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Review Article

Recent Advances in Nickel Catalysts for Deoxygenation of Triglycerides and Fatty Acids: A Review

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ABSTRACT

The increasing global demand for fossil fuels and their negative environmental impact have spurred considerable interest in developing renewable fuel sources. Biodiesel, derived from vegetable oils and animal fats, shows great promise as an alternative fuel due to its similar combustion characteristics to fossil fuels. However, the high oxygen content of biodiesel leads to technical challenges in engines. To address this, catalytic deoxygenation has been developed to convert the fatty acids in vegetable oils into second-generation liquid hydrocarbons. This process offers a viable method for the transportation industry to produce biofuels from renewable feedstocks. Palladium (Pd) and platinum (Pt) are commonly used deoxygenation catalysts, known for their excellent performance, but their high cost limits their use in large-scale industrial applications. Nickel (Ni), a non-noble metal, has emerged as a more cost-effective alternative, demonstrating significant catalytic activity for deoxygenation. This review explores the latest advances in the use of Ni catalysts for the deoxygenation of triglycerides and fatty acids. Key focus areas include the deoxygenation process, the role of Ni catalysts, and recent innovations in combining Ni with auxiliary materials to enhance performance. Additionally, the review examines how catalyst loading impacts the deoxygenation efficiency of triglycerides and fatty acids. This study provides crucial insights into the performance of Ni catalysts with different supports, offering a solid foundation for future research into biofuel production using Ni-based catalytic systems.

Keywords: Active metal, catalyst loading, catalyst, deoxygenation, mesoporous materials, metal oxides

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INTRODUCTION

The twenty-first century is marked by a heavy reliance on fossil fuels to meet industrial and domestic energy demands. This includes the use of coal, oil, and natural gas, which are predominantly used to generate electricity and power transportation. Despite the advancements in renewable energy technologies, the demand for fossil fuels continues to grow due to their high energy density and cost-effectiveness. However, the use of these fuels raises concerns about environmental sustainability and the need to transition towards cleaner and more sustainable energy sources. It was reported that 44×10^{15} kg of atmospheric carbon was produced each year by fossil fuels consumption, which is 400 times the yearly carbon standard set by the primary productivity of the present world biota (Jeng et al., 2021). The continued dependence and usage of fossil fuels has caused a severe impact on the environment, leading to increased awareness and high demand for sustainable and renewable energy to replace fossil fuels (Jeng et al., 2021). This environmental issue is very worrying and has received the world's attention through international agreements like the Paris Climate Agreement, which 190 parties signed in January 2021.

Biofuels offer significant benefits in the energy sector, playing a key role in the global transition to sustainable and renewable energy. One of their main advantages is reducing greenhouse gas emissions. Unlike fossil fuels, which release carbon stored for millions of years, biofuels are made from biomass that absorbs CO_2 during growth, creating a more balanced carbon cycle (Prasad & Ingle, 2019). The International Energy Agency (IEA) highlights biofuels as an important tool in lowering CO_2 emissions and combating climate change (Jeng et al., 2021).

Biofuels also enhance energy security by diversifying energy sources. They can be produced from various feedstocks, including agricultural and forest sources, as well as specific energy crops (Wang et al., 2023). This reduces dependence on imported fossil fuels, boosts rural economies by creating jobs, and increases national energy security. The U.S. Department of Energy emphasizes the potential of biofuel production to support rural development and energy independence (Schwab, 2016). The future of biofuels is promising due to technological advancements and strong policy support. Innovations in advanced biofuels, like cellulosic ethanol and algae-based fuels, address many limitations of first-generation biofuels. These advanced biofuels, derived from non-food biomass, offer higher energy yields and reduced environmental impact (Popp et al., 2016). They are expected to play a crucial role in helping the transportation sector reduce its carbon footprint and meet renewable energy targets (Darda et al., 2019).

Biofuels can be generated as an alternative to fossil fuels for energy production by means of deoxygenation (Osman et al., 2023). The process of deoxygenation in vegetable oil is often linked to the cracking of hydrocarbon chains, resulting in the breaking of the chains and the removal of oxygen in the form of carbon dioxide and carbon monoxide through decarboxylation and decarbonylation processes. Previous studies have demonstrated that platinum (Pt) and palladium (Pd), which are noble metals, exhibit favorable performance in deoxygenation reactions (Janampelli & Darbha, 2018). The study by Raut et al. (2016) showed that the application of palladium supported on SBA-16 was highly effective in removing oxygen from fatty acids. Platinum and palladium catalysts have been found to possess excellent capacities in removing oxygen from the feed. Nevertheless, this application is unsuitable for industrial utilization because of the exorbitant expenses associated with its implementation (Wang et al., 2019). Figure 1 exhibits a timeline for the development of noble metal Ni catalysts in renewable energy alternative fuel production.

Remarkably, the Ni-supported catalyst was discovered to possess exceptional deoxygenation capabilities, resulting in a diesel selectivity of 80% (Hafriz et al., 2020). Ni-supported catalysts are preferred for hydrodeoxygenation (HDO) applications as they efficiently cleave C-O bonds and exhibit high activity and selectivity (Ambursa et al., 2021; Yang et al., 2019). These qualities make them an exceptional choice among 3d transition metals for the process of hydrodeoxygenation in biofuel production. Their extraordinary ability to cleave C-O bonds is a significant characteristic of Ni catalysts in HDO, which is vital for eliminating oxygen from molecules derived from biomass (Attia et al., 2020). Ni-supported catalysts exhibit exceptional performance in hydrogenation reactions. They also demonstrate high selectivity towards the desired deoxygenated products and minimize

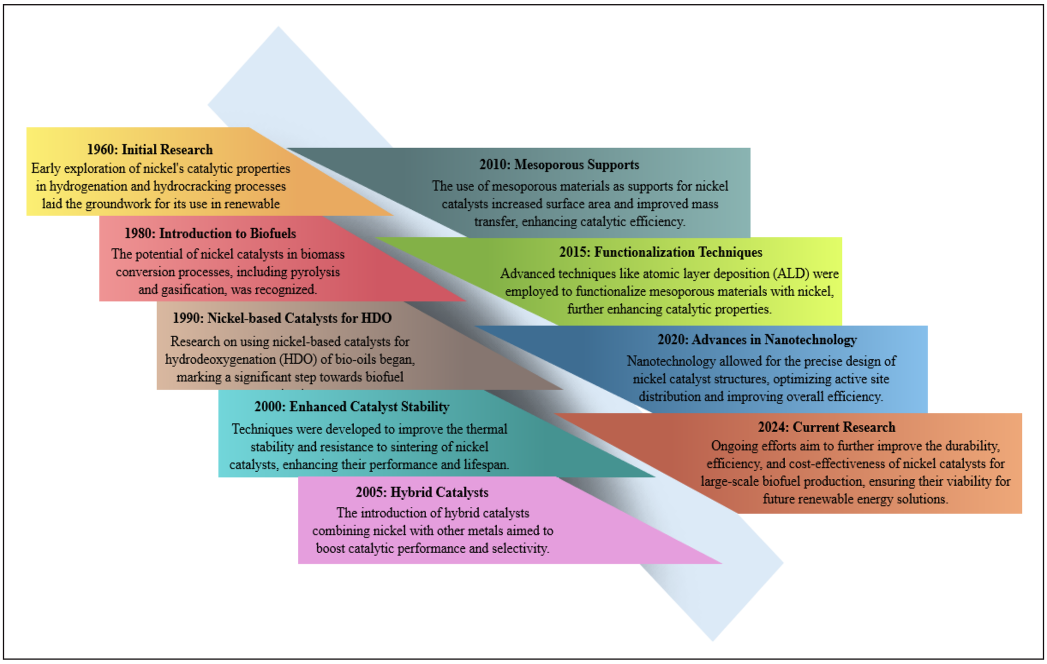


Figure 1. Timeline for the development of noble metal Ni catalysts in renewable energy alternative fuel production

undesired side reactions (Khalit et al., 2021). The ability to optimize activity and selectivity is crucial for efficiently utilizing different types of biomass feedstock. This makes Ni-supported catalysts highly flexible and versatile for various hydrodeoxygenation (HDO) applications (Yang et al., 2023).

Researchers have catalyzed palm fatty acid distillate deoxygenation using NiO/Al-SBA-15. Remarkably, NiO/Al-SBA-15 exhibited superior activity, producing 86% hydrocarbon with a notable preference for diesel fuel (Baharudin et al., 2019). This study strongly demonstrates that the presence of a Ni-supported catalyst significantly enhances the efficiency of oxygen removal. Therefore, the use of non-noble Ni catalysts to remove oxygen from triglycerides and fatty acids shows great potential as a cost-effective catalyst for producing biofuel.

Deoxygenation of Triglycerides and Fatty Acids

Deoxygenation is the simultaneous removal of oxygen by the processes of decarboxylation and decarbonylation of fatty acids, as depicted in Figure 2. This diagram demonstrates that direct decarboxylation removes the carboxyl group from the fatty acid by releasing carbon dioxide molecules, producing n-alkenes. Meanwhile, direct decarbonylation is a process that removes carboxyl groups by eliminating carbon monoxide and water molecules, resulting in the production of alkenes. Deoxygenation typically results in the formation of hydrocarbons with one less carbon atom than their initial fatty acids. Prior publications have extensively explored deoxygenation research involving acidic and basic catalysts applied to a range of fatty acids, triglycerides derived from different vegetable oils, and model oil molecules (Khalit et al., 2021). Most of the reaction conditions occurred within the temperature range of 230°C to 375°C in a hydrogen-rich atmosphere, with pressures ranging from 1 MPa to 11 MPa.

Although there has been a significant amount of research conducted in the past ten years on deoxygenation using environments with low levels of hydrogen and acid-catalyzed reactions, there are a few studies that specifically focus on the development of base and acid-base catalysts for selective deoxygenation in supercritical methanol solvent, which serves as a hydrogen source. This study primarily investigates the process of removing oxygen by using supercritical methanol as a hydrogen source, along with the assistance of base and acid-base catalysts.

Base-catalyzed deoxygenation has been proposed as a method to enhance the hydrolysis of triglycerides, causing them to break down into intermediate fatty acids (Romero et al., 2016). In a study by Tani et al. (2011), scientists used a MgO catalyst in a continuously stirred tank reactor to deoxygenate several bio-based feedstocks. The triglycerides underwent hydrolysis, catalyzed by the water in the feed, resulting in the initial breaking of the C-O bond and the formation of glycerol without fatty

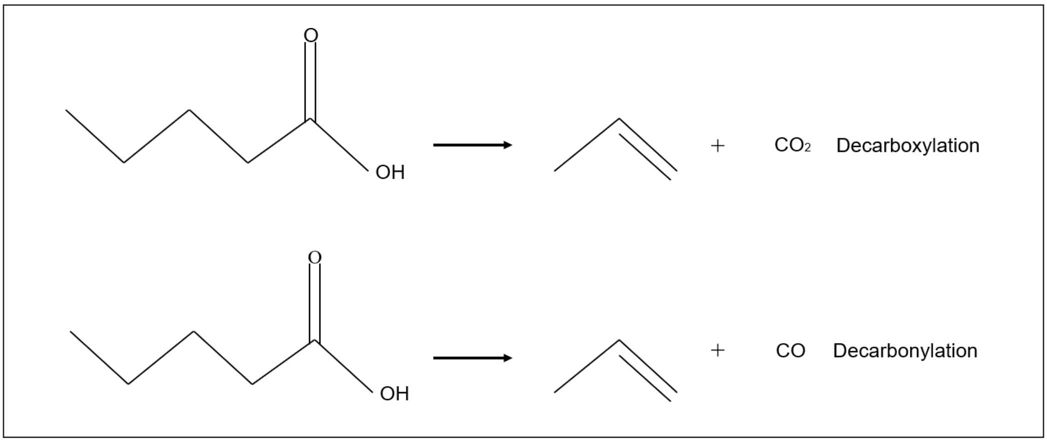


Figure 2. Deoxygenation routes (Bernas et al., 2010)

acids. Gaseous hydrocarbons such as propane are released as glycerol dehydrates, and water is continuously produced. Free fatty acids are generated during decarboxylation/decarbonylation. This is also in agreement with other researchers (Romero et al., 2016). The ß-elimination mechanism is speculated to facilitate triglyceride cleavage during deoxygenation over an acidic or basic catalyst, as shown in Figure 3. This figure represents all four of the main reaction processes that have been suggested for the breakdown of triglycerides. There is a scarcity of reports regarding the processes of ɤ-hydrogen transfer and direct deoxygenation reactions in typical deoxygenation reactions. Direct deoxygenation is a chemical process in which triglycerides decompose into intermediate compounds that are absorbed onto the surface of a catalyst without the formation of fatty acid intermediates. The ɤ-hydrogen transfer mechanism shows potential for generating hydrocarbons with minimal hydrogen usage. However, it is expected to be a more significant reaction mechanism for hydrocracking processes than deoxygenation (Rogers & Zheng, 2016). Ester carbonyl carbons with unique characteristics were detected during the process of removing oxygen from soybean oil using Ni-Mg-Al Layered Double Hydroxides at a temperature of 300°C. It was suggested that it could facilitate these processes in specific conditions by acting as an acid-base neutralizer.

Fatty acids undergo decarboxylation or decarbonylation to produce alkanes or alkenes. Research shows that basic catalysts are more effective for decarboxylation compared to acidic catalysts (Tani et al., 2011). For instance, using Ca(OH)2 and CaO, over 90% of n-C17 was produced from triolein oil deoxygenation CaO (Asikin-Mijan et al., 2016). Studies using MgO for palm oil deoxygenation without hydrogen also showed decarboxylation as the primary process, generating n-C15 and carbon dioxide (Tani et al., 2011). This aligns with research showing that deoxygenating triglycerides involves removing carboxyl groups and producing carbon dioxide and carbon monoxide (Morgan et al., 2012).

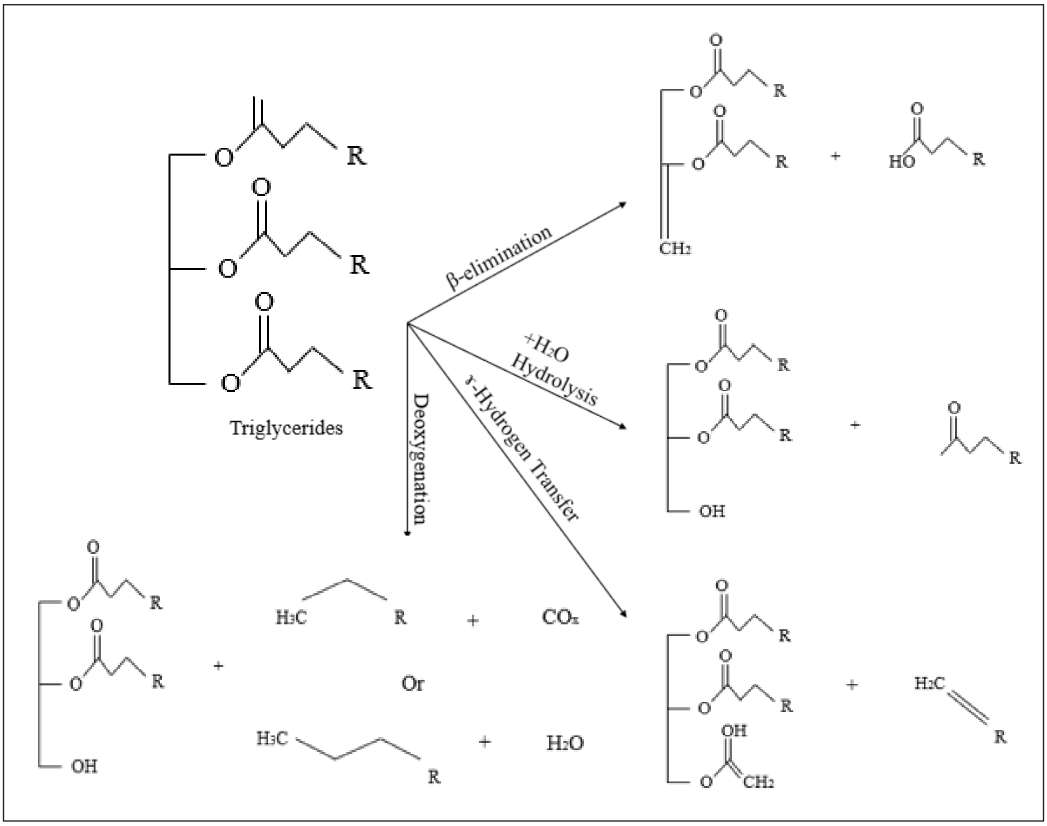


Figure 3. (a) β-elimination and (b) hydrolysis reactions (Rogers & Zheng, 2016)

However, acidic catalysts typically favor decarbonylation. For example, Co/SiO_2 and Co/Hβ catalysts promoted the decarbonylation of triglycerides, resulting in over 90% C_6/C_7 alkanes converting into heptanoic acid (Liu, Bie et al., 2014). In tristearin deoxygenation using acidic catalysts like Pt/C, Pd/C, and Ni/C, decarbonylated carbon monoxide was produced as a byproduct (Morgan et al., 2012).

Triglyceride deoxygenation catalysts fall into two main categories: noble and non-noble metals. Noble metals like platinum (Pt), palladium (Pd), and ruthenium (Ru) are highly effective and selective in deoxygenation processes but are too costly for large-scale use (Ouedraogo & Bhoi, 2020; Roldugina et al., 2018). Non-noble metals, such as nickel (Ni), cobalt (Co), and molybdenum (Mo), are more affordable and commonly used. Nickel, in particular, is known for its high catalytic performance and cost-effectiveness. It is often combined with other metals to improve stability and resistance to deactivation (Ambursa et al., 2021; Yang et al., 2019).

Metal hydrodeoxygenation (HDO) is favored for biofuel production due to its high selectivity in removing oxygen-containing groups while retaining the carbon in the feedstock, resulting in high yields of fuel-grade products (Cheah et al., 2022). Ni-supported catalysts are especially versatile and suitable for various biomass feedstocks, which is crucial for industrial applications where feedstock variability is common (Cheah et al., 2022).

HDO is also more cost-effective for large-scale production because non-noble metals like Ni and Co are much cheaper than noble metals (Li et al., 2018). Coating Ni catalysts with alumina or combining them with molybdenum further enhances their stability and performance (Majewski et al., 2021). Continued research on metal HDO catalysts allows for ongoing improvements, making the process more efficient and scalable (Ruddy et al., 2014). In conclusion, metal HDO, especially with non-noble metals like nickel, is a highly viable method for producing biofuels due to its selectivity, versatility, cost-effectiveness, and stability.

HDO Using Supercritical Methanol

Hydrodeoxygenation (HDO) is a key reaction that removes oxygen from bio-oil, converting it into hydrocarbons (Oyedun et al., 2019). A novel approach uses supercritical methanol as both a solvent and a hydrogen source. When methanol reaches its supercritical state (above 240°C and 8.1 MPa), it exhibits lower viscosity and higher diffusivity, enhancing its ability to penetrate biomass and react with oxygenated compounds in bio-oil (Tomic et al., 2015). Supercritical methanol breaks down into hydrogen (H2) and carbon monoxide (CO), or it directly donates hydrogen to help remove oxygen as water and methanol-derived byproducts, increasing the energy density and stability of bio-oil (Patil et al., 2011).

The use of catalysts, such as Ni, cobalt, and molybdenum, aids in breaking carbon-oxygen bonds during the HDO reaction. Supercritical methanol also serves as a hydrogen donor, reducing the need for external hydrogen sources (Park et al., 2021). This method operates at moderate conditions compared to conventional HDO, which requires higher temperatures and pressures (Ahamed et al., 2021). However, challenges include catalyst deactivation due to coke formation and metal sintering, as well as the need for precise control to prevent unwanted byproducts (Ahamed et al., 2021).

In conventional HDO, external hydrogen is supplied, typically requiring high temperatures (300–400°C) and pressures (up to 10 MPa) (Valle et al., 2019). In contrast, supercritical methanol generates hydrogen in situ, reducing the reliance on external sources and potentially lowering costs and simplifying the process (Shafaghat et al., 2019).

Deoxygenation Catalysts

Catalysts play a crucial part in deoxygenation. Different catalysts produce varying feedstock conversions and product selectivity (Rjeily et al., 2021). Catalysts utilized in deoxygenation can be categorized as homogeneous and heterogeneous, depending on their physical properties, as shown in Figure 4. As depicted in this picture, a homogeneous catalyst is characterized by mixing substrates and catalytic components in the liquid phase throughout a reaction. While homogeneous catalysts enhance the deoxygenation process, they exhibit inferior activity in comparison to heterogeneous catalysts. This is because homogeneous catalysts frequently encounter issues, including poor thermal stability as well as difficulty in recovering and reusing (Britovsek, 2012; Sudarsanam et al., 2018). On the other hand, heterogeneous catalysts, such as Ni-supported catalysts, provide enhanced stability, increased resistance to deactivation, and simplified separation from reaction mixtures, so offering them efficiency for industrial-scale processes (Navalikhina & Krylov, 1998; Ren et al., 2020). The conversion of stearic acid in supercritical water at a temperature of 400°C was found to be only 2 mol percent after a duration of 30 minutes. However, when Sodium hydroxide (NaOH) and Potassium hydroxide (KOH) were used as catalysts, the conversion increased to 13 mol percent and 32 mol percent, respectively (Watanabe et al., 2006). A study was carried out on several homogeneous catalysts, namely Manganese chloride ($MnCl_2$), Zinc Chloride ($ZnCl_2$), Cobalt chloride ($CoCl_2$), Copper sulfate ($CuSO_4$), Magnesium sulfate ($MgSO_4$), Sodium hydroxide (NaOH), and Potassium hydroxide (KOH). The investigation revealed that all of these catalysts enhanced the process of palmitic acid deoxygenation (Fu et al., 2011). After 18 hours at 370°C, sodium hydroxide had the highest deoxygenation activity in the group, producing 7.1 mol percent pentadecane. Their deoxygenation activity, however, remains significantly lower than that of other typical heterogeneous catalysts like Pt/C and Pd/C. Furthermore, homogeneous

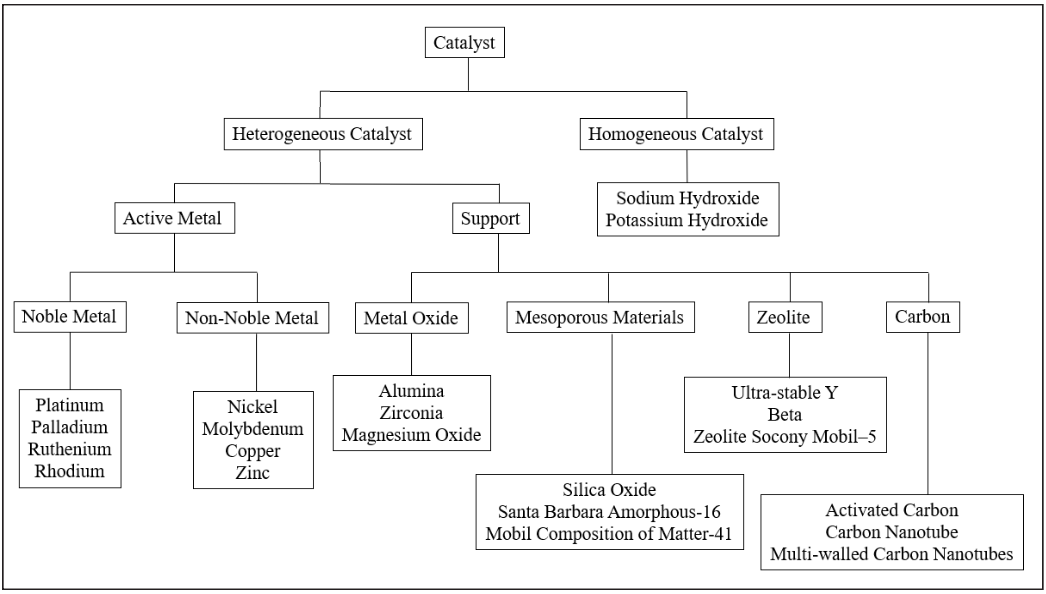


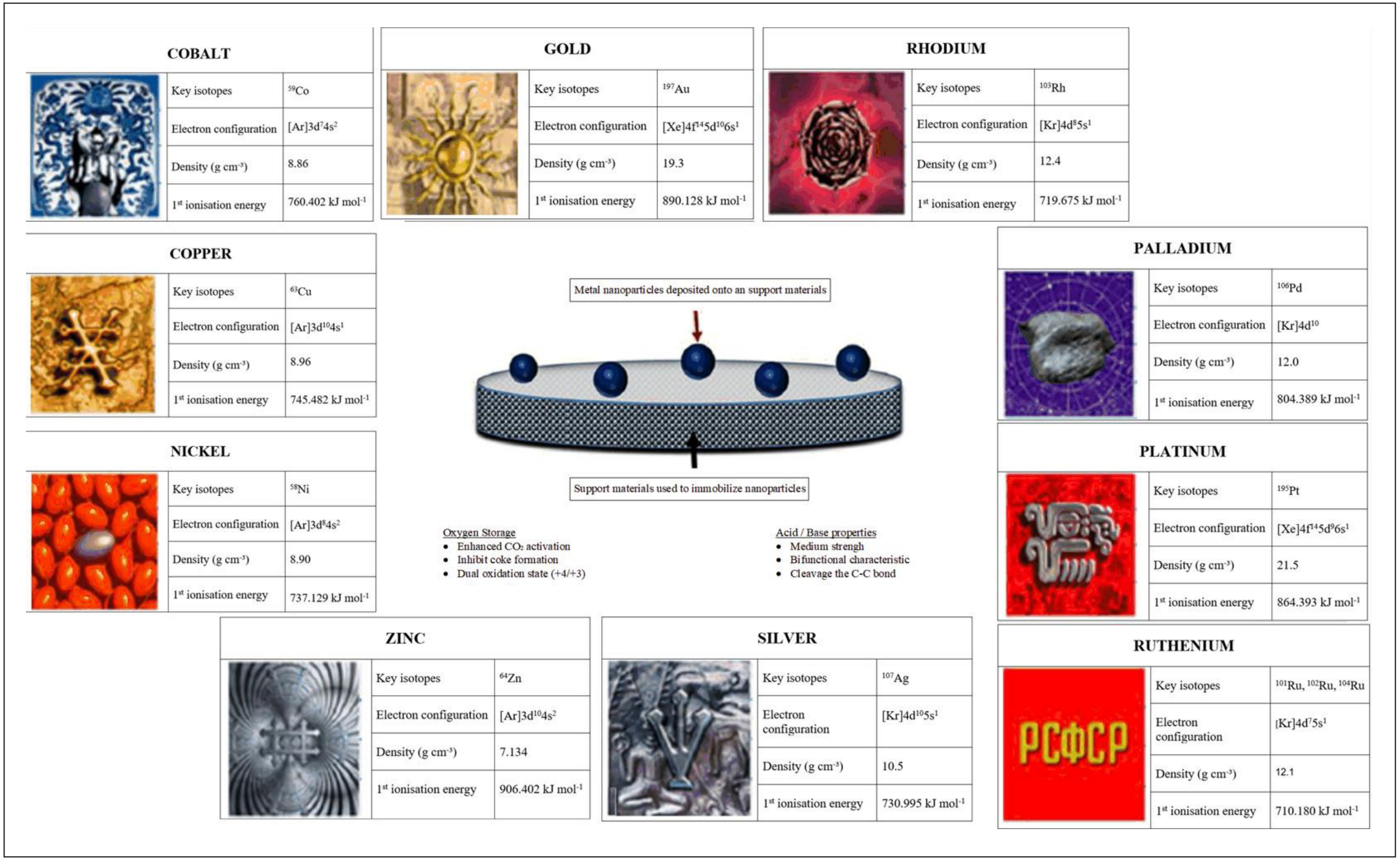
Figure 4. Classification of deoxygenation catalysts

catalysts are difficult to recover from feeds, so their reusability is limited. Heterogeneous catalysts, including metal-based catalysts consisting of support and doped active metals or metallic oxides, are widely developed and used in deoxygenation because of their exceptional performance. The supports used for dispersing and securing active metals or metallic oxides often possess a substantial surface area, and a significant number of them additionally exhibit deoxygenation activity (Ooi et al., 2019). The doped active metals can be categorized as noble and non-noble metals based on their scarcity and value.

Active Metals

The choice of active metal for deoxygenation is crucial as it has a substantial influence on both the quantity and the quality of the final product. Despite the extensive research conducted on deoxygenation, it has been constrained to catalysts. The predominant catalysts in this category are monometallic noble metals, including palladium (Pd), platinum (Pt), rhodium (Rh), and ruthenium (Ru). Figure 5 illustrates the transition metals, specifically iron (Fe), cobalt (Co), Ni, palladium (Pd), and platinum (Pt), which are classified in Groups 8–10 of the periodic chart, are recognized for exhibiting a high activity in hydrodeoxygenation (HDO) processes (Alkhoori et al., 2023; Jin et al., 2019). These transition metals exhibit excellent performance in cleaving the C-O bonds present in oxygenated compounds, which is crucial in the conversion of bio-oil into hydrocarbon fuels. Ni, a metal belonging to Group 10, has been extensively studied due to its cost-effectiveness and high catalytic activity in hydrodeoxygenation (HDO) reactions. Studies have demonstrated that Ni-supported catalysts successfully eliminate oxygen from various feedstocks produced from biomass, resulting in high-quality biofuels (Azman et al., 2021). The metal-support contact, which serves as the surface reaction mechanism, can strengthen the interaction between the support and the metal. The active metal nanoparticles are placed onto a support material to immobilize them. Table 1 displays the benefits and drawbacks of the active metals often employed for removing oxygen from triglycerides and fatty acids. From Table 1, platinum (Pt) and palladium (Pd) are very costly but show high activity and efficacy in hydrogen activation, favor carbon-carbon bond cleavage and are highly reactive in decarboxylation and decarbonylation reactions. Iridium (Ir) and rhodium (Rh) are also costly but give high selectivity in producing symmetrical ketones with poor selectivity toward deoxygenation and favor decarbonylation, respectively. Meanwhile, ruthenium (Ru) favors hydrodeoxygenation and possesses high selectivity in producing symmetrical ketones but has poor Ru/SiO_2 performance in producing deoxygenation products.

Remarkably, the deoxygenation activity was highest when Ni was used as a promoter, resulting in diesel selectivity of 80% (Hafriz et al., 2020). Ni catalysts possess numerous advantages over other non-precious metals employed in the production of biofuels, as shown in Figure 6. Ni catalysts' high activity and selectivity in cleaving carbon-oxygen



COBALT

Key isotopes	^{59}Co
Electron configuration	[Ar]3d^{7}4s^2
Density (g cm^{-3})	8.86
1st ionisation energy	760.402 kJ mol^{-1}

GOLD

Key isotopes	^{197}Au
Electron configuration	[Xe]4f^{14}5d^{10}6s^1
Density (g cm^{-3})	19.3
1st ionisation energy	890.128 kJ mol^{-1}

RHODIUM

Key isotopes	^{103}Rh
Electron configuration	[Kr]4d^{8}5s^1
Density (g cm^{-3})	12.4
1st ionisation energy	719.675 kJ mol^{-1}

COPPER

Key isotopes	^{63}Cu
Electron configuration	[Ar]3d^{10}4s^1
Density (g cm^{-3})	8.96
1st ionisation energy	745.482 kJ mol^{-1}

PALLADIUM

Key isotopes	^{106}Pd
Electron configuration	[Kr]4d^{10}
Density (g cm^{-3})	12.0
1st ionisation energy	804.389 kJ mol^{-1}

NICKEL

Key isotopes	^{58}Ni
Electron configuration	[Ar]3d^{8}4s^2
Density (g cm^{-3})	8.90
1st ionisation energy	737.129 kJ mol^{-1}

PLATINUM

Key isotopes	^{195}Pt
Electron configuration	[Xe]4f^{14}5d^{9}6s^1
Density (g cm^{-3})	21.5
1st ionisation energy	864.393 kJ mol^{-1}

ZINC

Key isotopes	^{64}Zn
Electron configuration	[Ar]3d^{10}4s^2
Density (g cm^{-3})	7.134
1st ionisation energy	906.402 kJ mol^{-1}

SILVER

Key isotopes	^{107}Ag
Electron configuration	[Kr]4d^{10}5s^1
Density (g cm^{-3})	10.5
1st ionisation energy	730.995 kJ mol^{-1}

RUTHENIUM

Key isotopes	^{101}Ru, ^{102}Ru, ^{104}Ru
Electron configuration	[Kr]4d^{7}5s^1
Density (g cm^{-3})	12.1
1st ionisation energy	710.180 kJ mol^{-1}

Figure 5. Active metals for fatty acid and triglyceride deoxygenation (Cheah et al., 2022)

Table 1

Main roles and limitations of reactive metals employed in deoxygenation studies (Cheah et al., 2022)

Elements	**Main roles**	**Limitations**	**References**
Platinum (Pt)	☐ High activity and efficacy in hydrogen activation ☐ Favors carbon-carbon bond cleavage ☐ Highly reactive in decarboxylation and decarbonylation	☐ Costly ☐ Gradually poisoned by carbon monoxide and sulfur-based functional groups and carbon laydown	Fu et al., 2011
Palladium (Pd)	☐ High activity and efficacy in hydrogen activation ☐ Favors carbon-carbon bond cleavage ☐ Highly reactive in decarboxylation and decarbonylation	☐ Costly ☐ Gradually poisoned by carbon monoxide and sulfur-based functional groups and carbon laydown	Cheah et al., 2022
Iridium (Ir)	☐ High selectivity in producing symmetrical ketones	☐ Costly ☐ Poor selectivity toward deoxygenation	Di et al., 2017
Rhodium (Rh)	☐ Favors decarbonylation	☐ Costly	Di et al., 2017
Ruthenium (Ru)	☐ Favors hydrodeoxygenation ☐ High selectivity in producing symmetrical ketones	☐ Poor Ru/SiO_2 performance in producing deoxygenation products	Di et al., 2017

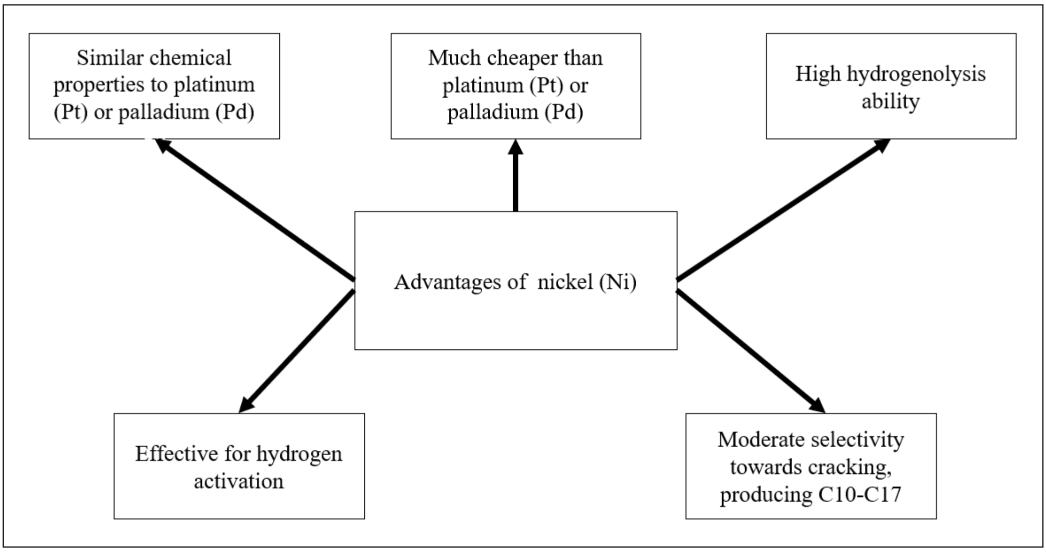


Figure 6. Advantages of non-noble Ni in biofuel production

(C-O) bonds in biomass-derived compounds is one of their most significant advantages. As a transition metal, Ni possesses exceptional catalytic characteristics for hydrogenation processes, which facilitate the effective elimination of oxygen and the conversion of bio-oil into valuable hydrocarbons (Ambursa et al., 2021; Yang et al., 2019). In addition, Ni catalysts are comparatively more cost-effective than noble metal catalysts like platinum

(Pt) and palladium (Pd), making them economically viable to produce biofuels on a wide scale.

Furthermore, Ni catalysts exhibit strong stability in the reaction conditions commonly used in HDO. They possess the ability to sustain their catalytic activity for extended durations and endure the elevated temperatures and pressures needed for optimal deoxygenation. This stability is important in continuous industrial operations, as catalyst lifespan has a direct impact on operational costs and efficiency (Shafaghat et al., 2019). In addition, Ni catalysts are versatile and can be used in various applications, such as supported Ni catalysts, Ni-supported alloys, and Ni on mesoporous materials (Khalit et al., 2021). This adaptability allows for the tailoring of catalytic characteristics to fulfill the specific requirements of a reaction and optimize performance. Moreover, Ni catalysts can perform in a variety of reaction environments, such as supercritical methanol. Ni catalysts are efficient in promoting the cleavage of methanol into hydrogen and carbon monoxide, which makes them valuable in operations where hydrogen is produced in situ. This enhances the overall efficiency of the HDO process as Ni catalysts can effectively promote hydrogenation reactions without the need for external hydrogen supplies (Shafaghat et al., 2019).

Progress in catalyst design has resulted in the development of bimetallic and multifunctional Ni catalysts that integrate the advantages of Ni with other metals or catalytic functions. For example, nickel-molybdenum (Ni-Mo) and nickel-cobalt (Ni-Co) catalysts have improved catalytic performance due to metal synergistic effects. These catalysts can enhance the efficiency of chemical reactions, the ability to selectively produce desired products, and the durability against deactivation caused by the creation of coke (Azman et al., 2021).

In conclusion, Ni catalysts offer numerous advantages in biofuel production. These include their exceptional catalytic activity, cost-effectiveness, ability to remain stable under reaction environments, versatility in form and function and compatibility with supercritical methanol environments. The presence of these qualities makes Ni catalysts a highly efficient and economically feasible choice for improving the efficiency and sustainability of the biofuel production process (Khalit et al., 2021).

Ni Loading

The presence of Ni has a substantial impact on both the speed of the reaction and the degree of selectivity or production of diesel-like hydrocarbons during deoxygenation (Hermida et al., 2015). Table 2 displays new research findings about the impact of Ni loading on the process of removing oxygen from vegetable oils and fatty acids. Liu, Zuo et al. (2014) conducted an experiment to investigate the deoxygenation of palm oil using a Ni/SAPO11 catalyst. The experiment involved adjusting the Ni loading from 2 wt.% to 9 wt.% and maintaining a temperature of 200°C and a hydrogen (H_2) pressure of 4 MPa.

The study's findings indicated that the Ni loading was augmenting from 2 wt.% to 7 wt.% led to an elevation in the production of liquid alkane and an enhancement in the selectivity of isomerization, with respective increases from 60% to 67.4% and from 46% to 61.5%. The absence of any significant impact on the production of liquid alkane was observed upon increasing Ni loading from 7% to 9 wt.%. Nevertheless, a notable enhancement in the selectivity of C1-C14 alkanes was observed. An isomerization selectivity of over 83% was achieved using 9 wt.% of Ni/SAPO-11.

Modifications in catalyst structures caused by metal loading can potentially impact catalytic activity. The influence of metal loading on deoxygenation processes is intimately related to various elements, including the preparation method, characteristics of support, and the metal's chemical state. The method of preparation has significant effects on the distribution and size of metal particles on the catalyst surface, which affects catalytic performance. Impregnation, co-precipitation, and sol-gel techniques can produce varied particle sizes and dispersion, which affects the accessibility of active sites for deoxygenation reaction (Li et al., 2015).

The stability of the catalyst and the dispersion of metal particles are significantly influenced by the characteristics of the support, including surface area, porosity, and the chemical composition of the support material (Liu, Bie et al., 2014). The catalytic activity is also influenced by the chemical state of the metal, which includes its oxidation state and the nature of its interaction with the support. The oxidation state can affect the metal's ability to activate and dissociate reactants, therefore influencing the efficiency of deoxygenation. The efficiency of metal-loaded catalysts in deoxygenation processes is determined by a combination of these parameters, emphasizing the significance of optimizing methods of preparation, carrier characteristics, and metal chemical states to attain desired catalytic performance (Wang et al., 2020).

According to the literature, Ni/HZSM-5 catalyst with a Ni loading of 7 wt.% demonstrated the best performance. The conversion rate for methyl hexadecanoate was 90%, and the selectivity for C5-C16 hydrocarbon was 83%. The observed phenomenon can be attributed to the synergistic interaction between the Ni metal and the HZSM-5 acid sites. Nevertheless, the concentration of NiO on the HZSM-5 surface was discovered to be significantly elevated and only became discernible for Ni loadings below 3 wt.%. In addition, the size of the NiO particles grew to 150 nm, and the catalyst surface experienced substantial agglomeration as the Ni loading reached 7 wt.% (Japar et al., 2020; Liu, Bie, et al., 2014). Hence, it is imperative to ascertain the ideal metal loading on the catalyst support to ensure that the catalyst's structure is conducive to catalyzing triglyceride deoxygenation.

Shi et al. (2023) studied the effect of the Ni loading amounts (2.5%–20%) on green diesel-like hydrocarbon production by H_2-free catalytic deoxygenation of oleic acid via Ni/MgO-Al_2O_3 catalysts. They revealed that Ni (10%)/MgO-Al_2O_3 catalysts exhibited

Table 2
Recent studies on the effects of Ni loading on the deoxygenation of fatty acids and vegetable oils

References	**Type of reactor**	**Fatty acid/ vegetable oil**	**Catalysts**	**Method of catalyst preparation**	**Reaction conditions**	**Conversion (%)**	**Hydrocarbon yield/selectivity (%)**
Zhou et al. (2021)	Fixed bed	*Jatropha curcas* oil	2Ni10W/AC 10Ni10W/AC 20Ni10W/AC	Carbothermal hydrogen reduction	T = 340°C P = 3 MPa RT = 5 h WHSV = 55.2 h^{-1} H_2/N_2 = 1 FR = 200 mL/min	-	10Ni10W/AC showed excellent performance with 94.5% of C15-C18
Maneechakr & Karnjanakom (2021)	Fixed bed	Waste palm kernel cake	5Ni/AC 10Ni/AC 15Ni/AC 20Ni/AC	Impregnation and pyrolysis	T = 550°C HR = 1000 °C/min Ultrasonic pretreatment time = 200 min	-	15Ni/AC demonstrated the best performance with 72% of hydrocarbon
Ameen et al. (2020)	Fixed bed	Rubber seed oil	3Ni/γ-Al_2O_3 12Ni/γ-Al_2O_3 15Ni/γ-Al_2O_3	Impregnation	T = 350°C P = 3.5 MPa WHSV = 1 h^{-1} H_2:oil ratio = 1000 N (cm^3/cm^3)	99.9	15Ni/γ-Al_2O_3 produced the highest hydrocarbon selectivity of 55.1 wt.%
Chen et al. (2019)	Fixed bed	Methyl laurate	6Ni/γ-Al_2O_3 8Ni/γ-Al_2O_3 10Ni/γ-Al_2O_3 12Ni/γ-Al_2O_3 14Ni/γ-Al_2O_3	Impregnation	T = 400°C P = 2 MPa FR = 50 mL/min	91.1	10Ni/γ-Al_2O_3 showed the best performance with 68.6% of C11
Sánchez-Cárdenas et al. (2016)	Batch	Oleic acid	3Ni/γ-Al_2O_3 5Ni/γ-Al_2O_3 7Ni/γ-Al_2O_3	Impregnation	T = 340°C P = 2.3 MPa H_2 RT = 5 h	70.1 79.4 84.8	7Ni/γ-Al_2O_3 gave the best performance with 69% of C17

*T=Temperature, P=Pressure, RT= Reaction Time, FR= Flow rate, HR=Heating rate, WHSV= Weight hourly space velocity, W= Tungsten, AC=Activated Carbon, Ni=Nickel excellent catalytic performance with the highest HCs (95.12%), olefin content (57.37%) and green biodiesel content (75.90%) due to mild Brønsted-rich acidity sites, lower reduction temperature, higher active metallic Ni species content, suitable oxygen vacancy content and favorable texture properties via the decarbonylation reaction pathway. Yang et al. (2022) investigated a one-step synthesis of highly active and stable Ni-ZrO_2 catalysts for the conversion of methyl laurate to alkanes with varied Ni loading of 5.28 to 16.15%. They found that 10Ni-ZrO_2 exhibits the best catalytic performance for obtaining alkane products with nearly 100% yield (99.4%), and the selectivity to C11 is as high as 87.6%. Jeon et al. (2022) studied the effect of Ni loading amount (5 to 30%) over Ni/MgO–Al_2O_3 on deoxygenation of non-edible fatty acid for green diesel production. They found that the 20% Ni/MG70 catalyst showed excellent deoxygenation performance owing to its highest number of Ni active sites, easier reducibility, and appropriate acidity. Khalit et al. (2021) synthesized and characterized Ni-supported activated charcoal using different loading of Ni (5 to 20%) supported on commercial activated charcoal. They revealed that the highest conversion of hydrocarbon was achieved when the reaction undergoes using Ni20%AC as a catalyst. The presence of the higher-loading active metal showed a high deoxygenation reaction by decarboxylation and decarbonylation pathways with a high hydrocarbon yield of 83% and high selectivity of n-C15 and nC17.

Catalyst Supports

The selection of catalyst support is vital in developing a catalyst as it influences the type of reaction pathway used, product distribution, and overall deoxygenation performance. Prior studies have determined the effect of the structural characteristics of catalyst supports on the interaction bonding between the active site and the support and the degree of active site dispersion on the support surface. Figure 7 depicts the metal oxides and mesoporous materials that researchers frequently use as catalyst support for deoxygenation. These materials are known for their exceptional heat and chemical resistance, making them suitable for use in demanding working circumstances. This diagram illustrates that catalyst support has an impact on the choice of reaction pathway, the distribution of products, and the total deoxygenation activity. The metal oxide has a relatively high specific surface area ranging from 200 m^2/g to 500 m^2/g. It also has high physical strength and is resistant to thermal and chemical degradation. Additionally, it can activate oxygenated compounds and has excellent oxygen storage capacity with intrinsic redox properties. However, their lower surface area with lesser uniform pore structure could restrict the availability and accessibility of active sites (Mortensen et al., 2011).

On the other hand, mesoporous materials have a high specific surface area ranging from 600 m^2 to 1000 m^2. They have large pore diameters ranging from 2 to more than 10 nm and moderate acidity. Furthermore, they possess a broad distribution of weak protonic

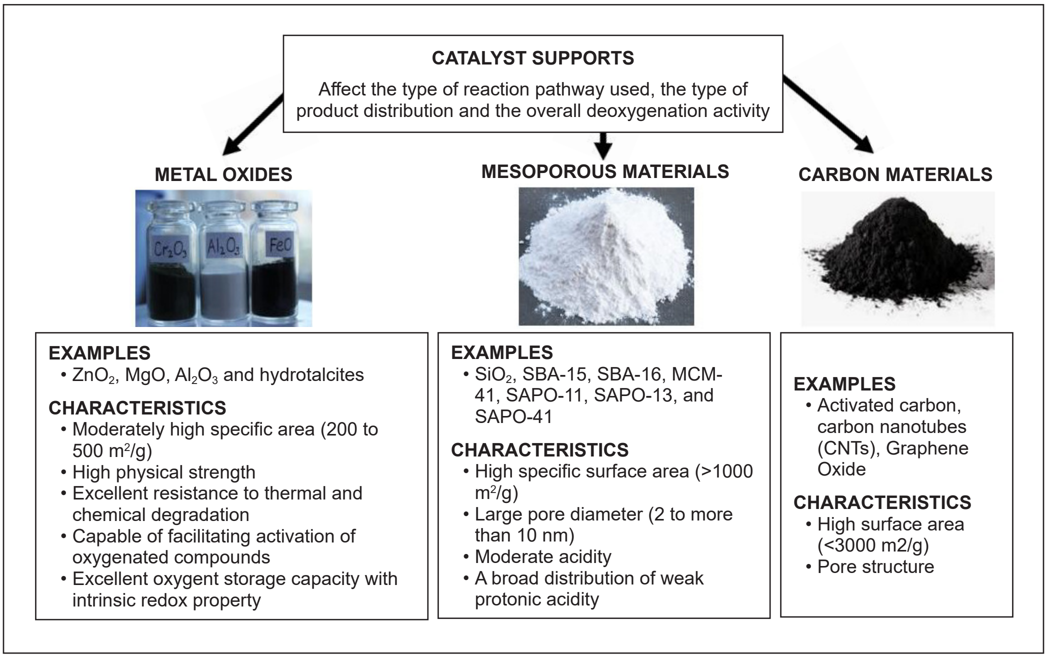


Figure 7. Catalyst supports that are frequently used in deoxygenation

acidity sites. These materials also have variable pore diameters and surface features that can be optimized for specific reactions (Corma, 2003). Nevertheless, mesoporous materials frequently have poorer thermal stability and mechanical strength than metal oxides, which can limit their use in harsh reaction conditions (Ren et al., 2012).

Studies on catalysts loaded with Ni on carbon support also have demonstrated excellent results in the production of biofuels, indicating their ability to improve catalytic performance and stability. Carbon catalysts impregnated with Ni are highly efficient in hydrodeoxygenation (HDO) processes, which are crucial for enhancing the quality of bio-oils to produce higher-quality biofuels. Carbon support possesses a large surface area and porous structure, assisting in the distribution of Ni particles and enhancing the accessibility of active sites. This structural advantage results in enhanced catalytic performance and greater conversion rates in biofuel production processes (Khalit et al., 2021). Despite that, carbon-supported catalysts face significant challenges, especially in terms of stability and durability, resulting in decreased catalytic activity (Zaman et al., 2022).

Future research on HDO catalyst development needs to focus on the utilization of hybrid supports that integrate the advantageous properties of the supports. For example, the integration of alumina and mesoporous silica in composite materials can take advantage of the heat resistance of metal oxides and the large surface area of mesoporous materials, leading to enhanced catalyst performance (Kankala et al., 2019) Furthermore, integrating mesoporous materials with metal oxides using methods such as atomic layer deposition might result in synergistic effects that improve catalytic capabilities and resistance to deactivation (Kankala et al., 2019). Nanotechnology advancements also provide the opportunity to develop catalyst structures precisely, optimizing the distribution of active sites and enhancing mass transfer characteristics, resulting in more efficient and durable catalysts. Integrating these materials in unique ways makes it possible to produce highly efficient, stable, and cost-effective HDO catalysts for biofuel production.

Table 3 summarizes recent studies on the effect of catalyst supports on the oxygen removal process from vegetable oils and fatty acids. In one study, rapeseed oil was treated using various supports—Silica oxide (SiO_2), Titanium dioxide (TiO_2), and Alumina (Al_2O_3)—saturated with 3.3 wt.% Ni and 15 wt.% molybdenum (Mo) at 260–300°C and 3.5 MPa in a fixed-bed reactor (Kubička et al., 2014). The researchers found that the physical properties of the supports, such as size, dispersion, and reducibility of the active

Table 3
Recent studies on the effects of catalyst-support on the deoxygenation of fatty acids and vegetable oils

References	Reactor type	Fatty acid/ vegetable oil	Catalyst	Reaction conditions	Findings
Khalit et al. (2021)	Semi-batch	Waste cooking oil	Ni/AC Ni/rGO Ni/Zeo	T = 350°C RT = 3 h	Ni/AC showed the best performance with 90% of hydrocarbon yield and 89% of n-(C15 + C17)
de Oliveira Camargo et al. (2020)	Batch	Oleic acid	Ni_2P/USY Ni_2P/H-ZSM-5 Ni_2P/Al-SBA-15	T = 300°C P = 5 MPa RT = 6 h	Ni_2P/Al-SBA-15 achieved the best performance with 42% of hydrocarbon (C10-C18)
Wang et al. (2020)	Reactor	Spirulina	NiO/SAPO-34 NiO/ZSM-5 NiO/USY NiO/γ-Al_2O_3 NiO/SiO_2	T = 270°C RT = 30 min	NiO/USY contributed to the best performance with 20.6% of hydrocarbon
Lee et al. (2020)	Semi-batch	Methyl palmitate	Ni/SiO_2 Ni-Al/SBA-15 Ni-Al/SBA-16 Ni-Al/KIT-6	T =280°C P =2 MPa RT = 2 h	Ni/Al-SBA-15 showed the highest selectivity of 77.7% of hydrocarbon
Liu et al. (2019)	Batch	Palmitic acid	Co/SiO_2 Co/γ-Al_2O_3 Co/H-ZSM-22	T = 260°C P = 2 MPa H_2 RT = 4 h	Co/HZP-22 gave the highest yield with 43.1% of n-C16
Raut et al. (2016)	Batch	Oleic acid, stearic acid, JCS oil	Pd/SBA-12 Pd/SBA-16	T = 300°C –325°C P = 0.5 MPa–3 MPa RT = 5 h	Pd/SBA-16 showed high catalytic performance

*T=Temperature, P=Pressure, RT= Reaction Time, Ni=Nickel, AC=Activated carbon, rGO= reduced graphene oxide, Zeo=zeolite, P, USY= ultrastable Y zeolite, SBA= Santa Barbara Amorphous, SAPO= Silicoaluminophosphate zeolite, ZSM-5= Zeolite Socony Mobil-5, SiO_2=silica oxide, KIT= Korea Advanced Institute of Science and Technology, JCS=*Jatropha curcas* seeds sites, significantly impacted product selectivity. Similar findings were reported by Chen et al. (2015), showing that product selectivity depends on the texture of the support, the interaction between active sites and support, and the strength of metal-support bonding.

Metal Oxides

Metal oxides are affordable and commonly used as catalysts and support materials for deoxygenating triglycerides and fatty acids (Tani et al., 2011). Their surface contains oxygen vacancies, which help activate fatty acids and aid in removing the carboxyl group, enhancing the catalytic reduction of oxygenated compounds. Studies have highlighted the potential of solid base materials as supports for oleic acid deoxygenation (Li et al., 2015). A series of Ni/MO-Al_2O_3 catalysts (with M representing metals like Mg, Ca, Ni, Cu, and Zn) were tested, and Ni/ZnO-Al_2O_3 was found to be the most effective for converting mono-unsaturated fatty acids into n-alkanes, due to its moderate base strength (Li et al., 2015). The optimal Zn/Al ratio improved Ni particle distribution, maximizing alkane production at 95.8% by preventing particle clumping (Chen et al., 2017).

CeO_2-based materials, known for their excellent oxygen storage capacity, were further enhanced by adding ZrO_2, improving thermal stability and reducibility (Deng et al., 2019). A Ce0.6Zr0.4O2 support achieved a 73.8% conversion rate and high decarbonylation selectivity in oleic acid deoxygenation (Shim et al., 2014). Basic supports perform poorly below 350°C (Arun et al., 2015), but Pt-WOx/Al2O3 showed complete conversion of oleic acid, with C18 selectivity increasing from 67.1% to 80.8% through hydrodeoxygenation and C17 formation via decarboxylation (Janampelli & Darbha, 2018).

Mesoporous Materials

Mesoporous materials have attracted significant attention for their large pore size, high specific surface area (600–1000 m²/g), and moderate acidity, which make them well-suited for use as high-performance deoxygenation catalysts. These materials exhibit a uniform pore size distribution (1.5–10 nm), which provides a greater number of active sites throughout the porous network. The wide range of pore sizes also facilitates access to these sites by larger organic molecules, making them more efficient for certain reactions (da Silva et al., 2019). SBA supports, particularly SBA-16, are favored for their stability and ability to accommodate large molecules due to their thicker walls and interconnected mesoporous structures, in contrast to the less stable MCM-41 (Arun et al., 2015). However, these materials often face challenges with structural instability under harsh hydrothermal conditions, leading to a steady reduction in surface area and the collapse of internal pore structures due to high temperatures and pressures, particularly when water is present.

SAPO molecular sieves, including SAPO-11, SAPO-31, and SAPO-41, are commonly used as selective catalysts for both isomerization and deoxygenation processes due to their unique hierarchical mesoporous structures. SAPO materials are noted for their high isomerization-to-cracking ratios, thanks to their weak protonic acidity located at the boundary between silica and the SAPO phase (Rabaev et al., 2015). SAPO-11 features 10-membered elliptical rings, while SAPO-31 has circular channels, both of which contribute to their efficiency in producing multibranched alkanes. SAPO-41, with slightly larger elliptical pores, also shows promise in these reactions (Zhang et al., 2017). However, SAPO-11 has been found to have limited hydrothermal stability, degrading when exposed to high temperatures and pressures above 200°C and 1.7 MPa, making it unsuitable for extended hydrodeoxygenation reactions (Herskowitz et al., 2013).

The impact of acidic support on reactions such as cracking, isomerization, and cyclization has been extensively studied. Supports like Pt/SAPO-11 and platinum-chlorinated alumina enhance catalytic performance by increasing the number of active sites and surface area, but they are prone to issues like carbon deposition, which can reduce catalytic efficiency over time. Studies have shown that under acidic conditions, paraffin intermediates formed during decarboxylation can undergo cyclization and isomerization, producing cyclic, branched, and alkyl aromatic hydrocarbons (Ahmadi et al., 2014). Despite the benefits, SAPO materials face challenges with stability, particularly under hydrothermal conditions, limiting their use in prolonged catalytic processes (Lutz et al., 2010).

Further research into mesoporous materials as catalyst supports has explored their performance in the removal of oxygen from compounds like guaiacol, with experiments using Ni-Re/SiO_2, Ni-Re/TiO_2, and Ni-Re/ZrO_2 under varying conditions. Ni-Re/SiO_2 showed greater selectivity for cyclohexane, although its performance in deoxygenation was lower than that of other supports (Herskowitz et al., 2013). Similar studies using palm oil and catalysts such as Ni/Al_2O_3, Ni/ZrO_2, and Ni/SiO_2 found that SiO_2-supported catalysts were less selective towards C15-C18 products. These findings suggest that while mesoporous materials offer large surface areas and moderate acidity, their catalytic efficiency in deoxygenation processes can be limited compared to metal oxide supports, which offer higher acidity and better stability under reaction conditions (Herskowitz et al., 2013).

Carbon Material

Recent advancements in deoxygenation catalysts have seen significant progress using carbon materials. Various metal catalysts supported on carbon, TiO_2, SiO_2, ZrO_2, and Al_2O_3 have been developed to aid fatty acid deoxygenation. Noble metal catalysts, such as Pd, exhibit excellent performance due to their unique electron structures. For instance, Pd/C achieved a 96% yield of n-alkanes from stearic acid in just 30 minutes (Ford et al., 2012). The support material significantly influences performance, with Pd/C showing a much higher decarboxylation rate than Pd/SiO_2 or Pd/Al_2O_3, likely due to carbon's strong adsorption capacity for fatty acids (Giordano et al., 2016). Noble metals like Pt, Ru, and

Rh on carbon supports also performed well, with Pd and Pt favoring decarboxylation and decarbonylation, while Ru was more effective in hydrodeoxygenation (Giordano et al., 2016).

Nickel-loaded carbon catalysts have also proven effective in biofuel production. For example, 15% Ni-loaded activated carbon yielded 72% hydrocarbons from palm kernel cake pyrolysis (Chen et al., 2023), and Ni/bio-AC achieved high conversion rates in fatty acid pyrolysis to biodiesel (Chen et al., 2023). Ni-Cu/C catalysts also demonstrated impressive results, producing 91% 2-methylfuran from furfural and 80% 2,5-dimethylfuran from 5-hydroxymethylfurfural under similar conditions (Fu et al., 2017).

Effect of Catalyst Loading on Triglyceride Deoxygenation

Catalyst loading plays a crucial role in chemical reactions by enhancing reaction rates, catalytic performance, activity, and stability (Gousi et al., 2020). A moderate excess of catalyst is typically recommended to account for possible poisoning from contaminants and carbon deposition (Kwon et al., 2011). However, excessive catalyst loading can increase cracking, leading to the formation of undesirable short hydrocarbons (Jeon et al., 2022).

Experiments using catalyst loadings of 1% to 9% showed that loadings between 1% and 5% improved deoxygenation efficiency due to a larger active surface area (Kwon et al., 2011). A 5% catalyst loading resulted in the highest % green diesel yield of 80% (Asikin-Mijan et al., 2020). However, loadings beyond 5% reduced the yield due to potential polymerization reactions (Kwon et al., 2011).

Further studies confirmed that 5% catalyst loading was optimal for deoxygenation of JCO into diesel-grade biofuel, with yields between 78% and 95% (Asikin-Mijan et al., 2020). Higher loadings, however, did not improve performance and led to side reactions such as CC-cleavage and the production of short hydrocarbons (Asikin-Mijan et al., 2020). Overall, 3% to 5% catalyst loading is optimal for deoxygenating triglycerides and fatty acids, providing high deoxygenation activity without excessive byproduct formation. Catalyst loadings beyond 5% tend to cause undesirable polymerization (Asikin-Mijan et al., 2020).

CONCLUSION

This paper highlights the strong potential of Ni-supported catalysts for producing high-quality biofuel through deoxygenation, thanks to their excellent catalytic properties and cost-effectiveness. However, issues such as fouling, sintering, and poisoning, as shown in Figure 8, can lead to catalyst deactivation. Therefore, developing new, durable, and affordable bifunctional catalysts with superior deoxygenation performance is essential. In summary, deoxygenation technology offers a practical solution for global biofuel production, supporting sustainable development goals 7 and 13, which focus on clean energy access and addressing climate change.

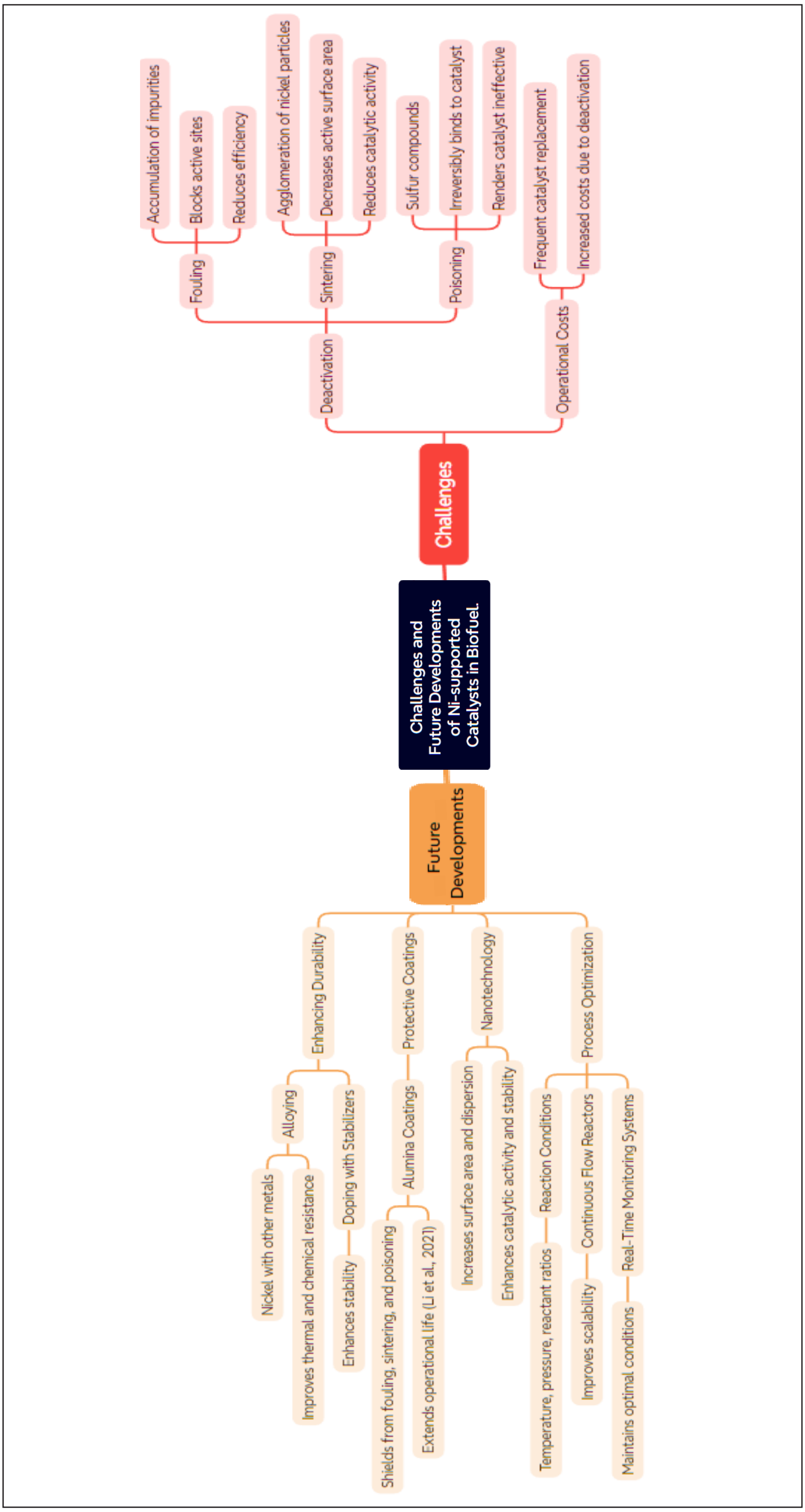


Figure 8. Challenges and future developments of Ni-supported catalysts in biofuel

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