of their reusability

The most promising technique is the reversible transesterification reaction, as depicted in Fig. 3. FAMES (biodiesel) and glycerol are produced by reacting fatty acids (oils) with alcohol in the presence of a suitable catalyst. A powerful base or acid, such as hydrochloric acid or sulfuric acid, or a strong base, such as sodium hydroxide, sodium methoxide, or potassium hydroxide, can be used as a catalyst [56]. Since alcohol is very weakly soluble in the oil phase and uncatalyzed reactions are exceedingly sluggish, the catalyst is typically used to initiate the reaction and serves as an alcohol solubilizer. The catalyst promotes the solubility of alcohol and, as a result, makes it possible for the reaction to advance at a tolerable rate [57]. One of the most crucial factors in the production of biodiesel is the catalyst's unique surface area. Because there are more active areas for reactions to occur on the catalyst's surfaces, a nanocatalyst with a better surface/volume ratio performs better. The transesterification reaction benefits from the high specific surface area of nanocatalysts. A popular nanocatalyst for the synthesis of biodiesel is calciumoxide because of its low cost and potent catalytic activity. CaO can therefore be used to make biodiesel either by itself or in combination with other substances. Other nanocatalysts used in the transesterification process include MgO, SrO, and ZnO. Once more, Fe<sub>3</sub>O<sub>4</sub>, CuFe<sub>2</sub>O<sub>4</sub>, activated carbon, and other substances can be utilized to improve the properties of these catalysts [58–65].

Transesterification involves utilization of catalysts for conversion of triglycerides to alkyl esters. Based on the phase and type of catalysts used, the process can be divided into.

- 1Homogeneous Catalysis
- 2Heterogeneous Catalysis

### 3.1. Homogeneous catalysis

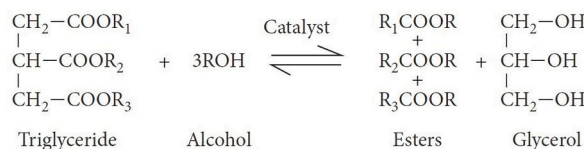
In homogeneous catalysis, a chemical which is in the same phase as the reaction system is used to catalyze a set of reactions. It is the most favored catalyst to be used in the synthesis of biodiesel because it is more convenient and takes short time to execute the reaction [66]. Homogenous catalysis can involve the use of either acid or base catalysts, with base catalysts being preferred over the former. Catalysts most preferred include alkali metal hydroxides and alkoxides, which operate at low temperatures to provide maximum yields [67].

#### 3.1.1. Base catalyst

Alkaline liquids such as alkali metal oxides including methoxides, sodium or potassium hydroxide, and carbonates are examples of homogeneous base catalysts. Base catalysts have a strong transesterification activity [68], however a notable downside is that oil with considerable FFA levels cannot be entirely converted into biodiesel and instead stays as soap in large volumes. The process may still be accelerated with an alkali catalyst up to 5% FFAs, but more catalyst is needed to make up for the catalyst that is lost to soap [69].

#### 3.1.2. Acid catalyst

In case of acid catalysts, Brønsted acids such as sulfonic and sulfuric acids, hydrochloric acid, catalyze the esterification reaction [70]. Alkyl esters are produced in exceedingly high yields using these catalysts. However, because of the slower reactions compared to



**Fig. 3.** Transesterification of triglycerides.

alkali-catalyzed reactions and the higher energy requirements, the procedure is economically burdensome [71]. Acid catalysis is capable of catalyzing both transesterification and esterification processes and is not influenced by the presence of FFA. The separation problems with homogeneous acid catalysis are identical to those with homogeneous base catalysis, notwithstanding these additional benefits.

### 3.2. Heterogeneous catalysis

In heterogeneous catalysis, the phases or states of catalysts differ from the reactants. These are the kinds of catalysts which frequently form active site during a reaction with their reactants [72]. It is remarkably effective and may be readily recycled as per the nature of catalyst. Based on the type utilized, heterogeneous catalysis is further classified into alkali-based, acid-based and enzyme-based catalyzed processes [73].

#### 3.2.1. Alkali/base catalyst

The use of heterogeneous basic catalyst seeks to get around limitations like saponification, which prevents extraction of glycerol from methyl ester phase when a homogeneous base catalyst is used. Under moderate circumstances, these catalysts also exhibit remarkable catalytic activity [74]. Other benefits of these catalysts include their non-corrosiveness, environmental friendliness, and ease of disposal. Additionally, they may be made to have better activity, greater selectivity, and a longer catalyst lifespan and are simple to remove from the reaction environment. Recent research demonstrated the successful use of heterogeneous based-catalysts made of various oxides, zeolites, and clay [75,76]. Commercialization of such catalysts is a significant challenge due to a series of major drawbacks, which include low FFA levels, limitations in diffusion, large molar alcohol to oil proportion, high cost when in comparison to conventional reactions, and catalyst syphoning stills [77]. Heterogeneous base catalysis employs a diverse range of metal-based oxides, such as alkali, alkaline earth, and transition metal oxides. Boron group-derived and waste-derived catalysts are other types of catalysts used in this type of catalysis [78].

#### 3.2.2. Acid catalyst

Heterogeneous acid catalysis is frequently performed using inorganic polymeric materials, which allow for simple separation from the reaction media, reusability, and the promotion of environmentally friendly technologies [79]. Compared to homogeneous acid catalysts, heterogeneous ones are less poisonous and corrosive and cause less environmental issues [80]. Numerous acid sites with varying degrees of Brønsted and Lewis acidity may be found in these catalysts. Application of these catalysts is constrained at some key points because nano porous inorganic oxides frequently exhibit low catalytic activity. These points include high solubility, low thermal stability, low surface area, obstacles in fabricating chemically antagonistic features, and low amounts of reactive sites [81].

#### 3.2.3. Enzyme-based catalyst

Enzyme-based catalysts, also called biocatalysts, are derived from living creatures which facilitate chemical processes keeping their own chemical makeup stable [82]. Extracellular and intracellular lipases are the two main types of enzyme-based biocatalysts that are often utilized in the manufacture of biodiesel. The enzymes extracted from microbial broth and then purified are known as extracellular lipases. In contrast, intracellular lipase stays either inside cell or in cellular walls that produce new cells. Bottlenecks such as generation of unwanted byproducts, such as soaps and pigments, which obstruct purification from glycerol and di- and mono-acylglycerols, can be minimized by using biocatalysts. The source of the enzyme and the process parameters affect how well the biocatalyzed transesterification process works. Contrary to chemical catalysts, a large spectrum of triglyceride sources can use biocatalysts, with FFA in the range of 0.5–80% [80]. Easy product removal, a process temperature ranging from 35 to 45 °C, absence of any byproducts, and the possibility to reuse catalysts are additional benefits of enzymatic biodiesel generation [83]. The complexity of separation and purification processes and their accompanying expense are limitation of utilizing extracellular enzymes as a catalyst. This can be reduced by utilizing microbial cells as whole-cell catalysts, resulting in satisfactory biodiesel production [84].

## 4. Nanomaterials in transesterification

Nanomaterials has been in spotlight in recent times due to various advantageous morphological and physico-chemical properties. Nanomaterials are particles which are approximately 1–100 nm ( $10^{-9}$  –  $10^{-8}$  m) in diameter and are much smaller than particle size of bulk materials which are usually in ranges of microns ( $10^{-6}$  m). Due to smaller size, these particles have various characteristic properties such as a high surface area-volume ratio, enhanced reactivity and photo-catalytic activity and low cytotoxicity [85]. This makes their utilization more advantageous over conventional materials. Usage of nanomaterials in transesterification has lately received a lot of emphasis for biodiesel generation due to excellent catalytic performance [86]. Compared to traditional catalysts, nanocatalysts have greater surface area, which improves their efficiency. Additionally, these catalysts have excellent saponification resistance, a high surface area-to-volume ratio, greater stability, and significant reusability [87]. Another key factor influencing the reaction conditions is designing nanocatalysts to be more stable at high temperatures, since this enhances the nanocatalyst's likelihood of being recyclable and reusable. As a result, many kinds of catalysts made of both organic and inorganic materials, such as ion exchange resin and enzymes, as well as oxides, and metal salts, are being produced. The creation of nanocatalysts made of polymers, zeolite, and carbon-based nanomaterials has recently attracted consideration from many researchers [88]. These catalysts primarily display appealing qualities of significant catalytic activity, shortening reaction time and lowering reaction temperature. Hence, production of biofuel will be significantly increased by the modification and manufacture of nanocatalysts. Numerous techniques,

including chemical vapor deposition, impregnation, precipitation, and electrochemical deposition can be used to create the newly formed nanocatalyst. Some noteworthy nanocatalysts that have been used in transesterification producing biodiesel include metal-oxide nanocatalysts such as CaO, ZnO, zeolites, hydrotalcites and nanobiocatalysts [89]. Some types of nanocatalysts can be seen in Fig. 4.

#### 4.1. Metal-oxide nanocatalysts

Out of all types, metal-oxide based nanocatalysts are the most preferred and accounts for a pivotal role in maximization of biodiesel production. Oxides of several metals such as Fe [90], Mg, Zn, Ca, etc. have been used for making of nanoparticles to be used for catalysis in transesterification.

Kumar et al. reported four types of calcium oxide (CaO) nanoparticles, namely, doped CaO, waste CaO, pure CaO and loaded CaO, which are used in transesterification of different kinds of feedstock oils [91]. Amalgamation of CaO nanoparticles via sol-gel method was done by Bharti et al. which was then used for catalysis in transesterification of feedstock oil extracted from soyabeans [92]. After optimization and synthesis in optimal conditions, the process gave a 97.61% biodiesel yield. The synthesized catalyst had a higher surface area/volume ratio than conventional CaO catalysts and had a reusability of up to 6 cycles with minimal drop in conversion efficiency. High concentrations of fatty acids can be readily converted by the CaO nanocatalyst, however long-term usage is not recommended owing to leaching and active site blocking. Doping with metal and metal oxides has the potential to curb the limitation. Higher yields of biodiesel were obtained by synthesizing CaO nanoparticles from discarded chicken eggshells and then using wet impregnation method to dope them with Cu and Zn [93]. On comparison, Zn/Cao was the most efficient with reusability of up to 7 cycles and a net conversion rate of 80%. Efficiency of Zn-doped CaO nanoparticles has also been studied by Ref. [20], which had a biodiesel production efficiency of 96.74% and could be reused for up to 5 cycles. Additionally, KF/CaO nanocatalysts with size ranges between 30 and 100 nm were manufactured by impregnation technique where CaO was doped [94]. Their efficacy in the synthesis of biodiesel using Chinese tallow seed as feedstock was assessed. According to this study, 96.8% of biodiesel was produced, demonstrating the nanocatalyst's potential for the biodiesel sector. CaO, as a nanocatalyst, has been broadly explored and analyzed for catalyzing the transesterification process. However, this catalyst has its own bottlenecks. Leaching which is mostly brought on by the catalyst being hydrolyzed, has a detrimental effect on the conversion efficiency. This leads to saponification, lowering the catalyst's overall catalytic activity and specificity. Comparative study of different metal-oxide catalyst can be seen in Table 4.

The nanocatalyst derived from MgO/MgAl<sub>2</sub>O<sub>4</sub> was described by Rahmani Vahid & Haghighi [103]. It was produced primarily using the combustion technique, succeeded by impregnation. The ideal fuel ratio was found to be 1.5, with 60.6 m<sup>2</sup>/g specific surface area and having an average pore diameter size of 6.3 nm. Further testing of the improved catalyst for generation of biodiesel revealed an efficiency of conversion of 95.7% and a reusability of 3–5 cycles. At 60 °C and methanol to oil ratio of 12:1, the creation of Li doped MgO nanocatalyst has been reported to have a yield of 93.9% [104]. Due to the addition of Li, these catalysts provide more basic site creation, which enhance the net production efficiency. Furthermore, using CaO and MgO nanoparticles studied the synthesis of biodiesel using cooking oils as the feedstock [105]. When compared to MgO nanocatalysts, CaO nanoparticles significantly increased the biodiesel yield.

Production of fatty acid methyl ester (FAME) was achieved by transesterifying palm oil involving catalysis by TiO<sub>2</sub>–ZnO

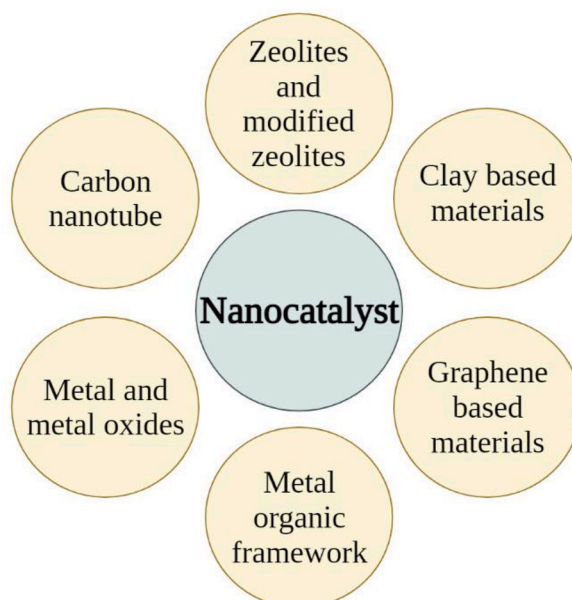


Fig. 4. Various types of nanocatalysts.

**Table 4**  
Biodiesel production using different metal-oxide catalyst.

Metal-oxide catalyst	Yield (%)	Reference
CaO–CeO <sub>2</sub>	95	[95]
Fe <sub>3</sub> O <sub>4</sub>	99.8	[96]
CaO	96	[97]
CaO–Al <sub>2</sub> O <sub>3</sub>	98.64	[98]
TiO <sub>2</sub> –MgO	86	[99]
MgO/MgFe <sub>2</sub> O <sub>4</sub>	91.2	[100]
MgO–ZnO	83	[101]
CaO–ZrO <sub>2</sub>	92.1	[102]

nanocatalyst. Ti replaces Zn in the lattice structure, creating deformities and thereby making the catalyst more stable and increasing its catalytic activity [106]. Sulfate-doped TiO<sub>2</sub> catalyst was reported to be efficient in refining petroleum and in production of biodiesel with cooking oil used as feedstock. Hematite was used to provide SO<sub>4</sub>/Fe–Al–TiO<sub>2</sub> catalyst its magnetic properties, while alumina served as a buffer layer and the catalyst, on transesterifying cooking oil, had a 96% conversion efficiency [107]. Doping of potassium tartrate on TiO<sub>2</sub> nanocatalyst increased the basicity of the catalyst, leading to a net production of 98.9% on transesterification of linseed oil [10]. Alves et al. showed a comparison analysis of Fe/Cd along with Fe/Sn oxide nanoparticles having magnetic properties to transesterify soybean oil [108]. Fe/Sn oxide nanoparticles outperformed with an 84% yield of biodiesel, exhibiting the highest catalytic activity out of the duo. In addition to having remarkable resistance at high temperatures, these nanoparticles further promote effective reaction between free fatty acids and alcohol (specifically methanol), increasing output. The nanocatalysts and related reports discussed here provide sufficient evidence to derive the resolution that nanocatalysts curated using multiple metal oxides are among the most effective heterogeneous catalysts for producing biodiesel using a variety of feedstocks.

#### 4.2. Carbon nanocatalysts

In accordance with various structures and sizes of the generated nanocomposite materials, nanocatalysts made from carbon materials such as carbon nanotubes, graphene as well as reduced graphene oxides have demonstrated superior physico-chemical characteristics [109].

Carbon nanotubes (CNTs) are reported to be an excellent catalysts, and beneficial applications in fuel cells and other electro-catalytical devices, due to various structural, mechanical, thermal, and biocompatibility properties. Generally, CNTs are made from graphite sheets wrapped into cylindrical shapes, measuring in nanometric dimensions, high surface area, and are exceptionally biocompatible in nature. Enzymes such as, lipases immobilized or conjugated with CNTs (or functionalized multi-walled carbon nanotubes - MWNTs) were shown to have better activity and stability. The ester hydrolysis of MWNT-immobilized lipase and free lipase in an aqueous media was studied over time, and increased amounts of aldehydes, carboxylates, and peroxides were reported as the end products [110]. Sulphonated multiwalled CNT was used as a nanocatalyst in Guan et al. research to *trans*-esterify triglycerides [111]. For the esterification of fatty acid derived from palm, Shuit et al. reported employing sulfonated multiwalled carbon nanotubes (s-MWCNT) [112]. Loading of ZnO nanoparticles on microwalled carbon nanotubes (MWCNT) led to decrement of surface area and increment of active sites, enhancing rate of reaction and mass transfer [113].

For the synthesis of biodiesel, some researchers have further employed lipase enzymes which have been immobilized rather than carbon nanotubes because of their improved thermal strength and recyclable nature. Fan et al., described the fabrication of multi-walled CNTs that were loaded with Fe-oxide, coupled with polyamidoamine dendrimers, and then anchored over *Burkholderia cepacia* lipase (BCL) [114]. Due to the surface-linked dendrimers, the designed catalyst demonstrated increased catalytic activity, producing an overall yield of 92.8% and outstanding recyclability to an approximate of 20 subsequent cycles. Nanocatalysts made of biochar are very effective for catalyzing transesterification and is primarily made via pyrolysis and carbonization of biomass derived from various feedstock. Using the carbonization approach, more carbon-based nanocatalysts can easily generated from fly ash and sugar-based nanomaterial [115]. However, biochar based nanocatalysts have low thermal stability and have lengthened time of reaction. Graphene oxide also shown improved fatty acid conversion due to more surface area and unique functional groups. TiO<sub>2</sub>/reduced graphene oxide nanocomposites were created by Verma et al. via transesterification of used cooking oil. Under optimal conditions, the catalyst had a conversion efficiency of 98% and reusability of up to 3 cycles [116].

#### 4.3. Magnetic nanocatalysts

Magnetic nanocatalysts may be produced from a variety of materials, such as metal oxides, iron, cobalt, nickel, and platinum alloys, which are used in a variety of ways [117]. Magnetic nanoparticles (MNPs) have several favorable characteristics: higher molecular weight and smaller size (thus, a good ratio of surface area to volume); quantum characteristics; and capacity to transport other chemicals (such as drugs). MNPs also have a variety of possible applications in biofuel industries, like the synthesis of sugars using lignocellulosic stuff by using immobilized enzymes [118]. MNPs can be utilized as enzyme immobilization support, or used to attach other catalytic nanoparticles, making them ideal nanoparticle for use in several applications. One major advantage of using 'MNPs-enzymes' is the possibility of collecting the enzyme complex using magnetic field, and reusing it more than once. By using a high-frequency magnetic field, these nanocatalysts may also be utilized for hydrogenation, photooxidation, and inductive heating.



**Table 5**  
Different types of nanomaterials used for biodiesel synthesis.

No.	Type of feedstock	Type of nanomaterials	Experimental conditions	Biodiesel Yield (%)	References
1	Wild mustard seed oil	Lanthanum titanium dioxide (LaTiO <sub>3</sub> ) nanoparticles	MeOH/oil ratio (4:1), catalyst (100 mg), reaction time (60 min), 80 °C	92.21	[132]
2	Canola oil	αFe <sub>2</sub> O <sub>3</sub> 1-x/ZnO x nanoparticles	MeOH/oil ratio (11.25:1), ultrasonic power of 278.46 W, irradiation power time 29.22 min, catalyst (47.24%), 65 °C	94.21	[133]
3	Waste cooking oil (WCO)	TiO <sub>2</sub> nano-catalyst	MeOH/oil ratio (10:1), 0.01% TiO <sub>2</sub> nano-catalyst coupled with 0.3% NaOH, reaction time (60 min), 60 °C	95	[134]
4	Mango seed ( <i>Mangifera indica</i> ) extracts	ZnO nanocatalyst	MeOH/oil ratio (7:2), 10 mL of ZnO nano catalyst, reaction time (60 min), 50 °C	85	[135]
5	Waste cooking oil (WCO)	<i>Candida antarctica</i> Lipase B immobilized on a magnetic hybrid sol-gel nanocomposite	MeOH/oil ratio (4:1), catalyst (1 g), reaction time (30 h), 40 °C	96	[136]
6	<i>Citrus medicaseed</i> oil	Green CuO nanoparticles	MeOH/oil ratio (8:1), catalyst 0.18%, reaction time (120 min), 85 °C	93	[137]
7	Canola oil	ZnO nanocatalyst	MeOH/oil ratio (11.19:1), ultrasound irradiation time (31.98 min), nanocatalyst (3.17%)	90.16	[138]
8	Waste palm cooking oil (WPCO)	Calcium oxide with a strontium ion additive (Sr–CaO)	MeOH/oil ratio (9:1), 5%w/w catalyst, reaction time (3 h), 80 °C	99.33	[139]
9	Waste cooking oil (WCO)	Heterogeneous nanocatalyst-based magnetic polyaniline (MPANI) decorated with cobalt oxide	MeOH/oil ratio (10:1), MPANI to Co <sub>3</sub> O <sub>4</sub> ratio of 1:1, 0.2 g catalyst, reaction time (240 min), 90 °C	93	[140]
10	Sunflower oil	ZnO nanoparticle	MeOH/oil ratio (20:1), catalyst (4.7%), reaction time (3 h), 70 °C	75	[141]
11	Olive oil	Magnetite nanoparticles-immobilized lipase from <i>Bacillus subtilis</i>	MeOH/oil ratio (12:1), catalyst (1% w/v), reaction time (1 h), 37 °C	45	[142]
12	Oleaginous fungal lipid from <i>Fusarium solani</i> (FS12)	Bimetallic gold-silver core-shell nanoparticle (Au@Ag NPs)	MeOH/oil ratio (20:1), 3% Au@Ag NPs, reaction time (30 min), 70 °C	91.28 ± 0.19	[143]
13	<i>Zanthoxylum armatumseed</i> oil	Silver oxide nanoparticles	MeOH/oil ratio (7:1), catalyst 0.5%, reaction time (2 h), 90 °C	95	[144]
14	Waste cooking oil (WCO)	CaO nanocatalyst from <i>Acalypha indica</i>	MeOH/oil ratio (11.8:1), catalyst (2.4%), reaction time (70 min), 63.7 °C	94.74	[145]
15	<i>Terminalia chebula</i> oil	CuO nanoparticles (from <i>T. chebula</i> leaves)	MeOH/oil ratio (9:1), catalyst (3%), reaction time (60 min), 60 °C, stirring speed of (650 rpm)	97.1	[146]
16	Palm oil	Magnetic mesoporous solid acid catalysts (Fe <sub>3</sub> O <sub>4</sub> @SBA-15-NH <sub>2</sub> -HPW)	MeOH/oil ratio (20:1), catalyst 4%, reaction time (5 h), 150 °C	91	[147]
17	Lipids from <i>Chlorella pyrenoidosa</i>	<i>Aspergillus niger</i> (KP001169) lipase enzyme immobilized into Fe <sub>3</sub> O <sub>4</sub> nanoparticles	MeOH/oil ratio (4:1), immobilized lipase (300 mg), reaction time (6 h), 45 °C, stirring (400 rpm)	90	[148]
18	Waste cooking oil (WCO)	Lanthanum phosphate foam heterogeneous nanocatalyst (LaPO <sub>4</sub> foam)	MeOH/oil ratio (5:1), LaPO <sub>4</sub> (2.5%), reaction time (120 min), 90 °C	91	[149]
19	Non-edible seed oil of <i>Chamaerops humilis</i>	CoO nanoparticles	MeOH/oil ratio (6:1), catalyst (0.54%), reaction time (180 min), 60 °C	92	[150]
20	Microalgal oil ( <i>Chlorella vulgaris</i> )	CaO nanoparticles	MeOH/oil ratio (9:0.6), catalyst (4%), reaction time (4 h), 80 °C	67	[151]
21	Waste seed oil of <i>Citrus aurantium</i>	Zirconium oxide	MeOH/oil ratio (6:1), catalyst (0.5%), reaction time (120 min), 87.5 °C	94	[152]
22	<i>Jatropha curcas</i> seed oil (JCSO)	Lipase- polydopamine (PDA)-TiO <sub>2</sub> nanoparticles	MeOH/oil ratio (6:1), NPs concentration (10%), reaction time (30 h), 37 °C, water percentage (0.5%)	92	[153]
23	Waste cooking oil (WCO)	Graphene oxide and bimetal zirconium/strontium oxide nanoparticles (GO@ZrO <sub>2</sub> -SrO)	MeOH/oil ratio (4:1), material ratio of (1:0.5 w/w) of GO: ZrO <sub>2</sub> -SrO, reaction time (90 min), 120 °C	91	[154]
24	Waste cooking oil (WCO)	Tungstophosphoric acid nanoparticles	MeOH/oil ratio (12:1), catalyst 3% (w/w of oil), reaction time (5 h), 65 °C	91	[155]
25	Waste cooking oil (WCO)	Magnetic nano-sized solid catalyst derived from bio-waste <i>Citrus sinensis</i> peel ash (CSPA)@Fe <sub>3</sub> O <sub>4</sub>	MeOH/oil ratio (6:1), catalyst (6%), reaction time (3 h), 65 °C	98	[156]
26	<i>Pongamia pinnata</i> raw oil	Fe <sub>3</sub> O <sub>4</sub> nanoparticles impregnated eggshell (CES-Fe <sub>3</sub> O <sub>4</sub> )	MeOH/oil ratio (12:1), catalyst (2%), reaction time (2 h), 65 °C	98	[157]
27	Blend of neem and rubber seed oils (NO-RSO)	Nanoparticles from elephant-ear tree ( <i>E. cyclocarpum</i> ) pod husk	MeOH/oil ratio (11.4:1), catalyst (2.96%), reaction time (5.88 min), microwave heating power (150 W)	98.77 ± 0.16	[158]
28	Microalgae oil	Fe <sub>3</sub> O <sub>4</sub> /ZnMg(Al)O solid base catalyst	MeOH/oil ratio (12:1), catalyst (3%), reaction time (3 h), 65 °C	94	[159]

(continued on next page)

Table 5 (continued)

No.	Type of feedstock	Type of nanomaterials	Experimental conditions	Biodiesel Yield (%)	References
29	Waste cooking oil (WCO)	Magnetic $\text{SO}_4/\text{Fe}-\text{Al}-\text{TiO}_2$ solid acid catalyst	MeOH/oil ratio (10:1), magnetic catalyst (3%), reaction time (2.5 h), 90 °C	96	[107]
30	Waste scum oil	$\text{TiO}_2$ nanoparticles	MeOH/oil ratio (9:1), catalyst (0.9%), reaction time (90 min), 60 °C, stirring speed (300 rpm)	93.5	[160]
31	Rapeseed oil	Potassium impregnated $\text{Fe}_3\text{O}_4-\text{CeO}_2$ nanocatalyst	MeOH/oil ratio (7:1), catalyst (4.5%), reaction time (120 min), 65 °C	96.13	[161]

Magnetic nanoparticles also act as carriers for enzymes, which will be more manageable in a magnetic field range. Rai and colleagues utilized magnetic nanoparticles to immobilize the lipase enzyme, which they then used to make biodiesel using palm oil [110]. The biocatalyst was quite stable and catalytically active, and it was possible to detach immobilized enzyme by using a magnetic field for five consecutive batches, while retaining >80% activity.

#### 4.4. Zeolite nanocatalysts

Zeolite materials are being used more often now than ever before. Strong acidic sites, a large surface area, specificity, and distinctive molecular sieving features are recognized for these compounds' potential catalytic activity [119]. Nanozeolites are hydrophobic in nature with large external surface areas, and their great solubility in both polar and non-polar solvents facilitates improved enzyme access to the substrate [120]. Synthetic-based materials are far more widely employed in commercial sectors than natural zeolite materials. ZSM-5, X, Y, and beta zeolites are commercially accessible synthetic zeolites that are primarily employed in the manufacturing of biodiesel [121].

Using NaX zeolite loaded with KOH, Xie et al. demonstrated the transesterification of soybean oil [122]. To gain the highest yield of production, this zeolite was synthesized and examined using various compositions, and the net biodiesel yield was 85.6%. Sunflower oil was *trans*-esterified using CaO nanoparticles and NaX zeolite under optimal circumstances, producing 93.5% biodiesel [123]. The utilization of NaY as well as KOH/NaY zeolites as catalysts in the transesterification of lipid extracted from *Madhuca indica* has been reported by another group [124]. These zeolites have a conversion rate of 98.9% and may be recycled and used repeatedly. CaO/-NaY- $\text{Fe}_3\text{O}_4$  is a nanomagnetic-based zeolite catalyst that was reported to exhibit outstanding stability at high temperatures and easy separation method. With canola oil used as feedstock material, upon analysis of various metrics, the zeolites showed a conversion efficiency of 95.37% [125].

ZSM-based nanocatalysts are being synthesized and used for catalyzing transesterification. ZSM-5, also known as Zbio was modified to form mesoporous zeolites. Due to the availability of acid active sites, ZBio has more activation energy. Biodiesel production of 87% was attained using these zeolites and was greatly influenced by the higher energy of activation [126]. Thus, the above discussions may be used to draw the conclusion that functionalizing zeolites with nanocatalysts will open new possibilities for scaling-up biodiesel production by transesterification of numerous feedstock materials [127].

#### 4.5. Nanobiocatalysts

Biocatalysts used in transesterification conventionally use intracellular and extracellular enzymes such as lipase to enhance biodiesel output. However, these catalysts are laden with bottlenecks such as complex methods of purification, low reusability, and relatively unstable nature of free enzymes. To combat such issues, immobilizing enzymes on solid substructures is considered a novel approach for better biodiesel yields. Nanoparticles can be effective support for enzyme immobilization due to increased Brownian motion and less steric and conformational constraints. Nanobiocatalysts employed in transesterification constitute two main components: nanocatalyst part containing nanoparticles to be used as support; and biological part comprising of the immobilized enzymes [128]. Based on the nature of nanocatalyst, nanobiocatalysts can be sub-divided into inorganic nanobiocatalysts, magnetic nanobiocatalysts, carbon scaffold-based nanobiocatalysts and mesoporous nanobiocatalysts.

Due to larger surface area, existence of more active sites, and superparamagnetic characteristics, magnetic nanomaterials (MNP) are one of the most often employed materials as a nanocatalytic component of nanobiocatalysts (NBCs). Ferrite colloids such as maghemite ( $\gamma\text{-Fe}_2\text{O}_3$ ) and magnetite ( $\text{Fe}_2\text{O}_3$ ) are the most often employed magnetic nanomaterials [90,129]. To facilitate the attachment of enzymes for immobilization, these nanomaterials' surfaces are typically further modified with certain functional groups, such as amides, alcohols, etc. Using the solvothermal technique, Touqeer et al. developed  $\text{Fe}_3\text{O}_4$  NPs which were covalently immobilized with lipase extracted from *Aspergillus terreus* AH-F2 [1]. The NBCs were then utilized to catalyze transesterification of wasted cooking oil to make biodiesel.

One of the most often used inorganic nanocarriers for the fabrication of the NBCs is composed of graphene oxide, carbon nanotubes, silica ( $\text{SiO}_2$ ), and alumina ( $\text{Al}_2\text{O}_3$ ), among others. The availability of different functional moieties such as COO- groups, OH groups, and epoxy groups allows for effective lipase immobilization without the need of unusual cross-linking. Graphene oxide was used by Li et al. for immobilization of lipase and the catalyst had a hydrolytic effectiveness of 80%, validating graphene oxide as a useful nanomaterial for NBCs [130]. One of the most recent advanced classes to appear in academic manuscripts as a nanoscaffolds for immobilizing enzymes needed to produce NBCs is metal organic frameworks. Possessing characteristics such as surface porosity, optimum surface

area, these have been proven to be amazingly effective in immobilizing a variety of enzymes for use in transesterification [131]. Some of the recently published work on using different types of nanomaterials for biodiesel synthesis, different types of feedstock, experimental conditions, and biodiesel yields are highlighted in Table 5.

## 5. Reusability and recovery of nanoparticles

The most important benefits of utilizing heterogeneous nanocatalysts in the biodiesel manufacturing process are their reusability and recovery. The nanocatalyst is used in several cycles during these processes to create biodiesel, and it is recovered and reused at every step. Chemical methods are commonly used to recover nanocatalysts. Heterogeneous catalysts make it simple and rapid to recover both the desired end product and byproduct from the reaction mixture. With this form of catalyst, a washing step is not necessary. Several advantages, such as quick and simple separation from the reaction mixture and quicker mixing of the reactants and catalyst, were suggested for the esterification process utilizing nanocatalysts.

## 6. Techno-economic analysis

As a medium-term substitute for petroleum-based diesel fuel, biodiesel—which is defined as the monoalkyl esters of long chain fatty acids—has drawn significant attention from all over the world. Oils can be transesterified with short-chain alcohols like methanol to create biodiesel, or free fatty acids can be esterified to create biodiesel (FFA). Glycerol is the lone consequence of the transesterification reaction, which is occurring in the presence of an appropriate catalyst like an alkali or an acid. The key benefits of biodiesel are that it is generated locally from renewable resources and emits less carbon monoxide, particulates, and sulfur dioxide. Besides from being biodegradable, non-toxic, and safer to handle, it also produces 78% less carbon dioxide. The primary drawbacks are its high cost, increased nitrogen oxide emissions, and worries about how it would affect the longevity of diesel engines when utilized in their purest form.

## 7. Challenges and prospects

Excellent surface properties, small size, biocompatibility (for most types), ease of synthesis, proven applications in a wide variety of fields, are some of the highly useful traits of nanomaterials. However, there is a flipside of using such nanomaterials – nanotoxicology, which is under the lens, with the possible effects on flora and fauna. The main attributes of nanomaterials, which makes them extremely useful are also a possible cause of concern that can directly or indirectly trigger numerous cytotoxic impacts in humans. In addition, the majority of metal-oxide nanoparticles are now known to be extremely harmful to specific organs, if entered in the human body. The studies have highlighted hazardous effects of nanoparticles, using Wistar rodents models, where it showed fatal changes in the liver, extended platelet count, and delicate disturbance. It has likewise been discovered that titanium was bio-aggregated in the liver, lungs, and mind of uncovered rodents, causing neuro-behavioral changes in the creatures and, additionally, organ wounds. However, the negative effects of extensive usage or release of nanoparticles in the environment is not yet completely studied, which is still a matter of concern. Therefore, there is a widespread concern about not just the advantages of these incredibly helpful particles yet in addition their harmful effects, especially when applied in large scale applications, such as, biofuel-biodiesel production. These contradicting opinions and possibilities provides worldwide experts an opportunity to propose and study long term effects, and how to avert negative impacts of using nanomaterials, while benefiting the society.

In light of the review, future investigations can greatly outperform the current biodiesel production restrictions. Creating a strategy for the large-scale use of non-edible feedstock oils and investigating ways to boost the production of biomass derived from algae, which contains a significant amount of oil. Developing nanocatalysts with multifunctional active sites that can endure disintegration during the *trans*-esterification reaction while also minimizing the impact of leaching. Nanomaterials and polymeric materials are used to functionalize carbonaceous and zeolite-based catalysts in order to increase their catalytic activity, specificity, and stability. Using structural modification based on the shape and size of nano-based materials to improve the kinetics of the reactions involved in the generation of biodiesel. developing statistical methods for efficient large-scale reaction condition optimization. Finally, technological Development is required to scale up the reaction process in order to reduce mass transfer resistance, high energy consumption, and by-product utilization. boosting the possibility of preserving the ideal reaction conditions and so raising the production of biodiesel.

## 8. Conclusions

Biodiesel production holds an enormous potential to cause a paradigm shift in global energy sources and can lead to a re-imagination of recycling of resources, products, and residues in light of sustainability. This can help to achieve eco-friendly routes of consumption and make the vision of a global circular bioeconomy a reality. However, scale-up of biodiesel production is laden with bottlenecks such as low output, dilemma of food vs feed and lack of enough statistical know-how to fully exploit the potential of biofuels. Transesterification is the popularly preferred route for production of biodiesel due to its economic viability and sustainability, coupled with minimal drawbacks and a high output. But, the biggest limitation is transesterification is a slow proceeding process and the conventional catalysts used to fasten the process often pose newer set of challenges.

Usage of nanoparticles as catalysts present a new advent in biodiesel production that can curb the existent bottlenecks. In this review, the various nanocatalysts and their role in enhancing biodiesel output via transesterification have been explored and discussed in detail. The unique characteristics of the nanocatalysts include a high surface area-to-volume ratio, varied shape, enhanced activity,

and specificity of the catalyst. Numerous functional groups can be used to significantly alter nanocatalysts, increasing their overall acidic character, reducing leaching of active metals, and increasing number of active sites for the catalytic process. The quality and output of biodiesel can be enhanced by using nanocatalyst in the right ratios to increase various reaction parameters.

As per the review, future studies can focus on up-scaling of biodiesel production from waste-derived and biomass-derived feedstocks, using nanomaterials. Synthesis and functionalization of nanocatalysts having minimal leaching and disintegration and possessing various active sites is another area of consideration and research. Finally, technical Development is required to scale up the reaction process in order to reduce mass transfer resistance, high energy consumption, and by-product utilization. With further research, advanced technological methods need to be devised to fully understand and avail the effectiveness of biodiesel in meeting global energy requirements in a renewable and environmentally sound manner.

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### Author contribution statement

All authors listed have significantly contributed to the development and the writing of this article.

### Data availability statement

Data included in article/supp. material/referenced in article.

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