

Review

Microwave-Absorbing Catalysts in Catalytic Reactions of Biofuel Production

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Abstract: Catalytic reactions in producing biofuels often face issues such as low product yield, low selectivity to preferred products and serious environmental issues which leads to the exploration of green technologies. Microwave technology is one of the green technologies that is widely applied in the field such as medical, food, signal processing or navigation, and has been reviewed for its potential in the catalytic reactions for biofuel production. With the application of microwave technology, its unique heating mechanism consists of magnetic field energy and electric field energy that enables the selective heating of materials, allowing rapid reaction and enhancement of catalytic performance of catalysts. In general, this review has discussed on the fundamentals and mechanisms of microwave technology with an in-depth discussion on the application of microwave-absorbing catalysts for biofuel production, especially in ammonia synthesis, bio-oil and 5-HMF production as well as methanation. Lastly, the challenges and future prospect of microwave-absorbing catalysts are included as well.

Keywords: microwave-absorbing; microwave technology; catalytic reactions; green technology; catalysts synthesis; bioenergy



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1. Introduction

The application of green technology has been widely studied and investigated in recent years, especially in biofuel production. The environmental issues, unsatisfactory conversion rate and product yield, as well as the rapid depletion of non-renewable sources have urged the society to seek a sustainable solution. Biofuel as one of the substitutes for non-renewable energy often involved the application of catalysts to optimize various chemical processes. Catalysts can be categorized into heterogeneous and homogeneous catalysts. Heterogeneous catalysts were focused on in this review for their inexpensive operational cost, high robustness and the convenience of separating the catalysts from the products. Numerous studies have reported the optimum performance of such catalysts and they are commercialized in industries. With the intention of improving catalytic reaction conditions, the research direction has shown interest in exploring non-conventional techniques like green technologies such as microwave, non-thermal plasma, photocatalysis and ultrasonication to study their mechanisms due to the advantages over conventional technologies.

Microwave heating has attracted the attention of society due to its different heating mechanisms compared to conventional heating, and is a useful tool across industries like food, pharmaceuticals, analytical chemistry, material synthesis and agriculture. The subjected material can be heated directly via microwave heating because of the unique mechanism of microwave heating, enabling them to interact with the targeted material

at a molecular level through an electromagnetic field [1]. One of the advantages is that microwave heating allows negligible convective or conductive heat losses as compared to conventional heating. The efficient heating shows that microwave is energy-saving as the volumetric heating of materials performed requires a relatively short processing time than conventional heating.

With the outstanding benefit of microwave heating, the active sites of catalysts or entire catalysts can be heated selectively to enhance the catalytic performance. In the case of microwave-unabsorbing material, selective heating of catalyst will result in the heat transfer to organic solvents and eventually increases the conversion yield [2]. Other than the conversion rate, Tashima et al. (2016) have shown promising results for microwave-assisted catalytic reactions, where the kinetic rate by microwave irradiation was higher than conventional heating by 1.15 times [3]. The interaction of materials with microwave irradiation is significantly influenced by the behaviours of materials. Materials that reflect microwave irradiation are conductors while materials that absorbed little amount of microwave irradiation are low dielectric loss materials. Some catalysts were discovered to possess good microwave-absorbing properties that subsequently enhance the efficiency of microwave-assisted catalytic reactions, which are known as high dielectric loss materials. The absorption of microwave energy is calculated based on the dielectric loss factor and electric field strength [1]. Therefore, the exploration of microwave-absorbing catalysts is important to maximize the efficiency of both catalysts and microwave irradiation in the reaction.

In this review, the mechanisms of microwave technology related to catalytic reactions and the synthesis of catalysts are discussed. Specific types of microwave-absorbing catalysts were critically reviewed along with their respective applications in biofuel, such as methanation, bio-oil production and glycerol dehydration. The significance of microwave-absorbing catalysts is included to provide insights on methods to enhance the development of this technology in various applications and aid in sustainable production.

2. Microwave Irradiation

2.1. Fundamentals of Microwave Irradiation

Microwaves are a form of electromagnetic waves consisting of two perpendicular components (electrical and magnetic fields) that their electromagnetic spectrum fall between radio waves and infrared [4]. The microwave frequencies are ranged from 0.3 to 300 GHz which correspond to the wavelength of 1 m to 1 mm. Microwave is generally utilized in phones, radar signals, navigational applications, satellite communications, food preparation, drying materials or medical treatments. In chemical industries, the microwave irradiation is mainly used for materials heating purposes, where the materials' responses varied due to the magnetic and electric fields associated with microwaves [5]. As microwave irradiation is mainly utilized for telecommunications, the regulations of the wavelengths for microwave equipment in research, industrial, domestic and medical field are required and the major operating frequency is 2.45 ± 0.05 GHz for most of the countries [6,7].

To generate microwave irradiation, the basic components required for industrial microwaves are the generator, waveguide and applicator. The usage of generator is the main component to convert the electrical energy into microwave energy and it often composed of transformer, solenoid and magnetron tube. Microwave irradiation is emitted from magnetron tube, while solenoid is used to wrap the magnetron tube for regulating the microwave power and high voltage is produced using transformer [8]. The waveguide is used to guide the microwave to the applicator, usually built up of aluminum due to its lightweight. The applicator is a place where microwave irradiation meets the load or samples to be investigated.

Several important factors have to be considered in microwaves are the frequency used, product mass, ionic content, dielectric properties, specific heat and density of the target

object. The properties of materials as regard to microwave irradiation are described as followed. Their complex permittivity (ϵ^*) is described in Equation (1) [9]:

$$\epsilon^* = \epsilon' - j\epsilon'' = \epsilon_0 (\epsilon_r' - j\epsilon_{eff}'') \quad (1)$$

where ϵ' is the real term to quantify the material's ability in storing electrical energy (also known as dielectric constant) and ϵ'' is the imaginary term of a loss factor that the material's ability in dissipating electrical energy (also known as dielectric loss); while the complex permeability (μ^*) is described in Equation (2):

$$\mu^* = \mu' - j\mu'' \quad (2)$$

where μ' is the real term to symbolic the amount of magnetic energy stored in the material and μ'' is the imaginary term of the magnetic energy that can be converted into heat energy. The ratios of imaginary to real terms for the complex permittivity and permeability are stated as below:

$$\tan \delta = \epsilon'' / \epsilon' \quad (3)$$

$$\tan \delta_\mu = \mu'' / \mu' \quad (4)$$

where $\tan \delta$ is the loss tangent (for complex permittivity) and $\tan \delta_\mu$ is the magnetic loss tangent (for complex permeability) [9]. The dielectric constant and dielectric loss varied with respect to the type of materials, which subsequently influence the loss tangent.

As compared to radio waves, microwave has higher frequencies which contains higher energy. From literatures, the higher microwave frequency significantly affects the penetration depth of microwave irradiation and their relationship can be expressed using wavelength and penetration depth as shown in following equation [10].

$$D_p = (\lambda/2\pi) \cdot [(\epsilon')^{1/2} / \epsilon''] \quad (5)$$

where D_p represents the depth of penetration which the incident energy absorbed is 63%, λ represents the wavelength of microwave, and π is the value of 3.1415.

The power loss density per unit volume, P , for both electric field and magnetic field heating are stated in Equations (6) and (7), respectively.

$$P = \omega \cdot \epsilon''_{eff} \cdot \epsilon_0 \cdot E_{rms}^2 \quad (6)$$

$$P = \omega \cdot \mu''_{eff} \cdot \mu_0 \cdot H_{rms}^2 \quad (7)$$

where ω is the angular frequency (also equals to $2\pi f$, where f is frequency), ϵ''_{eff} is the effective dielectric loss factor, ϵ_0 is the permittivity of free space, E_{rms} is the local value of electric field for Equation (6); while μ''_{eff} is the effective magnetic loss factor, μ_0 is the magnetic permeability of vacuum and H_{rms} is the local value of the magnetic field [11].

By exposing product to the microwave irradiation, the product mass is a concern especially for material heating. As the heat exchange is solely depending on the product mass, makes no difference for microwave heating and conventional heating through the definition of isobaric heat capacity ($\Delta Q = m \cdot C_p \cdot \Delta T$), and changing only based on temperature difference and C_p [12]. However, both methods possess varied heating efficiencies and lead to different energy consumption. The properties of the object being microwaved can influence the efficiency and uniformity of microwave irradiation. Ionic content of the subject influences the occurrence of ionic conduction when irradiate with microwaves while the dielectric properties of subject governs on the ability of a material to interact with the electromagnetic field of microwave [13].

2.2. Mechanism of Microwave Irradiation

Catalytic reaction performed through microwave irradiation has gained much attention due to their outstanding performance and enhanced reaction efficiency. The mech-

anisms of microwave irradiation are the main factor for their wide range of application in chemical processes. The energy absorbed during the process was dissipated as heat, hence the microwave heating is internal rather than external which different from the conventional heating methods. The even internal heating resulted by the conversion of electromagnetic energy into heat at molecular level is believed to assist in the enhancement of catalyst stability and decrement of coke formation [14].

In general, the heating mechanisms of microwave energy consist of electric field heating and magnetic field heating, which are governed by different mechanisms. For electric field heating, there are two major mechanisms, namely dipolar polarization and ionic conduction, while the combination of these two mechanisms is interfacial polarization [15]. The dipolar polarization is a mechanism involves the rotation of polar molecules aligning themselves with microwave's electric field, where the heat and friction were generated due to the continuous alignment of polar molecules as the electric field was regularly oscillating as shown in Figure 1. As for ionic conduction, the electric current is produced due to the oscillation of ions in forth and backward due to the electric force of microwaves. The produced current undergoes internal resistance, in which the collisions between the charged molecules with their neighboring molecules heat up the materials [16].

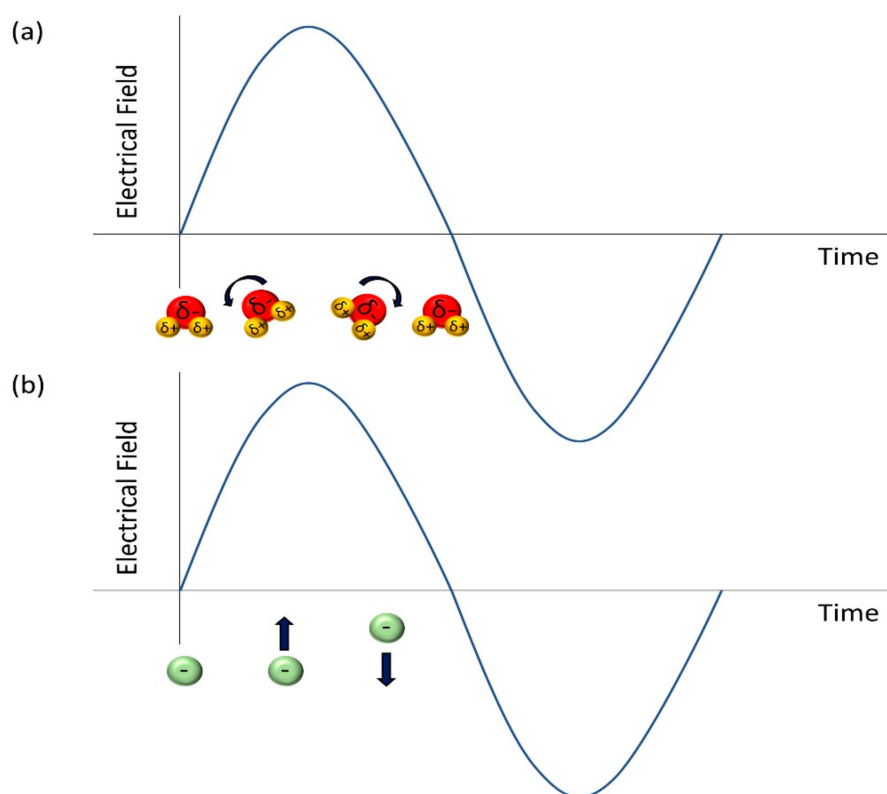


Figure 1. Electric field heating mechanisms under microwave. (a) Dipolar polarization which involves the rotation of polar molecule to align itself with electric field. (b) Ionic conduction that shows an ion moving back and forth if the molecule is charged.

However, another dominant mechanism known as magnetic losses would be advantageous to the selective heating of particular catalyst zones, due to the losses of eddy currents, hysteresis and residual loss occurred. Heat generation was observed during hysteresis losses as the magnetic dipoles of the materials could not keep pace with altering magnetic poles leading to a friction. Eddy current losses occur when there are resistances towards the circulating currents within the magnetic materials created by the generated electromagnetic field; while residual losses refers to other types of losses other than hysteresis and eddy current losses [17].

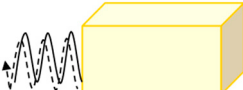
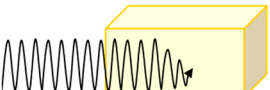
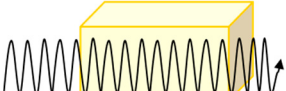
3. Catalysts Synthesis

Microwave irradiation is applicable in a wide range of application. One of the applications being an assisting technology for a process or an independent technology in synthesizing catalysts, which the processes assisted can be chemical vapour deposition, chemical reduction even biomediated process etc. [18–21]. It was proven that the processing period is shorten by integrating microwave into biomediation of plant extracts with gold or silver nanoparticles for pollutants removal. The favourable results obtained through the mentioned studies have shown the flexibility of microwave as a viable technology and advancement of process. It is also often promoted as a green technology compared to the traditional process due to efficient energy conservation, the improved effectiveness of process, product quality and product yield [22].

Exposing catalysts to microwave not only helps in heating up the object, the microwave irradiation also leads to the movement of atoms where the angle of the atoms of catalyst will change and open up the active sites. It is also observed that the materials become porous and specific pore volume or surface area increases after being exposed to microwave irradiation [20,23,24]. With more pores generated, the gas or liquid flow passing by the catalysts results in higher reaction rates with the catalysts as compared to non-microwave irradiated catalysts. Therefore, catalyst synthesis using microwave or other technologies assisted by microwave is concluded to enhance the catalytic properties of catalyst.

As microwave heating is commonly known as dielectric heating, the contribution of magnetic field is not noticeable or widely studied compared to dielectric heating. The material with higher magnetism was claimed to absorb more microwaves from past literature [25]. The ability of material in absorbing the microwave depends on the type of magnetic material, which some able to reproduce the magnetic field even after the magnetic field is removed like ferromagnetic materials. The common types of ferromagnetic are iron (Fe), nickel (Ni), cobalt (Co), and most of their alloys. The other type of materials such as paramagnetic and diamagnetic materials have lower magnetism than ferromagnetic, as it cannot sustain the magnetism once the field was taken away and later resist magnetism from the magnetic field. Good examples for diamagnetic are plastics, natural substances (carbon, water, wood), gold (Au), copper (Cu) and silver (Ag); while the common types for paramagnetic materials are magnesium (Mg), molybdenum (Mo), lithium (Li) and tantalum (Ta) [26]. Despite the type of magnetic materials, materials can be categorized in three major types according to their interaction with microwaves, namely reflector, absorber and transparent. The properties of materials according to the mentioned type are tabulated in Table 1.

Table 1. The properties of materials interacting with microwaves [27,28].

Categories	Interaction with Microwave	Material Characteristics	Type of Materials	Penetration
Reflector or Opaque		Conductor	Steel, aluminum, copper, silver	None as microwave are reflected and no energy transfer.
Absorber ¹		Lossy insulator	Water, charcoal, silicon carbide	Partial to total as the microwave are absorbed and exchange of electromagnetic energy takes place.
Transparent		Low loss insulator	Polytetrafluoroethylene (PTFE), alumina-based ceramics, corundum, fused quartz, Teflon, glass ² , alumina ² , silica ² , magnesium oxide ²	Total where the transmittance of microwave occurs without energy transfer.

¹ The dielectric properties of materials govern the amount of microwave absorption; ² The materials appear to be transparent to microwave at room temperature but absorb microwave and couple with them effectively after being heated to particular temperature.

From Table 1, catalyst required for biofuel production often focus on the absorber as type of materials as the materials that reflects microwave totally will not be transmitting any energy and probably only the surface of materials is heated, which do not contribute completely to the catalytic reaction. As for transparent materials, the absence of energy transfer could not contribute to the catalytic reaction as well. It was reported the absorber type of materials could have produced “microwave hotspot” while being irradiated under microwave, leading to the higher temperature of catalyst compared to the surrounding medium (e.g., liquid) [29]. In addition, the dielectric properties of the materials are the main factors in manipulating the amount of microwave being absorbed by the catalyst. Every type of materials possesses their own dielectric properties, which are further influence the reactivity of the catalyst and subsequently affect the product yield at the end of process.

The catalyst synthesis using microwave is simple, time-saving and efficient as the synthesis of catalyst can be performed through exposing the materials used as catalyst under microwave irradiation to form reactive catalyst or a homogenous distribution of catalyst on the supports. Several studies showed that catalyst synthesized through microwave irradiation has uniform distributed active components over the supports, where the active sites can be easily accessed by the reactants and subsequently increase the yield of the end products. The strong metal-support interaction was observed in the Ru-M_xC (M = Mo, Co, Cr) catalyst with acidified multi-walled carbon nanotubes as support in the study of Wu et al. using only household microwave oven at high temperature [30]. For the Pt nanoparticles supported on carbon as the fuel cell catalyst, the TEM results showed that the narrow particle size distributions of Pt nanoparticles on the surface of carbon indicating the uniform dispersion of catalyst on support, even with 20 wt% of Pt [31]. The enhanced properties of catalysts were reported as Ru-M_xC catalysts able to achieve current densities of 500 mA cm⁻² with a great stability of 1000 h and improved catalytic activity of catalysts for Wu et al. and Shams et al., respectively [30,31]. The adjustment of some parameters such as microwave power, irradiation time, and the mode of microwave irradiation were required to synthesize the catalysts at their optimal functionality. Among these parameters, short synthesizing time is the key factor of microwave being one of the popular technologies as this avoid excessive generation of impurities during the reaction and energy-efficient. Approximately 2 min was required to synthesize catalyst using microwave either as sole technology or assisting technology in past studies [32,33].

4. Applications

Microwave technology can be used not only for catalyst synthesis, but in various field as it enhanced the processes in terms of reaction rates, product yield and energy efficiency. It's unique heating mechanism favours its selection over conventional heating techniques. To give a brief idea, the comparison between microwave-assisted heating and conventional heating is tabulated in Table 2.

Table 2. Comparison between microwave-assisted and conventional heating [34,35].

Microwave-Assisted Heating	Conventional Heating
Rapid and uniform heating	Slow heating
Shorter preparation time	Longer preparation time
Lower thermal inertia	Higher thermal inertia
Heat transferred through in-core volumetric heating at molecular level	Heat transferred by conduction, convection or radiation

Several studies for the bio-oil production, methanation, methane reforming, sugar conversion to 5-hydroxymethylfurfural (5-HMF) and ammonia synthesis performed via microwave-assisted technology and conventional techniques have been presented in Table 3 to compare the experimental conditions and findings. The application of mi-

microwave technology related to catalytic reactions or catalysts synthesis are discussed in following sections.

Table 3. Comparison studies between microwave-assisted technologies and conventional technologies.

Application	Feed	Experimental Conditions	Method	Findings	Reference
Bio-oil	Switchgrass	Power: 750 W Temperature: 400 °C Catalyst: 10 wt% K ₃ PO ₄ + 10 wt% Bentonite	Microwave-assisted catalytic pyrolysis	Reduced water content in bio-oil with increased BET surface area of biochar using microwave-assisted pyrolysis. Average heating rate: 141 °C/min Heating time: 2.83 min BET surface area: 76.29 m ² /g Micropore area: 44.56 m ² /g Pore volume: 0.0332 cm ³ /g	[36]
	<i>Chlorella vulgaris</i> and high-density polyethylene (HDPE)	Feedstock mixing ratio: 1:1 Absorbent addition: 40% activated carbon (AC) Power: 800 W	Microwave-assisted co-pyrolysis	The promotion of CO, H ₂ O or CO ₂ was observed with the addition of AC. Oxygen/Nitrogen-containing compounds: 28.79%/20.8% Hydrocarbons content: 48.88% Alcohols: 14.6%	[37]
	Algae	Feed-to-susceptor ratio: 1:1 Power: 450 W Catalyst: ZSM-5 Temperature: 600 °C	Microwave-assisted co-pyrolysis	Pyrolysis char used a susceptor and pyrolysis of algae was rich in phenolic derivatives. Hydrocarbons obtained were ranging from C6 to C30. Bio-oil yield: 45 wt% Gas yield: 35 wt% Biochar yield: 20 wt%	[38]
	Switchgrass	Temperature: 400 °C Catalyst: 10 wt% K ₃ PO ₄ + 10 wt% Bentonite	Conventional pyrolysis	Longer heating time to reach desired temperature with poor biochar properties. Average heating rate: 14 °C/min Heating time: 28.81 min BET surface area: 0.33 m ² /g Micropore area: 2.01 m ² /g Pore volume: 0.0068 cm ³ /g	[36]
	Switchgrass	Temperature: 300 °C to 500 °C Heating rate: 10 °C/min Feed amount: 1 kg	Conventional pyrolysis	Bio-oil yield: ~28 wt% to ~32 wt% Gas yield: ~176 L to ~271 L Biochar yield: ~30 wt% to ~40 wt%	[39]
	White ash	Temperature: 300 °C to 500 °C Heating rate: 10 °C/min Feed amount: 1 kg	Conventional pyrolysis	Bio-oil yield: ~38 wt% to ~43 wt% Gas yield: ~154 L to ~225 L Biochar yield: ~28 wt% to ~39 wt%	[39]

Table 3. Cont.

Application	Feed	Experimental Conditions	Method	Findings	Reference
Methanation	CO ₂ , H ₂ , He	Temperature: 300 °C Molar ratio of CO ₂ /H ₂ /He: 1/4/5 Ni: 30 wt% Ce: 20 wt%	Microwave-assisted hydrothermal synthesis	Addition of Ce enhanced the catalyst activities and microwave promoted Ni dispersion on support. CH ₄ selectivity: 98.0% CO ₂ conversion: 52.9%	[40]
	CO ₂ , H ₂ , N ₂	Temperature: 325 °C Ratio of CO ₂ /H ₂ /N ₂ : 1/4/4 Flowrate: 70 mL/min Ni: 20 wt%	Microwave-assisted synthesis	Low temperature of H ₂ pretreatment allows more Ni active sites. CH ₄ selectivity was well-maintained from 200 °C to 400 °C. CH ₄ selectivity: 99.3% CO ₂ conversion: 91.6%	[41]
	CO, H ₂ , N ₂	Temperature: 300 °C Pressure: 1 MPa Ratio of CO/H ₂ /N ₂ : 1/1/3 Heating rate: 3 °C/min	Microwave-assisted solution combustion	Catalyst with large specific surface area and small Ni particles was obtained. CH ₄ selectivity: 96.2% CO ₂ conversion: 95.7%	[42]
	CO ₂ , H ₂ , N ₂	Temperature: 350 °C Ratio of CO ₂ /H ₂ /N ₂ : 1/4/4 Flowrate: 70 mL/min Ni: 20 wt%	Impregnation	Low temperature of H ₂ pretreatment allows more Ni active sites. CH ₄ selectivity was well-maintained from 200 °C to 400 °C. CH ₄ selectivity: ~99% CO ₂ conversion: 84.3%	[41]
	CO ₂ , H ₂ , He	Temperature: 50–200 °C Ratio of CO ₂ /H ₂ : 1/4 Catalyst amount: 200 mg Flowrate: 20 mL/min	Sol-gel, aerosol, impregnation	Low methanation production at 350 °C but maximum was achieved after annealing at 450 °C. CH ₄ selectivity: 100% @200 °C CH ₄ yield: 2.05 μmol _{CH4} /g _{cat} /s	[43]
	CO ₂ , H ₂ , N ₂	Temperature: 250–450 °C Molar ratio of H ₂ /CO ₂ /N ₂ : 36/9/10 Catalyst amount: 0.1744 g Flowrate: 250 mL/min	Impregnation	Catalyst synthesized through impregnation has poorer performance due to the Ni ⁰ size. CH ₄ selectivity: ~94% @450 °C CO ₂ conversion: 70.0% @450 °C	[44]
Methane reforming	CH ₄ , CO ₂	Specific power: 90 W/g Ratio of CH ₄ /CO ₂ /Ar: 1/1/2 Space velocity: 200 h ^{−1}	Microwave dry reforming	Insignificant changes and negligible carbon deposition on catalyst after 50 h stability test. CH ₄ conversion: > 95% CO ₂ conversion: > 95% H ₂ /CO ratio: ~1	[45]
	CH ₄ , CO ₂	Temperature: 800 °C Molar ratio of CO ₂ /CH ₄ : 0.5, 1, 1.5, 2 N ₂ flowrate: 60 mL/min Volumetric hourly space velocity: 2.4 L/(g.h)	Microwave dry reforming	10% of Fe ₂ O ₃ addition led to maximum performance of dry reforming reaction with good catalyst stability. CH ₄ conversion: 90.8% CO ₂ conversion: 95.2% H ₂ /CO ratio: 0.92	[46]
	CH ₄ , CO ₂	Power: 560 W Ratio of CH ₄ /CO ₂ /N ₂ : 1/1/3 Gas flowrate: 250 mL/min	Microwave dry reforming	Decreasing CH ₄ /CO ₂ ratio and increasing microwave power improve CH ₄ and CO ₂ conversions. CH ₄ conversion: ~80% CO ₂ conversion: ~60% H ₂ /CO ratio: 1.3	[47]

Table 3. Cont.

Application	Feed	Experimental Conditions	Method	Findings	Reference
	CH ₄ , CO ₂	Temperature: 800 °C GHSV: 33,000 mL/g _{cat} .h Catalyst amount: 0.9 g	Conventional dry methane reforming	Energy efficiency using this method was lower by 10% compared to microwave heating reactors. CH ₄ conversion: ~65% CO ₂ conversion: ~70% H ₂ /CO ratio: 0.85	[48]
	CH ₄ , CO ₂	Temperature: 800 °C Pressure: 1 atm	Dry methane reforming	The reaction can be improved by altering pressure and ratio of oxidant/methane. CH ₄ conversion: 85% CO ₂ conversion: 90%	[49]
	CH ₄ , CO ₂	Temperature: 950 °C Gas hourly space velocity: 8570 h ⁻¹ Sample mass: 300 mg	Dry reforming of methane (magnetic induction)	Magnetic catalyst used to improve CH ₄ dry reforming. CH ₄ conversion: 70% CO ₂ conversion: 80% H ₂ yield: 75% CO yield: 85%	[50]
Sugar conversion	Fructose	Using [Ch]Cl:CA Microwave heating: 2 min Temperature: 120 °C Solid/liquid ratio: 0.05	Biphasic systems with aqueous and organic phase assisted with microwave	The purity of 5-HMF remained after the repetition of 3 cycles process, reusing acidic deep eutectic solvents. HMF yield: 91.0%	[51]
	Corn stalk, rice straw and pine wood	Irradiation time: 2–6 min	Direct conversion using ionic liquids assisted with microwave	The yield of products obtained within 3 min of processing time, very efficient process. HMF yield: 45–52%	[52]
	Cellulose	Time: 3.5 min Power: 400 W Ionic liquid: [Bmim]Cl	Direct conversion in ionic liquids assisted with microwave	Microwave was proven to have synergetic effects on the cellulose conversion. HMF yield: 51.4%	[53]
	Glucose	Time: 10 min Temperature: 200 °C Medium: 50:50 w/w% 1-hexyl-3-methyl imidazolium chloride-water mixture	Direct conversion in ionic liquids-water mixture	Synergistic effect was observed through the addition of protic solvents. HMF yield: 53%	[54]
	Glucose	Temperature: 100 °C Catalyst amount: 0.5 g Time: 6 h	Direct conversion in ionic liquids	The catalyst porosity has to be optimized according to the medium used for maximum production. HMF yield: 64%	[55]
	Fructose	Medium: 3:1 of methylisobutylketone: 2-butanol Feed amount: 1 g Time: 6 h	Direct conversion in solvents	Good yield obtained using similar experimental condition for waste potato biomass too. HMF yield: 50 wt%	[56]
Ammonia synthesis	H ₂ , N ₂	Temperature: 260 °C Ambient pressure Catalyst amount: 1.2 g	Microwave-assisted catalytic synthesis	Low temperature and pressure required for this method are energy-saving compared to Haber-Bosch process. NH ₃ production rate: 1313 µmol _{NH3} /g _{cat} .h	[57]
	H ₂ , N ₂	Temperature: 320 °C Pressure: 0.65 MPa H ₂ /N ₂ ratio: 1 Time: 11 min	Microwave-assisted catalytic synthesis	Quick catalyst recovery compared to Haber-Bosch technology. NH ₃ production rate: 0.04 g/g _{cat} .h	[58]

Table 3. Cont.

Application	Feed	Experimental Conditions	Method	Findings	Reference
	H ₂ , N ₂	Temperature: ~260 °C Pressure: 0.1 MPa	Microwave-assisted catalytic synthesis	Ce-promoted catalyst enhances production and microwave activates stable molecules. NH ₃ production rate: 1.18 mmol/h.g _{cat}	[59]
	H ₂ , N ₂	Temperature: 300 °C Pressure: 10 bar	Catalytic synthesis	Efficient synthesis rate obtained through Co-Mg-O solid solution supported LiH catalyst, with Co nanoparticles. NH ₃ production rate: 19 mmol/g/h	[60]
	H ₂ , N ₂	Temperature: 350 °C Pressure: 7 atm Molar ratio of H ₂ /N ₂ : 1/1	Catalytic synthesis	The ammonia synthesis rate altered according to the composition of catalyst. NH ₃ production rate: 0.397 mmol/g _{cat} .h	[61]

Note: GHSV: gas hourly space velocity. @: at.

4.1. Bio-Oil Production

At the current situation, the importance of biomass as energy or chemical source has been look upon to for the global energy transition in achieving net zero emissions. Biomass valorization allows the biomass to be transformed into biofuels and value-added chemicals such as plastic or textiles. Biofuels especially liquid biofuels, can be produced through thermochemical conversions such as transesterification, pyrolysis or gasification and biological route like fermentation. These conventional techniques are applied for commercial-scale production, yet there is room for improvement in the productivity. The idea of incorporating microwave with these established techniques is to enhance the efficiency of process in terms of energy and time, whereby benefits such as convenient implementation without agitation, even internal heating of big biomass particles, volumetric scale heating and short processing time [62].

The range of liquid biofuel includes biodiesel, bio-oil and bioethanol, which can be extracted from high oil yield or sugar yield of biomass, respectively. Bio-oil is a liquid product that can be produced through pyrolysis and utilized as feedstocks of chemicals and materials, despite using in the boiler for combustion and co-firing, diesel engines and gas turbines [63]. As the composition of bio-oil varies according to the processing condition and biomass type which subsequently influences the properties of bio-oil, an upgradation might be required prior to the direct application in engines.

Microwave-assisted pyrolysis is reported to enhance the bio-oil composition and the surface properties of biochar [62]. In bio-oil composition, the major components found are hydrocarbons, ketones, phenols, esters, acids, alcohols etc. as tabulated in Table 4. A study showed that overall enhancement of bio-oil and biochar properties using microwave-assisted catalytic pyrolysis. It was reported that total amount of 7.7 wt% water content in the bio-oil was decreased with the increment of bio-oil yield and decrement of bio-oil viscosity [36]. High hydrocarbons content was found using co-pyrolysis with the highest yield of hydrocarbon ranged from C7 to C12, following by C16 and >C18, and least amount of ketones in the obtained bio-oil [37]. High composition of hydrocarbons in bio-oil represents the better quality of bio-oil to be processed into biodiesel, renewable diesel, or aviation fuels. Zero formation of hydrocarbons was noticed in the study of Wang et al. using cellulose as the feedstock, with a high phenol-rich bio-oil yield, ~55 wt%, was obtained [64]. This could be the type of catalyst used in the process that influenced the bio-oil composition. As for the biochar properties, surface area is one of the aspects being analysed through Brunauer-Emmett-Teller (BET) method which can be utilised as another source of fuel. Using microwave-assisted technology, the BET surface area of biochar was

increased by 75.97 m²/g as compared to conventional heating [36]. Another study reported even higher char's surface area using ZSM-5 catalysts, ranging from 118–125 m²/g while obtaining considerably high bio-oil content as final product [38].

Table 4. The bio-oil production using microwave-assisted technologies.

Process	Feedstock	Catalyst/Absorbent	Properties/Composition	Yield	Remarks	Reference
Catalytic pyrolysis	Switchgrass	30 wt% clinoptilolite	pH: 4.19 Water content: 21.77 wt% Viscosity (40 °C): 6.11 cP	36.2 wt%	BET surface area (biochar): 76.3 m ² /g	[36]
Catalytic pyrolysis	Cellulose	Fe/Modified HZSM-5	Phenols: 6.23% Oxiranes: 5.45% HCs: 0% Esters: 5.21% Ketones: 12.79% CAs: 25.50% Furans: 23.06% SCs: 21.76%	54.85 wt%	Biochar yield: 14.46 wt% Biogas yield: 23.78 wt% Coke yield: 6.91 wt%	[64]
Catalytic pyrolysis	Cellulose	Fe-Ni/Modified HZSM-5	Phenols: 20.86% Oxiranes: 14.15% HCs: 3.96% Esters: 0.73% Ketones: 11.94% CAs: 26.96% Furans: 21.40% SCs: 0%	51.86 wt%	Biochar yield: 14.46 wt% Biogas yield: 26.33 wt% Coke yield: 7.35 wt%	[64]
Catalytic pyrolysis	Corn stover	10–30% Na ₂ CO ₃	Water content: 53.09–61.49% pH: 3.92–4.62 Dynamic viscosity: 3.31–4.05 mPa.s (for 500 and 700 W) Phenols: 33.26% Furans: 14.11% Acids: 4.77% Guaiacols: 7.05%	41 wt%	Microwave: 700 W for compositions and yield.	[65]
Catalytic pyrolysis	Waste cooking oil	CaO from crab shell	Total aromatics relative content: 54.89% Cycloalkenes relative content > 4.07%	67 wt%	Biogas yield: 30 wt% CH ₄ and H ₂ formation being promoted.	[66]
Catalytic pyrolysis	Torrefied corn cob	Fe modified biochar (from rice husk)	Phenol: 0.455–0.704 mg/mL bio-oil (~0.16–0.24 mg/g biomass) Cresol: 0.09–0.239 mg/mL bio-oil (~0.03–0.08 mg/g biomass)	~33–35 wt%	The yield varies according to Fe amount	[67]
Co-pyrolysis	Microalgae and HDPE	Activated carbon	HCs: 48.88% Alcohols: 14.6% Amines: 7.16% Acids/esters: 5.74% Ketones: 0.42% Nitriles: 6.02% Phenols: 0% Others: 17.18%	-	31.02% of C7–C12, 22.3% of C16, 18.4% of > C18 hydrocarbons were obtained.	[37]
Co-pyrolysis	Microalgae and waste cooking oil	Phosphorus-doped biochar	C ₅ –C ₁₆ aliphatics: 30.58% C ₁₆₊ aliphatics: 2.62% Mono-aromatics: 52.35% Poly-aromatics: 6% Nitriles: 3.38% Alcohols: 5.17%	47.63%	No n-heterocyclics, amides, esters, phenols in bio-oil, but these components presented in the bio-oil produced using biochar.	[68]

Table 4. Cont.

Process	Feedstock	Catalyst/Absorbent	Properties/Composition	Yield	Remarks	Reference
Catalytic pyrolysis	Microalgae	Fe ₂ O ₃ with graphite	Phenol: 3% Ketone: 23% Aromatic compounds: 3% Esters: 9% Acids: 14% Nitrogen-cont. compound: 23% Alcohol: 8%	24.9%	Optimal ratio for Fe ₂ O ₃ with graphite is 3:7	[69]
Catalytic pyrolysis	Microalgae	ZMS-5 with graphite	Phenol: 4% Ketone: 14.5% Aromatic compounds: 10% Esters: 5% Acids: 14% Nitrogen-cont. compound: 11.5% Alcohol: 3%	23.8%	Optimal ratio for Fe ₂ O ₃ with graphite is 5:5	[69]
Catalytic pyrolysis	Peanut shells	Mixture of peanut shells and activated carbon	Other HCs: 18.85% Aromatic HCs: 15.08% Alcohols: 12.14% Phenols: 51.19% Ketones: 10.73%	25.97%	Ratio of catalysts to peanut shells: 12.5% Biochar: 34.5% Syngas: 39.53%	[70]
Catalytic co-pyrolysis	Mixture of waste polyethylene and algae	ZSM-5 catalyst	Aliphatic HC: 27% Cyclic aliphatic HC: 10% Aliphatic oxygenates: 22% Monoaromatic HC: 27% Polyaromatic HC: 8.6% Phenolics: 5%	40 wt%	Char surface area: 125 m ² /g	[38]
Catalytic co-pyrolysis	Mixture of waste polypropylene and algae	ZSM-5 catalyst	Aliphatic HC: 30% Cyclic aliphatic HC: 13% Aliphatic oxygenates: 44% Monoaromatic HC: 5.8% Polyaromatic HC: 2.7% Phenolics: 5%	45 wt%	Char surface area: 121 m ² /g	[38]
Catalytic co-pyrolysis	Mixture of waste expanded polystyrene	ZSM-5 catalyst	Aliphatic HC: 32% Cyclic aliphatic HC: 12% Aliphatic oxygenates: 43% Monoaromatic HC: 5.1% Polyaromatic HC: 0.8% Phenolics: 7%	65 wt%	Char surface area: 118 m ² /g	[38]

The CAs: Carboxylic acids; SCs: Saccharides; HCs: Hydrocarbons; HHV: Higher heating value; HDPE: High density polyethylene.

Besides hydrocarbons, the existence of phenols and esters allows bio-oil to be partially replaced petroleum fuels after separating oxygenates from the bio-oil [71]. Particular types of phenols were reported to aid in the oxidation stability, such as phenols with alkyl side chains on ortho and para position of phenylic hydroxyl group [72]. The usage of activated carbon in catalytic pyrolysis generates high content of phenolics while co-pyrolysis can generate bio-oil with no content of phenols and high yield of hydrocarbons [37,70]. In the case of Su et al. study, usage of biochar leads to formation of phenols, amides, esters and n-heterocyclics but no formation of these components were observed using phosphorus-doped biochar as catalyst [68]. However, the bio-oil yield was not affected much using both raw bio-char or phosphorus-doped biochar in the study. Another factor that influences the composition of bio-oil is the type of feedstock. A hydrocarbon-rich bio-oil was produced

using stillingia oil (triglyceride-based biomass) while bio-oil mainly consisted of oxygen-containing compounds (acids and phenols) was generated using *Camellia oleifera* shell (lignocellulosic biomass) [67]. In short, microwave has improved the composition of bio-oil while the type of catalyst and feedstocks used in the process contribute to the bio-oil composition and yield.

4.2. Methanation

Methanation is a process involved converting carbon monoxide and carbon dioxide (CO_x) into methane through hydrogenation. This process is important to produce the substitute or synthetic natural gas from sustainable source as the alternatives of natural gas. Carbon dioxide as a greenhouse gas can be converted into valuable fuels which will largely reduce the environmental impact and fulfill the high demand of natural gas at the same time. In order to produce synthetic natural gas, the reaction involved is names as Sabatier reaction or CO_2 methanation, as shown in Equation (8).



This process requires the input of hydrogen to produce methane, and the $\Delta H_{298\text{K}}$ of the reaction is -165.15 kJ/mol [40].

Microwave technology is involved in the catalyst development for methanation, especially catalyst works under low temperature methanation. To perform methanation, noble metals have been the best candidate as catalyst that can perform well under low temperature with high efficiency, but the high cost of noble metals was not economical and practical to be utilized for industries. Ni as one of the non-noble metals, is a good substitution of noble metal catalysts due to comparable activities and relatively affordable cost. From Table 5, microwave was proven to enhance the dispersity of Ni nanoparticles on the support as compared to impregnation method in the study of Song et al. Besides, the Ni catalysts synthesized through microwave-assisted technique showed enhanced methanation activity of the catalyst under low temperatures (325°C) and maintained high selectivity towards CH_4 in the range of 200 to 400°C [41]. The development of natural kaolin-based Ni catalyst using microwave-assisted hydrothermal method also possessed higher CO_2 conversion and CH_4 selectivity, 7.5% and 79.9°C , respectively. This is due to the Ni particles were highly dispersed with comparatively smaller crystalline size on the catalyst [40]. Other than Ni, the mesoporous silica KCC-1 synthesized by microemulsion system integrated with microwave-assisted hydrothermal method was reported to have high BET surface area, high number of basicity and oxygen vacancy. A total 5-fold higher methanation activity was observed in mesoporous silica KCC-1 than MCM-41 and SiO_2 as the mentioned aspects were directly related to the catalytic performance [73].

Table 5. Catalyst synthesized using microwave-assisted technologies for CO_2 methanation.

Catalyst Synthesized	Process	Textural Properties/Composition	Yield	Remarks	Reference
Ni/ Al_2O_3	Microwave	O: 40.50 wt% Al: 39.93 wt% Ni: 19.57 wt% Ni particle size: 10 nm Ni reduction degree: 90.4% Ni dispersion: 25.3%	CO_2 conversion: 91.6% CH_4 selectivity: 99.3%	Temperature: 325°C Durable stability for 72 h Reduced by H_2 at 450°C Surfactant: Polyvinyl pyrrolidone (PVP)	[41]
Ni-Ce/metakaolin	Hydrothermal	Ni: 26.57 wt% NiO crystallite size: 24 nm BET surface area: $31.18 \text{ m}^2/\text{g}$ Pore volume: $0.1532 \text{ cm}^3/\text{g}$ Average pore size: 19.65 nm	CO_2 conversion: 52.9% CH_4 selectivity: 98% CH_4 yield: 51.9%	Temperature: 300°C Ce-promoted in catalyst synthesizing Durable stability for 48 h	[40]

Table 5. Cont.

Catalyst Synthesized	Process	Textural Properties/ Composition	Yield	Remarks	Reference
Mesoporous silica KCC-1	Microemulsion coupled with hydrothermal	BET surface area: 773 m ² /g Total pore volume: 1.2195 cm ³ /g Pore distribution: 4–6 nm and 20–25 nm Particle size: 200–400 nm Basic sites concentration: 586	CO ₂ conversion: 48.7% CH ₄ selectivity: 98% CH ₄ yield: 38.9%	Temperature: 449.85 °C Durable stability for 90 h	[73]
Ni/mesocellular silica foam	One-pot for mesocellular silica foam; incipient wetness impregnation for Ni	Surface area of support: 913 m ² /g Pore volume of support: 0.98 cm ³ /g Ni particle size: 4.5 nm Si/O: 0.56 Ni/Si: 0.014 Ni: 5 wt%	CO ₂ conversion: 62–77% CH ₄ selectivity: 94–97%	Synthesized from rice husk ashes Cyclohexane as swelling agent Temperature: 350 °C Durable stability for 20 h	[74]
Ni/La-Sm-CeO ₂	Sol-gel	Ni particle size: 12.3 nm Ni dispersion: 7.9% Pore volume: 0.08 cm ³ /g CeO ₂ crystalline size: 7.2 nm BET surface area: 40.3 m ² /g Ni: 12.1% Ce: 14.4% O: 61.8% C: 7.0% Na: 0.9% La: 1.0% Sm: 2.8%	CO ₂ conversion: 53% CO selectivity: 5% @500 °C (0 at 300 °C) CH ₄ selectivity: 100% CH ₄ yield: 59.9–61.6%	Temperature: 300 °C Durable stability for 20 h	[75]
Ni/La-Pr-CeO ₂	Sol-gel	Ni particle size: 10.1 nm Ni dispersion: 9.6% Pore volume: 0.09 cm ³ /g CeO ₂ crystalline size: 8.0 nm BET surface area: 45.8 m ² /g Ni: 11.3% Ce: 13.6% O: 61.7% C: 7.6% Na: 1.4% La: 1.0% Pr: 3.4%	CO ₂ conversion: 55% CO selectivity: 2.5% @500 °C (0 at 300 °C) CH ₄ selectivity: 100% CH ₄ yield: 62.0–63.4%	Temperature: 300 °C Durable stability for 20 h	[75]
Ni/La-Mg-CeO ₂	Sol-gel	Ni particle size: 9.1 nm Ni dispersion: 10.7% Pore volume: 0.07 cm ³ /g CeO ₂ crystalline size: 7.5 nm BET surface area: 38.8 m ² /g Ni: 14.3% Ce: 13.4% O: 62.5% C: 6.4% Na: 0.2% La: 1.1% Mg: 2.1%	CO ₂ conversion: 49% CO selectivity: 4.5% @500 °C (0 at 300 °C) CH ₄ selectivity: 100% CH ₄ yield: 58.2–61.8%	Temperature: 300 °C Durable stability for 20 h	[75]
Ni-Al ₂ O ₃	Combustion with urea (fuel)	BET surface area: 186.1 m ² /g Average pore diameter: 3.6 nm Dispersion: 5.2% Maximum Ni surface area: 34.6 m ² /g	CO conversion: 95.7% CH ₄ selectivity: 96.2%	Temperature: 300 °C Lifetime test: 200 h	[42]

Despite using microwave in synthesizing catalyst, the structure of catalyst support can be synthesized using microwave. In the study of Paviotti et al., the production of mesoporous silica foam through microwave-assisted hydrothermal sol-gel process allowed the homogenous distribution of Ni catalyst with big proportion of them in the mesopores interacting with the pore-walls [74]. The synthesizing time was significantly shortened using microwave irradiation from >20 h (using conventional hydrothermal treatment) to 5 h. Other supports such as $\text{La}_2\text{O}_3\text{-Pr}_2\text{O}_3\text{-CeO}_2$, $\text{La}_2\text{O}_3\text{-Sm}_2\text{O}_3\text{-CeO}_2$ and $\text{La}_2\text{O}_3\text{-MgO-CeO}_2$ prepared using the same method had displayed high selective methanation using Ni catalysts [75]. The basic sites were improved and oxygen vacancies were increased with the presence of Mg^{2+} , La^{3+} , and Sm^{3+} into CeO_2 to reinforce the supports, subsequently increased the rate of methanation reaction at low operating temperature. The reinforcement had enhanced the stability of the catalyst as well. From the studies discussed, catalysts synthesized using microwave-assisted technologies have improved the physical properties of catalysts and leads to higher conversion rate of methanation.

4.3. Methane Reforming

Methane reforming such as dry reforming of methane (also known as CO_2 reforming) and methane steam reforming are the routes to produce synthesis gas (syngas) for power applications such as other fuels production (methanol and diesel fuel), electricity generation via Organic Rankine Cycle or steam turbine, industrial thermal oil or steam production as well as heat production for industrial purposes. Different types of methane reforming involve different reactants to obtain the same components as products. Equation (9) is the reaction of dry reforming of methane, which the greenhouse gases, CO_2 and CH_4 were reacted to obtain CO and H_2 ; while Equation (10) displayed the reaction of methane steam reforming.



Dry reforming of methane is a highly endothermic reaction due to the strong thermal stability of both CO_2 and CH_4 , as the bond-dissociation energies of C=O-O and $\text{CH}_3\text{-H}$ are very high (532 kJ/mol and 435 kJ/mol, respectively) [76]. Hence, high energy input is required for the conversion of CO_2 and CH_4 . Both reactions were endothermic reactions, but methane steam reforming generates lower CO content-syngas that accounted for less calorific value, with lower energy input than that of dry reforming of methane [77].

In methane steam reforming, one of the focuses for study is the catalysts with different supports which prevent the deactivation of catalyst, improving the methane conversion efficiency and stability of catalyst. The catalyst synthesized via microwave-assisted technologies is tabulated in Table 6 while the methane reforming assisted with microwave is presented in Table 7. The occurrence of carbon deposition on catalyst's surface at 300–100 °C has led to the catalyst deactivation, therefore the optimization of catalyst fabrication is much needed [24]. The study of Wattanathana et al. has reported that the platinum doped on calcined cerium complex with cubic fluorite structure through microwave-assisted wetness impregnation showed good catalytic activity and reduced coke formation with the increasing amount of platinum doped [78]. Another focus for methane steam reforming is the utilization of microwave to overcome the limitation of heat transfer to the catalytic volume due to the endothermicity of methane steam reforming [79]. It was reported that the energy consumption is lower in microwave-assisted process compared to electrolyzers. With the optimized microwave reactor configuration, the energy efficiency of microwave-assisted methane steam reforming was increased by 23% compared to classical one and the CH_4 equilibrium conversion was achieved at 750 °C using Ni-based catalytic monolith [80].

Table 6. Catalyst synthesized using microwave-assisted technologies for methane reforming.

Catalyst Synthesized	Process	Textural Properties/ Composition	Yield	Remarks	Reference
Pt/CeO ₂	Wetness impregnation	BET specific surface area: 39.58 m ² /g Average particle size: 21 nm Carbon formation: 1.75 mmol/g	CH ₄ conversion: 71.4%	10% of Pt was doped Durable stability for 6 h	[78]
NiCo-MgAl ₂ O ₄	Two-step combustion	BET specific surface area: 35 m ² /g (fresh) and 33.5 m ² /g (used) Mean pore diameter: 28.45 nm (fresh) and 24.60 nm (used) Total pore volume: 0.26 cm ³ /g (fresh) and 0.21 cm ³ /g (used) Metal crystallite size: 19 nm (fresh) and 22 nm (used) Ni dispersion: 5.30% Lattice strain: 0.19 Mg: 20.87 wt% Al: 53.65 wt% Ni: 18.9 wt% Co: 7.08 wt%	CH ₄ conversion: 99.3%	Temperature: 750 °C Microwave power: 800 W CH ₄ :H ₂ O feed ratio = 1:1.12 Low carbon deposition (0.09% weight loss) after 15 h 0.0035 mg lamentous carbon deposited	[24]
Ni-MgAl ₂ O ₃	Two-step combustion	BET specific surface area: 41.69 m ² /g (fresh) and 36.2 m ² /g (used) Mean pore diameter: 23.11 nm (fresh) and 21.08 nm (used) Total pore volume: 0.24 cm ³ /g (fresh) and 0.191 cm ³ /g (used) Metal crystallite size: 29 nm (fresh) and 32 nm (used) Ni dispersion: 3.50% Lattice strain: 0.77 Mg: 20.84 wt% Al: 59.96 wt% Ni: 19.19 wt%	CH ₄ conversion: 97.4%	Temperature: 750 °C Microwave power: 800 W 0.0059 mg lamentous carbon deposited	[24]
Ni-Co/MFI zeolite	Hydrothermal	BET specific surface area: 380 m ² /g Specific micropore surface: 379 m ² /g Total pore volume: 0.194 cm ³ /g	CH ₄ conversion: 97.0% CO ₂ conversion: 99.0% H ₂ yield: 98.0% CO yield: 94.0%	Temperature: 950 °C	[81]
* Biochar	Pyrolysis	BET specific surface area: 39 m ² /g Microporous specific surface area: 26.18 m ² /g Total pore volume: 0.129 cm ³ /g Micropore volume: 0.021 cm ³ /g Average pore size: 1.322 nm K: 1.351 wt% Fe: 0.234 wt% Ca: 1.237 wt% Mg: 3.107 wt% Al: 0.244 wt% Na: 0.431 wt%	CH ₄ conversion: ~100% CO ₂ conversion: ~100%	Temperature: 800 °C Energy efficiency: 49.2%	[82]

Note: * used for combined reforming of methane by CO₂ and H₂O.

For CO₂ reforming, the complete transformation of CO₂ and CH₄ into syngas can be achieved with 68.4% and 96.8% conversion, respectively, under 6 kW microwave capacity [83]. A total conversion up to 90% was achieved with the 45–60 W of microwave power

input and no catalyst deactivation in the operation (6 h), using the mechanical mixture of Ni/Al₂O₃-SiC and Ni/SiC [76]. The mechanical mixture of SiC to the common catalyst, Ni/Al₂O₃ is required as Ni/Al₂O₃ cannot be heated up by microwave, which the resulted catalyst demonstrates to be cheap, stable and good candidate for the microwave-assisted CO₂ reforming process. Another study showed that Fe-based catalysts remained good catalytic activity in a stability test (around 50 h), with negligible carbon deposition after the long period [45]. The oxidized activated carbon as the microwave receptors/catalyst was reported to be not suitable for microwave-assisted CO₂ reforming compared to activated carbon, due to the presence of the oxygen surface group that reduces the catalytic activity of the carbon [84]. Except the influence of surface groups, microporosity is essential for the good catalyst made from carbon. Both methane reforming pathways require catalyst fabrication to either withstand the microwave power and remain the catalytic performance or to conduct the process with high energy efficiency.

Table 7. Methane reforming using microwave-assisted technologies.

Process	Catalyst/Absorbent	Textural Properties/Composition	Yield	Remarks	Reference
Methane steam reforming	Ni/CeO ₂ -Al ₂ O ₃ on a SiC monolith	Average pore diameter: 4.6 µm Specific surface area: 10.0 m ² /g SiC: 87.5 wt% Ni: 1.7 wt% Al ₂ O ₃ : 0.5 wt% CeO ₂ : 10.8 wt%	Equilibrium in H ₂ yield and CH ₄ conversion @800 °C (GHSV: 3300 h ⁻¹) and 850 °C (GHSV: 5000 h ⁻¹)	Pressure: 1 bar S/C: 3 Energy efficiency: 55% @1300 W (Power of microwave) Energy consumption: 3.8 kW/Nm ³ H ₂	[79]
Methane steam reforming	Ni/CeO ₂ -Al ₂ O ₃ on a SiC monolith	Pore distribution with radius: 3 nm Thickness of catalytic layer: 200 µm	Equilibrium in H ₂ yield and CH ₄ conversion @750 °C (GHSV: 5000 h ⁻¹)	Pressure: 1 bar S/C: 3 Energy efficiency: 73% Power of microwave: 400 W Energy consumption: 2.5 kWh/Nm ³ H ₂	[80]
CO ₂ dry reforming of methane	Fe/SiC	Specific surface area: 32.118 m ² /g (fresh) and 27.443 m ² /g (after 50 h) Pore size: 5–8 nm and small amount of 0–2 nm micropores ¹ C: 0.46; 22.16 ¹ O: 1.85; 35.29 ¹ Mg: 0; 2.88 ¹ Al: 3.36; 2.93 ¹ Si: 10.84; 33.87 ¹ Ca: 0.73; 0 ¹ Fe: 82.77; 0.22	CH ₄ and CO ₂ conversions: 85% H ₂ /CO ratio: ~1	Specific microwave power: 72 W/g Durable stability for 50 h Carbon deposition: ~0.78 wt%	[45]
Methane dry reforming	Fe-rich char from corn stalk	BET specific surface area: 150.46 m ² /g (fresh) and 139.18 m ² /g (during test) Total pore volume: 0.326 cm ³ /g (fresh) and 0.313 cm ³ /g (during test) Micropore volume: 0.309 cm ³ /g (fresh) and 0.269 cm ³ /g (during test) K: 0.956 wt% Fe: 7.126 wt% Ca: 0.443 wt% Mg: 1.523 wt% Al: 0.189 wt% Na: 0.367 wt%	CH ₄ conversion: 90.8% CO ₂ conversion: 95.2%	Fe ₂ O ₃ addition of 10% Temperature: 800 °C Syngas content: 88.79% H ₂ /CO ratio: 0.92 Durable stability for 160 min	[46]

Table 7. Cont.

Process	Catalyst/Absorbent	Textural Properties/Composition	Yield	Remarks	Reference
Methane dry reforming	Char from corn stalk	BET specific surface area: 30.86 m ² /g (fresh) and 25.98 m ² /g (during test) Total pore volume: 0.175 cm ³ /g (fresh) and 0.179 cm ³ /g (during test) Micropore volume: 0.137 cm ³ /g (fresh) and 0.116 cm ³ /g (during test) K: 0.740 wt% Fe: 0.052 wt% Ca: 0.505 wt% Mg: 1.606 wt% Al: 0.262 wt% Na: 0.030 wt%	CH ₄ conversion: ~57% CO ₂ conversion: ~80%	Temperature: 800 °C Syngas content: 88.79% H ₂ /CO ratio: 0.87 Durable stability for 160 min	[46]
Methane dry reforming	Ruthenium-doped SrTiO ₃ perovskite	SrTiO ₃ crystallite size: 29.8 nm Semi-quantitative weight percentage: 77.8 wt% BET specific surface area: 8 m ² /g Total pore volume: 0.026 cm ³ /g	CH ₄ conversion: ~99.5% CO ₂ conversion: ~94%	7 wt% of ruthenium doped H ₂ /CO ratio: ~0.9 Temperature: ~940 °C CH ₄ :CO ₂ vol.% feed ratio = 45:55 (maximized CH ₄ conversion)	[85]
Dry reforming of methane	Ni/SiC	-	CH ₄ conversion: 80% CO ₂ conversion: 90%	Temperature: 800 °C Short-term stability test for 6 h	[76]
Dry and bi-reforming of methane	Co-Mo/TiO ₂	BET specific surface area: 36.4 cm ² /g Uniform size distribution: 50–100 nm	CH ₄ conversion: 81% CO ₂ conversion: 86%	H ₂ /CO ratio: 0.9 Durable stability for >50 h	[86]
Dry and bi-reforming of methane	Cu-Mo/TiO ₂	-	CH ₄ conversion: 76% CO ₂ conversion: 62%	H ₂ /CO ratio: 0.8 Durable stability for >60 h	[86]
Methane dry reforming	Wood-derived activated carbon	BET specific surface area: 937.99 m ² /g Specific micropore surface: 353.65 m ² /g Specific pore volume: 0.61 cm ³ /g Specific micropore volume: 0.20 cm ³ /g	CH ₄ conversion: ~80.0% CO ₂ conversion: ~60.0%	Microwave power: 560 W CH ₄ /CO ₂ /N ₂ = 1:1:3 Total gas flow of H ₂ /CO ratio: 250 mL/min H ₂ /CO ratio: 1.3	[47]

¹ The element took from two different points from the fresh catalyst. GHSV: Gas hourly space velocity. @: at.

4.4. Sugar Conversion to 5-Hydroxymethylfurfural (5-HMF)

5-hydroxymethylfurfural (5-HMF), a furan-based compound, is a key intermediate for biofuel and fine chemical production which can be used to produce solvents, polymers, liquid fuels, plastics and pharmaceuticals etc. [87]. It can be derived from biomass and directly synthesized through the acid-catalyzed dehydration of C6 carbohydrates, fructose and glucose [88]. The other route to generate 5-HMF is hydrolyzing polysaccharides, trisaccharides or disaccharides into corresponding C6 monosaccharides and subsequently perform dehydration in single or biphasic system [51]. The isomerization of glucose from the biomass carbohydrate to fructose is required before converting it to 5-HMF.

The application of microwave is studied for the production of 5-HMF as the unique heating mechanism allows the quick conversion of components into 5-HMF in a short processing time. Table 8 has summarized the studies discussed within this section with their respective findings. The study of Zhang and Zhao have demonstrated the high

yield of 5-HMF (~60%) from lignocellulosic biomass, which only processed for 3 min under microwave irradiation (~400 W) using CrCl_3 as the catalyst [52]. Another study has reported 5 min of microwave irradiation time led to the maximum yield of 5-HMF in aqueous media while no obvious increment observed by extending the processing time up to 15 min [89]. Such short processing period showed that this process is an energy- and cost-effective process. Some novel catalysts have been studied for the mentioned reaction, such as sulfonated carbon microsphere catalyst and polyoxometalates for the production of 5-HMF. One of the advantages for these catalysts are high recyclability as sulfonated carbon microsphere catalyst can be reactivated using 1 M sulphuric acid solution whereas polyoxometalates can be recovered using diethyl ether with negligible loss of catalytic efficiency [90,91].

Besides, the ionic liquid (also known as green solvent) is a good medium to be utilized with microwave-assisted catalytic reaction for the production of 5-HMF. The usage of ionic liquid, [BMIM]Cl with the catalyst, ZrCl_4 showed synergistic effect with microwave irradiation in generating 5-HMF, and the catalytic activity still remained after several usage [53]. The study of Paul and Chakraborty was in agreement with the study of Liu et al. as both studies reported the application of ionic liquid as medium for an efficient process [53,92]. The combination of $\text{AlCl}_3 \cdot 6\text{H}_2\text{O}$ and [BMIM]Cl was successful to produce a total 5-HMF yield of 59.8 wt% at 150 °C in 20 min using corn starch as the feedstock [93]. Slight difference of 5-HMF yield was observed using waxy corn starch and high amylose corn starch as feedstock, which the later yielded lower amount of HMF compared to the previous. Another type of ionic liquid, [TMG]BF₄ as catalyst, was claimed to be effective to produce 5-HMF from microcrystalline cellulose [94]. However, recycle study has to be done for ionic liquid as it is highly expensive as compared to conventional organic solvent despite its wide application in various fields. Other than ionic liquids, acidic deep eutectic solvents (DES) were utilized to improve the production of 5-HMF. The study of Morais et al. have reported that DES was applied as both solvents and catalysts, which [Ch]Cl:CA with the ratio of 1:1 was used to obtain around 82.4% of 5-HMF yield. The recovery of 5-HMF from the DES medium was performed using bio-based solvent, enabling the recycle use of DES medium for other cycle of extraction up to 3 times [51].

Table 8. Sugar conversion using microwave-assisted technologies.

Process	Feedstock	Catalyst/ Absorbent	Yield	Remarks	Reference
Biphasic systems with aqueous and organic phase	Fructose	¹ Acidic deep eutectic solvents	HMF yield: 91.0%	Using [Ch]Cl:CA Microwave heating: 2 min Temperature: 120 °C Solid/liquid ratio: 0.05	[51]
Direct conversion using ionic liquids	Corn stalk, rice straw and pine wood	$\text{CrCl}_3 \cdot 6\text{H}_2\text{O}$	HMF yield: 45–52% Furfural yield: 23–31%	Irradiation time: 2–6 min	[52]
Direct conversion	Fructose	TiO_2 nanoparticles	HMF yield: 3.4% (commercial TiO_2), 25–54%	Microwave power: 300 W Temperature: 120–140 °C Time: 5–20 min	[89]
Direct conversion	Glucose	TiO_2 nanoparticles	HMF yield: 22.1–37.2%	Temperature: 120–140 °C Time: 2 or 5 min	[89]
Direct conversion	Sucrose	TiO_2 nanoparticles	HMF yield: 12.0–21.0%	Temperature: 120–140 °C Time: 5 or 10 min	[89]
Direct conversion	Cellobiose	TiO_2 nanoparticles	HMF yield: 14.5–18.7%	Temperature: 120 °C or 140 °C Time: 5 min	[89]
Direct conversion	Maltose	TiO_2 nanoparticles	HMF yield: 10.7–14.1%	Temperature: 120 °C or 140 °C Time: 5 min	[89]

Table 8. Cont.

Process	Feedstock	Catalyst/ Absorbent	Yield	Remarks	Reference
Direct conversion in water	Fructose	Sulfonated carbon microsphere catalysts	HMF yield: 88.3 mol%	Power: 60 W Temperature: 186 °C Time: 10 min Energy efficiency: 0.147 mmol/kJ	[90]
Direct conversion in DMSO-water	Chitin	Polyoxometalates: $H_4[SiW_{12}O_{40}]$	HMF yield: 23.1%	Solvent: 67% DMSO-water Temperature: 200 °C Time: 3 min	[91]
Direct conversion in ionic liquids	Cellulose	ZrCl ₄	HMF yield: 51.4%	Time: 3.5 min Power: 400 W Ionic liquid: [Bmim]Cl	[53]
Rapid catalytic conversion	Lignocellulosic Sunn hemp fibres	CuCl ₂	HMF yield: 26.8%	Temperature: 160–200 °C Time: 46 min Ionic liquid: [Bmim]Cl	[92]
Catalytic dehydration	Corn starch	AlCl ₃ ·6H ₂ O	HMF yield: 59.8 wt%	Solvent used: DMSO/[Bmim]Cl Temperature: 150 °C Time: 20 min	[93]
Direct conversion	Microcrystalline cellulose	Ionic liquid: [TMG]BF ₄	HMF yield: 28.63%	Temperature: 132 °C Time: 48 min Catalyst loading: 0.44 mg/mg	[94]

¹ Acts as both catalyst and solvent. DMSO: dimethyl sulfoxide.

4.5. Ammonia Synthesis

Ammonia, NH₃ as one of the most consumed chemicals, is commonly used as fertilizer, refrigerant gas and utilized in the production of pesticides, textiles, plastics, explosives and more. Around 80% of the ammonia produced by the industry is utilized as fertilizer and the society is constantly exposed to ammonia as it can be found in lots of household or industrial strength cleaning solutions [95]. Since ammonia consists of 3 hydrogen atoms and 1 nitrogen atom, the ammonia is currently being discussed for its potential in the significant energy transition to clean energy, which is hydrogen. A report of the potential roles of ammonia in hydrogen economy has been released by U.S. Department of Energy, reporting the usage of ammonia as hydrogen carrier during delivery to cut cost from using pipelines in transporting the hydrogen from central hydrogen production [96]. With the huge market demand, alternatives to Haber-Bosch process (conventional ammonia synthesis method) such as photochemical synthesis, electrochemical synthesis and microwave-assisted synthesis are explored to overcome the low product yield. Among these techniques, microwave-assisted synthesis allows selective heating at microwave-absorbing catalyst particles instead of heating the reaction gases, N₂ and H₂ [59].

Several aspects are considered essential as a good synthesis technique, for example high yield of end product, low energy consumption/less energy intensive, flexibility on the production scale, operation at mild condition and etc. Using microwave reactor to synthesis ammonia, the process can be performed in low temperature and pressure with the additional advantage, flexible to produce in small-scale distribution. Several studies related to ammonia synthesis via microwave-assisted technologies was shown in Table 9. The reporting of ammonia production at ambient pressure with low temperature, 300 °C is 10 times better using supported catalyst (e.g., Fe/Al₂O₃) compared to the bulk catalyst containing only single type of metal [97]. It was reported that a total amount of 0.25 g ammonia/g catalyst/day could be produced using Ru/MgO catalyst that fits for on-demand ammonia produced in small scale without high production cost [58]. Mild operating condition is performed for such yield, using microwave frequency of 2.45 GHz for 11 min and the operating temperature of 320 °C under ambient pressure. Higher metal ratio in catalyst also contributes to high stability, catalytic activity and energy efficiency due

to higher dispersity of Ru, preventing the sintering of catalyst. Relevant study conducted by Araia et al. is coherent with the statement of previous study as the increment of Cs and Ru loading in Cs-Ru/CeO₂ catalyst led to higher production of ammonia [57]. However, the ammonia synthesis was enhanced by increasing the operating pressure and reducing the ratio of H₂/N₂ under microwave irradiation [98]. From the mentioned study, Wang et al. reported that the ratio of H₂/N₂ influences the ammonia production, and the increment of gas hourly space velocity (GHSV) also leads to improvement of ammonia production rate. In short, optimization of parameters for ammonia synthesis, especially operating temperature, pressure and gas flowrate inclusive of ratio of gases injected have to be performed to gain the maximized ammonia production.

Table 9. Ammonia synthesis using microwave-assisted technologies.

Process	Catalyst	Yield	Remarks	Reference
Catalytic synthesis	Cs promoted Ru/CeO ₂	NH ₃ production rate: 1.18 mmol/h.g _{cat}	Temperature: ~260 °C Pressure: 0.1 MPa	[59]
Synthesis using non-plasma microwave system	Fe/Al ₂ O ₃	NH ₃ production rate: 128 µmol/g/h	Power: 300 W Temperature: 300 °C Ambient pressure 20 wt% of Fe is used	[97]
Synthesis using fixed microwave frequency	Ru/MgO	NH ₃ production rate: 0.25 g _{amm} /g _{cat} /day	Temperature: 320 °C Time: 11 min Microwave frequency: 2.45 GHz Ambient pressure 10 wt% of Ru is used	[58]
Catalytic synthesis	Cs-Ru/CeO ₂	NH ₃ production rate: 1313 µmol _{NH3} /g _{cat} .h	Temperature: 260 °C Ambient pressure 2 wt% of Cs and 4 wt% of Ru are used	[57]
Catalytic synthesis	Cs-Ru/CeO ₂	NH ₃ production rate: 0.04 g/g _{cat} .h	Temperature: 320 °C Pressure: 0.65 MPa H ₂ /N ₂ ratio: 1 Stable for 6 cycles of startup-shutdown operation	[98]
Catalytic synthesis	Fe promoted Co/γAl ₂ O ₃	NH ₃ production rate: 53.9 × 10 ⁻⁸ g ⁻¹ s ⁻¹ Total NH ₃ production: 441.5 × 10 ⁻⁸ mol	Temperature: 600 °C Pressure: 1 atm 0.5 wt% of Fe is used as promoter	[99]

5. Challenges and Future Prospect

Numerous studies have indicated that microwave irradiation is advantageous compared to conventional production techniques from the aspect of product yield, efficiency and wide range of applications. However, there are some challenges faced using microwave technology which need to be studied. Suitable catalysts have to be utilized as some catalysts are not reactive to microwave irradiation. Therefore, the selection of catalysts has to be specific, as microwave-absorbing catalysts are preferred to further enhance the process. The type of catalysts not only affects the interaction of catalyst with the microwave, but influences the coke formation/deposition on the surface of catalyst as well. The resulting coke deposition on catalysts' surface will hinder the microwave heating rate, which is proven in the study of Mohamed et al. [100]. For the process to be performed at mild condition, the ratio of metal used to produce catalysts have to be optimized to improve the stability of catalyst and maintain the catalytic performance. The fabrication of suitable catalysts is one of the challenges in microwave-assisted technology, where a catalyst that accelerates

the process and acts as a microwave absorbent at the same time should be applied to interact with microwave irradiation for the internal heating effects on the reactants. For the commercial-scale production, the large catalytic volume is another limitation in microwave technology. This is because the penetration depth of microwave to absorb matters is limited when the production capacity is scaled-up. Such phenomenon will result in the heating of reactants/absorbing matters by convection instead of the microwave dielectric heating for those positioned in the center of the reactor. Besides, the heat loss in large catalytic volume also needs to be considered which leads to the adjustment of power density. Heating up a large catalytic volume requires that a safe and reliable process setup, as well as robust reactor design to handle elevated temperature, have to be developed for safety purposes.

Despite the challenges stated above, microwave technology is continuously explored to deal with the mentioned issues for their advantageous offered in the applications. In future, more studies on the suitable type of microwave reactors in performing large catalytic volume are needed to obtain similar process efficiency as small-scale operation. One of the important aspects is the future need for microwave-absorbing catalysts, which able to withstand the extreme condition and absorb the electromagnetic energy for reaction. With the world moving forward to green fuel, microwave-absorbing catalysts are required for important reaction such as microwave plasma. For such process, a robust catalyst that being able to be reused and maintained their catalytic reactivity at high temperature with high stability is essential as most of the catalysts can be deactivated under high temperature. The extension studies of microwave technology can be linked with plasma technology in biofuel field, where this technology will be beneficial in various applications as well.

6. Conclusions

The application of microwave-absorbing catalysts in biofuel production have showed significant improvement in product yield, properties of char produced and high selectivity to preferred products with maximum energy efficiency and fast reaction period. The advantageous of microwave technology is based on the optimized reaction condition and applicable in bio-oil production, ammonia synthesis, 5-HMF production, methanation and methane reforming process. The major factor in influencing the success of microwave-assisted catalytic reaction is the catalyst, where the process is easily affected by the catalyst's stability and catalytic performance. However, more commercial scale studies are required for microwave technology to be established in the field of biofuel as the current application of microwave is mainly focusing on radar signals, satellite communications, medical treatments or households (food preparation). The microwave technology also has the potential to be incorporated with plasma technology in balancing the green energy transition with energy security through important reactions, leading to a more sustainable future.

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