

Review

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An overview of the catalytic properties of metal-organic frameworks and their applications in organic reactions, polymer synthesis and biodiesel production

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Abstract

Anticipation of the reduction of environmental pollution and achieving renewable fuels, leads to paying outstanding attention to biodiesel production. Biodiesel, which is referred to as renewable fuel, is obtained as a product of esterification of vegetable oils, food waste oil, animal fat, algae, etc. One of the most important factors in the production of biodiesel is the characteristic of the utilized catalysts. Several acidic and basic catalysts have been recognized for the synthesis of biodiesels with dissimilar active site dispersity and catalytic stability. Metal-organic frameworks, which are expanding as new catalysts with multiple capabilities, are being used in various industries. These catalysts have unique properties such as high porosity, high active specific surface, etc., which leads to their remarkable efficiency. In this study, the discussion focused on the use of metal-organic frameworks for biodiesel production, as well as the recent reports on the use of chromium metal-organic frameworks (Cr-MOFs), cobalt metal-organic frameworks (Co-MOFs), copper metal-organic framework (Cu-MOFs), and zirconium metal-organic frameworks (Zr-MOFs) as acid catalysts.

Keywords Biodiesel, Acid catalysts, Cr-MOFs, Co-MOFs, Cu-MOFs, Zr-MOFs

1. Introduction

Providing green sources of energy to reduce air pollution and prevent climate change is one of the requirements of researches [1, 2]. The other important reason for replacing fossil fuels is using renewable energy sources [3, 4]. Biodiesels, as green fuels, are free from sulfur and aromatic compounds and the emission of carbon monoxide and hydrocarbons is lower due to their burning [5]. There are many reports on the production of biodiesels by esterification of fatty acids from different resources such as palm, soybean, canola, vegetable raw materials, and even

waste cooking oil [6-9]. One of the effective factors in this reaction is the type of utilized catalyst. Several heterogeneous and homogeneous catalysts with acidic and basic roles have been reported in the synthesis of biodiesels [10, 11]. Recently, the use of nanocatalysts such as metal oxide, metal-organic frameworks, etc. are being developed in the production of biodiesel [12-15]. Metal-organic frameworks, which are an important category of nano compounds, have significant properties. High specific surface area, high porosity, and nanopores are the most important factors in the unique properties of these



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compounds. Many applications of metal-organic frameworks have been reported, such as biological properties, gas absorption, and catalytic properties in the synthesis of organic compounds. In general, metal-organic frameworks are synthesized from organic and metal ligands. In addition to the aforementioned factors (high porosity and high specific surface area), metal plays an important role as a Lewis acid in the catalytic applications of metal organic frameworks [16, 17].

In addition, MOFs could be recommended for the synthesis of biodiesels due to their uniform pore size and controllable functional groups [18].

Nikseresht et al. used phosphotungstic acid encapsulated in Fe (III) based metal-organic framework for biodiesel production by an ultrasound bath at room temperature [19]. The maximum yield they achieved was 96% with ethanol and n-butanol. Their results show the effect of the structure and the composition of the catalysts and the type of alcohol on the transesterification efficiency [19]. In this study, the recent developments and reports on using chromium metal-organic frameworks (Cr-MOFs), cobalt metal-organic frameworks (Co-MOFs), copper metal-organic frameworks (Cu-MOFs), and zirconium metal organic frameworks (Zr-MOFs) as catalysts in the production of biodiesels were discussed.

affected materials. MOF compounds have been used as catalysts in various fields [20-23]. For example, some of them including the polymerization process, oxidation process, and synthesis of heterocyclic compounds, they are reviewed below.

2.1. Polymerization process using MOFs as catalyst

One of MOF compounds' most important catalytic applications is their use in the polymerization process [24]. There have been many reports in this regard. Below are some examples in this field. Fig 1 shows the MOF compounds containing zinc and chromium, which were synthesized by Liu et al. and used as catalysts in the polymerization of Alkenes [25].

As mentioned, metal-organic frameworks have high catalytic properties due to their high specific surface area, which leads to an increase in the contact surface with the affected materials.

The combination of MOF containing zirconium and benzene tricarboxylic acid (BTC), named Zr-BTC (MOF-808- Fig 2), is another example that can polymerize alkenes [26].

2. Catalyst activity of metal organic framework

As mentioned, metal-organic framework has high catalytic properties due to their high specific surface area, which leads to an increase in the contact surface with the

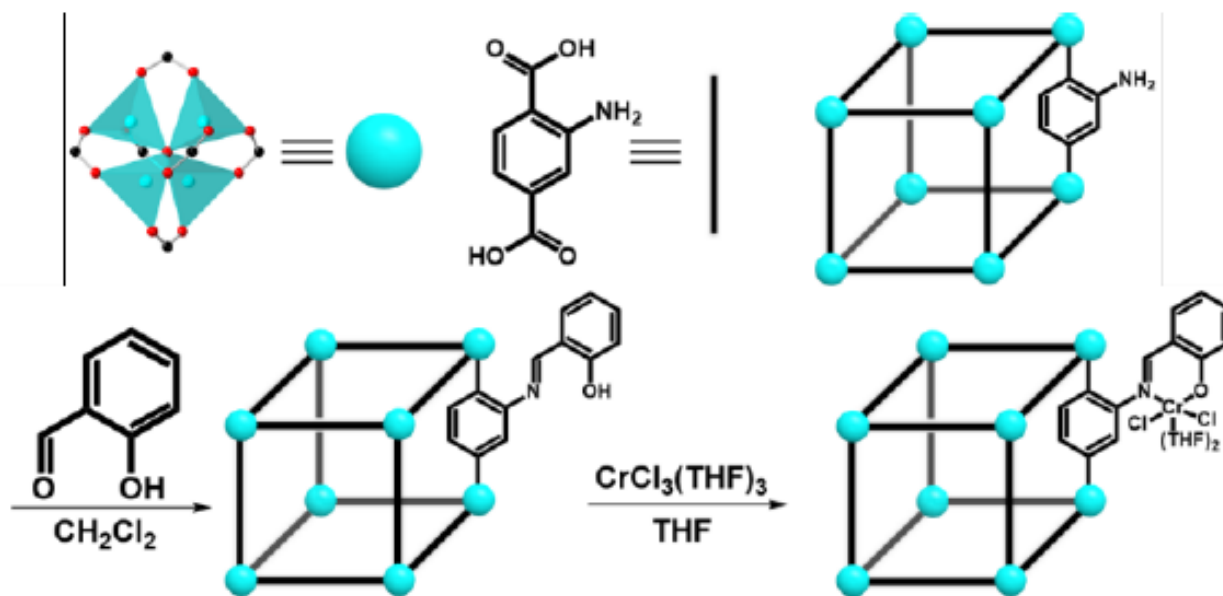


Fig 1. Structure of zinc /chromium MOF compounds with catalyst activity in polymerization of alkenes

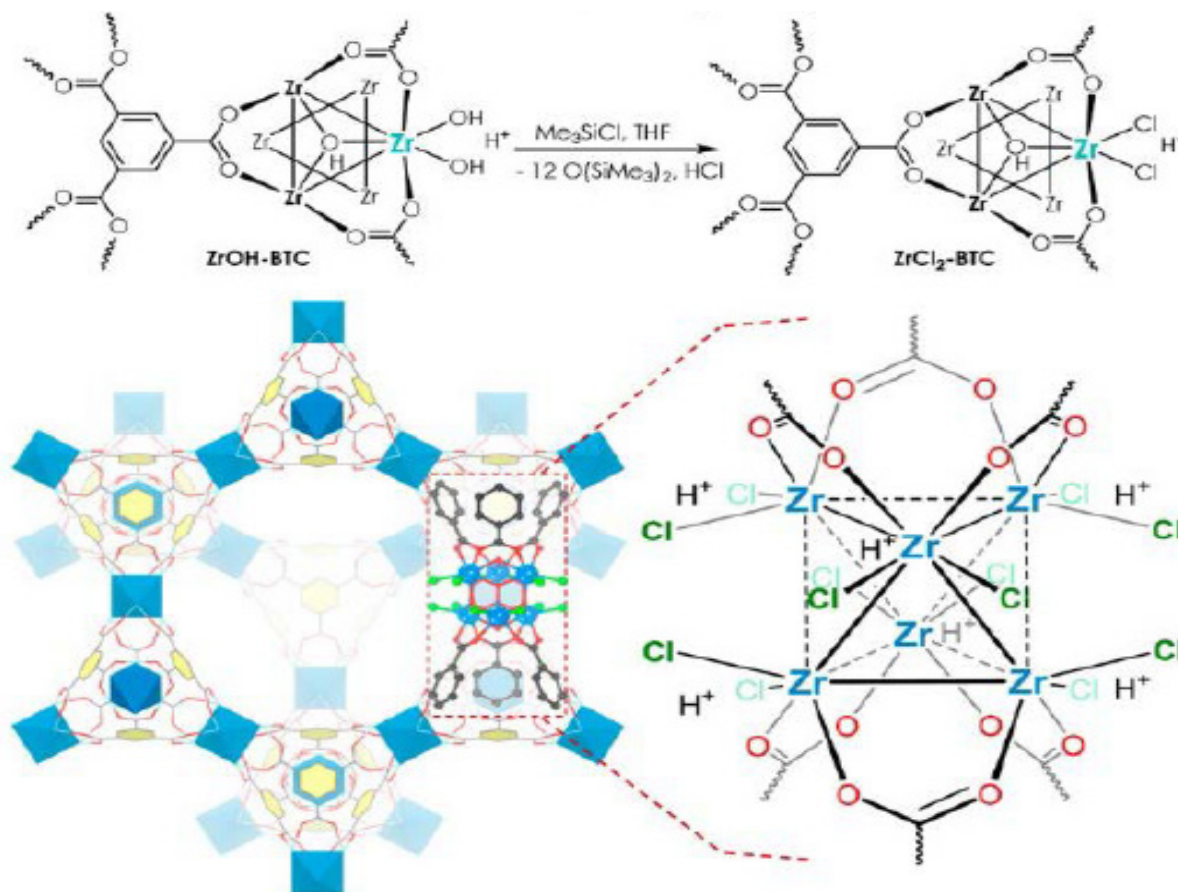


Fig 2. Structure of MOF-808 with catalyst activity in polymerization of Alkenes

Zn-based MOF, which has an octahedral structure, is capable of ethylene polymerization, and high-density PE and yield high molecular weight have been reported for it. Zn-based MOF (Fig 3), which has an octahedral structure, is capable of ethylene polymerization, and high-density PE with high molecular weight has been reported for using it as a catalyst. Another feature of this composition is the ability of cation exchange with Ti, Cr, Co, Ni, Fe, and V as shown in Fig 3 [27-30].

Polymerization of dienes is another process in which MOF compounds can be used as catalysts. The MOF containing cobalt (Fig 4) is an example previously reported for 1,3-butadiene or isoprene polymerization. The highest stereoselectivity (>99%) is reported as one of the advantages of this catalyst [28].

The polymerization of Acrylamides and Methacrylates is another example of the polymerization process in which MOF compounds are used as catalyst [31-35]. For example, the MOF shown in Fig 5, which contains iron and glycine, has been used as a catalyst in the polymerization of N,N-dimethylacrylamide [31].

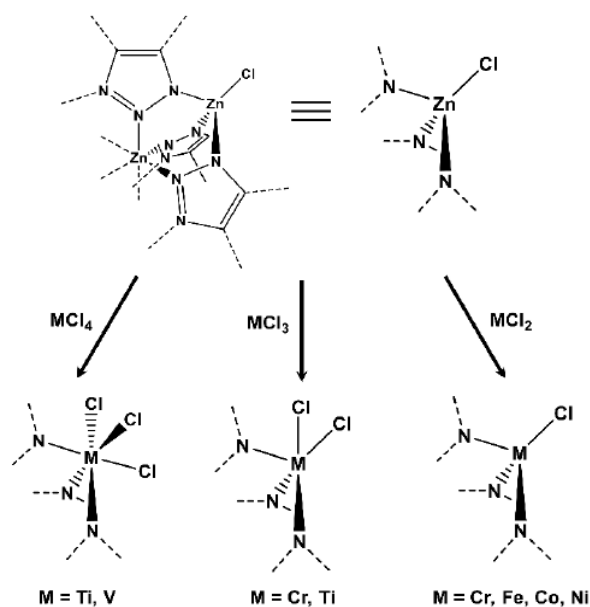


Fig 3. Structure of Zn-based MOF with catalyst activity in polymerization of alkenes with cation exchange ability

2.2. Oxidation process using MOFs as catalyst

Several oxidation processes, such as CO oxidation, water oxidation, C–H oxidation, etc. have been reported, and using MOF compounds as catalysts has led to increased efficiency and reduced reaction time.

In 2017, a study was conducted for CO oxidation by CuBTC MOF. In this study, CuBTC MOF was synthesized using Copper (II) nitrate trihydrate and 1,3,5-benzene tricarboxylic acid (BTC). In the oxidation process, thermal activation of CuBTC MOF under inert Ar gas, O₂, H₂, and CO (Fig 6) was also investigated [36].

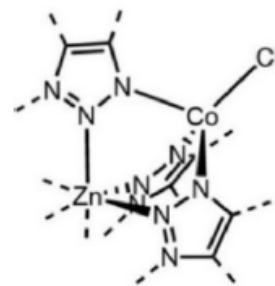


Fig 4. Structure of Co MOF with catalyst activity in polymerization of dienes

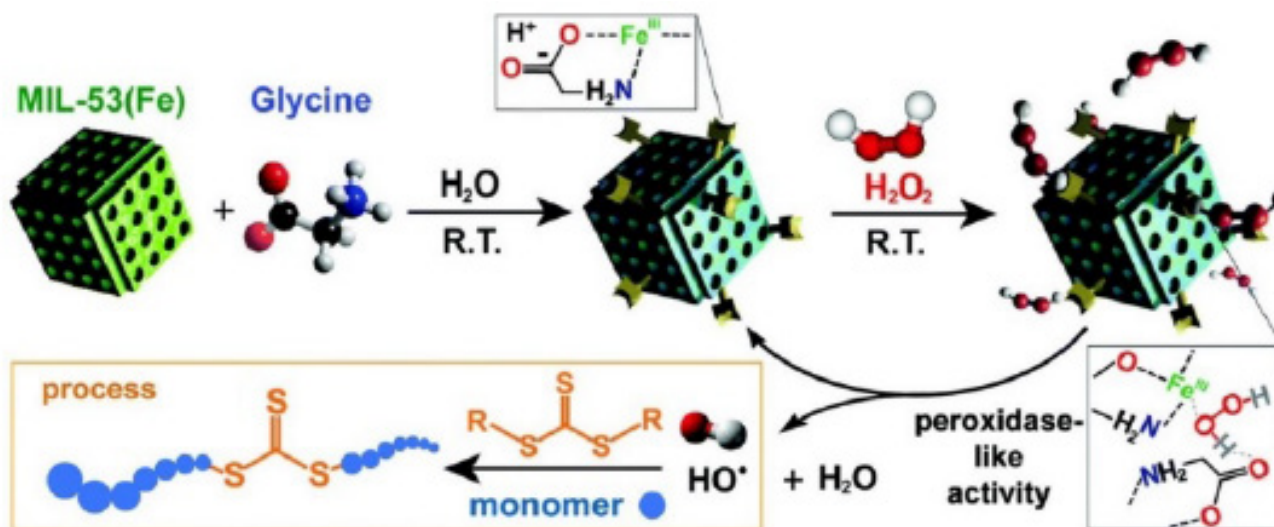


Fig 5. Synthesis of Fe/glycine MOF with catalyst activity in polymerization of N,N-dimethylacrylamide

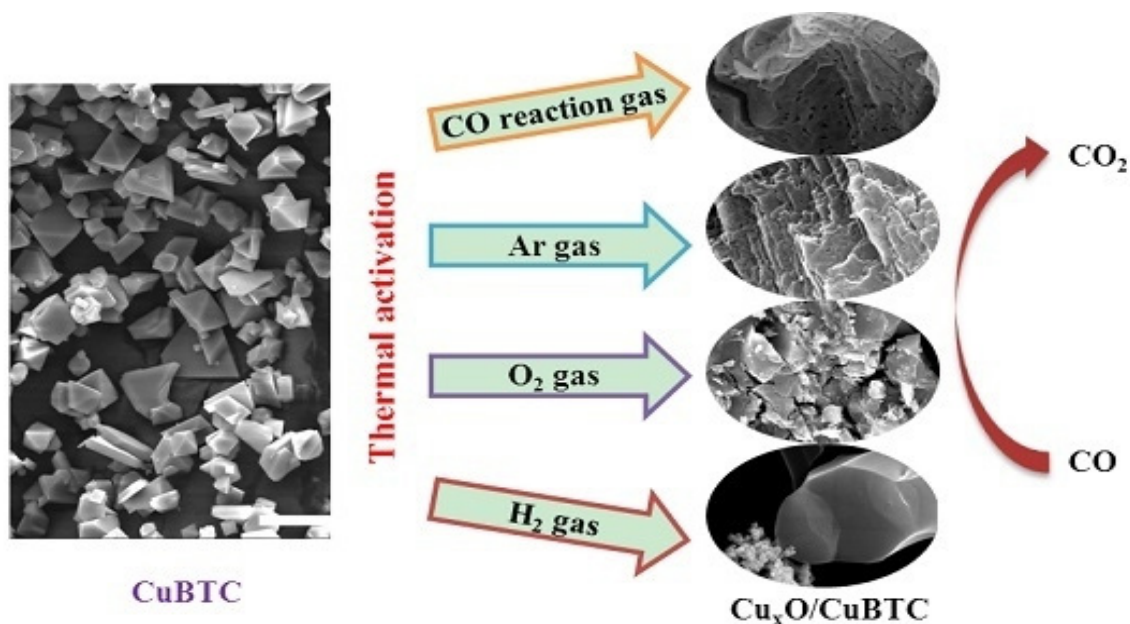


Fig 6. Thermal activation of CuBTC MOF under inert Ar gas, O₂, H₂, and CO and CO oxidation

Based on the above results, it was found that the highest activity is obtained when activating CuBTC in CO reaction gas since more pores and effective Cu₂O are formed during thermal activation [36].

Water oxidation, which is one of its most important applications in electrochemistry, has an important place in electrocatalysts. A new Ni/Fe-MOF-graphene nanocomposite (Fig 7) was synthesized by Yuan Wang et al. and evaluated in this context [37].

The obtained results indicated the high activity of Ni/Fe-MOF-graphene nanocomposite in water oxidation and its stability.

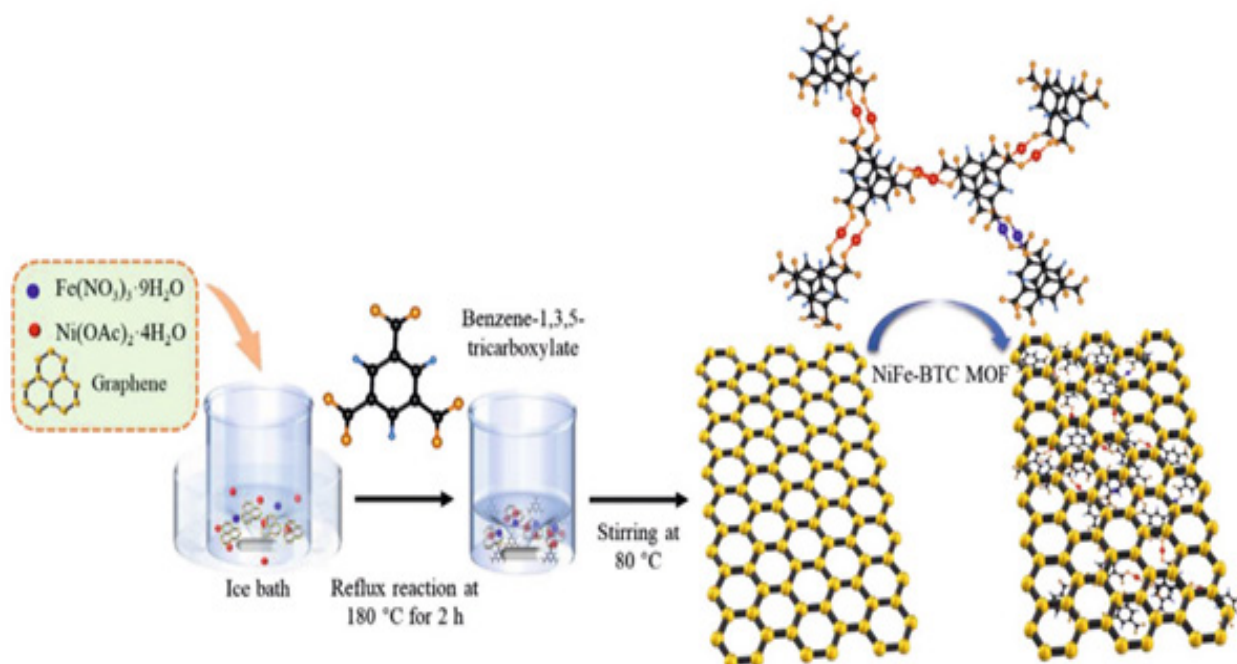


Fig 7. Synthesis of Ni/Fe-MOF-graphene nanocomposite with water oxidation application

In a 2022 study, two MOF compounds were synthesized, one containing Cu and the other containing Fe. In this study, using isotopic tracking, the catalytic properties of compounds in CH-based oxidation processes such as olefin epoxidation, carbonylation, and hydroxylation were investigated, the results are given in the tables of Fig 8 and Fig 9 [38].

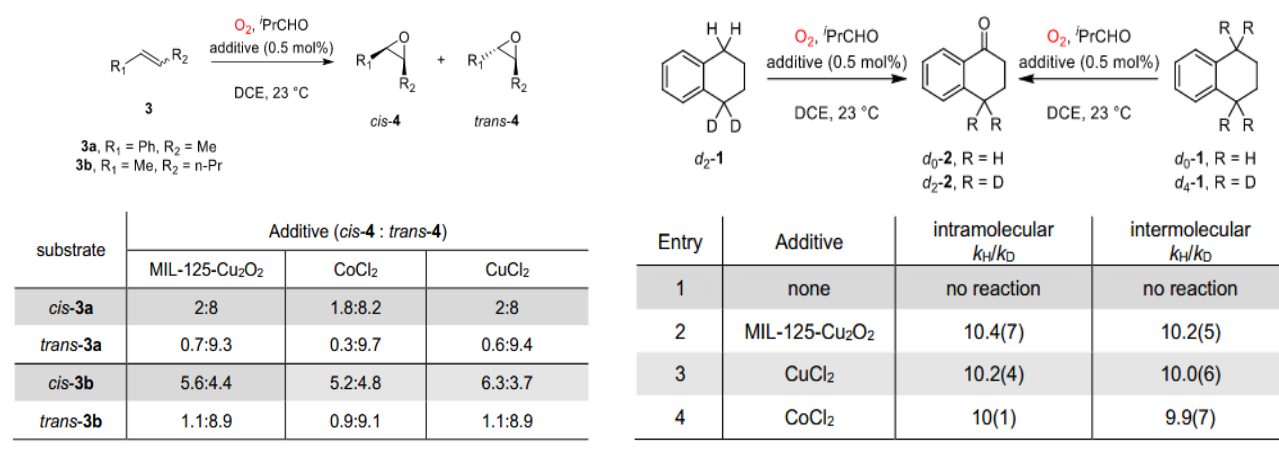


Fig 8. Olefin epoxidation and carbonylation using copper-MOF

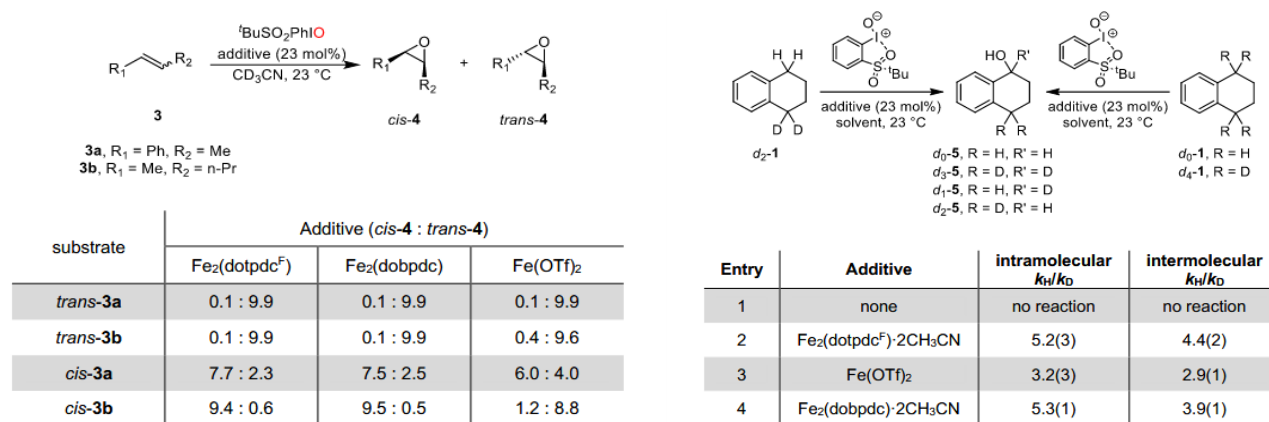


Fig 9. Olefin epoxidation and hydroxylation using Fe-MOF

2.3. Synthesis of heterocyclic compounds using MOFs as the catalyst

Heterocyclic compounds that contain one or more heteroatoms in the ring structure have a high position in organic chemistry. One of the most important uses of these compounds is their medicinal and biological properties. In general, it can be said that most medicines contain heterocyclic compounds [39-42]. Catalyst plays an important role in the synthesis of heterocycles and provide new methods in their synthesis [42-44]. In this context, MOFs have been introduced as catalysts, and some of their recent applications in the synthesis of heterocycles are given below.

In 2022, pyrazole derivatives were synthesized by Asiri et al. using zirconium magnetic MOF (Fig 10) [45].

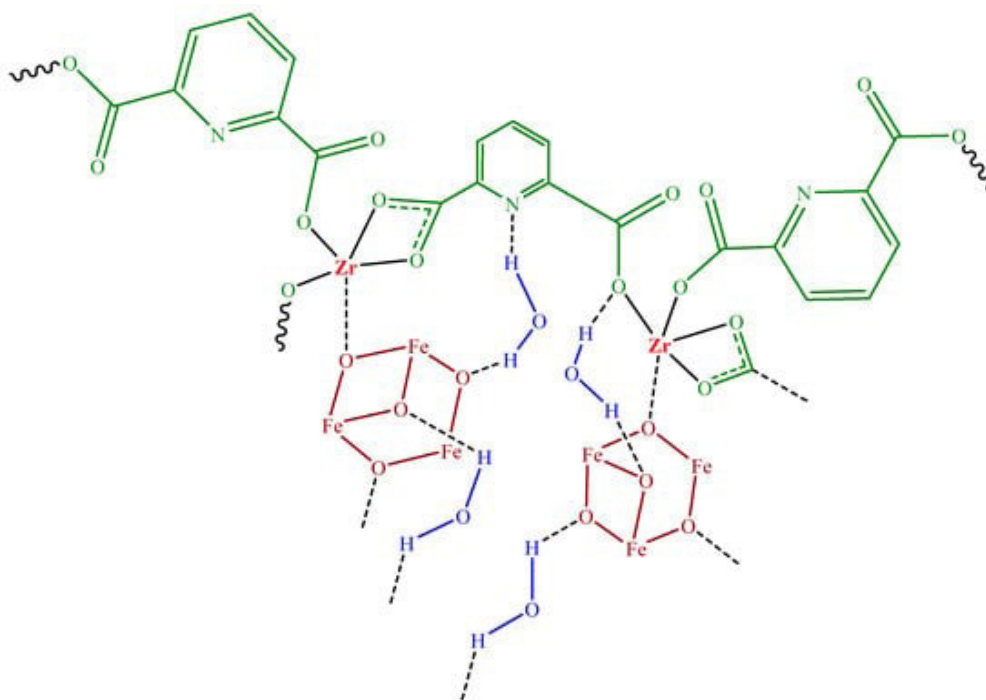


Fig 10. Structure of zirconium magnetic MOF with catalytic and biological activity

The zirconium magnetic MOF synthesized in this study had biological properties such as antimicrobial and catalyst properties [45].

The Ta-MOF nanostructures Fig 11 was another MOF composition that was used for the synthesis of new 1,4-dihydropyran derivatives [46].

The high efficiency of the products, reducing the time of derivatives synthesis, and reusing up to 6 times without any reduction in efficiency are the characteristics of this catalyst [46].

Fe₃O₄/Zn-metal organic framework magnetic nanostructure (Fig 12) was another compound that was reported as an antibacterial agent and a strong catalyst in the synthesis of another group of heterocycles named spiro [indoline-pyranopyrimidines] derivatives [47]. The spiro[indoline-pyranopyrimidines] derivatives synthesized in this study are shown in Fig 13 [47].

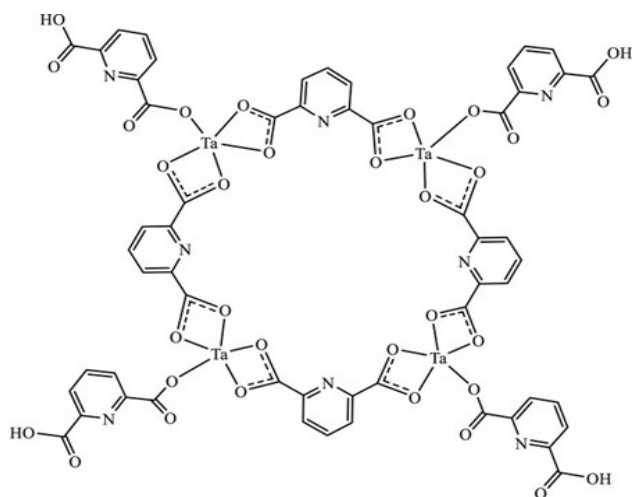


Fig 11. Structure of Ta-MOF nanostructures used as a catalyst in the synthesis of 1,4-dihydropyran derivatives

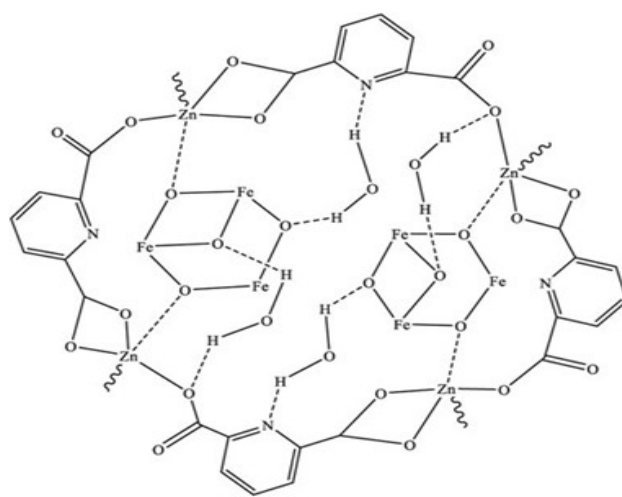


Fig 12. Structure of Fe₃O₄/Zn-metal organic framework magnetic nanostructure with catalytic and biological activity

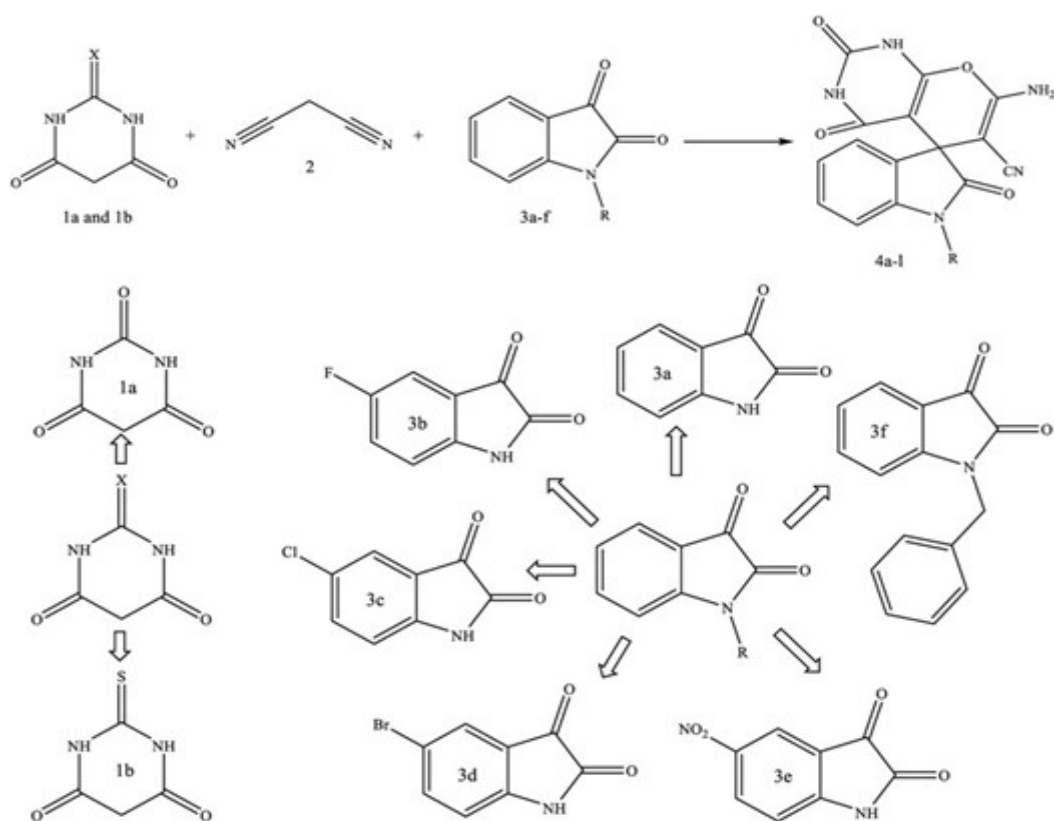


Fig 13. Synthesized spiro[indoline-pyranopyrimidines] derivatives using Fe₃O₄/Zn- metal-organic framework magnetic nanostructure

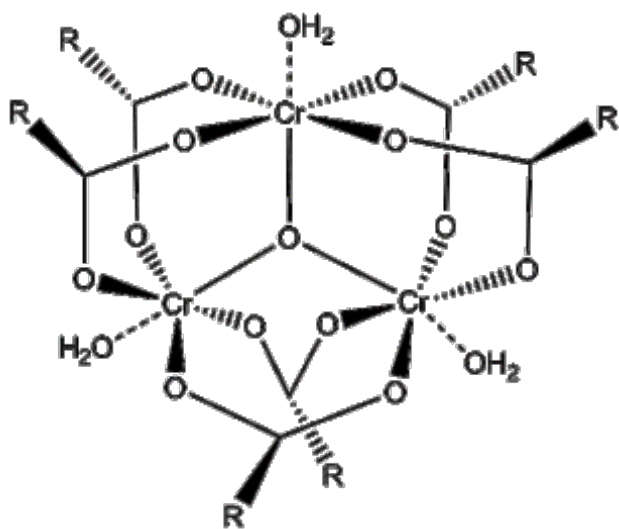


Fig 14. Structure of Cr-Tp MOF

In this study, the synthesis of new derivatives, high efficiency of products in short time, and the ability to reuse the catalyst up to 5 times without a significant decrease in efficiency have been reported among its advantages [47]. Therefore, MOF compounds, as mentioned, can be a suitable choice as catalysts in the production of biodiesels due to their high thermal stability and high specific surface area.

3. Chromium metal-organic framework (Cr-MOF) as a catalyst in biodiesel production

Chromium is one of the intermediate elements of the periodic table and the first element of the group of six in the periodic table. One of the most important uses and properties of chromium is its protection abilities against rust and corrosion [48, 49].

Chromic acid, which contains chromium in the highest oxidation state (+6), is used in various industries and laboratories such as electroplating, photographic industry, and wood preservative to protect against decay and insect infestation [50-52]. Based on the reports on the biological properties of chromium and its compounds and complexes, it could demonstrate antibacterial activity, anticancer activity, and antioxidant activity [53-55]. Several metal-organic frameworks have been synthesized using chromium. There are some reported properties for these metal-organic frameworks, for instance, photocatalytic detoxification in water, removal of antibiotics, catalytic applications, synthesis of heterocycles, and organic dye adsorption [56-59]. The recent reports depicted the use of chromium metal-organic formwork as a Lewis acid catalyst in biodiesel production. Fig 14 shows the structure of Cr-Tp MOF utilized for biodiesel production by Calophyllum inophyllum oil [60].

The structure of this metal-organic formwork, in which terephthalate was used as a ligand, was confirmed using spectroscopic data such as Fourier-transform infrared (FT-IR), scanning electron microscopy (SEM), energy-dispersive X-ray spectroscopy (EDS) and X-ray diffractive (XRD) [60]. Reusability of up to 10 times and a high conversion percentage of oil to biodiesel (~93%) were the most important advantages of this catalyst [60]. Cr-EDTA-MOF was another synthesized novel compound that was used as a green catalyst in biodiesel production after confirmation of its structure by powder X-ray diffraction (PXRD), Brunauer–Emmett–Teller method (BET), SEM, EDS, FT-IR, and X-ray mapping techniques. In the reported esterification reaction using this catalyst, oleic acid, palmitic acid, and methanol were used (Fig 15) and an efficiency of over 90% was achieved [61].

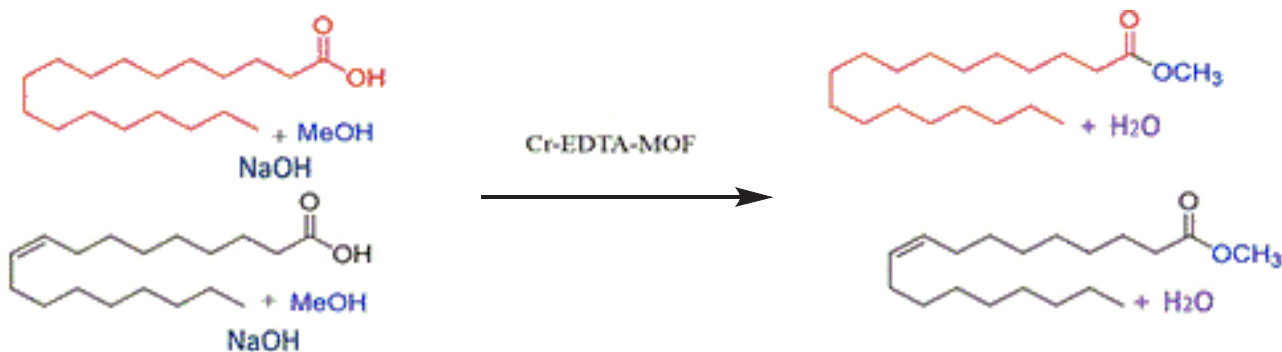


Fig 15. Cr-EDTA-MOF in biodiesel production from oleic acid and palmitic acid

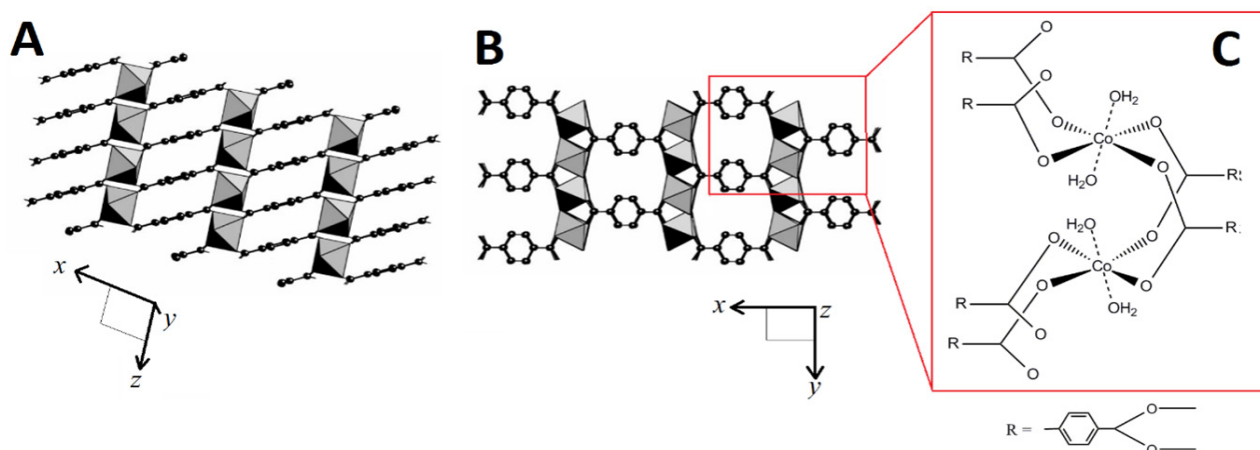


Fig 16. Structure of Co-Tp MOF

The High surface area of synthesized Cr-EDTA-MOF (234.55 m²/g) could lead to the use of a small amount of catalyst. Besides, this catalyst has shown the properties of easy separation, being recyclable and reusable without a significant decrease in catalytic activity (reusable 4 times with acceptable efficiency) [61].

4. Cobalt metal-organic framework (Co-MOF) as a catalyst in biodiesel production

Cobalt is one of the most important intermediate elements of the periodic table. This element and its compounds have shown many catalytic and biological properties such as antibacterial, antileishmanial, antioxidant, enzyme inhibition, and anticancer [62, 63]. In organic chemistry, cobalt and compounds containing cobalt such as its complexes are known as strong catalysts in the synthesis of organic and heterocyclic compounds [64, 65]. Also, cobalt compounds have depicted strong catalytic properties in Fischer-Tropsch synthesis [66] and were used as a clean and efficient technology for the destruction of organic pollutants [67]. Metal-organic frameworks containing cobalt have displayed strong catalytic properties in the synthesis of organic compounds [68], high-performance asymmetric supercapacitors [69], high-performance electrocatalysts for H₂O₂ production [70], and selective electrooxidation of glycerol coupled with hydrogen production [71].

In 2020, Marso et al. synthesized metal-organic forms containing cobalt and terephthalate (Fig 16). After confirming their structures by spectroscopic FTIR, SEM, EDS, and XRD techniques, they were used as catalysts in saponification reaction and biodiesel production using methanol, and Calophyllum inophyllum oil [60]. Reusability of up to 10 times and a high conversion percentage of oil to biodiesel (~93%) were the most important advantages of this catalyst [60].

Another cobalt (II) metal-organic framework was synthesized in 2018, using Co(NO₃)₂·6H₂O, 1,2-di-(4-pyridyl)-ethylene and 5-Nitroisophthalic acid by hydrothermal method [72]. X-ray diffraction, melting point, IR, and elemental analysis techniques were used to determine the molecular structure of the synthesized catalyst. Then, the synthesized MOF was used as an acid catalyst under ultrasonic conditions to produce biodiesel using methanol and Erythrina mexicana oil. Gas Chromatography (GC) method, ¹H, and ¹³C NMR spectroscopy were used to determine and quantify the oil composition. Finally, a total yield of 83.7% was reported using cobalt (II) metal-organic framework and Erythrina mexicana oil with composition listed in Table 1 [72].

Table 1. Fatty acid composition of Erythrina mexicana oil

No.	Fatty acid methyl esters	Relative percentage*
1	Methyl palmitate	15.2 ± 0.3
2	Methyl oleate	4.8 ± 0.1
3	Methyl stearate	37.2 ± 1.6
4	Methyl linoleate	21.3 ± 0.5
5	Methyl arachidate	0.98 ± 0.002

5. Copper metal-organic framework (Cu-MOF) as a catalyst in biodiesel production

Copper is a metal that is in group d and the first element of group 11 of the periodic table of elements. Copper is the most conductive metal after silver, and is widely used in electrical industries such as electric wires because it is cheaper than silver [73].

Unlike fish and invertebrates, whose main blood component is hemoglobin, which is made of iron, the main blood component of mollusks and crustaceans is hemocyanin (containing copper) [74]. Copper is essential for all plants and animals and is present in a variety of enzymes, including cytochrome oxidase and the copper-zinc-containing enzyme superoxide dismutase [75]. In addition, the copper element and its compounds, including copper oxides and complexes containing copper, have shown significant biological properties such as anticancer activities, antitumor agents, antioxidant activity, and antimicrobial activity [76-79]. The use of copper in organic chemistry as a catalyst has been reported especially in Ullmann coupling reactions [80]. Metal-organic frameworks containing copper have also been developed and have been conducted in organic chemistry as a catalyst in reactions such as oxidation and the oxidative cross-coupling of amines and alcohols, click reaction, Biginelli reaction, and Knoevenagel reaction [81-83]. Adsorption of organic pollutants, drug delivery systems, cytotoxic activity, and battery-supercapacitor-type hybrid energy storage devices are reported as capabilities of the newly synthesized copper MOFs [84-87].

In 2020, waste cooking oil and new copper and calci-

um-based organometallic frameworks as an acid catalyst were used in the esterification reaction and production of biodiesel [88]. The synthesized catalyst was identified and confirmed by XRD, SEM, thermogravimetric analysis (TGA), FT-IR, and BET. The esterification and transesterification of oil was performed and the specification of the produced biodiesel (with efficiency reported in Fig 17) was according to the ASTM standards. [88].

In the mentioned study, optimal biodiesel yield was reported to be 84.5 (vol%) and biodiesel yield decreased up to 7% after 3 catalyst- cycles [88].

Tresia Pangestu et al, synthesized a new copper-based metal-organic framework (with a surface area of 1085.72 m²/g) using copper sulfate and benzene-1,3,5-tricarboxylic acid (BTC- Fig 18). The synthesized MOF had thermal stability up to 303°C and the produced CuBTC-MOF was used to produce biodiesel from palm oil [89].

In their study, the optimal amount of 0.04 g of catalyst, palm oil, and methanol transesterification was used and the efficiency was reported to be 91% [89].

Xanthoceras sorbifolia Bunge oil and methanol were utilized for biodiesel production by ILe@Cu@MOF as a catalyst. The stages of MOF synthesis and biodiesel production are shown in Fig 19 [90].

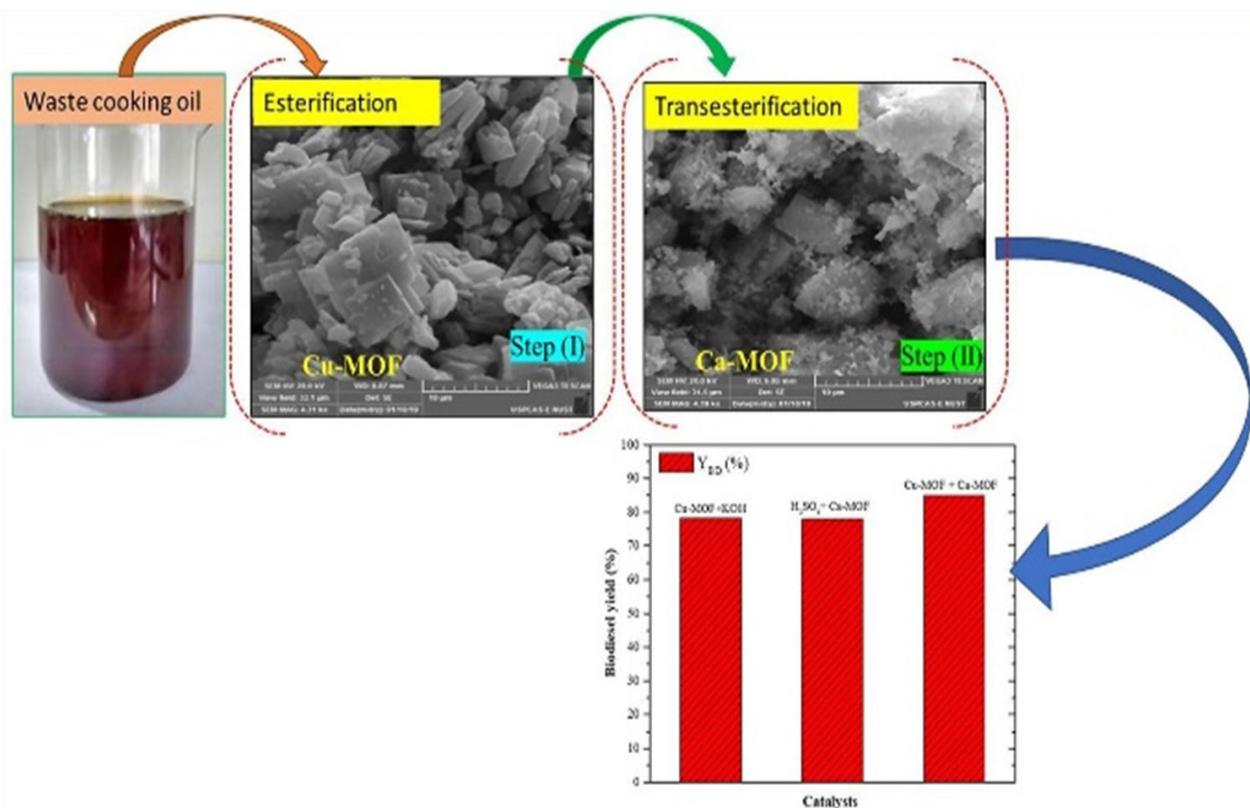


Fig 17. The esterification and transesterification of waste cooking oil by different catalysts

An efficiency of 82.85% was reported utilizing optimal conditions of 3 wt% catalyst, molar ratio of methanol to oil 35:1, and temperature of 50°C for 4 hr. Besides, biodiesel production efficiency was decreased to 73.4% after 5 times recycling of the catalyst [90].

6. Zirconium metal-organic framework (Zr-MOF) as a catalyst in biodiesel production

Zirconium is the 40th element of the periodic table and is known by the Zr symbol. Zirconium is lighter than steel and its hardness is similar to copper, and corrosion resistance can be considered as one of its most important features [91, 92]. The most important uses of zirconium in the industry are in pottery as a refractory material [93], in the jewelry industry as a gemstone [94], in the chemical industry for piping in corrosive environments [95], in the medical industry in the manufacture of restorative devices and medical prostheses, such as knee and hip joints, as well as in the dental field for dental implants and crowns

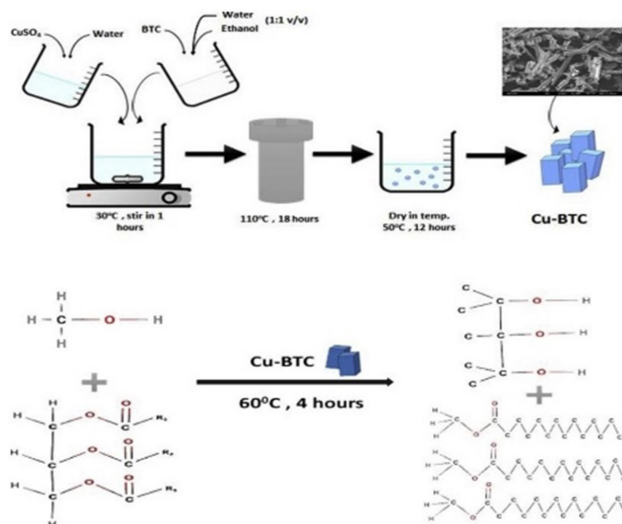


Fig 18. Synthesis of Cu-BTC MOF and transesterification of palm oil

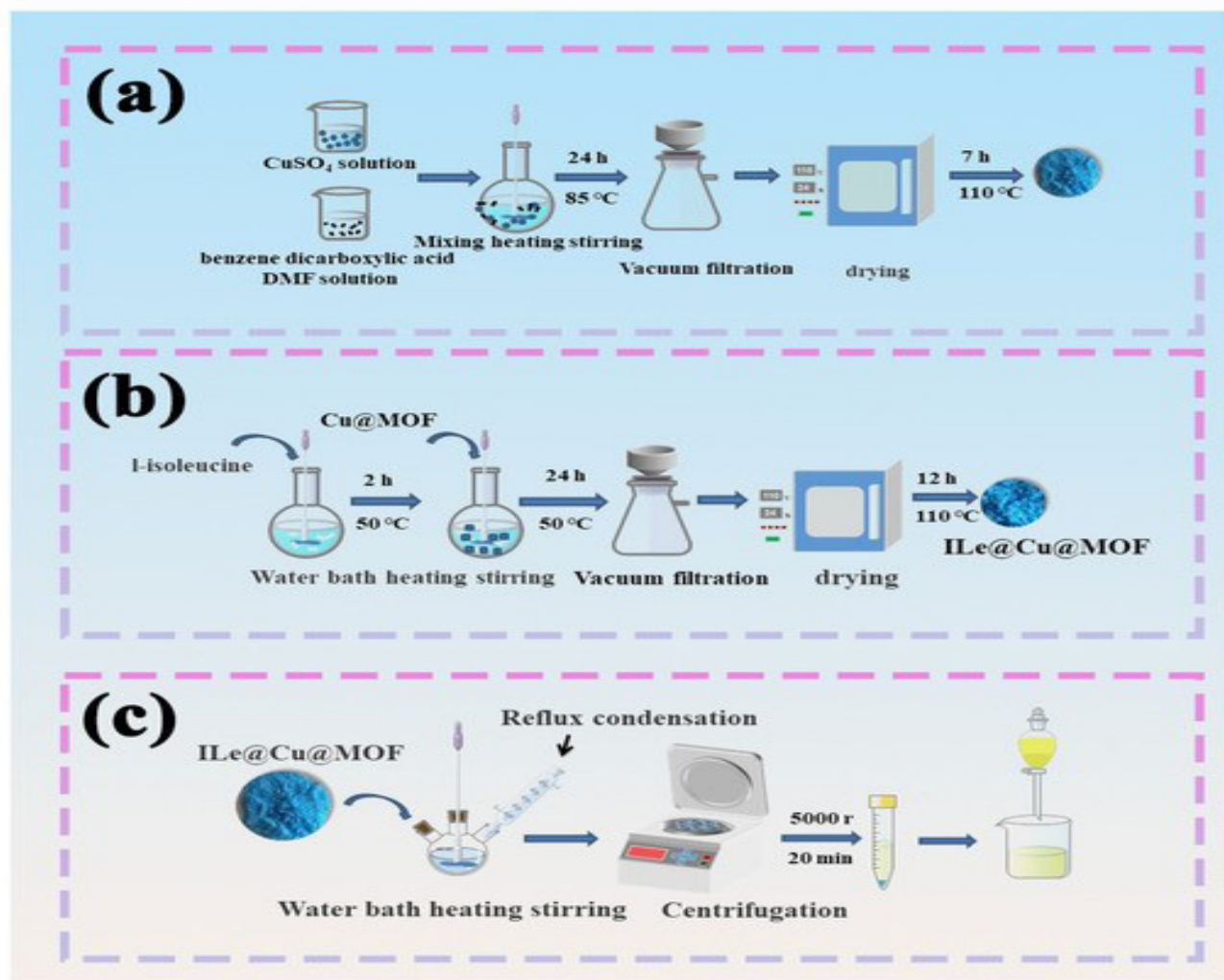


Fig 19. a: Synthesis of Cu@MOF catalyst; b: Synthesis of ILe@Cu@MOF; c: Biodiesel production

[96-99]. Biological properties such as anticancer activity, blood anticoagulant, antimicrobial, etc. have been reported from compounds containing zirconium [100, 101]. The catalytic role in photocatalytic reactions can be called the most important catalytic property of zirconium [102, 103]. From other reported catalytic properties of zirconium and its compounds, biodiesel production, [104], diesel soot oxidation [105], synthesis of organic and heterocycles compounds, etc. can be mentioned [106].

The first compound of zirconium MOF was synthesized and reported in 2008 named UiO-66 (Fig 20). Strong oxygen-zirconium bonds have been mentioned as the factor of its stability [107].

So far, many MOF compounds containing zirconium have been synthesized and reported. Several properties such as

supercapacitive [108], photoelectrochemical [109], catalytic properties [110], application in environment [111] etc. have been reported for zirconium MOFs. In general, these unique properties can be attributed to the highly stable, high thermal stability, and high specific surface area of zirconium MOFs [111, 112].

Zirconium MOF and synthesized MOF compounds containing UiO-66 can be named as catalysts that have been reported the most in biodiesel production [113]. In general, The proposed mechanism for esterification and biodiesel production using UiO-66 is given in Fig 21 [114]. Table 2 shows some of these catalysts, the used raw materials, reaction conditions, reusability, and yield reported for them.

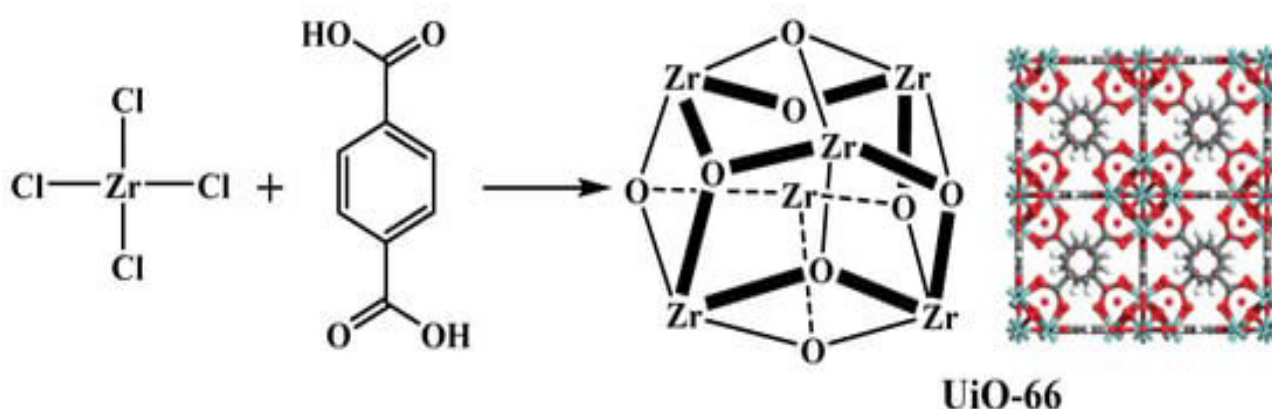


Fig 20. Synthesis of UiO-66

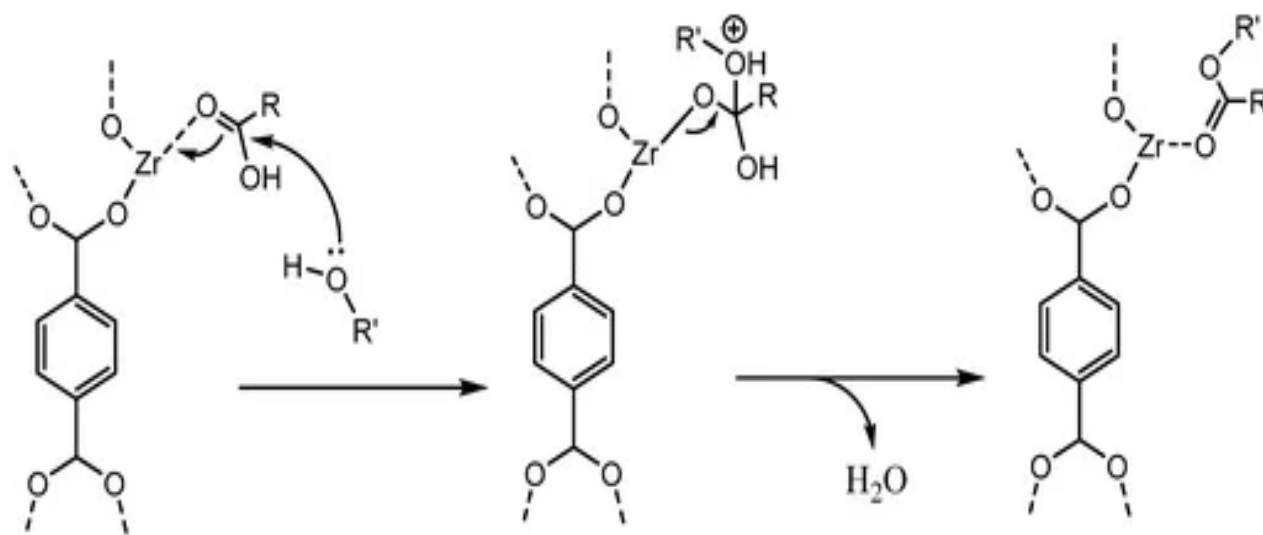


Fig 21. Proposed mechanism for esterification and biodiesel production using UiO-66

Table 2. Production of biodiesel using zirconium MOFs and MOF compounds containing UiO-66

Entry	Catalyst	Raw Materials	Reaction conditions (Time, Temperature, Catalyst amount, Molar ration (Acid(Oil):Alcohol))	Reusability	Yield (Y%) or Conversion (C%)	Ref
1	ZrSiW/UiO-66	Oleic acid + Methanol	4 h, 150 °C, 8%, 1:20	4 cycles, C = 88.9%	C = 98.0%	[115]
2	Ce-BDC@HSiW@UiO-66	Oleic acid + Methanol	4 h, 130 °C, 0.2 g, 1:30	6 cycles, C = 76.9%	C = 81.5%	[116]
3	K-PW ₁₂ @UiO-66(Zr)	Oleic acid + Methanol	4 h, 75 °C, 5%, 1:20	10 cycles, no significant loss	C = 90%	[117]
4	UiO-G	Oleic acid + Methanol	2 h, 70 °C, 8%, 1:12	4 cycles, C = 66.6%	C = 91.3%	[118]
5	UiO-66(Zr)-NH ₂	Oleic acid + Methanol	4 h, 60 °C, 6%, 1:39	4 cycles, C > 50%	C = 97%	[119]
6	10SA/UiO-66(Zr)	Oleic acid + Methanol	4 h, 25 °C, 6%, 1:39	6 cycles, C = 83%	C = 94.5%	[120]
7	FDCA/SA-UiO-66(Zr)	Oleic acid + Methanol	24 h, 60 °C, 6%, 1:40	6 cycles, Y > 90%	Y = 98.4%	[121]
8	Ca ²⁺ /UiO-66(Zr)	Oleic acid + Methanol	4 h, 60 °C, 6%, 1:39	5 cycles, Y = 84%	Y = 98%	[122]
9	AIL@NH ₂ -UiO-66	Oleic acid + Methanol	6 h, 75 °C, 5%, 1:14	6 cycles, C = 90.42%	C = 95.22%	[123]
10	AILs/HPW/UiO-66-2COOH	Soybean oil + Methanol	6 h, 110 °C, 10%, 1:35	5 cycles, C > 80%	C = 95.8%	[124]
11	PW ₁₂ @UiO-66	Soybean oil + Methanol	4 h, 75 °C, 0.2 g, 1 g:5.5 ml	4 cycles, no significant loss	C = 91.1%	[125]
12	PSH/UiO-66-NO ₂	Jatropha oil + Methanol	4 h, 70 °C, 4%, 1:25	3 cycles, C = 77.14%	C = 97.57%	[126]
13	Lipase/Zr-MOF/PVP	<i>Ricinus communis</i> oil + Methanol	12 h, 50 °C, 2 mg, 1:3	7 cycles, C = 66%	C = 83%	[127]
14	HPW/UiO-66-NH ₂	Euphorbia Lathyrus L. oil + Methanol	8 h, 180 °C, 3.5%, 1:40	4 cycles, no significant loss	Y = 91.2%	[128]

5. Conclusion

In this study, some recent reports were discussed on organic compounds synthesis, polymers synthesis, and biodiesel production using chromium metal-organic frameworks (Cr-MOFs), cobalt metal-organic frameworks (Co-MOFs), and copper metal-organic frameworks (Cu-MOFs) as acid catalysts. High ability as acid catalysts was depicted for metal-organic frameworks in organic synthesis, polymer synthesis, and biodiesel production. High efficiency, reusability, and being green and friendly to the environment were among the capabilities of the use of Cr-MOFs, Co-MOFs, Cu-MOFs, and Zr-MOFs as catalysts reported in biodiesel production. Therefore, it can be concluded that the development and synthesis of new MOFs based on chromium, cobalt, copper, and zirconium could lead to achieve valuable catalyst for green fuel production using different oils.

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Declarations

Ethics approval and consent to participate

Not applicable.

Consent for publication

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Competing interests

The authors declare that they have no competing interests.

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