Chapter 13
Application of Nanotechnology in Biofuel Production

Jahanvee Mitra, G. K. P. Srilekha, Nilesh Wagh, and Jaya Lakkakula

Abstract One of the greatest challenges the world is facing is the indiscriminate consumption of energy resources due to population explosion. Thus, alternative resources to produce renewable and biodegradable biofuel were necessary to meet the crisis of fossil fuels. Nanotechnology has attracted a greater deal of attention from many researchers because of nanomaterials possessing characteristics like small size, large surface area, and the most important excellent catalytic activity that give them great potential in the production of biofuels. Several nanomaterials act as nanocatalysts and play a vital role in catalytic degradation for bioethanol production and catalyze the transesterification and esterification reaction for extraction of biodiesel from edible and nonedible oils. Nanomaterials associated with microbial enzymes and immobilized onto various forms of lipases have been explored for improved biodiesel production. Nanoadditives have been explored to enhance biogas production. Hence, this chapter aims to review the innovative and outstanding applications of nanomaterials in the field of biofuel production.

Keywords Nanotechnology · Biofuel · Nanocomposites · Nanoparticles · Nanocatalyst · Nanotubes

13.1 Introduction

Fossil fuels, like oil, coal, and natural gas, are the primary sources of energy in various industries. Since fossil fuels are widely consumed by many countries around the world, the demand for petroleum and other fossil fuels increased exponentially (Chow et al. 2003). This led to volatility in the prices of petroleum and environmental concerns along with the rapid depletion of nonrenewable resources (Sagar and Kartha 2007). To overcome these issues, significant numbers of studies were conducted to produce biofuels in a sustainable, efficient, and economically viable way. Biofuels, including biodiesel and bioethanol, are produced using organic

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matter, also known as biomass, such as soybean, rapeseed, sugarcane, cottonseed oil, etc., algal biomass, and even organisms (Luque et al. 2008; Chowdhary et al. 2020; Khan et al. 2020). Over the past few decades, uses of biofuels, with low carbon footprint, have immensely altered the environmental and financial conditions of various industries such as transport, agriculture, cottage industry, and household. The potential substitution of biofuels with depleting fossil fuels seems to be reliable because of its cost-effective and environmentally safe nature (Gowen 1989).

The conventional downstream production of biofuels from feedstock was popular; however, because of its high-energy input and production costs, it had many drawbacks. To overcome these limitations, a widely emerging technology was introduced, which is known as nanotechnology (Mandotra et al. 2018). Nanotechnology deals with particles at atomic scale which are known as "nanoparticles" that lie between the range of 1 and 100 nm and are made of different types of materials (Navya et al. 2019). Though these particles are small, their properties are much more advance than particles that are larger in size, due to their high surface-to-volume ratio, stability, and feasibility. Nanotechnology has been applicable in various fields, including biomedical, automobile, agriculture, architecture, etc., and is slowly emerging in the field of industrial biotechnology (Averback 2004).

Over the past few years, various nanomaterials, such as nanotubes, nanocatalysts, nanocomposites, nanoparticles, nanofibers, etc., are being used to convert crop oils or algal biomass into fatty acid methyl ester (FAME) or biodiesel. Being smaller in size and having a high surface-to-volume ratio, they immobilize enzymes easily and generate high catalytic effect, which boosts the conversion of biomass to biofuels (Yiu and Keane 2012; Ren et al. 2011). Transesterification is the process in which chemical reactions lead to the conversion of crop or algal oils into FAME, also known as biodiesel. Oils are extracted from crops, algae, animals, etc. which react with alcohol under the influence of heterogeneous catalysts (Zhang et al. 2013). This process has several specifications like reaction time, reaction temperature, optimal molar ratio of oil and alcohol, concentration of moisture and free fatty acids, and catalytic dosage (Sarno and Iuliano 2019). Usually, the enzymes used during the process of transesterification become inactive due to the presence of substrates or formation of by-products (Kumari et al. 2009). However, with the help of different nanomaterials, the enzyme system can be stabilized also, due to their high thermostability and efficiency, and low-cost input nanoparticles can be replaced with traditional methodologies used in biodiesel production (Verma et al. 2013).

Wen et al. performed an experiment in which biodiesel was produced from Chinese tallow seed oil using KF/CaO nanocatalyst. KF/CaO nanocatalyst was synthesized (impregnation method) and characterized using TEM, BET, and XRD. It was observed that the size of the pores of the catalyst was between 30 and 100 nm (TEM), with a surface area of 109 $m^2 g^{-1}$ and an average per size of 97 nm (BET), as well as the formation of a new crystal $KCaF_2$ in the catalyst (XRD). Consecutively, Chinese tallow seed oil was transesterified using KF/CaO under optimal conditions like reaction temperature of 65 °C and an alcohol-oil molar ratio of 12:1. Under optimal conditions, the catalyst usage was between 1% and 5% w/w of oil; however, at 4% specifically, the biodiesel yield was found to be maximum. The production of biodiesel was 96.8% due to high catalytic activity and stability, because of the large pore size of the KF/CaO catalyst (Wen et al. 2010). Similarly, biodiesel was produced using date palm seed oil with the help of a CaO-Fe_3O_4 nanomagnetic catalyst. Ali et al. prepared CaO-Fe_3O_4 nanomagnetic catalyst using chemical precipitation method and characterized it. Further, it was reported that calcination of CaO-Fe_3O_4 leads to the formation of calcium ferrite and the presence of iron components of calcium ferrite. To produce high amounts of biodiesel, a batch reactor was utilized for transesterification of date palm seeds oil under optimal conditions. These optimal conditions included 300-min reaction time, 65 °C reaction temperature, 10 wt% of CaO-Fe_3O_4 catalyst, and 20:1 molar ratio of methanol/oil. Under optimal conditions, biodiesel yield obtained from date palm seeds oil with the help of CaO-Fe_3O_4 nanomagnetic catalyst was around 69.7% (Ali et al. 2017).

The main objective of the chapter is to give a broad and thorough understanding of the studies conducted by various researchers to produce biofuels using nanotechnology. It primarily discusses the use of nanotubes, nanocatalysts, and nanocomposites for the conversion of biomass or waste feedstock into fatty acid methyl ester (FAME) or biodiesel. Furthermore, it gives a detailed outline of the transesterification processes yielding varying results with the help of different nanomaterials. The nanotechnological application to produce enhanced and efficient biodiesel or FAME yield, low-cost input, and environment-friendly biofuel production has been critically reviewed.

13.2 Application of Nanotechnology in Biofuel Production

13.2.1 Nanotubes

Nanotubes are one of the most widely used nanostructures in the field of industrial biotechnology due to its low toxicity, high efficiency, and feasibility. For the production of biofuels, nanotubes are functionalized with different substances to enhance their yielding capacity. This makes nanotubes a highly promising and potential nanostructure for the green synthesis of biofuels.

The depletion of petroleum has increased the demand for other sources of fuel especially green sources such as vegetable oil or animal fats. Biodiesel is the most economical, nontoxic, and biodegradable fuel. An experiment was conducted in which sodium titrate nanotubes acted as activators in the transesterification reaction of processed cooking oils to produce biodiesel. The morphology and microstructures were observed using BET and TEM, whereas the chemical bonds were studied by FT-IR spectroscopy. It was revealed that sodium titrate nanotubes of average diameter 5.37 nm and length varying from 50 to 80 nm (TEM) showed the presence of water molecules on titanate structures (FT-IR) and surface area of 120 $m^2 g^{-1}$ (BET). The catalyst activity was observed at different conditions for 2 h, and it was found that 95.9% of biodiesel was yielded at 80 °C. Zaki et al. (2019) reported that with an increase in sodium titrate nanotubes (catalysts) and the amount of cooking oil, the biodiesel production increased. Hence, the utilization of cooking oil for biodiesel production was very cost-effective, and the output (biodiesel) was found to be 1.2 times greater than the input (cooking oil) in which energy means highly productive (Zaki et al. 2019).

In recent years, significant consideration has been given to the production of biodiesel using various green sources like animal fats or vegetable oil. Also, the use of functionalized multiwalled carbon nanotubes (MWCNTs) has a huge contribution to the production of biodiesel. A comparative study was made in which a carboxylated MWCNT (MWCNT- COOH) and butylamine MWCNT (MWCNT- BA) were used for immobilization of *Candida antarctica* lipase B (CALB) for the enzymatic production of biodiesel using rapeseed oil. The morphological characteristics of MWCNTs were observed, which revealed that the diameter of functionalized MWCNTs was less than 20 nm (TEM), and the presence of carbon, nitrogen, and oxygen was confirmed (XPS) which led to the increased of the absorptivity of nanotubes. It was observed that a maximum yield of 92% was obtained when MWCNT-COOH was used, whereas the yield decreased to 86% when MWCNT-BA was used. Rastian et al. (2016) reported that immobilization of CALB stabilized the reaction and increased the output of biodiesel (Rastian et al. 2016). Lately, the production of biodiesel via transesterification reaction using refined vegetable oil, as the primal matter, has been known to be the most common approach. To examine the efficiency of sulfuric acid-regenerated MWCNTs as an activator to produce biodiesel using palm fatty acid distillate (PFAD), an experiment was conducted. The MWCNTs were 40–60 nm in diameter and 1–2 μm in length which was measured using SEM and FT-IR spectroscopy. It was observed that the as-synthesized MWCNTs produced less biodiesel as compared to the acid-regenerated MWCNTs. This was because the regenerated MWCNTs were cut into shorter tubes which enhanced their ability for esterification reaction. It was stated that acid regeneration of MWCNTs increased their catalytic activity, thus leading to a yield of 93.5% under certain conditions. Hence, acid-regenerated MWCNTs were found to be a very efficient catalyst to produce biodiesel using PFAD (Shuit and Tan 2015).

Considering the global environment, it is necessary to center the renewable energy sources and the production of biofuels. Transesterification reaction of oil feedstock using lipase as the catalyst is the most favorable method for the production of biodiesel. In another experiment, superparamagnetic multiwalled carbon nanotubes (MWCNTs) loaded with iron oxide and functionalized with polyamidoamine (PAMAM) were immobilized on *Burkholderia cepacia* lipase (BCL) and further used for biodiesel production. TEM revealed the dimension of around 40 nm of modified MWCNTs, whereas FT-IR spectroscopy was used to observe the lipase immobilization on MWCNTs. It was observed that biodiesel yield was maximum in the presence of water (2%), the temperature was in the range of 25–35 °C, and the ratio of oil to methanol increased from 1:2 to 1:5. Fan et al. (2016) reported that BCL-immobilized m-MWCNTs-PAMAM were the most effective catalyst to produce biodiesel using transesterification. Thus, magnetized BCL-

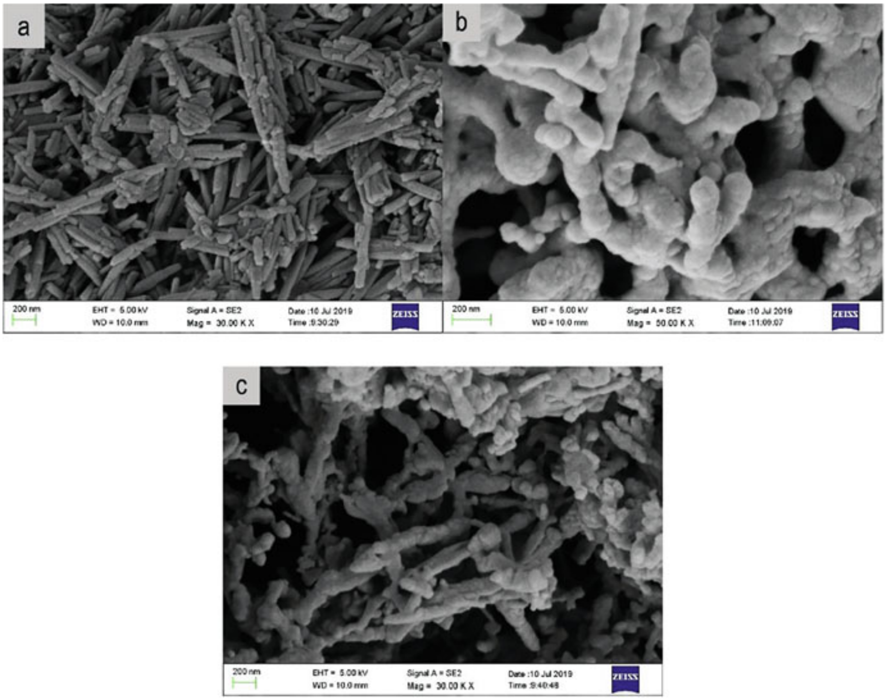

Fig. 13.1 SEM images of different samples: (**a**) HNTs, (**b**) HNTs-Ca, (**c**) HNTs La-Ca

mMWCNTs-PAMAM are proved to be the most promising nanobiocatalysts for increased production of biodiesel (Fan et al. 2016).

Halloysite is known to be a magnificent nanomaterial and has a nanotube-like structure with a layer of aluminosilicate. It has been strongly used in water purification, energy storage, and separation of gas. The nanocavity structure resists the mass transfer and the very effective catalytic activity of the halloysite. In this experiment, functionalized halloysite nanotubes (HNTs) with La-Ca (lanthanum and calcium) bimetallic oxide were utilized for the catalysis of transesterification reaction producing biodiesel. The catalyst HNTs/La-Ca of diameter 18.35 nm and crystalline phase structure was characterized using SEM (Fig. 13.1) and XRD, respectively. It was observed that along with La_2O_3 and CaO, HNTs-La/Ca catalysts were highly efficient and produced a yield of 97.5%. Lin et al. (2020) noted that the halloysite nanotubes when functionalized gave the highest productivity with 88.7% yield at the fifth reused cycle. Thus, a promising and favorable stimulus for the transesterification reaction for effective biodiesel production was identified (Lin et al. 2020).

Immobilization of various forms of lipases has been very helpful for biodiesel production; lipases can be immobilized both covalently and non-covalently, but the covalent immobilization was proved to be more stable and efficient. An experiment was conducted in which *Candida antarctica* lipase B (CaL-B) was immobilized covalently on carboxylated SWCNTs and used to produce biodiesel. SWCNTs of 0.8–1.6 nm (inner diameter) and 1–2 nm (outer diameter) and length of 5.33 μm (observed under TEM) were utilized for this experiment. It was observed that the biocatalyst used could convert 83.4% of oil in 4 h at 35 °C retaining more than 90% after ten reusable cycles. Bencze et al. (2016) noted that covalent immobilization of enzyme was very favorable and stable for the biocatalyst since it generated conformational changes in the protein. Thus, a very active and useful catalyst to produce biodiesel was advanced (Bencze et al. 2016).

Extensive use of nanoparticles especially magnetic carbon nanotubes (m-CNTs) has been observed for the immobilization activity due to their attributes like immense surface area, chemical stability, porosity, and elevated heat conductivity. In this experiment, *Rhizomucor miehei* lipase (RML) is immobilized onto magnetic MWCNTs modified with polyamidoamine (PAMAM) dendrimer and used for the transesterification reaction of waste vegetable oil. The synthesized catalyst was studied using TEM and FT-IR which revealed the diameter of MWCNTs to be around 40 nm, and the presence of PAMAM and immobilization of RML was confirmed. After immobilization of the enzyme, the recovery activity reached 2808%, and the reaction activity went up 27 folds when compared with free enzymes. Fan et al. (2017) noted that 94% of the waste oil could get converted into biodiesel when PAMAM-m-MWCNTs immobilized with RML were used as catalysts in the transesterification reaction. A very efficient, stable, and reusable catalyst was developed which produced high-quality biodiesel (Fan et al. 2017).

Recently, the use of a solid acid catalyst containing ion-exchange resins, sulfated oxides, mobile composite material number 41, and Nafion has been reported. But they have a disadvantage that the acid side of the catalyst easily gets reduced by hydration of acidic hydroxyl groups (OH). The sulfonation and MWCNTs were further characterized, and it was noted that MWCNTs have poor dispersibility in methanol (UV-vis spectra) and average poor diameter (12.3 nm) and surface area (92.37 $m^2 g^{-1}$) (BET). An effective protonic acid catalyst was prepared by grafting MWCNTs with SO_3H (sulfonation), then treating it with sulfuric acid, and further incorporating it with ultrasonication. The catalytic activity was observed to produce biodiesel. It was examined from the experiment that the optimum condition for synthesis was 10 wt% $(NH_4)_2SO_4$ solution with 10 min of ultrasonication treatment at a temperature of 235 °C. Shuit et al. (2015) reported that this method of sulfonation for catalyst production was very feasible, less toxic, and economical. Also, the produced catalysts had good thermal stability and dispersibility. In conclusion, s-MWCNTs prepared from $(NH_4)_2SO_4$ solution was advanced technology for production of biodiesel (Shuit et al. 2015).

Nanotubes and nanorods are the most promising and advanced one-dimensional nanostructures which are widely used as catalysts for different applications. One of the types of nanotubes is kaolinite nanotubes which possess a very large surface area and porous structure. An experiment was conducted in which kaolinite nanotubes were synthesized and doped with potassium ions (K+/KNTs) and further used for catalysis in the transesterification reaction. The doped nanotubes were characterized using XRD and BET techniques. It was observed the results after experimenting 4 h using 6 wt% of K^+/KNTs catalyst that 99.4% of biodiesel was extracted from the sunflower oil. A novel catalyst was developed with 14.5 nm pore diameter and 7.43 mmol OH/g basicity which could produce biodiesel with high quality and properties (Abukhadra et al. 2020).

The most frequently used feedstock for biodiesel production is food-grade crops, yet there are some issues related to it like seed oil containing high free fatty acid (FFA) that cannot be converted into biodiesel in the presence of catalyst since saponification can occur. Thus, whenever a high FFA content oil is used as feedstock, biodiesel was produced using an acid catalyst. In this experiment, MWCNTs functionalized with sulfonated organosilane were used to fabricate biodiesel from high FFA seed oil. With a rise in the temperature, the mass of MWCNTs decreased (TGA), and the functionalization of acidic groups on MWCNTs added stability to it. Macawile et al. (2020) reported that conversion of high FFA accommodating oil into biodiesel was a two-step process and was very feasible in the presence of SO_3H-MWCNTs acid catalyst. Thus, a very efficient acid catalyst was successfully developed (Macawile et al. 2020).

Nanotubes are widely used in the production of biofuels; one of the most common types of nanotubes used is carbon nanotubes. Goh et al. (2012) filled single-walled carbon nanotubes (SWCNT) with iron oxide to obtain magnetic single-walled carbon nanotubes (mSWCNT). The enzyme amyloglucosidase (AMG) was immobilized on these mSWCNT (through physical adsorption and covalent immobilization) to hydrolyze starch, a biomass prototype used in this study. The carbon nanotubes were characterized using TEM and SEM. It was confirmed that SWCNTs of size 20–50 nm (in a bundle) (SEM) were loaded and covered with Fe_2O_3 nanoparticles (TEM). Also, SEM images showed adsorbed AMG over mSWCNT. It was determined that mSWCNT showed decreased enzymatic activity, which helps in less alteration in the structural conformation (CD) of AMG, eventually making it possible to recycle AMG for further use. It was also proved that AMG-immobilized mSWCNT increases the formation of AMG and other enzyme complexes, hence increasing the production of biofuel (Goh et al. 2012).

13.2.2 Nanocatalysts

13.2.2.1 Metal Oxides

With an increasing amount of research on biodiesel production using nanocatalyst, Gurusamy et al. (2019) conducted a study on *Ulva lactuca* seaweed to elevate the production of biodiesel in the presence of TiO_2-ZnO nanocomposite catalyst. The nanocatalyst was prepared using the coprecipitation technique and consecutively characterized using TEM which revealed that the size of nanocatalyst of around 12 nm and XRD confirmed its amorphous nature. The conversion of hydroxydecanoic acid to fatty acid methyl ester (FAME) was performed under

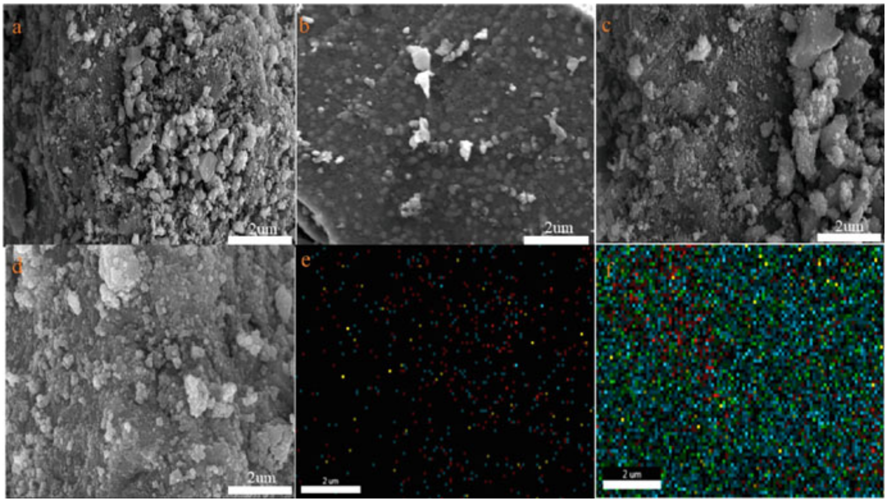

Fig. 13.2 (**a**) The SEM images of ZnO-TiO2–500 °C, (**b**) the SEM images of La3+/ZnO-TiO2–500 °C, (**c**) the SEM images of La3+/ZnO-TiO2–600 °C, (**d**) the SEM images

optimum and mild conditions, i.e., 4 wt% catalyst, 60–80 °C reaction temperature, and 4 h of reaction time. It was noted that a biodiesel yield of 82.8% was attained upon conversion from hydroxydecanoic acid to FAME. Furthermore, authors used the leftover algal biomass to synthesize silver nanoparticles (AgNPs) to study its antimicrobial activity (Gurusamy et al. 2019). Another study was conducted using waste cooking oil as biomass to produce biodiesel with the help of La^{3+}/ZnO-TiO_2 photocatalyst. Guo et al. (2021) prepared La^{3+}/ZnO-TiO_2 photocatalyst through the sol-gel method and characterized it. It was found that TiO_2-ZnO nanocomposite of size 8–11.5 nm (TEM), calcination temperature of 400–600 °C (TG), and crystal structures were observed on the surface using SEM-EDS (Fig. 13.2). Initially, the experiment was conducted under optimal conditions like reaction temperature of around 35 °C, 4 wt% of catalyst dosages, an ethanol-oil ratio of 12:1, UV irradiation, and reaction time of 3 h which resulted in 96.14% conversion of waste cooking oil to ethanol through photocatalytic esterification. Consecutively, transesterification of waste cooking oil was accomplished with the help of NaOH as a catalyst. Although after five cycles of this experiment it yielded 87% biofuel, this method was proved to be more stable and sustainable (Guo et al. 2021).

Like the previous experiments, Lin et al. (2020) performed an experiment using functional CaO/Au nanocatalyst for the production of biodiesel from soybean oil. However, in this experiment, Au (III)-polluted waste eggshell was used to synthesize CaO/Au nanocatalyst and further use it for biodiesel production. To achieve this goal, the eggshell powder was prepared and used for the synthesis of CaO/Au nanocatalyst; furthermore, the catalyst was characterized. It was observed that a thick layer of Au nanoparticles was found over the surface of CaO (XRD) making the particle size up to 2–5 nm (TEM); also, EDS confirmed even distribution of Au nanoparticles on CaO. The transesterification process was performed with the help of methanol and soybean oil in a molar ratio of 12:1, 1 wt% catalyst concerning soybean oil, 70 °C reaction temperature, and 3 h of reaction time. The results obtained reported a total biodiesel yield of 88.9% and reusability up to five times without degradation in its efficiency (Liu et al. 2021).

The transformation of biomass into biofuels can be achieved using magnetic nanoparticles. Mapossa et al. (2020) conducted a study using $NiFe_2O_4$ and $Ni_{0.3}Zn_{0.7}Fe_2O_4$ magnetic nanoparticles to convert soybean oil into biodiesel. $NiFe_2O_4$ and $Ni_{0.3}Zn_{0.7}Fe_2O_4$ were synthesized by combustion reaction with the help of a conical reactor. The crystallinity of $NiFe_2O_4$ and $Ni_{0.3}Zn_{0.7}Fe_2O_4$ nanoparticles was found to be 55% and 72%, respectively (XRD), and the size of the nanoparticles was confirmed to be in the range of 13–20 nm. The experiment was performed under controlled optimal conditions, i.e., 2 wt% catalyst dosage, reaction temperature of 180 °C, a reaction time of 1 h, and oil-to-alcohol molar ratio of 1:12. The biodiesel yield was found out to be 94%; this procedure was conducted due to its efficiency, stability, and high activity, and hence this method can be helpful in the immediate future (Mapossa et al. 2020). Similarly, another experiment was conducted by Guo et al. (2012) in which a magnetic solid base catalyst made up of Na_2SiO_3 and Fe_3O_4 nanoparticles were used for converting cottonseed oil to biodiesel or FAME. Firstly, the synthesis of catalysts was carried out by depositing Na_2SiO_3 and Fe_3O_4 nanoparticles; consecutively, catalyst showing high catalytic activity was obtained at 350 °C calcination temperature, 2 h of aging time, Si/Fe molar ratio of 2.5, and calcination time of 2.5 h. The nanocatalyst was characterized by VSM and TEM to determine its magnetic strength and morphology, respectively. It was found out that Fe_3O_4 nanoparticles were spherical ball-like structures of size 20 nm (TEM) with high magnetic strength (VSM). Under optimal conditions like 60 °C reaction temperature, 100 min reaction time, 1:7 oil-to-methanol molar ratio, 5 wt% catalyst concentration, and 400 rpm stirring speed, the biodiesel yield was analyzed. A yield of 99.6% was obtained with a very good recovery rate of Na_2SiO_3/Fe_3O_4 nanocatalyst (Guo et al. 2012).

Recently, Santha et al. (2019) produced an experiment in which sustainable biofuel was produced, using CuO nanoparticle-based heterogeneous nanocatalyst, with the help of biogenic waste. Transesterification was carried out on waste cooking mustard oil using CuO nanoparticles as a catalyst which were fabricated by the coprecipitation method. The formation of CuO nanoparticles of size 13 nm and morphology resembling facets broken flower was confirmed using XRD and FESEM, respectively. EDAX was used to check the presence of impurities in the CuO catalyst, after which the transesterification process was carried out. During this process, certain optimal conditions were maintained (temperature 70 °C, time 2.5 h, and centrifuged at 4200 rpm for 15 min) to separate biofuels from by-products. A FAME yield of 88% was achieved through the ultrasonic reactor method, and the presence of methyl and ester groups was confirmed using FT-IR (Santha et al. 2019). Another experiment, in which potassium fluoride (KF) was infused on CaO/NiO, was performed to study the conversion of waste cottonseed oil to biodiesel. 20 wt% KF was impregnated on CaO/NiO through the wet impregnation method and consecutively characterized by TEM, SEM, XRD, and BET. Based on the characterization, it was revealed that rhombus-shaped cluster particles of the catalyst of size 150 nm were formed (TEM and SEM); also the surface area of CaO/NiO decreased with an increase in KF impregnation (BET). Now, the transesterification process was carried out at 65 °C, for 4 h, with 5 wt% catalysts and methanol-to-waste cottonseed oil molar ratio of 15:1 to convert waste cottonseed oil to FAME. Kaur and Ali (2014) reported biodiesel yield of more than 96.5% and reusability up to four times without any degradation in its efficiency (Kaur and Ali 2014). The production of renewable energy has captivated many researchers due to the diminishing fossil fuel reserves. Biodiesel is the most environmentally safe fuels because of its properties like clean, nontoxic, and aroma-free nature. In this experiment, the CaO nanocatalyst was synthesized through the sol-gel method to churn out biodiesel. It was observed that the surface methodology using CaO nanocatalyst was very efficient and 97.61% yield was obtained. Further, it was reported that the conversion of soybean oil to biodiesel was observed to be 60 °C for 2 h with 3.675 wt% of the catalyst. Thus, CaO as nanocatalyst showed the highest rate of conversion in the transesterification as compared to the other nanocatalyst (Bharti et al. 2019).

In the following study, a palm kernel shell was used for the synthesis of highly mesoporous activated carbon by hydrothermal-assisted carbonization (HTC) for an enhanced catalytic activity. Waste cooking oil was used as a biomass prototype, to which the activated carbon acts as a degrading agent. Furthermore, the HTC-based activated carbon was infused with K_2CO_3 and CuO (through wet impregnation) to carry out esterification and transesterification processes. Using different characterization methods, the presence of K_2CO_3 and CuO nanoparticles was determined (FESEM) also, and the formation of a broad band in the range of 1366 cm^{-1} and 1346 cm^{-1} was determined (FT-IR). Abdullah et al. (2021) reported an increased production of biodiesel when 4 wt% of the catalyst was added, while 6 wt% of catalyst did not show any changes; however, it disturbed the mixing process between oil, methanol, and catalyst. The catalyst helped in increasing the contact between the active sites on the catalyst and reactant for esterification and transesterification processes (Abdullah et al. 2021).

Pollution is the main problem all over the world, and one of the main causes of pollution is excess use of fossil fuels (Chowdhary and Raj 2020). New techniques are being introduced to produce nonpolluting fossil fuels that would be safe for the environment and available on a large scale. One such technique is the use of seed oils like sunflower, soybean, date seed, etc. An experiment was conducted in which recycled cooking oil was used to produce biodiesel via transesterification reaction with nano-CaO and nano-MgO as catalysts. The nanocatalysts were synthesized by the sol-gel method. It was observed that nano-CaO was more efficient as compared to nano-MgO due to the high contact area of CaO. Tahvildari et al. (2015) stated that biodiesel yield was enhanced when both the catalysts were used together. Nano-MgO was not a very efficient catalyst when used individually due to its basic affinity, but when combined with CaO, it gave highly promising results (Tahvildari et al. 2015). The fuel prices have gone unreasonably high due to an increase in demand and decrease in amount. To overcome this issue, alternative fuels that are easily producible and renewable are being synthesized using green sources like vegetable oils. Waste cooking oil is one of the feedstocks used for biodiesel production since it cannot be used for human consumption and has low cost and high availability. MgO nanocatalysts were produced using the coprecipitation method and used in the synthesis of biodiesel from waste cooking oil. The nanostructured phase and characteristics were observed using UV-vis and FT-IR spectroscopy. The MgO nanoparticle size and energy bandgap (Mg-O) were observed to be 7.86 nm and 5.84 eV (FT-IR and UV-vis). Ashok et al. (2018) investigated the biodiesel yield of 93.3% with 2 wt% of MgO catalyst at a temperature of 65 °C. Based on the experiment, it was inferred that MgO nanocatalyst was very effective in producing high-quality biodiesel (Ashok et al. 2018).

Characteristics like magnetically controlled drug delivery, sensors, memory storage devices, and catalysis make magnetic nanoparticles highly promising in the field of biofuel production. Also, they can be recovered and reused which makes them cost-effective too. In this experiment, the Fe_3O_4 nanoparticles were modified with tetraethyl orthosilicate (TEOS) and 3-chloropropyl trimethoxysilane (CPTMS) and then immobilized with different amines for transesterification of soybean oil for biodiesel production. Characterization of the modified nanoparticles was done using FTIR, SEM, and TEM spectroscopy. The particle sizes were revealed to be 45 and 65 nm (SEM and TEM), and the immobilization of amines was confirmed through FT-IR spectroscopy. A yield of 96% was observed at 160 °C within 3 h with 6% of the modified nanoparticles used as catalysts. Farzaneh et al. (2018) reported that the nanoparticle Fe_3O_4@SiO_2@CPTMS@amine (nanocatalyst) was highly stable and reusable because of which they were very efficient in the production of biodiesel. Thus, functionalization and modification of the nanoparticles increased their efficiency and activity rate (Farzaneh et al. 2018).

The use of renewable sources of energy due to the excessive use of petroleum and depletion of fossil fuels has become indispensable. Biofuels are produced using various bioresources and thus do not possess any kind of harm to the environment. An experiment was conducted in which microalgae *Chlorella vulgaris* was used to produce fatty acid methyl esters (FAMEs) type of biodiesel. Superparamagnetic few-layer graphene oxide and Fe_3O_4 (MGO) immobilized with lipase (ROL) were used as nanobiocatalysts in the transesterification reaction. The characterization of graphene oxide and its functionalization with 3-aminopropyl triethyoxysilane (AP) and glutaraldehyde (GA) was studied using SEM, XRD, and FT-IR spectroscopy. The graphene oxide layers were characterized by AFM. It was observed that the highest amount of biodiesel conversion occurred when ROL/MGO-AP-GA was used as a nanocatalyst. Tahir et al. (2020) reported that ROL/MGO-AP-GA was the most efficient catalyst even after reusability and converted microalgae bio-oil into biodiesel effectively. Thus, the functionalization of MGO improved their characteristics like loading capacity, thermal stability, and storage stability and successfully produced a large amount of biodiesel from microalgae (Nematian et al. 2020).

In the following experiment, biodiesel was produced by electrolysis from two microalgae strains, namely, *Chlorella vulgaris* and *Spirulina platensis*, using CaO/KOH-Fe_3O_4 and KF/KOH-Fe_3O_4 as magnetic nanocatalysts. SEM and XRD were used to characterize the nanocatalysts, and the sizes were measured to be 55.91 nm and 42 nm, respectively. Electrolysis technique was conducted using two graphite electrodes, and the production of biodiesel at various conditions like weight percentage of catalyst, reaction time, and the ratio of alcohol was observed. It was depicted that the optimum condition for biodiesel production was at 25 °C using 1.5 wt% of catalyst at a molar ratio of 1:6 (methanol/oil). Farrokheh et al. (2020) submitted a report stating that *Chlorella vulgaris* showed better results in the yield as compared to the *Spirulina platensis* microalgae and that the KF/KOH-Fe_3O_4 were highly efficient nanocatalyst since they took less processing time and saved raw material. Thus, the method of electrolysis was highly potential; it reduced the transesterification reaction time and increased the efficiency of biodiesel (Farrokheh et al. 2020). Production of biodiesel through the transesterification reaction of vegetable oils and fats with methanol is the most acquainted and efficient technique. Among the vegetable oil, castor oil is the highly produced oil in India and is known for its high viscosity and density. In this experiment, castor oil was used to produce biodiesel in the presence of a heterogeneous Ni-doped ZnO nanocatalyst. The structural and functional characterization of Ni-doped ZnO nanocatalyst was done using AFM and XRD spectroscopy. It was observed that the Ni-doped ZnO increased the catalytic activity due to the surface area, and a maximum yield of 95.20% was noted. Baskar et al. (2018) reported that the optimum conditions for the biodiesel yield through castor oil were 55 °C for 60 min, 11 wt% of catalyst, and 1: 8 ratio of oil/methanol. Consequently, the nanocatalyst was highly promising to produce biodiesel from castor oil which is a low-cost feedstock (Baskar et al. 2018).

The nanocatalytic technology has played a vital role in the synthesis of biodiesel through a transesterification reaction. They are well known for increasing the efficacy of biodiesel production from various vegetable oils. The nanocatalysts modify the surface morphology for efficient production, whereas other catalysts such as carbonates, alkalis, zeolites, etc. do not actively modify the surface at the atomic scale. An experiment was conducted in which magnesium oxide nanocatalysts were used for biodiesel production through surface structural modifications. The prepared nanocrystalline-MgO particles underwent in situ aberration-corrected TEM, and the XRD technique was used to study other structural properties. Following the in situ AC-TEM results, it was observed that vacancies were devised at oxygen sites in the plane connecting Mg atoms. Thus, Gai et al. (2009) submitted a report stating that the vacancies between adjacent Mg-Mg atoms were the active sites and responsible for high biodiesel synthesis (Gai et al. 2009).

The destructive effect of pollution and high use of nonrenewable and toxic fossil fuels has laid a foundation for an alternative energy production method that is less toxic and detrimental to the environment. Presently many new techniques are being identified to produce biofuels through various green sources, one of which is the transesterification reaction of vegetable oils with the application of heterogeneous catalysts. A study was made to examine the efficiency of two different nanostructured oxides and their activity for biodiesel production. The nanoparticles synthesized were characterized using BET and SEM analysis. The size of nanoparticles was examined to be 15–55 nm (SEM). Parameters like acidity/basicity and surface area (BET) were analyzed, and it was observed that the highest activity was related to surface basicity, not surface area. Further, it was reported that heterogeneous catalysts were dependent on temperature and only CaO was active at 70 °C converting 98% into biodiesel, whereas all other oxides were active at 150 °C. Thus, it was detected that in catalysis the effect of surface basicity was superior to that of particle size, and it was successful in advancing the transesterification reaction (Do Nascimento et al. 2012).

During the past decades, the production of methane-rich biogas through the method of anaerobic digestion (AD) has been established greatly. Scientists have recognized microalgae as a highly promising feedstock to produce biogas. Also, recently, the application of nanotechnology for improved biofuel production on a large scale is being appreciated. In this experiment, the potential of *Chlorella pyrenoidosa* (microalgae) in the presence of α-Fe2O3 nanoparticles (IONPs) for biogas production was studied. The IONPs were characterized using TEM and SEM spectroscopy. It was observed that there was an increase in the growth of microalgae in the presence of IONPs and biomass composition was enhanced. Rana et al. (2020) examined from the biochemical methane potential test that biogas productivity was improved with IONP supplementation. A rise of 25.14% with 22.4% enhanced methane content was reported. Consequently, microglial biogas production was a total success (Rana et al. 2020).

13.2.2.2 Metals

In recent years, waste materials are being used to produce biofuels to maintain sustainability and efficiency of biofuels at the same time. Ganesan et al. (2020) produced biofuel from *Jatropha* oil with the help of silver waste generated from discarded X-ray films. Silver nanoparticles (AgNPs) of size 60–70 nm (FESEM) were synthesized by green synthesis using oak gall's extract and *Camellia sinensis* extract. Using various characterization techniques, the absence of impurities in AgNPs (EDX) and crystallite size of AgNPs (25 nm) was confirmed (XRD). Moreover, the catalytic cracking process was initiated by maintaining catalytic cracking conditions, i.e., the temperature range was kept between 400 and 500 °C at a fixed WHSV (= hourly mass feed flow rate/*catalyst* mass). The oil was then passed through a condenser (0 °C), because of which gaseous products, as well as liquid products, were formed and collected. The obtained products were analyzed using the GC analysis technique, based on which it was revealed that 90% *Jatropha* oil was converted to 85% biofuel and 79% gaseous product, i.e., gasoline (Ganesan et al. 2020).

Biomass is known to be the premier green source of renewable energy and supplies 14% of the total energy demand. This is because of its abundant nature in all the areas like agriculture, forest residues, and unwanted plants (weed). An experiment was conducted in which two weed plants *Cannabis sativa* and *Parthenium hysterophorus L.*as biomass for biodiesel production were used in the presence of nanocatalysts Co and Ni. The structural details of the nanocatalyst were measured using SEM and XRD spectroscopy. Gasification of *Cannabis sativa* led to the extraction of 12% gas, 53% oil, and 34% biochar, whereas *Parthenium hysterophorus L.* extracted 17% of gas, 44% oil, and 38.36% biochar. Tahir et al. (2020) reported that *Cannabis sativa* was more efficient and produced 53% more biodiesel as compared to *Parthenium hysterophorus L.* Hence, the production of biodiesel from unwanted plants like weeds had a high potential to avail the energy demands (Tahir et al. 2020). Unwanted weed biomass from cultivated fields was found to be potential biomasses in the biofuel production. This experiment was performed to check the activity of nickel and cobalt nanoparticle-based nanocatalyst on the lignocellulosic part of weed biomass. Ali et al. (2020) used different weed plants like *Carthamus oxyacantha*, *Asphodelus tenuifolius*, and *Chenopodium album* to produce biodiesel, biochar, and biogas using nanocatalyst. Initially, nickel and cobalt nanoparticles were produced using hydrothermal method and characterized TEM, XRD, and SEM. It was found that nickel and cobalt nanoparticles of size 2–90 nm (SEM), when used for the gasification of weed biomass, resulted in the yield of 18% biochar, 23.75% biogas, and 57.5% bio-oil which was further analyzed for transesterification. GC-MS results revealed that the biodiesel sample accommodated 65.47% esters, which demonstrates the improved quality of biodiesel (Ali et al. 2020).

Recent works have proved magnetic nanoparticles to be highly proficient in catalysis. They have emerged on a large scale due to their properties like magnetic separation from the reaction medium and surface area. The aim of this experiment was an extraction of biodiesel from soybean oil using magnetic mixed iron/cadmium (ICdO) and iron/tin oxide (ISnO) nanoparticles as catalyst. No significant difference in the catalytic activity of both the catalysts was detected, which led to an equal yield of biodiesel. Alves et al. (2014) reported that 90% of fatty acid could be converted into biodiesel using ISnO and ICdO as catalysts and that these catalysts could be magnetically recovered and reused. Hence, they were recognized as catalysts showing high potential in the generation of biodiesel and were also cost-effective (Alves et al. 2014). Researchers have recognized bimetallic nanoparticles as highly promising metal nanoparticles for the production of biodiesel. They have certain characteristics and better reactivity which make them a suitable catalyst for the transesterification reaction. This study is about the synthesis of bimetallic gold-silver core-shell nanoparticles (Au@AgNPs) and their application in biodiesel synthesis from sunflower oil. The characterization of the catalyst Au@AgNPs was done using TEM, energy dispersive X-ray (EDX) analysis. It was observed that when silver particles were deposited on the gold nanoparticles, it showed high catalytic activity in the transesterification reaction of sunflower oil. Banerjee et al. (2014) reported that the required optimum conditions for biodiesel extraction from sunflower oil are 65 °C for 2 h with catalyst concentration of 5%. The results depicted that Au@Ag core-shell nanoparticles were of high potential as a catalyst for transesterification of sunflower oil (Banerjee et al. 2014).

The demand for biodiesel is seen to be increasing worldwide because of the increase in vehicle population and industrialization. To subdue this problem, biofuels like biodiesel and bioethanol were prepared as an alternative for fossil fuels. An experiment was directed in which a magnetically separable zinc ferrite nanocatalyst was used to produce biodiesel from waste cooking oil. The structural features of nanocatalyst were studied using XRD, FT-IR, and SEM. The average crystalline size was revealed to be around 74 nm (XRD). The results of FT-IR spectra showed the presence of H-O-H vibrations. A maximum yield of 98.6% of biodiesel in transesterification reaction using $ZnFe_2O_4$ nanocatalyst was observed at 60 °C temperatures for waste cooking oil. Ashok et al. (2019) reported that zinc ferrite nanocatalyst was successful in producing biodiesel efficiently through the transesterification reaction of waste cooking oil and is a non-spontaneous reaction (Ashok and Kennedy 2019). In developing countries like India, it has become very necessary to evolve some alternative sources for fuels. Biodiesel and bioethanol production has reached great heights due to their renewable and nontoxic nature. An experiment was conducted in which esterification of palmy fatty acid for biodiesel production was done in the presence of immobilized lipase on magnetic nanoparticles. The structural and morphological characteristics were studied using FT-IR and TEM spectroscopy. Gupta and Rathod (2020) reported 82.47% conversion due to esterification of palm fatty acid into biodiesel with lipase-immobilized magnetic nanoparticles. The nanoparticles were reusable up to five cycles giving a yield of 80.19%. Immobilizing the nanoparticles with lipase increased its efficiency to 85%. Hence, these particles were having great potential as catalysts for producing high-quality, cost-effective, and pollutant-free biodiesel from soybean oil (Gupta and Rathod 2020).

The most suitable biological resource for biodiesel production is microalgae. They possess certain characteristics such as photosynthetic efficiency by harvesting solar radiations, allowing for rapid growth rate and the production of biomass in large quantity. In this study, the impact of carbon/nitrogen (C/N) on microalgal growth and application of nanoparticles for biodiesel extraction. Three types of microalgae species *Chlorococcum* sp., *Scenedesmus* sp., and *Euglena* sp. were isolated, and a comparative study was made in the presence of different carbon sources and titanium (Ti) nanoparticles. The morphological characterization of Ti NPs was done using SEM analysis. Among all the carbon sources used, it was identified that sucrose was the most suitable microalgal biomass enhancement. An increase in biomass from 3.5 g/L to 5.02 g/L and lipid content from 58 to 60% after the addition of Ti nanoparticles was observed. Khanra et al. (2020) reported that the optimum concentration of Ti nanoparticles was 15 ppm with the harvesting potential of 82.46% and the most effective algae for biodiesel production was determined to be *Chlorococcum* sp. Hence, according to the results, the Ti nanoparticles were highly promising and most viable to produce biodiesel from microalgae feedstock (Khanra et al. 2020). The current advancement in processes to produce cost-effective and biodegradable fuel energy with the use of magnetic nanocatalyst has been accepted worldwide. In this experiment, a magnetic nano-sized solid catalyst was produced with the help of *Citrus sinensis* peel ash (CSPA)@Fe_3O_4 and used to produce biodiesel from waste cooking oil. The structural and morphological characteristics were examined using TEM, XPS, XRD, and FT-IR spectroscopy. The average particle size was 12–15 nm (TEM). The chemical composition of the

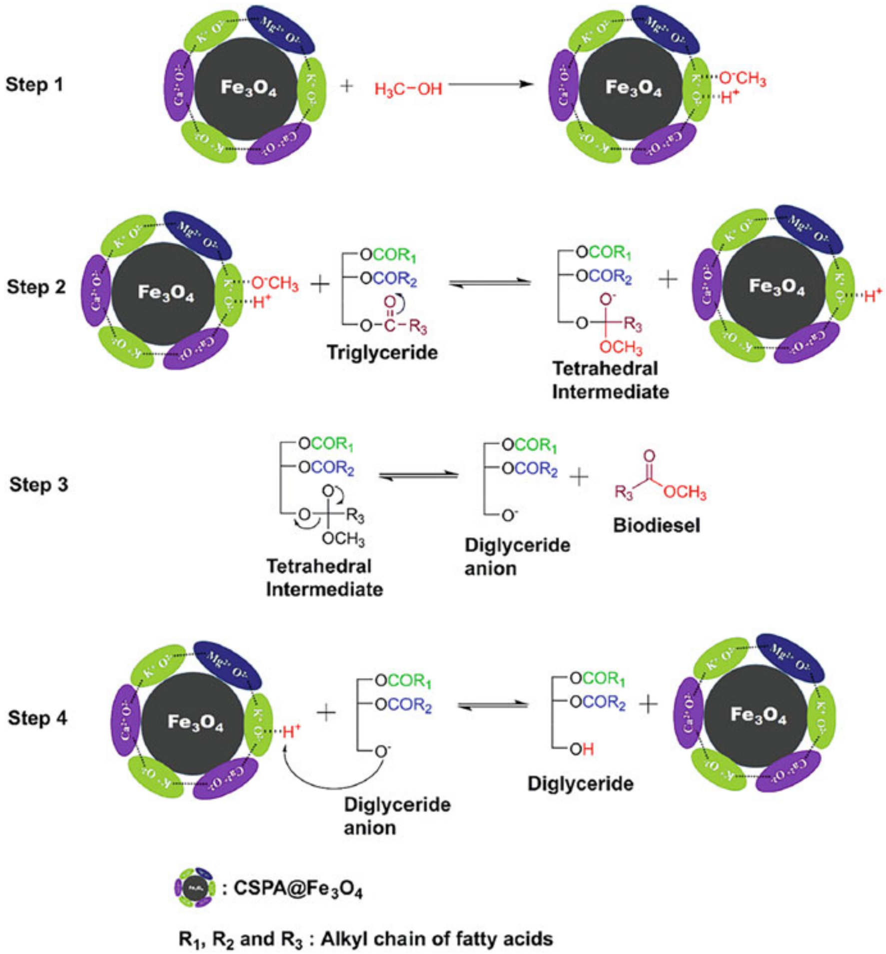

Fig. 13.3 Probable mechanism of CSPA@Fe_3O_4-catalyzed transesterification of WCO

catalyst was observed to be Ca, K, C, and O(XPS). Also, the presence of O-H group was revealed in the catalyst (FT-IR). It was observed that the size, shape, and surface properties of the catalyst were enhanced due to its core-shell structure. Changmai et al. (2021) noted that the synthesized catalyst could give a yield of 98% in the transesterification reaction (waste cooking oil) (Fig. 13.3) and also that the magnetic catalyst was easily recoverable. Therefore, this method using iron oxide nanoparticles was a great success and showed excellent activity in biodiesel production (Changmai et al. 2021).

Microalgae are considered as best source for biodiesel production since it has a high value of triglycerides and polysaccharides and shows increased growth rate in any environmental conditions. A study was made in which microalgal biomass was produced in which lignocellulosic biomass (LCB) was the carbon source and then further biodiesel was extracted from it using iron nanomaterials. The size of iron nanoparticles was observed to be 3.347 nm (SEM). Two types of cultures *Dictyococcus* sp. and *Coelastrella* sp. each 44% and 52% were grown from which FAME was extracted. Sakthi Vignesh et al. (2020) reported that the two microalgae each produced a yield of 90 and 28%, respectively. The lipid content was enhanced by nitrogen starvation for better results. Thus, the product obtained through bio-iron nanoparticles was evaluated for its activity, and quality was found to be highly efficient (Sakthi Vignesh et al. 2020).

Similarly, another study by Ashok et al. (2021) demonstrated the use of waste cooking oil to produce biodiesel with the help of magnetically recoverable magnesium-substituted zinc ferrite nanocatalysts. The characterization techniques revealed that the nanocatalyst of size 8–10 nm (HR-SEM) had a high magnetic moment and saturation magnetization property which is necessary for the separation of the catalyst from the reaction medium (VSM). Furthermore, it was revealed that at standard conditions such as 3 wt% of nanocatalyst dosage, 18:1 methanol-oil ratio, a reaction time of 30 min, and 65 °C reaction temperature, the biodiesel yield increased to 99.9%. Also, the magnetically separated nanocatalyst could be recovered up to ten cycles with 94% yield of biodiesel (Ashok et al. 2021).

13.2.3 Nanocomposites

Nanobiotechnology is a branch of biotechnology that has promising applications in all aspects of science and technology. Enzymes are the biological catalysts that enhance the speed of any reaction, but recent studies have evolved their function by immobilizing it with any type of nanomaterials. This study is about how immobilization of different nanomaterials like nanoparticles, nanocomposites, and nanofibers on enzymes can be considerate for the stabilization and production of biofuels. Different nanomaterials along with enzyme immobilization were characterized using BET, AFM, XRD, and, FT-IR spectroscopy analysis. Verma et al. (2013) noted that enzyme activity was increased after immobilization because nanomaterials have a large surface area and thus load a large quantity of enzymes on them. Also, because of their high tensile strength, they cannot break easily. Hence, this research leads to an establishment of a novel nanobiocatalytic technique that was very suitable to produce biodiesel (Verma et al. 2013).

Another way of producing biodiesel from biomass is through an electrocatalytic process which involves the use of electrodes made from nanoparticles. This experiment was performed to transform waste palm oil to biodiesel with the help of Pt-Ni/NiO/Ni_2O_3 nanocatalyst-based electrodes. Pt-Ni/NiO/Ni_2O_3 nanocatalyst was synthesized and characterized using TEM, XRD, DTG, TG, FT-IR, and Raman analysis. It was found that nanocatalyst of size 50 nm with quasi-spherical morphology consisted of a bridge between Pt and Ni/NiO/Ni_2O_3 nanoparticles (TEM), and the presence of nickel oxides was confirmed by FT-IR, whereas Raman analysis helped in the confirmation of the formation of Pt and Ni-Ni oxide nanoparticles. Sarno et al. (2020) performed transesterification of palm oil into biodiesel using low voltage and temperature, i.e., 10 V and 40 °C, respectively, in a two-phase system with less amount of nanocatalyst. The palm oil was treated at 240 °C for 7 days with a reaction time of 3 h, and a high yield of biodiesel was observed. A biodiesel yield of 96.2% was obtained, proving it as a substitute in the production of biodiesel in a sustainable way (Sarno et al. 2020). Likewise, a heterogeneous base nanocatalyst was used to produce biodiesel from vegetable oil. In this experiment, Se-doped ZnO nanocatalyst was used to carry out a transesterification process to convert vegetable oil to FAME and eventually into biodiesel. Se-doped ZnO nanorods were prepared by mechanothermal method and characterized using XRD, XPS, HR-TEM, and FE-SEM. XRD confirmed the presence of doped Se in ZnO crystal, while the rod-shaped structure of Se-doped ZnO nanoparticles of an average diameter of 50 nm was determined using FE-SEM and HR-TEM, respectively. Rao et al. (2021) performed a transesterification process under optimal conditions such as 65 °C temperature, 5 wt% catalyst concentration, 1:20 oil-to-methanol volume ratio, and 3 h of reaction time. A total biodiesel yield of 94.7% was obtained, along with which it was found that the catalyst was recyclable up to five cycles without any degradation in the FAME yield (Rao et al. 2021).

With the arrival of new technologies, various methods are used to convert fatty acids into biofuels or fuel additives. One of these technologies is the use of nanocomposites as heterogeneous catalysts for sustainable production of biodiesel and fuel additives. Peixoto et al. (2021) prepared organo-sulfonic aryl-silica nanoparticles (by post-grafting methods) as a highly active heterogeneous catalyst for the esterification of free fatty acids (FFA), levulinic acid (LA), and aldol condensation of furfural. Firstly, characterization of nanoparticles was studied using XPS, TEM, SEM, and FT-IR (average size of 10–100 nm), the structure (uniform spheres) and presence of oxygen, carbon, and silica on the surface of nanoparticles were confirmed. Furthermore, in the case of esterification reactions of FFA and LA, the temperature was kept at 120 °C for 2 h with 10 wt% of catalyst dosage and reused for 6 and 10 cycles, respectively. On the other hand, for the aldol condensation reaction, the temperature was maintained around 65 °C for 2 h. Based on the results, it was revealed that esterification of LA showed 100% biodiesel yield with 83.2% yield even after ten cycles. Moreover, the aldol condensation reaction of furfural with 3-methyl furan produced a high yield within 10 min of reaction proving its high catalytic effect (Peixoto et al. 2021).

The following study was conducted on cottonseed oil to produce biodiesel using nanocrystalline lithium impregnated calcium oxide as a nanocatalyst. Li^+ ions were impregnated over CaO by the wet impregnation method; additionally, their structural and chemical characteristics were studied using XRD and TEM. These techniques helped confirm the size (50 nm) and nanostructure of the catalyst. Furthermore, the transesterification process was carried out under optimal conditions, i.e., reaction temperature of around 35 to 65 °C, time of 0.5–8 h, catalyst dosage of 5 wt%, and oil-to-methanol molar ratio of 1:12. Along with these, moisture content of up to 15 wt% and free fatty acid content up to 6 wt% were maintained. Kumar and Ali

(2010) reported that when the moisture content and free fatty acid content were kept high, the production of biodiesel from cheap feedstock was found to be more, making it possible to reduce the production cost of biofuels (Kumar and Ali 2010).

Fatty acid alkyl ester (FAME) is not only renewable but also biodegradable and less hazardous. Lipases are recognized to be an efficient catalyst especially lipase deriving out of yeast. In this work, *Magnusiomyces capitatus* yeast was used as a source of lipase. The *M. capitatus* A4C extracellular lipase was an extraordinary biocatalyst that could catalyze both the esterification and transesterification reactions. $CuSO_4.5H_2O$ hybridized with ECL was used for biodiesel production, and its structural analysis was done with the help of SEM and FT-IR. The morphological changes that occurred after the hybridization of $CuSO_4.5H_2O$ were observed (SEM). The results observed were 89.7% conversion of triglycerides into FAME while 98.7% of oleic acid into FAME in an equal time. Baloch et al. (2021) reported that the ECL-$CuSO_4$ hybrid was preferable for the esterification reaction as compared to the transesterification. So, the biocatalyst produced from *M. capitatus* yeast had a very promising effect on biodiesel production as an alternative for depleting fossil fuels (Baloch et al. 2021).

As a result of increased population and industrialization, it has been observed that the need for energy sources is also increasing. To meet this increasing demand, new techniques and sources are being established to produce renewable, nontoxic energy. Transesterification is one of the techniques utilized to generate fatty acid methyl esters (FAME) used as biodiesel. This reaction is usually catalyzed by nanomaterials and is the most suitable catalyst to produce biodiesel. In this experiment, transesterification of soybean oil was achieved with the influence of CaAlSi mixed oxide nanoparticles. The structural and morphological characteristics of oxide nanoparticles were examined using XRD, FT-IR, and SEM spectroscopy. It was observed that CaAlSi nanoparticles were effective in catalyzing the transesterification reaction of soybean oil. Farzaneh et al. (2017) submitted a report which described the most suitable condition for biodiesel production through soybean oil was 60 °C in the presence of 6% catalyst (oxide nanoparticles). It was a successful attempt that resulted in a 95% yield of biodiesel within 8 h. Also, it was analyzed that the catalysts were heterogeneous and reusable (Farzaneh et al. 2017). Fatty acid methyl ester (FAME) is one biodegradable and nontoxic biofuel prepared by the researchers to meet the crisis arising due to the lack of petroleum and other fossil fuels. Techniques like transesterification and esterification in the presence of nanocatalysts especially oxides have reached a high success in biodiesel production. In this study, the zeolite imidazolate framework (ZIF-8) was hybridized with potassium (KNa/ZIF-8) that further catalyzed the transesterification reaction of soybean oil for biodiesel production. The KNa/ZIF-8 catalyst was characterized using FT-IR, XRD, and SEM spectroscopy. It was observed that after the addition of potassium to the Na/ZIF-8, the basicity was increased and so the activity of the catalyst. Saeedi et al. (2016) reported that KNa/ZIF-8 was the finest catalyst to fabricate biodiesel due to its high basic nature and surface area. The optimum conditions identified for a yield of greater than 98% were 10:1 ratio for methanol/oil for 3.5 h. Hence, the catalyst was highly stable and active showing excellent results for the production of biodiesel through soybean oil (Saeedi et al. 2016). Waste frying oil (WFO) is a new feedstock found by scientists that have a high capability for biodiesel production when treated in the presence of certain nanocomposites as the catalyst. This study is about the synthesis of CaO-KOH-Al_2O_3 nanocomposites and examines their role in biodiesel production from canola oil. Different methods like coprecipitation, impregnation, sol-gel, and MW-assisted solution combustion synthesis (M-SCS) were used for the synthesis of nanocomposites. The structure and function of synthesized nanocomposites were examined using SEM, XRD, BET, and FT-IR analysis. It was observed that the nanocomposites formed by the M-SCS method showed greater basicity and thus activity. 86% yield of biodiesel was noted when this nanocomposite was used as the catalyst. Nayebzadeh et al. (2019) reported that the nanocomposites synthesized by M-SCS were more simple, economical, and active as compared to others. They increased porosity, surface area, and stability and thus were very effective in the production of biodiesel (Nayebzadeh et al. 2019).

Traditionally used heating systems had many disadvantages like the need for high temperatures, energy loss, and cost. To overcome these scientists found a technique known as microwave irradiation; it gained high success due to its proficiency in completion of chemical reaction in a short period. An experiment was conducted in which KOH/$Ca_{12}Al_{14}O_{33}$ nanocatalyst was prepared using microwave irradiation for biodiesel production. Interestingly, yield of biodiesel increased from 30.2% to 93.4% with an increase in the amount of catalyst and reaction time from 20 to 60 mins. Heydari et al. (2018) reported that KOH/$Ca_{12}Al_{14}O_{33}$ nanocatalyst successfully converted many triglycerides into biodiesel since they had a large pore size and surface area. Therefore, calcium aluminate was proved to be a highly promising nanocatalyst to produce biodiesel, especially through the microwave irradiation method (Heydari et al. 2018). Biodiesel is a type of renewable energy with low sulfur and aromatic content and is used widely as a green fuel. It can be derived from edible as well as nonedible vegetable oils and animal fats. *Capparis spinosa* is one of the vegetable oils which can be comprehensively generated and is available in all climates and areas. In this experiment, biodiesel was fabricated from *Capparis spinosa* oil in the presence of a heterogeneous catalyst nanozeolite NaX. The structural characteristics of the NaOH/NaX catalyst were examined, and yield of 90.81% was observed at optimal conditions. Helmi et al. (2021) reported that *Capparis spinosa* seeds contain 30–50% convertible oil in them and the optimum temperature for transesterification is 60 °C with methanol-to-oil ratio of 1:6.7 and the weight of catalyst to be 2.3 wt%. Therefore, *Capparis spinosa* oil is the most effective feedstock producing magnificent quality of biodiesel, and NaOH/NaX catalyst is a nonheterogeneous catalyst of high potential and activity (Helmi et al. 2021).

Lately, the Earth's natural resources are seen to be diminishing with increasing demand and industrialization. This is the fundamental cause of developing new approaches for the production of biodiesel. The efforts of researchers have recognized and developed several techniques that can use pyrolysis, microemulsions, and vegetable oils for biodiesel synthesis. An experiment was conducted in which they used five different types of calcium oxide-based catalysts along with gold nanoparticles (AuNPs) as nanocatalysts for the production of biodiesel through transesterification. SEM and XRD analyses were used for the characterization of CaO-based/Au nanoparticles. The gas chromatography results revealed that the biodiesel produced with AuNP-supported CaO catalyst was better than that produced by conventional CaO catalyst. It was further reported that oil conversion of about 90–97% was observed at an optimum temperature of 65 °C with 3 wt% of catalyst and also that the reusability of the catalyst was ten times which makes it the most efficient and economical (Bet-Moushoul et al. 2016).

Metal nanoparticles are extensively used nanoparticles in different sectors of industries. Nowadays, metal nanoparticles are used for the production of biofuels from biomass. Recently, in 2019, Laskar et al. (2020) experimented using ZnO-supported silver nanoparticles (AgNPs) to produce biodiesel from palm oil. Initially, ZnO@AgNPs were synthesized by the homogenous precipitation method, wherein polyethylene glycol acted as a surfactant and reducing agent and examined different aspects using TEM, NMR, BET, and XRD. Based on the results obtained by the characterization of ZnO@AgNPs, the deposition of Ag on ZnO nanoparticles (XRD), the size of ZnO (85 nm) and AgNPs (23 nm), and the surface area of 28.13 m^2/g (BET) were confirmed. The transesterification process was carried forward at 60 °C, for 60 min, with oil-to-methanol molar ratio of 10:1 and 10 wt% of catalyst. A FAME yield of 97% was reported; moreover, the catalyst recyclability tests confirmed the reusability of ZnO@AgNPs up to five cycles without any loss in its efficiency (Laskar et al. 2020).

13.3 Conclusion

Most of the experiments mentioned in the above chapter focused on the production of biodiesel using various types of biomasses. Currently, researchers have observed that nanoparticles are seen to be replacing the traditional heterogeneous catalysts. A great deal of attention was toward the application of nanotechnology for the efficient production of biodiesel. Several nanomaterials such as nanotubes, nanoadditives, and nanocomposites were proven to be the most effective nanocatalyst for the catalysis of transesterification and esterification reaction of biodiesel production. A higher amount of yield and an excellent quality of biodiesel were extracted using nanomaterials as seen in Table 13.1. Certain characteristics of the nanocatalysts like small size, large surface area, high rate of activity, stability, and reusability make them suitable catalysts for the reactions. Therefore, the sustainable and cost-effective synthesis of biodiesel was only possible due to the use of nanocatalysts of various forms catalyzing the reactions of various green sources of biomass like vegetable oils and microalgae.

Table 13.1 The amount of yield obtained using different biomass prototypes for the production of biodiesel using various nanomaterials

Type of nanomaterial	FAME yield (%)	Biomass	Size	Characterization	References
Carbon nanotubes (MWCNTs)	85–98	Soybean oil	D = 40–60 nm	XPS, FT-IR, TEM	Fan et al. (2016)
Sulfonated MWCNTs			D = 40–60 nm, L = 1–2 μm	FT-IR, Raman spectra, BET	Shuit et al. (2015)
CaO nanocatalyst			8 nm	SEM, TEM, XRD, FT-IR, BET	Bharti et al. (2019)
Fe_3O_4 nanoparticles			45–65 nm	FT-IR, XRD, VSM, SEM, TEM	Farzaneh et al. (2018)
NiFe2O4 and Ni0.3Zn0.7Fe2O4 magnetic nanoparticles			13–20 nm	XRD, FT-IR, GC, SEM, TEM	Mapossa et al. (2020)
Magnetic nanocatalysts			Cadmium (228.80 nm) Tin (189.99 nm) iron (259.95 nm)	XRD, Raman spectroscopy, UV-vis	Alves et al. (2014)
CaO/au nanocatalyst			2–5 nm	XRD, EDS, FESEM, TEM, XPS	Liu et al. (2021)
CaAlSi mixed oxide nanoparticles			48–54 nm	XRD, SEM, FT-IR	Farzaneh et al. (2017)
Nanostructured sodium-zeolite imidazolate framework with potassium (ZIF-8/K)			D = 1–1.5 nm	FT-IR, XRD, TGA, SEM	Saeedi et al. (2016)
Mg-substituted zinc ferrite nanocatalyst	82–100	Waste cooking oil	8–10 nm	EDX, DRS, VSM, XRD, FT-IR, HR-SEM	Sakthi Vignesh et al. (2020)
Zinc ferrite nanocatalyst			8.55–13.04 nm	XRD, FT-IR, DRS, SEM	Ashok and Kennedy (2019)
Sodium titanate nanotubes			D = 5.37 nm, L = 50–80 nm	XRD, TEM, BET, FT-IR	Zaki et al. (2019)
La^{3+}/ZnO-TiO2 photocatalyst			8–11.5 nm	TG, XRD, SEM-EDS, HRTEM, BET, UV-vis, Raman spec	Guo et al. (2021)

CaO and MgO heterogenic nanocatalyst			65–70 nm	XRD, SEM	Tahvildari et al. (2015)
Magnesium oxide nanocatalyst			7.86 nm	XRD, UV-vis, FT-IR,	Ashok et al. (2018)
Magnetic nanocatalyst			12–13 nm	TEM, XRD, FT-IR, XPS	Changmai et al. (2021)
K2CO3-CuO nanocatalyst			2–17 nm	XRD, FESEM, EDX, FT-IR, TCD, BET	Abdullah et al. (2021)
Sulfonated MWCNT	82–98	Palm oil	D = 40–60 nm, L = 1–2 μm	SEM, FT-IR	Shuit and Tan (2015)
Halloysite nanotubes			D = 17.47 nm	XRD, SEM, XPS	Lin et al. (2020)
Magnetic nanoparticles			10–20 nm	FT-IR, BET, TEM, UV-vis	Gupta and Rathod (2020)
Zinc oxide-supported silver nanoparticles			23–85 nm	XRD, TEM, BET, HNMR, GC	Laskar et al. (2020)
Pt-Ni/NiO/Ni2O3 nanoparticles			2–3 nm	XRD, TG, DTG, Raman spec, FT-IR, TEM	Sarno et al. (2020)
Oxide nanoparticles	85–98	Other oils	15–55 nm	XRD, BET, zeta potential, SEM	Do Nascimento et al. (2012)
Plasma-functionalized MWCNT			D = 10–20 nm, L = 10–30 μm	XPS, TEM, BET	Rastian et al. (2016)
Ni-doped ZnO nanocatalyst			35.1 nm	AFM, XRD, VSM	Baskar et al. (2018)
Silver nanoparticles			60–70 nm	XRD, UV spec, EDX, FESEM	Ganesan et al. (2020)
SO3H-MWCNTs			D = 20–30 nm, L = 10–30 μm	SEM, FT-IR, TGA, BET	Macawile et al. (2020)
NaOH/NaX nanoheterogeneous catalyst			70.44 nm	XRD, SEM, BET	Helmi et al. (2021)

(continued)

Table 13.1 (continued)

Type of nanomaterial	FAME yield (%)	Biomass	Size	Characterization	References
Graphene oxide magnetic nanobiocatalyst	82–99	Algae	20–30 nm	AFM, FT-IR, XRD, BET, zeta potential	Nematian et al. (2020)
Titanium nanoparticles			15 ppm Ti nps	SEM	Khanra et al. (2020)
CaO/KOH, Fe3O4 magnetic nanocatalysts			42–55.91 nm	SEM, BET, FT-IR	Farrokheh et al. (2020)
TiO2-ZnO nanocomposite catalyst			~ 12 nm	XRD, FT-IR, TEM, EDAX	Gurusamy et al. (2019)
Iron nanoparticles			3.347 nm	SEM, BET, FT-IR	Ashok et al. (2021)
Iron oxide nanoparticles			<50 nm	UV-vis, XRD, TEM	Rana et al. (2020)
Gold-silver core-shell nanoparticle	83–100	Sunflower oil	12 nm	UV-vis, TEM, XRD, FT-IR	Banerjee et al. (2014)
Carbon nanotubes (SWCNTs)			D = 1–2 nm, L = 5–33 μm	TEM, HNMR spectra	Bencze et al. (2016)
Kaolinite nanotubes			D = 14.5 nm	XRD, BET, TEM	Abukhadra et al. (2020)
CaO-based/au nanoparticles				XRD, SEM	Bet-Moushoul et al. (2016)
Magnetic MWCNTs	80–95	Vegetable oil	40 nm	TEM, XRD, FT-IR	Fan et al. (2016)
Se-doped ZnO nanocatalyst (nanorods)			50 nm	XRD, XPS, HRTEM, FE-SEM	Rao et al. (2021)
Magnesium oxide nanocatalysts			15–19 nm	SEM, TEM, XRD, XPS	Gai et al. (2009)
CaO/NiO Nanocatalyst	96–100	Cottonseed oil	1.8 μm	XRD, TEM, SEM	Kaur and Ali (2014)
Na2SiO3 and Fe3O4 nanoparticles				VSM, TEM	Guo et al. (2012)

Nanocrystalline lithium-ion impregnated calcium oxide			50 nm	XRD, TEM, BET, FESEM	Kumar and Ali (2010)
Nanocatalysts (co, Ni)	50–60	Unwanted weed	20–50 nm	SEM, XRD, FT-IR	Tahir et al. (2020)
Nanocatalyst nickel and cobalt			2–90 nm	XRD, TEM, SEM	Ali et al. (2020)
KOH-CaO-Al_2O_3 nanocomposites	86–93	Canola oil		XRD, FT-IR, SEM	Nayebzadeh et al. (2019)
KOH/calcium aluminate nanocatalyst			18–30 nm	XRD, BET, FT-IR, SEM	Heydari et al. (2018)
Nano-entrapped lipase (ECL)	98–100	Acids		FT-IR, TGA, SEM, XRD	Baloch et al. (2021)
Organ sulfonic aryl-silica nanoparticles			10–100 nm	XPS, FT-IR, TEM, SEM	Peixoto et al. (2021)
Magnetic carbon nanotubes	–	Starch	20–50 nm (bundle)	FT-IR, TEM, SEM, Raman analysis, ICP-OES	Goh et al. (2012)
CuO nanoparticles	88	Biogenic waste	13 nm	XRD, EDAX, FESEM, FT-IR	Santha et al. (2019)
Nanoparticles, nanofibers, nanosheets, nanopores	–	–	Pore size 4–40 nm	BET, AFM, XRD, TEM, FT-IR	Verma et al. (2013)

SEM scanning electron microscope, *TEM* transmission electron microscope, *XRD* X-ray diffraction, *XPS* X-ray photoelectron spectroscopy, *FT-IR* Fourier transform infrared spectroscopy, *BET* Brunauer-Emmett-Teller, *UV-vis* UV visible spectroscopy, *EDX/EDAX/EDS* energy-dispersive X-ray spectroscopy, *AFM* atomic force microscopy, *ICP-OES* inductively coupled plasma atomic emission spectroscopy, *H-NMR* proton nuclear magnetic resonance, *TGA* thermogravimetric analysis, *VSM* vibrating sample magnetometer, *GC* gas chromatography.

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