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Strontium Oxide Nanoparticles for Biodiesel Production: Fundamental Insights and Recent Progress

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ABSTRACT: Biodiesel is a renewable and environmentally friendly alternative to fossil fuels. Despite nearly 3 decades of research in the field of biodiesel, there remains major obstacles for large-scale production. In the search for an active, selective, and reusable solid base catalyst, strontium oxide (SrO) is emerging out as a preferred choice for the transesterification reaction under various methods of activation. SrO exhibits the highest activity among processable alkaline earth metal oxides as a result of its strong basicity. SrO nanoparticles (NPs) and hybrids showed improved performance. Recent progress achieved in the development of synthetic methods of SrO NPs is reviewed. Advantages of SrO-based nanocomposites for biodiesel production are discussed. Finally, potential support materials for enhancing the catalytic performance of SrO NPs with commercial implications are elaborated.

1. INTRODUCTION

Ever since the disclosure of the first patent in 1993 on the use of biodiesel as an alternative to fossil-based engine oil by Koracs,[1] enormous progress has been made in the development of processes for biodiesel production. Ever increasing interest of research in this area is seen in Figure 1, depicting the number of publications (with the Web of Science search keyword "biodiesel") over the years. Within a span of 27 years, there are nearly 53 000 publications on biodiesel production.

In comparison to various catalysts for biodiesel production, the significance of the use of a strontium oxide (SrO) catalyst for the transesterification reaction is its exceptionally high activity (with rate enhancements of several orders of magnitude). In addition, SrO-based catalysts are flexible with respect to chemical composition and structural changes, leading to desired property enhancement under diverse reaction conditions, offering the unique possibility of easy commercialization.[2] Tariq et al. reviewed the transesterification processes using homo- and heterogeneous catalysts for the biodiesel.[3] The key reviews on the subject of biodiesel production using a SrO catalyst are listed in Table 1.

It can be seen in Table 1 that there are only two reviews on the subject dealing with biodiesel production and SrO catalysts reviewed only up to 2011. However, neither of the two deals with the development of synthetic strategies for nano SrO catalysts and their exploitation for the sustainable biodiesel production process, which is the subject of the present review. Thus, to fill the gap in the literature, the objective of the present review is not only to summarize the key aspects of the synthesis, characterization, and exploitation of nanoparticles (NPs) of SrO as catalysts for biodiesel production, from the conversion diverse feedstock, but also to synthesize new knowledge based on the authors expertise in the field and to provide directions for future research in this field.

One of the recent pilot plant studies for biodiesel production [116 L of biodiesel from 202 L of waste cooking oil (WCO) in 15 h, of which 12 h is consumed for separation and purification after the transesterification process] is based on continuous ultrasonic reactors operating at 20 kHz in two steps. With a short residence time of 0.93 min throughout the entire process, a high yield of biodiesel (93.8 wt %) was obtained from WCO as the feedstock and KOH (1.0 wt %) as the catalyst. Because the process was carried out at ambient temperature, the electricity consumed is significantly reduced (0.072 kWh/L of biodiesel).[4,5] Replacement of a liquid alkali catalyst especially by solid base nanocatalysts makes the biodiesel process sustainable.

1.1. Biodiesel as a Potential Alternative Transportation Fuel.

Limited crude petroleum resources and increasing awareness of the environmental impact of fossil fuels motivate the search for alternative fuels. Biodiesel is considered to be an important renewable energy source because of its potential to fulfill energy demands, reduce greenhouse gases, and mitigate global warming.[6,7] To obtain this renewable fuel, organic precursors have to undergo a catalyzed chemical reaction called transesterification.

Major chemical components of plant oils and animal fats are triglycerides. Triglycerides consist of different fatty acid compositions directly influencing the quality of the biodiesel.[8] The chemical structure of triglycerides is illustrated in Scheme 1.

Production of biodiesel from triglycerides involves the chemical process of transesterification, wherein the triglyceride is converted into esters. During the transesterification, also

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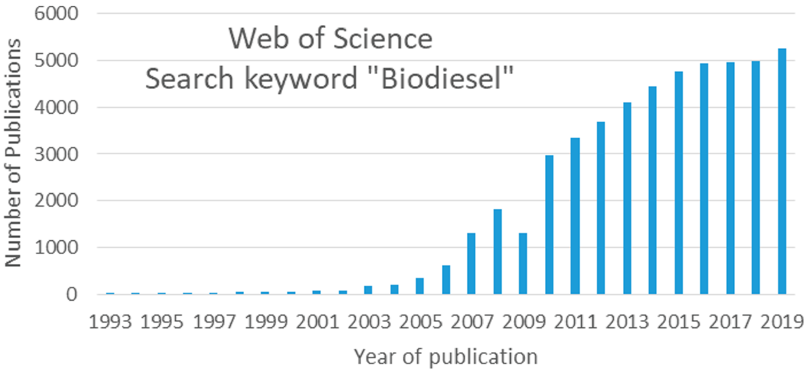
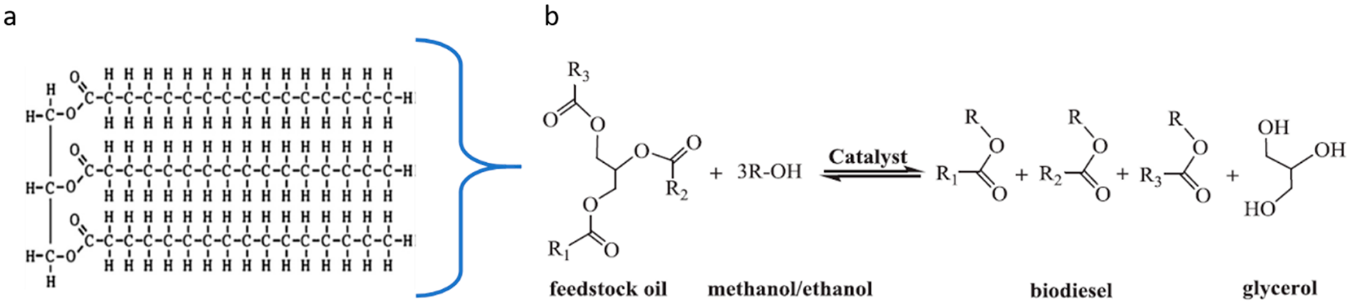


Figure 1. Plot depicting the increase in the number of publications in the research area of biodiesel production as a function of the year of publication.

Table 1. List of Key Reviews in the Field

topic reviewed	year up to which the topic is reviewed	reference
use of microwave, sonochemical, and conventional heating as methods of activation of the transesterification of oils, cooking oils, microalgae, castor, and *Jatropha* seeds using SrO, KOH, and NaOH as catalysts for biodiesel production	2011	2
biodiesel production through the transesterification of edible and non-edible oils using homogeneous (NaOH and KOH) and heterogeneous (CaO, MgO, SrO, ZnO, La_2O_3, and Mg–Al hydrotalcite) catalysts; the properties of the biodiesel product were examined using gas chromatography, GC–MS, HPLC, NMR, and FTIR	2011	3

Scheme 1. Schematic Illustration of (a) Triglyceride and (b) Its Transesterification Reaction with Alcohol To Form Biodiesel



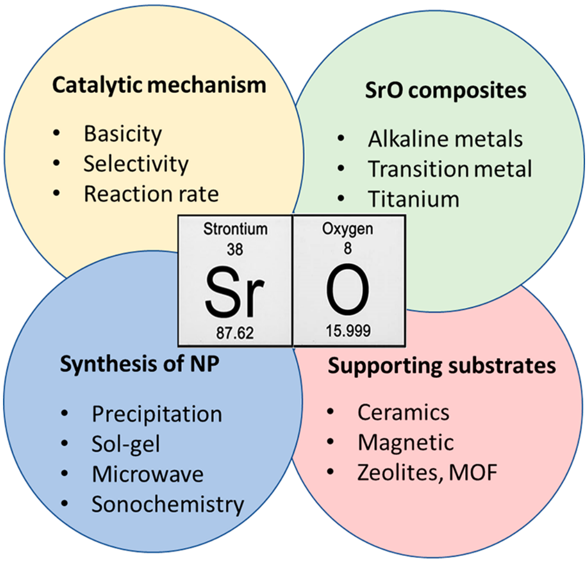
called alcoholysis, the organic group (R1, R2, and R3) from an ester is exchanged with the organic group (R) of an alcohol, forming fatty acid alkyl ester (FAAE, biodiesel) as the reaction product and glycerol as the reaction byproduct (Scheme 1).[9,10]

This review aims to present recent progress in the utilization of SrO as a catalyst for biodiesel production. The performance of the catalyst is significantly impacted by several important aspects, namely, (1) catalytic mechanism of SrO, (2) methods of synthesis of SrO NPs, (3) structure and properties of SrO-based composites, and (4) nature of the support materials (Scheme 2).

2. CATALYTIC MECHANISM OF CATALYSTS FOR BIODIESEL PRODUCTION

The role of a catalyst in the conversion of oils and fats is crucial for effective transesterification rates, with biodiesel yields matching industrial demands. The catalysts used for this reaction can be divided into two groups, homo- and heterogeneous catalysts, and subdivided into acid or base catalysts. Homogeneous catalysts are in the dissolved state and act in the same phase as that of the reactants and products. They are commonly classified into acid and alkali catalysts. They are currently the most widely used catalysts for large-scale production of biodiesel, owing to their low cost and faster conversion of oil to biodiesel.[11]

Scheme 2. Major Strategies for the Development of a SrO-Based Catalyst for Biodiesel Production



2.1. Heterogeneous Catalysts. Heterogeneous catalysts, where the catalyst is in a different phase than the reactants and products, offer significant benefits. Unlike homogeneous catalysts, heterogeneous catalysts can be separated from the solvent for reuse. They usually result in faster and more effective reactions at lower temperatures.[12] On the other hand, a higher molar ratio of alcohol/oil is generally required for heterogeneous catalysts.[13] Other concerns include catalyst deactivation over prolonged cycles, inaccessibility to the active sites, and issues related to kinetics, phase separation, and mass transfer. Therefore, further comprehensive research is required to develop effective and adaptable materials for heterogeneous transesterification processes.

Heterogeneous catalysts can be divided into acid and base catalysts. The catalytic mechanism of each catalyst type and the number of sequential processing steps involved for biodiesel production are different.[2,14] In heterogeneous acid catalysis, triglyceride is adsorbed on the surface of the catalyst by protonation of carbon in the carbonyl group, which is to be attacked in turn by alcohol in the liquid phase.[15]

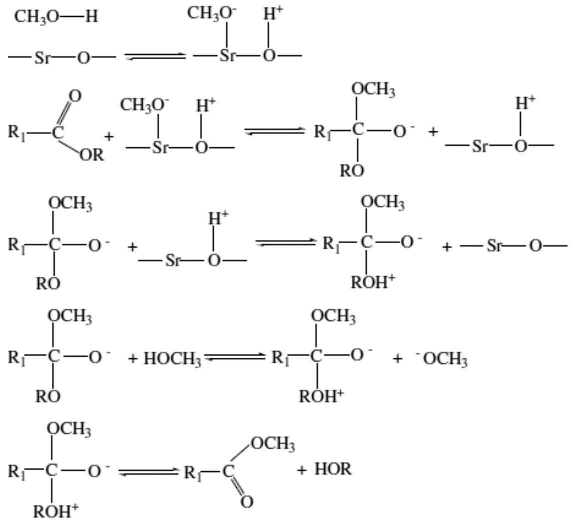
Heterogeneous base catalysis is preferred over acid catalysis in terms of activity. On the other hand, base catalysts are more sensitive to water, moisture, and adverse free fatty acids (FFAs).[16]

2.2. Catalytic Mechanism of Alkaline Earth Metal Oxides. Among solid catalysts, alkaline earth metal oxides possess the highest Lewis basicity. As a result of their low solubility in alcohol, they are top candidates for high yield biodiesel production.[17,18] The electronegativity of the conjugated metal cation induces the attractive force of attaching the oxygen anion to the conjugated metal cation.[13,19] Moreover, the structure of metal oxides involves positive metal ions (cations, M^{2+}) and negative oxygen ions (anions, O^{2-}), which correspond to Lewis acid and Lewis base sites, respectively. The stronger basic sites occur at low coordination defects, corners, and edge sites or on high Miller index surfaces.[20] The surface of the solid catalyst is heterogeneous at the atomic/molecular level, with each of the surface active sites being coordinatively unsaturated to different extents. Because each of these active sites are distinguishable by their number of nearest neighbors, they exhibit completely different catalytic activities.[21,22]

The basic surface oxide anions provide sufficient adsorptive sites for alcohols, on which the O−H bonds break into methoxide anions (CH_3O^-) and hydrogen cations (H^+) (see Scheme 3).[16] The methoxide anions then react with the triglyceride molecules to form the corresponding fatty acid methyl esters (FAMEs).[23]

Among the alkaline earth elements, beryllium (Be) and radium (Ra) cannot be used as catalysts because of their carcinogenicity and radioactivity, respectively.[24] The order of catalytic activity of the remaining alkaline earth oxides is barium oxide (BaO) > strontium oxide (SrO) > calcium oxide (CaO) > magnesium oxide (MgO).[19] Among alkali earth metal oxides, CaO is the most intensively studied oxide for the transesterification reaction the last 2 decades owing to its relatively high basic strength, low solubility in methanol, abundance, and environmental friendliness.[25] Tremendous research was performed on the use of CaO as a catalyst for the transesterification reaction.[26−33] Renewed interest in CaO is due to its non-expensive and abundant precursors, such as constructional lime,[34,35] mollusk shell,[36−38] egg shell,[39−44] and limestone.[45] However, severe leaching of CaO was observed after the first run, hindering its reusability and long-term durability.[46] Moreover, the basicity and, hence, the catalytic activity of CaO still remain low relative to SrO and BaO.[47,48] Despite the high activity of BaO in the transesterification reaction, its toxicity and high solubility in methanol make BaO unsuitable for biodiesel production.[22,19] While Ca and Mg are traditionally used in solid base catalysis for the transesterification reaction, with the stronger basicity and exceptionally high reaction rates under certain fields of activation (microwave/ultrasound), SrO is a better catalyst candidate for biodiesel production.

Scheme 3. Mechanism of SrO-Catalyzed Transesterification[a]



[a]This scheme was reprinted with permission from ref 16. Copyright 2007 Elsevier.

2.3. SrO as a Solid Base Catalyst for Biodiesel Production. SrO can catalyze many chemical reactions, such as oxidative coupling of methane,[49,50] selective oxidation of propane,[51] nitroaldol reaction and mixed Tishchenko reaction,[52] Michael addition,[53] and transesterification reaction.[16] SrO possesses the highest base site density among alkali earth oxides, with $H\_ > 26.5$, as obtained by the Hammett indicator method.[54] SrO is a suitable catalyst for the transesterification process as a result of its high basicity, strong base sites, relatively low lixiviation tendency in biodiesel, and high selectivity toward the transesterification reaction.[55] All of these parameters represent the contributing factors of the SrO catalyst as the choice for optimal biodiesel productivity.[56,57] For example, Koberg et al. showed that the transesterification of cooked oil to biodiesel in a microwave domestic oven can be completed using commercial SrO as the catalyst in only 10 s.[55] The short reaction time minimizes the input energy consumed for the biodiesel production. The catalytic reactivity of the active sites in the esterification of oleic acid with methanol increases, owing to the high thermal and chemical stability of the sulfate ions bonded on Sr cations.[58] Cyclability investigation showed that SrO could be recycled and reused 10 times, with only 2−3% activity loss observed on the 10th cycle.[59] The reason for this mass loss may be related to catalyst detachment pulverization during recycling. Catalytic activity of SrO is attributed to the surface reaction, and the mechanism is illustrated in Scheme 3. At first, surface O^{2-} extracts H^+ from CH_3OH to form surface CH_3O^-

Table 2. Published Data on Sr-Based Oxides, Mixed Metal Oxides, and Composite Catalysts for Biodiesel Production

catalyst	feedstock	reaction conditions	biodiesel yield (wt %)	reference
SrO	fried vegetable oils	catalytic bed system; *T*, 65 °C; 40 min; MeOH/oil molar ratio, 6:1; 5 wt % catalyst amount; flow rate of 100 mL/min	85	65
SrO	*Camelina sativa*	conventional heating; *T*, 60 °C; 120 min; MeOH/oil molar ratio, 12:1; 0.5 wt % catalyst amount	82	66
SrO	babassu oil and castor oil	conventional heating; *T*, 65 °C; 1 h; MeOH/oil molar ratio, 6:1; 1 wt % catalyst amount	97.2 and 96.4	64
SrO	olive oil	conventional heating; *T*, 65 °C; 15 min; MeOH/oil molar ratio, 6:1; 5 wt % catalyst amount	82	60
SrO	palm oil	conventional heating; *T*, 65 °C; 3 h; MeOH/oil molar ratio, 15:1; 3 wt % catalyst amount	∼92	67
SrO	soybean oil	conventional heating; *T*, 65 °C; 30 min; MeOH/oil molar ratio, 12:1; 3 wt % catalyst amount	95	16
SrO	babassu oil and coconut oil	fixed-bed reactor; RT (∼25 °C); 3 h; oil/MeOH weight ratio, 5:1; 1.5 wt % catalyst amount	98.54	68
SrO	waste cooking oil	microwave irradiation; *T*, 60 °C; 10 s; MeOH/oil molar ratio, 6:1; 1.8 wt % catalyst amount	99.8	55
SrO	castor seed and *Jatropha* seed	microwave irradiation; *T*, 60 °C; 5 min; 30 wt % catalyst amount; $CHCl_3$/MeOH, 1:2 (v/v)	99.95 and 99.7	59
SrO	*Nannochloropsis* sp.	microwave irradiation; *T*, 60 °C; 5 min; 30 wt % catalyst amount; $CHCl_3$/MeOH, 1:2 (v/v)	99.9	69
SrO	rapeseed oil	supercritical MeOH conditions; hydrothermal heating; *T*, 220 °C; 10 min; MeOH/oil molar ratio, 40:1; 1 wt % catalyst amount	>90	70
SrO	palm oil	ultrasound heating; *T*, 65 °C; 30.7 min; MeOH/oil molar ratio, 9:1; 2.5 wt % catalyst amount; 130 W; pulse on, 9 s; pulse off, 1 s	93.37	71
SrO	palm oil	ultrasound heating; *T*, 65 °C; 60 min; MeOH/oil molar ratio, 9:1; 3 wt % catalyst amount; 50% amplitude	95.2	47
		SrO Composites		
Cu/SrO (CuO, 10 wt %)	hempseed oil	hydrothermal heating; *T*, 180 °C; 3 h; MeOH/oil molar ratio, 12:1; 3 wt % catalyst amount; 3 MPa	96	72
MgO−SrO (5 mmol of Sr/g)	palm olein oil	conventional heating; *T*, 60 °C; 30 min; MeOH/oil molar ratio, 9:1; 5 wt % catalyst amount	97.3	13
Sr/MgO (2.5 mmol of Sr/g of support)	soybean oil	conventional heating; *T*, 65 °C; 30 min; MeOH/oil molar ratio, 12:1; 5 wt % catalyst amount	93	73
$Sr_xCa_{1-x}O$ (Sr/Ca molar ratio, 0.5)	palm oil	conventional heating; *T*, 65 °C; 30 min; MeOH/oil molar ratio, 9:1; 5 wt % catalyst amount	98.31	74
Sr/CaO (Sr, ∼17 wt %)	cottonseed oil and waste frying oil	conventional heating; *T*, 60 °C; 2 h; MeOH/oil molar ratio, 12:1; 3.5 wt % catalyst amount	97.3 and 96.7	75
SrO−CaO (Sr/Ca weight ratio, 1:6)	*Jatropha* oil	ultrasound heating; *T*, 65 °C; 30 min; MeOH/oil molar ratio, 10:1; 6 wt % catalyst amount; 210 W	95.4	76
$Sr/Mg_{0.72}Al_{0.2}$ oxide (Sr, 1.5 wt %)	rice bran oil	conventional heating; *T*, 60 °C; 3 h; MeOH/oil molar ratio, 20:1; 1.5 wt % catalyst amount	56	77
SrO−NiO	macaw oil (*Acrocomia aculeate*)	conventional heating; *T*, 65 °C; 5 h; MeOH/oil molar ratio, 9:1; 2 wt % catalyst amount	97	78
$Sr_3Al_2O_6$ nanocomposite	soybean oil	conventional heating; *T*, 60 °C; 61 min; MeOH/oil molar ratio, 25:1; 1.3 wt % catalyst amount	95.7	79
$SrAl_2O_4$	rapeseed oil	hydrothermal heating; *T*, 160 °C; 2 h; MeOH/oil molar ratio, 6:1; catalyst amount, 1.95 g	89	80
Sr cinder ($SrAl_2Si_2O_8$ and $Sr_5Al_8O_{17}$)	soybean oil	hydrothermal heating; *T*, 180 °C; 1 h; MeOH/oil molar ratio, 24:1; 4 wt % catalyst amount	97.1	57
		Loading of SrO on Porous Support Materials		
Ba−Sr/ZSM-5 (6 wt % Sr based on ZSM-5 and 4 wt % Ba based on the Sr weight catalyst amount)	sunflower oil	conventional heating; *T*, 60 °C; 3 h; MeOH/oil molar ratio, 9:1; 3 wt % catalyst amount; stirring, 500 rpm	87.7	62
$Ca_xSr_{1-x}O$/hydroxyapatite (2.5 mmol of Sr/g of support)	soybean oil	conventional heating; *T*, 70 °C; 5 h; MeOH/oil molar ratio, 9:1; 5.6 wt % catalyst amount	85	81
$Sr_xZn_{1-x}O$ (2.5 mmol of Sr/g of support)	soybean oil	conventional heating; *T*, 65 °C; 5 h; MeOH/oil molar ratio, 12:1; 5 wt % catalyst amount	94.7	82
Sr/ZrO_2 (Sr, ∼7 wt %)	waste cooking palm oil	hydrothermal heating; *T*, 113.7 °C; 89 min; MeOH/oil molar ratio, 30:1; 2.1 wt % catalyst amount	86.7	83
Sr_2TiO_4 (Sr/Ti molar ratio, 0.8)	soybean oil	conventional heating; *T*, 60 °C; 15 min; MeOH/oil molar ratio, 15:1; 1 wt % catalyst amount	98	84
SrO/Al_2O_3 (SrO, 2 wt %)	rapeseed oil	ultrasound emulsification; supercritical EtOH; 30 min; *T*, 350 °C (electric heating), 30 MPa; EtOH/oil molar ratio, 12:1; 2 wt % SrO amount; flow rate, 300 mL/min; 30 MPa	97.46	85
SrO/SiO_2 (Sr/Si molar ratio, 6)	olive oil	conventional heating; *T*, 65 °C; 10 min; MeOH/oil molar ratio, 6:1; 5 wt % catalyst amount	95	60
SrO/SiO_2 (Sr/Si weight ratio, 2)	*Nannochloropsis* sp.	microwave irradiation; *T*, 60 °C; 2 min; $CHCl_3$/MeOH, 1:2 (v/v)	99.9	86

Table 2. continued

catalyst	feedstock	reaction conditions	biodiesel yield (wt %)	reference
		Loading of SrO on Porous Support Materials		
SrO/SiO_2 (Sr/Si molar ratio, 6)	*Chlorella vulgaris* ESP-31, 22.7 wt %	conventional heating; *T*, 45 °C; 6 h; 5.5 wt % catalyst amount; MeOH/oil molar ratio, 424:1	81.8	87
$SrTiO_3$	palm kernel oil	hydrothermal heating; *T*, 170 °C; 3 h; MeOH/oil molar ratio, 20:1; 10 wt % catalyst amount	99.9	88
$SrZrO_3$	soybean oil	conventional heating; *T*, 60 °C; 3 h; MeOH/oil molar ratio, 12:1; 3 wt % catalyst amount	98	61
$SrZrO_3$ (Sr/Zr atomic ratio, 2:1)	waste cooking oil	conventional heating; *T*, 75 °C; 7 h; EtOH/oil molar ratio, 12:1; 5 wt % catalyst amount; stirring, 400 rpm	99.61	89

(step 2). Next, the carbon atom of the carbonyl group of the triglyceride molecule attracts a methoxide anion from the SrO surface of SrO to form a tetrahedral intermediate (step 3), where R_1 represents the chain of the alkyl group. Later, the tetrahedral intermediate picks up another H^+ from the surface of SrO (step 4). The tetrahedral intermediate can also react with methanol to generate methoxide anions (step 5). In the final step, rearrangement of the tetrahedral intermediate results in the formation of biodiesel (step 6).

Importantly, SrO, like other basic catalysts, is very sensitive to the presence of water and/or FFA that can be found in most low-grade feedstocks.[60] To overcome FFA and water contamination, the feedstock should be mixed with acidic materials, like zirconium oxide[61] or acid zeolites,[62] or washed with a low concentrated homogeneous base catalyst, like KOH,[57] prior to the transesterification reaction. SrO is also highly hygroscopic and has a strong tendency to react with CO_2 and water in air to form less-active $SrCO_3$.[63] Despite a similar catalytic activity between SrO and $Sr(OH)_2$, the later could be partially dissolved in the reaction medium, releasing OH^- ions, and can no longer fully act as a heterogeneous catalyst. To reactivate the catalyst, it must be calcined at temperatures higher than 1000 °C.[64]

Table 2 summarizes advances in biodiesel production using the SrO catalyst for the transesterification reaction as bulk, mixed metal oxide, or supported catalyst. The reaction conditions and product yields were highlighted.

Comprehensive analysis of the results highlighted in Table 2 reveals the four main varying parameters responsible for successful catalysis, namely, (1) catalytic mechanism of SrO, (2) methods of synthesis of SrO NPs, (3) structure and properties of SrO-based composites, and (4) nature of the support materials, as denoted in Scheme 1. The potential of the SrO catalyst with strong and high amounts of basic sites is capable of activating and converting diverse lipid feedstock to biodiesel in close to 100% yield. Owing to the great potential of SrO, a careful design of the catalyst will allow in the near future for movement from the laboratory scale to industrial and commercial biodiesel production, replacing the homogeneous base catalysts used currently in large-scale production plants.[90]

Two approaches are employed to increase the surface area of the catalyst and, thus, the number of active sites. The first approach involves synthesis and development of SrO NPs and composites. The second approach is loading SrO on high-surface-area supporting materials.[58]

2.4. SrO Nanocatalysts for Biodiesel Production.

The activity of the catalyst has been proven to be dependent upon the particle size. Smaller particles, in the nanometer scale, exhibit higher reaction rates than micrometric particles as a result of a higher external surface area. The nanocatalyst has higher selectivity as a result of its nanodimensional porosity. NP catalysts also hold reduced diffusion limitations. Likewise, nanocomposites have gained attention in biomass conversion to chemicals and fuels, because they possess a large surface area with enhanced interaction between the reactant and catalyst.[91] Moreover, the nanometric size of the catalyst provides shorter paths for the substrate to approach active sites, reducing the diffusion constraints significantly. As a result, in contrast to bulk SrO, where most of the mass is not active, nanocrystalline SrO is capable of producing biodiesel with 99% conversion of the triglyceride. In addition, studies have also shown that nanocatalysts demonstrate good recycling characteristics lasting up to five reuses.[92] Tremendous research efforts were invested in recent years to synthesize alkaline earth and transition metal oxide catalysts in nanometer size for the transesterification reaction of edible and non-edible feedstock, affording FAME conversion higher than 95% and reusability of several times.[92−102] It was shown that to obtain similar biodiesel conversion, the amount of nanocatalysts required is only 30% of that of bulk catalysts. More importantly, the reaction is less affected by the moisture of the oil and not influenced by the FAA content.[103,104]

3. METHODS OF SYNTHESIS OF SRO NPS

3.1. Traditional Precipitation.

Despite the unique properties of SrO as a base catalyst for biodiesel reaction, NPs of SrO are not easy to fabricate, mainly as a result of its extremely high melting point (2531 °C). It is more common to use strontium salts as precursors. Modification of these salts requires significantly lower temperatures (approximately 900 °C). Identification of the product is usually carried out by the characteristic X-ray diffraction (XRD) pattern. The XRD pattern of nano $SrTiO_3$ is shown in Figure 2a.[105] An effective and simple method to obtain metal oxide NPs is the route of chemical oxidation. In this method, Sr salt, usually $SrCl_2$, is dissolved in a strongly basic solution (achieved by the addition of NaOH up to a pH value of 12).[106,107] Hydrothermal synthesis is another method usually employed that combines pressure as a driving force for the forward reaction. In this method, a Sr salt, for example, $Sr(OH)_2$, is reacted with a strong reducing agent in a strongly basic solution. The solution is sealed in an autoclave and heated to approximately 200 °C for a few hours. The product precipitates out and can be separated subsequently from side products (salts) by centrifugation. This method results in peculiar shapes, such as prisms and flower-like crystals (panels b and c of Figure 2).[107] Torras and co-workers studied the immobilization of SrO catalysts on a membrane (in a catalytic membrane reactor). They reported that SrO was the strongest catalyst in conversion of sunflower oil into several types of methyl

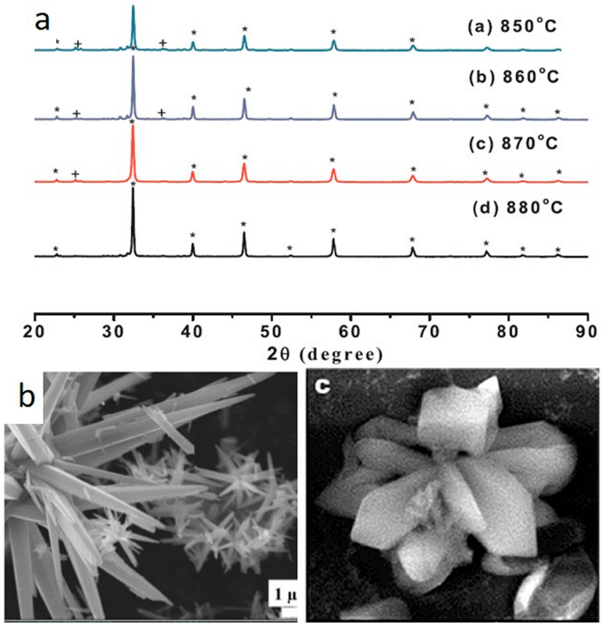


Figure 2. Structure and morphology of SrO NPs. (a) XRD pattern of the prepared catalyst, with $Sr(OH)_2$ calcined at 850, 860, 870, 880 °C. This figure was adapted with permission from ref 111. Copyright 2020 Elsevier. (b) FESEM images of the flower-like ZrO/SrO composite. This figure was adapted with permission from ref 107. Copyright 2017 Elsevier. (c) SrO catalyst particles immobilized on the polymeric matrix. This figure was adapted with permission from ref 108. Copyright 2019 Elsevier.

esters.[108] Another traditional method for NP fabrication involves the thermal annealing of strontium carbonate ($SrCO_3$) prepared by mixing $Sr(NO_3)_2$ and Na_2CO_3 by heating to 900 °C under an inert atmosphere in a flow furnace.

3.2. Sol−Gel. In the sol−gel method, strontium salt, typically hydrous $SrCl_2$, is dissolved in a basic solution (pH 8−9). The gelatinous mixture is then slowly converted into a colloidal suspension. Notably, we could not find SrO NPs produced by sol−gel, only its composite with other metals (Ti and Sn).[109] SrO NPs were also synthesized by less conventional methods (such as the wet chemical method and polymer precursor method) than microwave irradiation and sonochemistry.[110,111]

3.3. Microwave-Assisted Synthesis. Microwave-assisted synthesis provides rapid kinetics and uniform heating of reagents, leading to uniform nucleation and growth conditions. This bottom-up approach may result in a reduced particle size and better crystallinity and leads to the formation of novel metastable phases.[112−119] The use of microwaves has also been demonstrated to be suitable for anchoring NPs on solid supports, allowing for homogeneous dispersion of the NPs on the support.[117−123] It has been shown that microwave irradiation as short as 30 s is enough for the formation and deposition of the SrO precursor on SiO_2 beads ($SrCO_3$@SiO_2). Equimolar amounts of $Sr(NO_3)_2$ and Na_2CO_3 with SiO_2 beads were irradiated by a domestic microwave in water under vigorous stirring. The composite, $SrCO_3$@SiO_2, is further subjected to calcination at 900 °C for 3 h, leading to the decomposition of $SrCO_3$ and *in situ* deposition of SrO NPs on SiO_2 beads (SrO/SiO_2). The use of this composite as a catalyst for the conversion of waste cooking oil to FAME in a batch process under microwave irradiation allowed for an exceptionally high yield of biodiesel, 99.4 wt %, in just 10 s. Moreover, the catalyst could be easily separated and reused for 10 consecutive reaction runs without any significant activity loss, as shown in Figure 3D.[117] To significantly reduce the process cost by substantial reduction in the utilization of SrO, Tangy et al. used this catalyst as a fixed-bed catalyst in a continuous flow microwave reactor for the conversion of 2.46 L of waste cooking oil to biodiesel for 24.6 min. A conversion value to biodiesel as high as 99.2 wt % was achieved with the reactor packed with 15 g of SrO/SiO_2 catalyst.[119]

A unique structure−activity relationship is observed in the catalytic function of SrO/SiO_2 to produce biodiesel from cooking oil waste in batch as well as continuous flow microwave irradiation processes (Figure 3). The high-resolution scanning electron microscopy (HR-SEM) image of the ∼41 wt % SrO/SiO_2 catalyst prepared under the best process conditions is shown in Figure 3A. On the surface of the millimetric silica beads, a nanometric tube structure (average length of 139 nm and width of 50 nm of nanotubes) of SrO was formed. These structural features are analogous to the nanostructure on the surface of the lotus leaf (panels B and C of Figure 3), inducing a unique hydrophobic property. The nanotubular structure of SrO on SiO_2 beads in the SrO/SiO_2 composite is attributed to the exceptional catalytic function (activity and selectivity) for the effective conversion of waste cooking oil to biodiesel. Such nanostructures of SrO bound to the SiO_2 surface may have a water-repelling property, leading to stable and enhanced catalytic activity. Increasing the hydrophobicity of the metal−organic framework (MOF) support was proposed as an effective strategy for upgrading the performance of biodiesel.[124] Further, knowledge on the chemical composition and distribution of SrO particles on the millimetric SiO_2 bead support was obtained from scanning electron microscopy (SEM) with energy-dispersive X-ray analysis (EDXA), as shown in Figure 4. Uniform deposition of SrO on the surface of the millimetric silica bead is seen in the SEM−EDXA images of the SrO/SiO_2 catalyst (Figure 4). The spherical shape of the support beds (3−6 mm) remained intact, even after microwave irradiation (for 30 s, during catalyst preparation), followed by high-temperature calcination [at 900 °C for 3 h, for the decomposition of $Sr(CO_3)_2$ to SrO] (Figure 4A). Elemental mapping of Sr and O on the SiO_2 surface is shown in the images (panels C and D of Figure 4). The energy-dispersive spectroscopy (EDS) spectrum shows the elemental composition of the catalyst (inset of Figure 4B).[117]

3.4. Sonochemistry. Another exclusive technique for SrO NP synthesis is the use of sonochemistry, wherein the acoustic cavitation produces metal oxide NPs.[125,126] During the collapse of the bubble, extreme temperatures (5000−25 000 K)[127] are obtained, causing the breaking of chemical bonds. Because this collapse occurs in very short times (less than nanoseconds), high cooling rates (>1010 K/s) are also obtained. This high cooling rate hinders the organization and crystallization of the products, and the fast kinetics results in the formation of nanostructures.[128] Tangy et al. synthesized a composite of carbon dots (C-dots) and SrO NPs via sonication.[118] The fabrication of such a composite consists of subjecting a solution of $Sr(NO_3)_2$ and C-dots to ultrasonic irradiation for 30 min in a hot water bath at 50 °C, leading to the *in situ* deposition of C-dots on the surface of $Sr(NO_3)_2$. The composite was further calcined in an inert atmosphere at 650 °C for 4 h, resulting in the formation of a SrO−C-dot

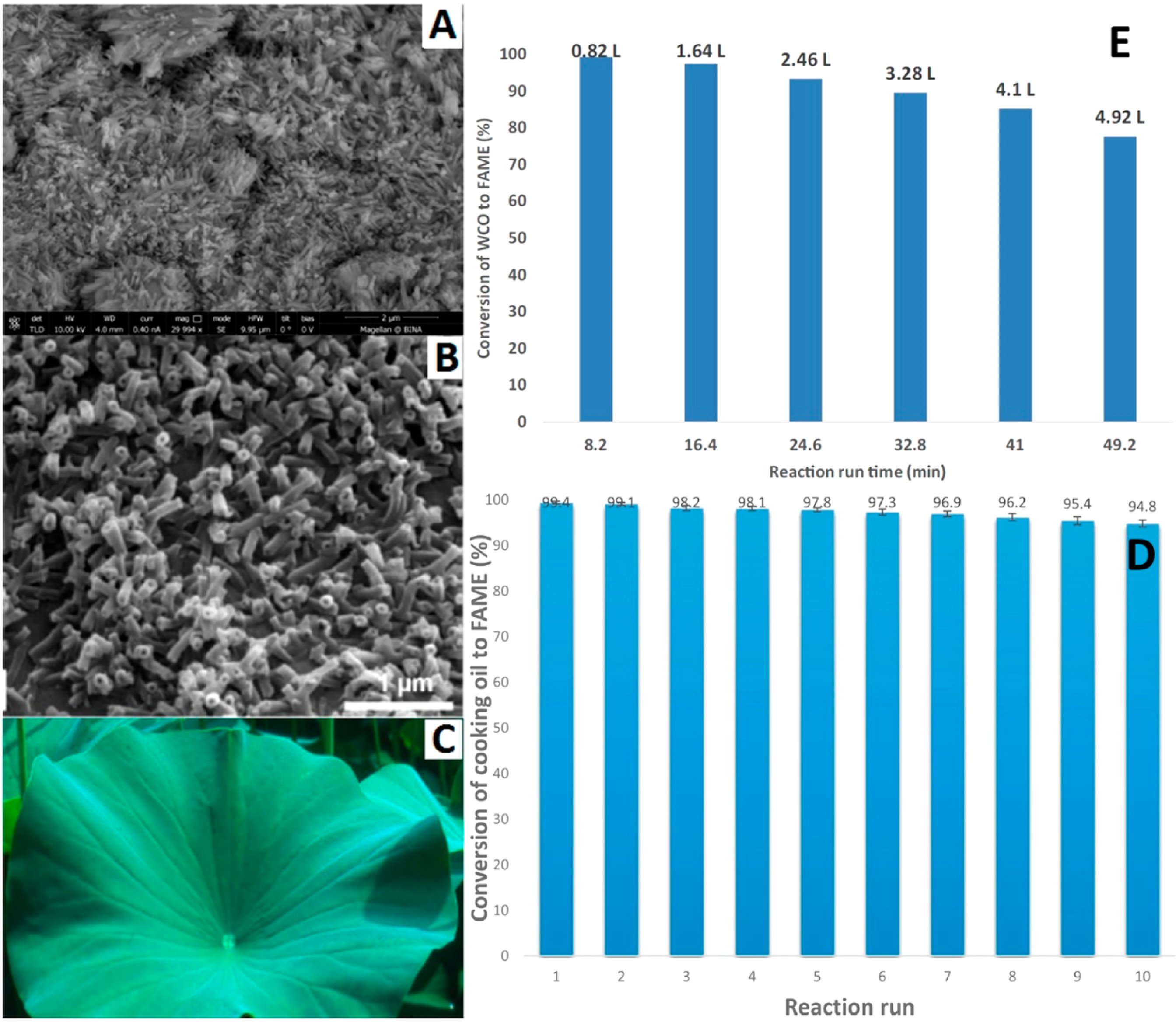


Figure 3. Structure−property relationship in the catalyst, SrO/SiO_2. (A) HR-SEM image of $SrO@SiO_2$ nanotubules of SrO on millimetric silica beads. (B) SEM image of the upper leaf side, indicating the nanotubules of wax. This figure was reproduced with permission from ref 117 Copyright 2016 American Chemical Society. (C) Digital image of lotus leaves exhibiting extraordinary hydrophobicity. Stable catalytic performance of the SrO/SiO_2 catalyst in a (D) batch and (E) continuous flow microwave-driven conversion of waste cooking oil to biodiesel. This figure was reproduced with permission from ref 119. Copyright 2017 Elsevier.

composite. A schematic illustration of the synthesis mechanism is presented in Figure 5. This carbon−metal oxide composite was used as a catalyst for the single-step conversion of *Chlorella vulgaris* microalgae to FAME in a domestic microwave oven and offered a lipid conversion value of 97 wt % and a FAME yield of 45.5 wt % in a reaction time of 2.5 min.[118]

4. SRO-BASED COMPOSITES

Composite materials have the potential advantage of benefiting from the positive possessions of each component of the composition. It is especially beneficial if the synergistic effect of the composed materials enhances the desired properties. Likewise, compositing Sr with other metals shows improved performance when used as a catalyst for biodiesel production. The composition can be achieved by solid mixing, sol−gel method, co-precipitation, or wet impregnation.

4.1. Alkaline Metal Composites.

The most widely used element for composite preparation is Ca, because it is the closest alkaline earth metal to Sr.[79] The composition of these two metals and their effect on stability, including the homogeneous contribution of leaching of active sites and the catalyst reusability, was investigated. At a Sr/Ca molar ratio of 0.5, a maximum conversion of 98.31% of palm oil to FAMEs was achieved. Significant improvement in the performance of Sr-doped CaO was reproduced in other publications, suggesting that the dramatic improvement can be achieved by a simple and non-expensive minor modification.[129,130]

Alkaline earth metals other than Ca and transition metals were also coupled with Sr. Shahbazi et al. synthesized a catalyst with different Sr−Mg molar ratios using the co-precipitation method. The basicity of the composite was found to be greater than that of bare SrO ($22.5 < H\_ \leq 27$ compared to $9.8 < H\_ \leq 15$). In optimized conditions (SrO/MgO ratio of 3:7),

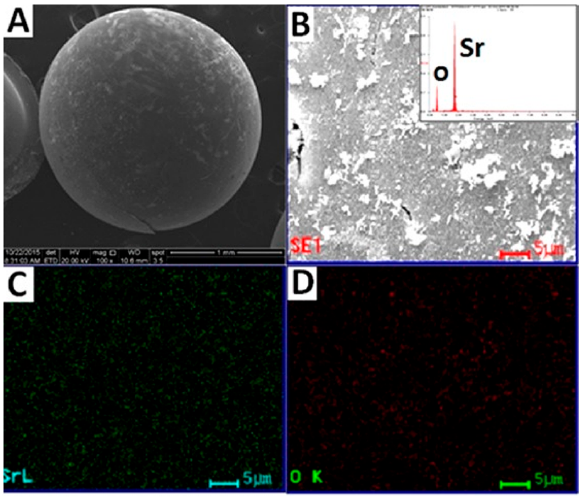


Figure 4. (A and B) SEM images, (inset of B) EDXA spectrum, and (C and D) elemental mapping of Sr and O of the SrO/SiO_2 solid base catalyst. This figure was adapted with permission from ref 117. Copyright 2016 American Chemical Society.

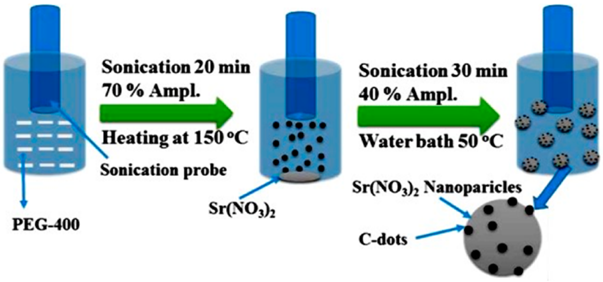


Figure 5. Schematic illustration of the sonochemical process producing a SrO−carbon nanodot composite. This figure was adapted with permission from ref 118. Copyright 2016 American Chemical Society.

87.49% biodiesel yield was obtained.[131] Similarly, Shalini et al. prepared a strontium titanate nanocatalyst through the sol−gel method using a polymer precursor to obtain $SrTiO_3$. Characterization of the pore size (7.6 nm), and total basicity (1.89 mmol/g) showed that the prepared catalyst has sufficient basic sites and enough surface exposure for heterogeneous catalysis, resulting in the highest 97.9% FAME conversion.[132]

4.2. Transition Metal Composites. Algoufi et al. investigated a SrO−Al alloy. Although Al is supposedly a weaker base than Sr, a mixture of Sr/Al of 0.5 exhibited similar basicity.[133] A glycerol conversion of 99.4% and a glycerol carbonate (GLC) yield of 100% were achieved while using this composite catalyst. Ambat et al. found that a 3:1 Sr/Al ratio demonstrated the best performance in 5% tetrahydrofuran (THF) co-solvent.[134]

de Abreu et al. composited SrO with Fe_3O_4 and $SrFe_2O_4$ to fabricate a magnetic composite catalyst for separation and reuse in babassu biodiesel production. Synthesized magnetite and strontium ferrite successfully catalyzed biodiesel with a 89.7% yield.[135] Ce and Sr composites showed successful catalysis as well.[136]

5. POROUS SUPPORT MATERIALS FOR SRO NPS

An intrinsic drawback of SrO as a catalyst is its low surface area. To increase the practical density of available active sites, SrO can be used in the form of NPs. However, introducing SrO NPs directly into the feedstock may cause several operational constraints in the process. First, although the amount of available catalytic sites increases, they could be easily blocked by intermediate products or glycerol, thus adversely affecting the reusability. Another concern regards the emulsification, leading to difficulty in catalyst separation, impeding industrial application.

5.1. Porous Ceramic Materials. Various porous supports, on which the catalyst NPs can be loaded, are examined to increase the active surface of the catalyst. Nevertheless, the exploitation of porous materials for biomass production must be carefully designed (see a typical pore size distribution in Figure 6a. The sites available for catalysis in these materials are mostly present inside the nanometric pores and inaccessible for the large feedstock molecules (triglycerides). Furthermore, they are densely packed, lowering their accessibility. As a result, mass transfer to the catalytic sites into the small zeolite pores is strongly hindered. On the other hand, it is increasingly realized that porous materials promise tremendous potential as support to nanomaterials enhancing the catalytic performance for biomass production. Support materials may influence the leeching of the catalyst active compound as well as lower its deactivation upon FFA attack. Porous supports for SrO NP can be classified as silicates and other porous oxides, ordered materials with a high surface area, such as zeolites and MOFs, and ferromagnetic materials used for magnetic separation.

SiO_2, TiO_2, Al_2O_3, and other metal oxides can form porous microstructures, demonstrating a high surface area (up to 600 m^2/g).[137−139] Moreover, the surface of porous silica can be modified to exhibit different properties. Grafting both Lewis acids and oxygen-containing molecules is easy as a result of the basic nature of the active component in the catalyst NPs. Grafting of SrO NPs to the SiO_2 support is shown in Figure 6b. Studies proved that SrO NPs loaded on porous oxides, such as Al_2O_3, SiO_2, and TiO_2, exhibit a better product yield in biomass conversion.[89,133,140]

Activated carbons were also used as a catalyst support. The high-surface-area material possesses many advantageous, including high chemical stability and cost effectiveness. Carbon-supported catalysts lead to an efficient transesterification process, prevent soap formation, and allow for recycling of the catalysts.[141]

An interesting approach to use supporting material to enhance the activiy of the catalysts is achieved by the functionalization process to form the complete catalyst. It is also important to study the various kinetic models from the reaction catalyzed by base catalysts in preparation for the scaling-up process. Understanding the effect of the support materials could assist in lowering the required activation energy and enable higher catalysis rates.[142]

5.2. Zeolites and MOFs. While high-surface-area materials boost the catalytic activity by increasing the available active sites of the catalyst, there is greater benefit using supports that themselves actively participate in the catalytic reactions. MOFs and zeolites might exhibit catalytic activity related to the metal centers or oxide moieties. For example, it was shown that Fe- and Cu-centered MOFs promote esterification and transesterification reactions.[143,144] Support materials can contribute to preserving the activity of the catalyst sites by increasing hydrophilicity. Hydrothermal stability of solid catalysts is a major challenge in the realm of designing industrial catalysts for biomass conversion.[117,124,145]

Because the reaction takes place in an aqueous environment, hydrophilicity affects accessibility of the substrate into the catalytic sites upon glyceride transesterification, as proven by Sun et al.[124] (Figure 6c).

Some zeolite crystals used as support materials also established catalytic activity toward the esterification reaction. Accordingly, large-pore zeolites [such as faujasite (FAU) and mordenite (MOR)] have been successfully examined for the esterification of fatty acids.[146] Moreover, impregnation of zeolites with alkaline earth oxides significantly increases the catalytic activity for substrate conversion.

An interesting example for a trinary zeolite−metal oxide composite was presented by Feyzi et al. To employ ZSM-5 zeolite [$Na_nAl_nSi_{96-n}O_{192}\cdot 16H_2O$ ($0 < n < 27$)] as the support material for SrO, the support was impregnated with an aqueous solution of

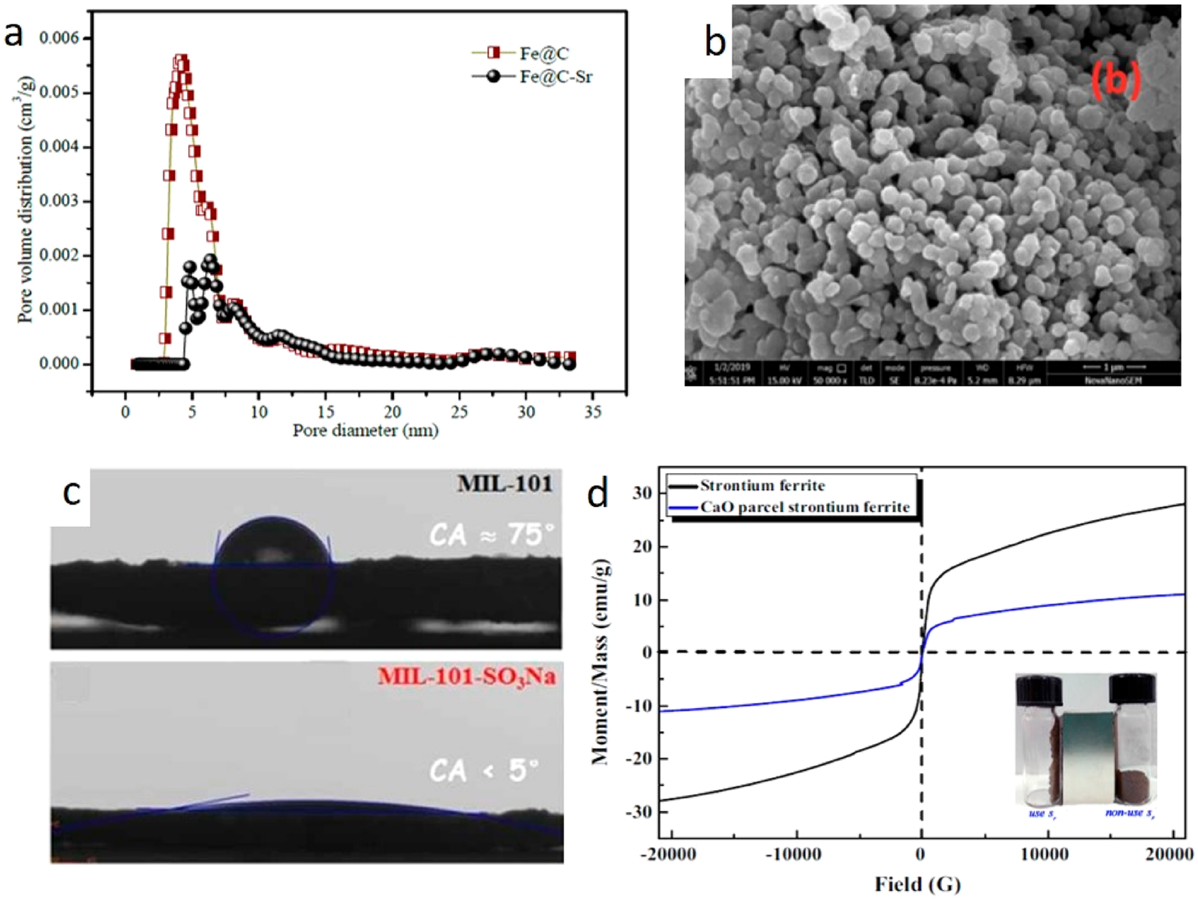


Figure 6. Effect of SrO loading on different support materials. (a) Typical nitrogen desorption−adsorption pore diameter distribution of SrO. This figure was adapted with permission from ref 147. Copyright 2020 Elsevier. (b) HR-SEM image of $SrTiO_3$. This figure was adapted with permission from ref 111. Copyright 2020 Elsevier. (c) Air−water contact angles of SrO supported by different MOFs. This figure was adapted with permission from ref 124. Copyright 2017 Royal Society of Chemistry. (d) Magnetic hysteresis curves of as-prepared SrO-based NPs on Fe_2O_3 and CaO coated on this composite. This figure was adapted with permission from ref 129. Copyright 2016 Elsevier.

$Sr(NO_3)_2$ with different loadings. When $Ba(NO_3)_2$ was added (1−7 wt % barium−strontium by weight), a Ba−Sr/ZSM-5 catalyst was obtained.[62]

5.3. Magnetic Supports for Recyclable Catalysis. An effective way to recover the nanocatalyst can be achieved by inducing the funcationality of ferromagnetism to the catalyst. As a result, the catalyst can be separated after the production process for reuse. Li et al. presented SrO loaded on an Fe@C support obtained by the carbonization of the MOF MIL−Fe (100) as a heterogeneous base catalyst (Fe@C−Sr) for the transesterification reaction. The maximum loading of SrO tested in this study was 30 wt %. However, an optimization study revealed better performance at only 4% SrO loading as a result of a higher surface area. The average pore diameter of Fe@C was increased from 4.37 to 7.07 nm (Fe@C−Sr) (Figure 6a). The saturation magnetization of Fe@C−Sr was raised to 86.76 emu/g compared to 61.42 emu/g of Fe@C, revealing that SrO addition enhances the magnetic strength of the catalyst and enhances catalyst separation efficiency (Figure 6d). Transesterification process parameters were optimized, and the biodiesel yield of 98.12% was achieved. Recyclability of Fe@C−Sr was observed all through the four cycles tested, with identical catalytic results similar to those of the fresh material.[147] For special industrial needs, SrO was also loaded on a polymeric membrane. This configuration allows for overcoming major problems related to catalyst recuperation and soap formation. Conversion can be increased by a combination of reaction and separation, using membranes with a specific pore size. The membrane Amberlyst 15, containing acid sites, was combined with basic sites of SrO. The configuration comprising the physical immobilization of the catalyst over the membrane reached a methyl ester yield of >90 wt %.[108]

6. FUTURE PERSPECTIVE

SrO is the most promising heterogeneous catalyst among processable alkaline earth metal oxides and demonstrated experimentally the highest catalytic activity. Possessing the highest basicity, it outperforms calcium and magnesium oxides. However, as of now, Ca and Mg oxides are more commonly used than SrO. Several problems impede its commercial exploitation, and thorough and comprehensive research is required to make SrO-based catalysts economically favorable. The surface area of bulk SrO is relatively low, which lowers the catalytic sites exposed for the transesterification reaction. Reducing the particle size to the nanoscale facilitates an increase in the catalytic active sites of the catalyst. On the other hand, synthesis of SrO NP is not an easy task as a result of its crystalline stability and high melting point. Both traditional and novel methods for SrO NP synthesis, including thermochemical, sol−gel, microwave irradiation, and sonochemistry synthesis, were presented. We presented the morphology of the products and compatibility to biodiesel production. To further improve the SrO performance, new approaches for nanofabrication should be employed. Thin-layer CVD, atomic layer deposition (ALD), laser ablation, and other advanced methods may enlarge the surface area and further improve the catalytic activity. Compositing Sr with other metals affects the chemical and physical properties of the catalyst. Composites have shown enhanced catalytic activity by altering the electronic state. Different components in the composite impact the desorption of the catalyst from the support (as a result of the weak support metal oxide interaction) and its deactivation upon FAA attacks. In this regard, a considerable number of alloys and composites need to be investigated further. Particular interest should be paid to lanthanides and other rare or precious metal doping. Advanced doping methods will allow for precise control on chemical states with very low dopant concentrations. Finally, grafting NPs of the heterogeneous catalyst on porous supports improves different process parameters of the transesterification reaction, with the main advantage of reusability gained by the microsize of the supports. Microscopic particles can be separated, physically or magnetically, after the reaction and reused for many cycles, dramatically reducing the production costs. Additionally, use of porous supports increases the specific surface area and chemical and physical stabilities and facilitates accessibility of catalytic sites to the substrate. Some supports, such as MOFs and zeolites, contain active sites and actively participate in the catalysis process. Rapid development must be made in the design of porous materials to optimize the potential of supporting materials for biodiesel production. Special attention is devoted on a wide range of MOFs, with variable structures and metal centers. Other chemical systems that should be considered are three-dimensional (3D) structures of nanocarbons, including graphene, carbon nanotubes (CNTs), and activated carbons, as support to SrO-based NP catalysts. Adapting recent innovation in different fields of materials science may improve SrO-based catalysis to reduce the biodiesel production cost, making the process profitable, sustainable, and environmentally friendly. This would result in a major leap toward achieving a global eco-friendly renewable and sustainable energy resource.

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Notes

The authors declare no competing financial interest.

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Asmita Dutta completed her bachelor's and master's degrees from Presidency University, Kolkata, India. Currently, she is a Ph.D. research student at the Chemical Sciences Department at Ariel University, Israel. She is studying mainly carbon nanodots and their application in energy and electrocatalysis applications.

Arie Borenstein is a lecturer in the Chemical Sciences Department at Ariel University. He received his Ph.D. from Bar-Ilan University, Israel, in the field of electrochemistry. He then pursued a postdoctoral term in University of California, Los Angeles (UCLA), where he developed nanomaterials for energy applications. In Ariel, Dr. Borenstein established the lab for nanocarbons and composites. His research focuses on development of carbon-based nanocomposites for energy systems, electrocatalysis, and carbon dioxide reduction.

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