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Review Article

Agricultural and Forest Residues towards Renewable Chemicals and Materials Using Microwave Liquefaction

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Microwave-assisted liquefaction is regarded as a promising thermochemical approach to produce renewable and sustainable chemicals and materials from lignocellulosic biomass. Agricultural and forest residues as sources of lignocellulosic biomass have great potential in this regard. With process optimizations, several biomass types have been subjected to liquefaction in different solvents with various catalysts. The products from recent microwave liquefaction with and without further fractionation have been thoroughly analyzed and used for the synthesis of biomaterials. Renewable chemicals, polyurethane foams with partial use of renewable raw materials, and phenolic resins have been the main products from microwave-liquefied products. Further research on microwave liquefaction mechanisms and scalable production should be enhanced to fully evaluate the economic and environmental benefits. This work presents an overview on achievements using liquefaction in combination with microwave energy to convert lignocellulosic biomass into value-added products and chemicals.

1. Introduction

Fuels, chemicals, and polymers derived from renewable and sustainable lignocellulosic biomass have attracted great interest because of the environmental issues caused by the extensive use of fossil fuels [1, 2]. Agricultural and forest processing residues are generally regarded as a green, renewable, and sustainable resource [3]. According to Zhang, in China, the bioenergy from agricultural and forest residues is made up of agriculture (74%) and forest (22%). The bioenergy potential of crop straw is equal to that of 201 million tons of standard coal (48.98%), followed by forestry residue (22%) and agricultural processing residues (12%), revealing that agricultural and forest residues have great potential for energy, chemical, and material production [4]. Generally, lignocellulosic biomass is mainly composed of cellulose, hemicellulose, and lignin, and their proportion differs in the types of forest and agricultural residues. For wood, the proportion of cellulose, hemicellulose, and lignin ranges from 25% (olive tree pruning) to 43.8% (spruce), 6.3% (spruce) to 28.5% (pinewood), and 16.2% (olive tree pruning) to 29.1% (red maple), respectively. As for agricultural residues, the proportion of the three components is 14.3% (corn fiber) to 43.1% (sugarcane bagasse), 16.8% (corn fiber) to 37.2% (barley hull), and 4.7% (corn pericarp) to 24.8% (bagasse), respectively [5]. Cellulose consists of a linear homopolysaccharide composed of β-D-glucopyranose units linked together by β-1-4 linkages (glucosidic bonds), and hemicelluloses are polysaccharides consisting of β-(1→4)-linked backbones. Lignin is composed of three different phenyl propane units, namely, syringyl alcohol, guaiacyl alcohol, and p-coumaryl alcohol [6]. The native chemical structures of lignocellulosic biomass make lignocellulosic biomass the source of sustainable products.

With the rapid development of thermochemical conversions of lignocellulosic biomass, the utilization of lignocellulosic biomass includes the fabrication of composites, either with or without nonbiobased feedstocks, to the generation of fine chemicals and renewable energy (Figure 1). Thermochemical conversion methods including combustion, gasification, pyrolysis, and liquefaction are common pathways in

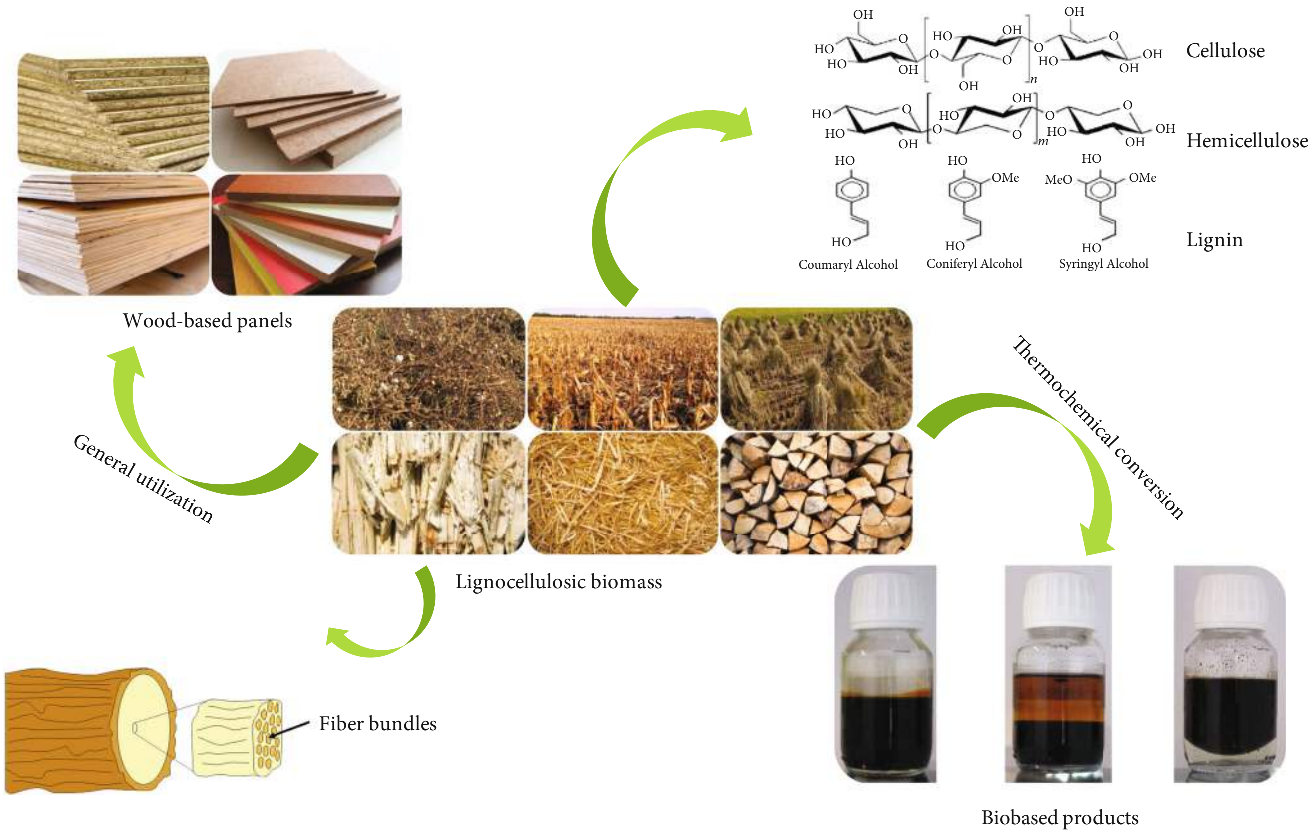

Figure 1: Products of lignocellulosic biomass.

converting lignocellulosic biomass to renewable energy and chemicals [7]. For comparison, liquefaction is generally carried out in organic solvents at a relatively low temperature to convert lignocellulosic biomass into small molecular substances; meanwhile, liquefaction products are directly used as ingredients or refinement for further usage [2].

This paper gives an overall report on lignocellulosic biomass sources and liquefaction reagents used in microwave liquefaction as well as process optimization. In addition, the analysis and applications of microwave-liquefied products are also summarized and discussed. This review mainly summarizes the recent research achievements on microwave-assisted liquefaction of lignocellulosic biomass and is anticipated to initiate further research to facilitate the production of renewable chemicals and materials from microwave liquefaction.

2. Microwave Liquefaction versus Conventional Liquefaction

The conventional liquefaction process of lignocellulosic biomass was carried out using conduction/convection heating by oil bath or heating mantle, which usually requires a long reaction time and yields a low liquefaction rate, resulting in low efficiency. In order to have a more complete reaction in conventional liquefaction, large amounts of solvents such as phenol have typically been used but are inconsistent with an environmentally friendly and sustainable concept [8–12].

Microwave irradiation is categorized as electromagnetic irradiation in the range of wavelengths from 0.01 m to 1 m with corresponding frequency of 300-0.3 gigahertz (GHz) [13]. Microwave heating is the direct coupling of microwave energy with molecules. Therefore, microwave heating occurs over the entire volume of material. Due to the advantages of microwave heating, microwave irradiation method have been applied to chemical synthesis [14], extraction [15, 16], gasification [17], pyrolysis [18], and liquefaction [19, 20]. Compared with conventional liquefaction, microwave-assisted liquefaction is more efficient. Previous research had shown that the application of microwave energy on liquefaction could enhance the liquefaction extent [19–22]. Previous research also indicated that microwave heating influenced the properties of the liquefied products; chemicals from microwave liquefaction processed much smaller average molecular weight compared with those from conventional liquefaction [21, 23].

3. Lignocellulosic Biomass Sources for Microwave Liquefaction

3.1. Biomass Type. Since Krzan et al. first applied microwave energy to liquefaction [19, 20], a variety of biomass types including sweetgum sawdust [8]; agricultural residues including bagasse, corn stover, rice straw, wheat straw, cotton stalk, and corncobs [24–26]; organosolv lignin of olive tree prunings [27]; *Sargassum polycystum* C. Agardh [28]; microalgae [29]; *Ulva prolifera* [30]; wheat straw alkali lignin

[31]; sugar beet pulp [32]; peony oil palm empty fruit bunch fiber [16]; oil palm empty fruit bunch cellulose [33]; poplar; Chinese fir; bamboo; hemp xyloid stem [22]; mixed softwood pellets [34]; banana pseudostem [35]; coconut fiber [36]; and grapefruit [37] have been studied as raw materials for microwave-assisted liquefaction for the production of renewable chemical platforms and/or biobased materials.

The chemical composition (cellulose, hemicellulose, lignin, and ash) and microstructure of the lignocellulose feedstocks greatly differed with each other, which may influence their liquefaction behaviors under microwave heating. Studies have shown that the conversion yield of corn stover, rice straw, and bagasse is variable [12, 24, 38, 39]. Xiao et al. [25] made a comparison of the liquefaction behaviors of different agricultural crop residues under the same microwave-assisted liquefaction conditions, and the results revealed that the liquefaction rate of rice straw was much lower than that of wheat straw, corn stover, and corncobs. Li et al. studied five types of microwave-liquefied biomass, and the results showed that poplar and Chinese fir were the most easily liquefied, followed by bamboo and hemp xyloid stem. While comparing with the other four feedstock materials, the rice straw exhibited the lowest liquefaction degree [22].

Research results on microwave liquefaction of bamboo showed that the liquefied bamboo residue rate could be less than 5% [40, 41]. Despite the high liquefaction rate of bamboo in different solvents under microwave heating, further study revealed that the higher percentages of thin cell wall tissues as well as lower cellulose and lignin contents resulted in a lower residue content. It indicated that the anatomical characteristic and chemical components could affect the microwave-assisted liquefaction process of bamboo [42, 43]. The difference in the conversion yield between the liquefaction of poplar, fir, and woody underbrush may also provide evidence that structure and chemical compositions can influence the liquefaction behaviors since significant differences exist between softwood, hardwood, and agricultural residues [40, 44–47].

Similar to the varying liquefaction results for different ligneous plants, the species of microalgae was also found to be an important factor. Other raw materials that have been studied and found to influence liquefaction conversion and product properties include type of waste paper and source of lignin [27, 28, 30, 48, 49].

3.2. Biomass Particle Size. The use of small particle size greatly improved the liquefaction efficiency and reduced the amount of the solvent required. Zheng et al. found that the liquefied residue content of sugar beet pulp with a particle size of 75-177 μm reduced much faster than that with a size of 177-841 μm [32]. Li found that when the particle size of poplar wood decreased from 0.38-0.83 mm to 0.18-0.25 mm, the liquefaction rate increased from 58.5% to 87.0% [45]. The results on microwave liquefaction of bamboo revealed that smaller size of feedstocks could shorten the time of liquefaction when the temperature was 120°C, which may be because the smaller the particles, the easier the full material can be accessed by the solvent and lignin recondensation that occurs when the temperature is 140°C [50].

3.3. Moisture Content. Moisture of the raw materials is another factor that can influence liquefaction behavior. The optimal moisture content of poplar wood for microwave liquefaction in phenol was 30-40% [45]. The relation between moisture content and liquefaction yield of *Ulva prolifera* showed that the liquefaction yield increased with increasing moisture content and then tended to decrease with a further increase in moisture and showed a maximum at 8%. This may be because the excessive moisture content caused a decrease in reaction temperature and solvent concentration, and the proper amount of moisture was beneficial to microwave absorption and hydrolysis. At the same time, water molecules are polar molecules; proper moisture content could enhance the ability of absorbing microwave energy and increase the heating rate of a wood liquefaction system [51].

4. Liquefaction Reagents for Microwave Liquefaction

Liquefaction reagents including solvents and catalysts are dominating factors influencing the liquefaction process [52]. Proper liquefaction reagents can make the feedstock particles more evenly dispersed in the solid-liquid system and promote the liquefaction reaction. Meanwhile, the liquefaction efficiency and product quality and applications are also largely dependent on the reagents employed.

4.1. Solvents. Compared with higher analogues, i.e., diethylene glycol, dipropylene glycol, and polyethylene glycol, simple glycols, i.e., ethylene glycol (EG) and propylene glycol (PG), showed higher liquefaction efficiency for the liquefaction of dry poplar sawdust using microwave energy. The liquefaction rates of simple glycols were 85% and 99% (EG and PG, respectively), while those of higher analogues were only 63-69% [53]. The liquefaction efficiency of wheat straw alkali lignin in alcohols was in the decreasing order of methanol, glycol, propanol, glycerol, alcohol, and butanol, which indicated that methanol was a preferable solvent. This finding was attributed to the larger dipole moment of methanol, resulting in a better absorbance of microwave energy [31]. According to the yield of liquid products, more yield indicates better liquefaction efficiency. The use of methanol and ethanol for the liquefaction of organosolv lignin showed the best liquefaction efficiency compared to that of butanol, ethanediol, and isopropanol. Meanwhile, the molecular weight of the generated chemicals was much different. The weight average molecular weight (Mw), number average molecular weight (Mn), and Mw/Mn of isopropanol were 1919, 388, and 4.95, respectively. The Mw of butanol and ethanediol possessed similar values, 1522 and 1517, but the Mw/Mn of ethanediol was narrower than that for butanol. The Mw of methanol and ethanol exhibited lower values (1164 and 1162, respectively) compared with butanol, ethanediol, and isopropanol. The reason may be that both methanol and ethanol are lower molecular weight alcohols, showing higher nucleophilic activity, and could promote the C-O-C cleavage of lignin in solvolytic reaction [54]. In terms of methanol, ethanol, water, polyethylene glycerol, and glycerol, with increasing the reaction temperature from 120°C, the liquefaction yield of bamboo in polyethylene glycerol and glycerol decreased, which revealed that polyethylene glycerol and glycerol were two desirable and energy-saving liquefaction solvents under lower reaction temperature using microwave energy [55].

As the concentration of methanol in glycerol increased, the content of 5-hydroxymethylfurfural and levulinic acid decreased, and the total content of sugar derivatives showed the same trend by increasing the methanol content. An apparent conclusion was that methanol could enhance the liquefaction efficiency of the feedstocks; on the other hand, it could also prevent further decomposition of the sugar derivatives [56].

In addition to the solvent type, the ratio of solvent to solid also has an effect on liquefaction yield. The overall solvent to solid ratio in a microwave liquefaction system was lower than that in conventional liquefaction. The optimal phenol to polar wood ratio for maximum liquefaction yield and economic efficiency was 5/2 [45]. As the solvent to coconut fiber increased from 3.3 : 1 to 5 : 1, the liquefaction rate increased from 56% to 89% and remained constant with further increase in the solvent proportion [36]. The liquefaction yield of varied solvents from different raw materials are shown in Table 1. As shown in Table 1, liquefaction using glycol and methanol as cosolvent exhibited excellent liquefaction rate compared to other solvents. Besides the types of solvents being able to influence the liquefaction rate, the concentration of solvents and the ratio of dual components solvents also have an effect on the liquefaction rate.

4.2. Catalyst. A catalyst has been used in almost every liquefaction process using either conventional or microwave heating. In the liquefaction of most lignocellulosic biomass using microwave energy, sulfuric acid has been identified as a prevailing catalyst [57–60]. A variety of acids (sulfuric acid, hydrochloric acid, phosphoric acid, and formic acid) have been used in the microwave liquefaction of the components of lignocellulosic biomass. The results confirmed that sulfuric acid was also a good choice in the liquefaction of lignin [61]. Sulfuric acid was found to be the most influential factor on the conversion of lignin compared to time and lignin concentration [60]. Furthermore, sulfuric acid was more efficient for the production of monophenolic products from liquefaction of lignin than the zeolite and FeS binary catalyst [62]. Sulfuric acid, sodium hydroxide, levulinic acid, and p-toluene sulfonic acid monohydrate (PTSA) (98%) were used in the microwave-assisted conversion of cellulose, and their efficiency was comparatively elucidated. The results indicated that sulfuric acid was a strong mineral acid catalyst and was proposed as the preferable catalyst for microwave liquefaction of cellulosic materials [63].

Kong et al. [64] studied the influence of Fe_2O_3-modified HY (Si/Al ratio 5.0) on the bio-oil liquefaction yield of *Ulva prolifera*. It was found that the doped Fe_2O_3 enhanced the total acid sites of the catalyst, which distinctly increased the bio-oil yield from 40.6 to 52.6%, demonstrating that the doped amounts of Fe_2O_3 to the HY (Si/Al ratio 5.0) obviously improved the catalytic performance for the liquefaction of *Ulva prolifera*.

The changes in the catalyst concentration or amount in the feedstock of a microwave liquefaction system also affect liquefaction behaviors. Previous research results revealed that the liquefaction yield of biomass first increased by increasing the catalyst concentration, while further increasing the concentration the liquefaction yield decreased [36, 46]. The proper concentration of sulfuric acid for most lignocellulosic biomass using microwave energy was 3-5%, which was relatively lower than that used for conventional liquefaction. For example, the optimized sulfuric acid concentration for the liquefaction of *Ulva prolifera* and Kraft lignin was 5% and 3%, respectively [30, 65].

5. Microwave Liquefaction Process Optimization

From the above, in the process of microwave liquefaction, we can conclude that raw materials, liquefaction reagents (solvents, catalysts) have significant effects on the conversion yield and the properties of the end products. On the other hand, the microwave power, liquefaction temperature, and time also affect the conversion yield and quality of the products.

5.1. Temperature and Time. Temperature and time are the main factors influencing liquefaction behaviors and properties of the liquefied products in the microwave heating system. The effect on the liquefaction yield of rice straw was in the decreasing order of reaction temperature, catalyst amount, solvent to solid ration, reaction time, phenol to glycerol ratio, and microwave power [38]. With microwave liquefaction of rice straw, temperature was identified as the most influential factor. A study on the microwave liquefaction of poplar wood sawdust also indicated that the most influential factor was temperature [44]. Research results on the microwave liquefaction of other lignocellulosic biomass also revealed that the most important factor influencing the liquefaction was temperature [24, 41, 58, 66, 67]. The depolymerization of ethanol organosolv lignin from bamboo with low-power microwave showed that temperature was more influential than reaction time [68]. The significant microwave liquefaction factor is temperature, and the reason may be that temperature enhances and attacks solvents and catalysts onto glycosidic linkages resulting in the decomposition of cellulose into small molecules [69].

The optimum liquefaction temperature and time for oil empty fruit bunch fiber and oil palm empty fruit bunch fiber cellulose was 160°C with 15 min and 175°C with 40 min [33]. The research results on the liquefaction of coconut fiber indicated that the optimal reaction temperature was 160°C [36].

Table 1: Liquefaction yield of varied solvents from different raw materials.

Feedstock	Solvent			Liquefaction rate (%)	Ref.
Poplar sawdust	Simple glycols		Ethylene glycol	85	[20]
			Propylene glycol	99	
	Higher analogues with glycols		Diethylene glycol	63	
			Dipropylene glycol	62	
			Polyethylene glycol	69	
Wheat straw alkali lignin			Butanol	10-12.5	[31]
			Alcohol	12.5-15	
			Glycerol	About17.5	
			Propanol	About 20	
			Glycol	20-22.5	
			Methanol	25-27.5	
Ethanol organosolv lignin (from bamboo culms)			Methanol	84.86	[54]
			Ethanol	84.22	
			Butanol	49.3	
			Ethanediol	38	
			Isopropanol	27	
Bamboo (*Phyllostachys pubescens*)			Glycerol	96.71	[56]
			PEG400#	74.1	
			Methanol	36.47	
			Ethanol	24.97	
			Water	17.74	
Corn stover			Ethylene glycol (EG)	92.47	[38]
			PEG400/glycerin (4 : 1)	92.42	
			Glycerin	72.89	
Pine (*Pinus* sp.) tree sawdust	Concentration of solvents	Concentration of methanol (%)	0	48.5	[55]
			25	55.4	
			50	62.7	
			100	76.3	
Peony seeds			Petroleum ether	23.8	[16]
			Hexane	24.0	
			Hexane+ethanol	24.4	
Fir sawdust		The mass ratio of PEG400/glycerol	0.5	About 82	[46]
			4.0	90.5	
Wood underbrush		Glycerol to EG ratio	1.0	79.3	[47]
			2.0	88.1	
			5.0	84.4	

The liquefaction yield of *Sargassum polycystum* C. Agardh gradually increased when the temperature was between 130 and 170°C and then slowly decreased when the temperature increased to 190°C. Similar to the effect of temperature, the whole reaction was also divided into two stages by reaction time; i.e., the liquefaction yield dramatically increased in the initial 15 min and tended to keep constant from 15 to 35 min [28]. Similar results on the relationship between temperature, reaction time, and liquefaction yield of bamboo have also been observed [58, 59].

From these results, it can be concluded that liquefaction yield is influenced by interactions existing between temperature and time. The conversion yield of rape straw remarkably increased and finally reached 87% (180°C, 10 min) by increasing the liquefaction temperature and prolonging the reaction time [67]. With the temperature at 120°C and isopropanol as the solvent, lignin was converted into aromatic compounds (mainly ethanone, 1-(4-hydroxy-3-methoxyphenyl), and ethanone, 1-(4-hydroxy-3, 5-dimethoxy phenyl)) and the yield of the liquid product was 45%. However, from 120°C to 180°C, the liquefaction rate gradually declined to 37% and the proportion of residues increased [70]. The above analysis indicated that the prevailing reaction temperature for most lignocellulosic biomass except for lignin was in the range of 150-180°C depending on the biomass type and reagents used and the preferable time was less than 1 h. Further

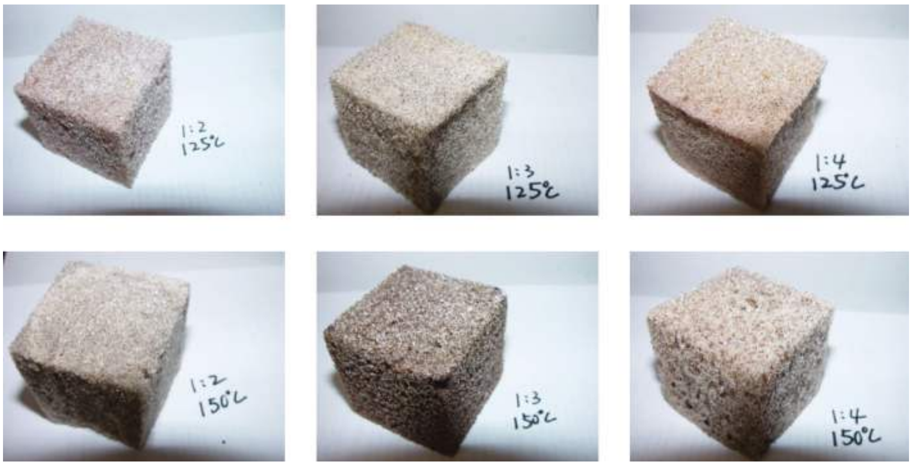

Figure 2: Photographs of polyurethane foams prepared from microwave-liquefied bagasse [60].

increases in temperature or prolonging the reaction time may result in a decrease in conversion yield and recondensation of the already decomposed fragments.

5.2. Microwave Power. As microwave power increases, the heating rate of the liquefaction system increases. With the orthogonal experiment (refers to an approach to analyzing a representative test site to achieve process optimization) [71] on microwave liquefaction of corn stover, Li et al. [39] found that microwave power was the most influential factor. Guo et al. [28] reported that with increasing microwave power from 200 W to 400 W, the liquefaction yield gradually increased but the liquefaction yield clearly decreased when the power was increased to 600 W. Chai and Tao [40] found that the residue content of liquefied bamboo first decreased and then increased as the microwave power increased. The minimum residue content of bamboo was as low as 3.4% when the maximum microwave power was 500 W. The yield of bio-oil from the liquefaction of *Ulva prolifera* increased as the microwave power was 200-600 W, while the yield decreased as the microwave power was 600-800 W. The decrease of the liquefaction yield with high microwave power may be because bio-oils could easily undergo recondensation [51].

6. Microwave Liquefaction Products and Utilization

Biopolymers are commonly considered as renewable, biodegradable, and ecofriendly materials. They can be obtained completely or partially from renewable resources and synthesized like traditional plastics [72]. Compared to polymers based on petroleum, biopolymers derived from renewable resources avoid fossil fuels and the relative amount of the net carbon released is much smaller [73].

For microwave liquefaction, the liquefaction products usually can be divided into two portions, the liquid and the solid parts. Generally, the liquefaction reaction mixtures will be filtered through filter paper, and solid parts are retained on the filter paper, and liquid parts are filtered through the filter paper. The chemical composition of the two parts is different.

By replacing the raw materials with liquefied products, it can not only increase the biodegradability of bioproducts but also lower the cost of production. There are different kinds of biobased polymer products from microwave liquefaction, which can be applied in many fields, such as biobased polyurethane foams, biobased adhesives, and cellulose-reinforced materials.

6.1. Integrated Utilization of Liquefied Liquid. In most studies, the liquid products from the microwave liquefaction of lignocellulosic biomass were analyzed and applied to polyurethane foam (PUR) and phenol resins depending on the solvents used. Various lignocellulosic biomass types such as poplar, southern pine, bamboo, bagasse, agricultural residues, and lignin have been microwave liquefied in alcohol solvents to produce PU foams. As shown in Figure 2, under different liquefaction conditions (the mass ratio of bagasse flour and biocomponent polyhydric alcohol was 1 : 2, 1 : 3, and 1 : 4 and the temperature was 125°C and 150°C), all the synthesized PU foams were of rigid type and the foam was darker in color with the addition of the liquefied bagasse [49, 60, 74–80]. The properties of the fabricated biobased foams from microwave-liquefied products were largely dependent on the biomass type, heating methods, and liquefaction conditions. Research results with corn stover revealed that the PU foams with the addition of microwave liquefaction materials had favorable biodegradability but poor thermal stability compared to those synthesized with conventional liquefaction. The poor thermal stability is mainly because the molecular weight of the microwave liquefaction product of corn stover is lower than that of the conventional liquefied product, resulting in a decrease in the degree of crosslinking of the synthesized polyurethane [26]. The application of the proper amount (less than 10%) of lignin fractionated from the microwave-assisted liquefied switchgrass into the PU matrix could improve the performances of PU foams in the apparent density, mechanical strength, and thermal stability [79]. By adding 20% biopolyol from microwave-liquefied rape straw, the biofoam cell diameter decreased by 90% compared with the PU without biopolyol from rape straw and the foam cell became more homogenous and finer, and the PU foams exhibited extremely low thermal conductivity and excellent mechanical strength. The further addition of 4% cellulose nanocrystals (CNCs) increased Young's modulus and compressive stress by 590% and 150%, respectively [80]. The use of liquefaction biopolyol with the presence of solid residue could enhance the thermal stability of the biofoams [81]. By adding the liquefied products of bagasse from high reaction temperature, the PU foams possessed better physical and mechanical performance compared with those prepared with liquefied products from low temperature. High polyhydric alcohol concentration in the liquefied bagasse resulted in higher thermal stability of the biofoams [60]. In the preparation of the rigid PU foams from microwave-liquefied lignin products without any pretreatment, by increasing the [NOC]/[OH] ratio from 0.6 to 1.0, the compressive strength of the foams increased [78]. By increasing the biopolyol proportion from microwave-liquefied lignin (from 25% to 50%), the apparent density, compressive strength, and thermal stability of the obtained biobased foams all increased [49]. Research also indicated that the lignin derivatives from the microwave-liquefied bamboo biopolyols could increase the density and thermal stability of the biofoams [74].

In addition to the application of the liquefied liquid in the preparation of PU foams, adhesives and polyether polyols have also been made from biopolyols from microwave liquefaction. Under the presence of MgO, phenolic compounds were obtained from microwave-assisted liquefaction of bamboo and were successfully applied to synthesizing fast-curing phenol-formaldehyde resins [82]. Biomass-based polyether polyols were prepared from the microwave-liquefied poplar products with double metal cyanide MMC as a catalyst [77]. The main compositions of bio-oil from microwave liquefaction are listed in Table 2. As shown in Table 2, bio-oil from different feedstocks showed different compositions. Under different liquefaction conditions, main compositions from the same feedstock are also different. Bio-oil from cotton stalk, rape straw, *S. polycystum* C. Agardh, *Ulva prolifera*, and *Nannochloropsis oceanica* has similar composition with different content.

6.2. Fractionation and Utilization of Liquefied Products.

The compositions of the microwave-liquefied products were complex due to the complex composition of the lignocellulosic biomass. Fractionation of the liquefied products was an efficient method to realize the high value of the liquefied products and expand their utilization. Xu et al. [57] successfully separated the liquid products from the microwave-liquefied lignocellulosic biomass into the phenolic rich fraction (phenolic compounds) and the sugar derivative fraction (biopolyols) via a simple method. With microwave-assisted liquefaction of bamboo, aromatic and monosaccharide derivatives were also successfully obtained with stepwise precipitation and extraction [83]. The content of glycoside compounds in the monosaccharides was 92%, and the content of the phenolic compounds in the aromatic fraction was approximately 82% [84]. The water-soluble fraction of the liquefied corn stover was extracted with hexane, chloroform, diethyl ether, and ethyl acetate. The results revealed that the content of levulinic acid isopropyl ester was higher in the chloroform and ethyl acetate, while the hexane and ether extract had a lower levulinic acid isopropyl ester content [85]. Lignin recovered from the microwave liquefaction of lignocellulosic biomass (moso bamboo, Chinese tallow tree wood, switchgrass, and pine wood) in a binary glycerol/methanol solvent retained the core structure of lignin and had high solubility in common solvents. The addition of the fractionated lignin from microwave liquefaction into a polylactide (PLA) matrix improved the tensile properties of PLA-lignin composites, and the PLA films with low lignin content possessed good UV light-resistant properties [86]. Detoxified cellulosic components and lignin fractions were successfully fractionated from microwave-liquefied decommissioned chromate copper arsenate- (CCA-) treated wood poles, and the metals were almost completely distributed in the spent liquor [87].

6.3. Characterization and Utilization of Liquefied Residues.

Research on microwave liquefaction of lignocellulosic biomass has also focused on process optimization and utilizations of the liquid products. In order to make full use of biomass to achieve close-loop utilization of the resource via microwave liquefaction, research on the characterization and potential utilization of liquefied residues was performed in several studies. Morphology, chemical structure, and thermal stability of the microwave-liquefied residues were thoroughly investigated to obtain a better understanding of the microwave liquefaction behaviors of biomass [43, 55, 88]. Further research found that the microwave-liquefied bamboo residues still retained the cell wall structure and tracing cellulose, and pure white cellulose fibers could be extracted from the residues with a mild bleaching process. Further treatment of the cellulose yielded nanofibrillated cellulosic fibers with lengths greater than 550 nm (Figure 3) [50, 89, 90].

7. Liquefaction Mechanism on Microwave Liquefaction

Previous research on the mechanism of microwave liquefaction is limited. In one study, the chemical reaction mechanism on microwave liquefaction in methanol was proposed [57]. Xie et al. proposed that selective liquefaction of bamboo cell wall components existed during microwave liquefaction based on the findings that lignin and extractives were more easily decomposed than cellulose, and the proposed selective liquefaction process has been used to produce high value cellulose nanofibers (Figure 4) [89, 91]. However, previous research did not provide any evidence to indicate if microwave energy affected the chemical reactions or selective liquefaction of cell wall components.

8. Conclusions

This report gives a review on recent research on microwave-assisted liquefaction of lignocellulosic biomass. Microwave

Table 2: Main compositions of bio-oil from microwave liquefaction.

Feedstock	Particle size	Microwave power (W)	Temperature (°C)	Time (min)	Solvent	Catalyst	Main compositions	Area (%)	Yield of bio-oil (%)	Ref.
Peony	20 mesh	80.92	80.92	3.72	Hexane +ethanol		Palmitic acid		34.49	[16]
							Stearic acid			
							Oleic acid			
							Linoleic acid			
							Linolenic acid			
Cotton stalk	60 mesh	500	150	30	Ethylene glycol	HZSM-5	Benzonitrile	2.30		[23]
							1-Propanone	5.86		
							Thiomorpholine	1.89		
							Cyclohexaneethanol	1.01		
							8-Heptadecene	2.10		
							Hexadecanoic acid, methyl ester	1.82		
							2-(Prop-2-enoyloxy) tridecane	2.93		
							9,12-Octadecadienoic acid, methyl	3.37		
							Oleic acid	33.07		
							1-Hexyl-1-nitrocyclohexane	6.13		
							9-Octadecenoic acid (Z)-, methyl ester	14.05		
							1-Cyano-4-(5-hexenyl)benzene	6.14		
							E-2-Octadecadecen-1-ol	3.33		
							Pentadec-7-ene, 7-bromomethyl-	3.22		
							2-(3-Hydroxybutyl)cyclooctanone	1.65		
S. polycystum C. Agardh	60 mesh	400	170	15	Ethylene glycol	H_2SO_4	Diethylene glycol	44.94	87.70	[28]
							Hexadecanoic acid, methyl ester	5.40		
							Methyl oleate	22.20		
							Octadecanoic acid, methyl ester	6.99		
							Squalene	1.35		
Ulva prolifera	60 mesh	600	165	30	Ethylene glycol	H_2SO_4	Diethylene glycol	38.51	93.17	[30]
							Phthalic acid, dipropyl ester	0.94		
							Phthalic acid, ethyl pentyl ester	0.90		
							Hexadecanoic acid, methyl ester	5.80		
							9-Octadecenoic acid (Z)-, methyl ester	27.50		
							Octadecanoic acid, methyl ester	5.81		
							9,15-Octadecadienoic acid, methyl ester	0.54		
							Eicosanoic acid, methyl ester	0.78		
							Phthalic acid, 2-ethylhexyl isohexyl ester	7.96		
							2,6,10,14,18,22-Tetracosahexaene	0.71		

Table 2: Continued.

Feedstock	Particle size	Microwave power (W)	Temperature (°C)	Time (min)	Solvent	Catalyst	Main compositions	Area (%)	Yield of bio-oil (%)	Ref.
Wheat straw alkali lignin	38 μm	300	160	5	Methanol	Ferric sulfate	Vanillin	11.23	55.22	[31]
							Acetovanillone	5.79		
							Vanillic acid	3.76		
							Coniferyl alcohol	2.38		
							2,6-Dimethoxyphenol	2.03		
							Syringaldehyde	13.38		
							4-Allyl-2,6-dimethoxyphenol	5.14		
							Acetosyringone	29.22		
							4-Hydroxy-3,5-dimethoxybenzohydrazide	2.63		
							Syringic acid	5.32		
							4-Hydroxy-2-methylacetophenone	8.37		
							2,3-Dihydrobenzofuran	5.27		
							Desaspidinol	2.45		
Ulva prolifera		600	180	30	Ethylene glycol	H_2SO_4	Benzenecarboxylic acid	4.32	84.81	[51]
							Diethyl phthalate	1.96		
							Hexadecanoic acid, methyl ester	0.94		
							Tridecanoic acid	13.32		
							Octadecanoic acid, methyl ester	2.03		
							9,12-Octadecadienoic acid	42.58		
							(R)-(-)-14-Methyl-8-hexadecyn-1-ol	1.21		
							Cyclopropaneoctanal	0.52		
Ulva prolifera	60 mesh		150	30	Ethylene glycol	8% Fe/HY*	5,6-Epoxy-2,2-dimethyl-3-heptyne	2.87		[65]
							2-Butanol, 3-methyl-, (S)-	0.76		
							Butanedinitrile	0.69		
							Thiomorpholine	1.05		
							Benzaldehyde, oxime, (Z)-	3.96		
							Hydrazinecarboxamide, 2-(phenylmethylene)-	1.09		
							1,1-Cyclohexanedimethanol	1.16		
							2-Amino-5-methylbenzoic acid	4.42		
							Benzene, [2-(1-propoxyethoxy)ethyl]-	3.34		
							Ethanone, 1-(2-benzothiazolyl)-	2.17		
							1-Diethoxymethyladamantane	3.00		
							1-Methoxy-1,4-cyclohexadiene	0.50		
							Benzonitrile	6.59		

Table 2: Continued.

Feedstock	Particle size	Microwave power (W)	Temperature (°C)	Time (min)	Solvent	Catalyst	Main compositions	Area (%)	Yield of bio-oil (%)	Ref.
							Cyclobutanecarboxylic acid, 2-methyl-, methyl ester	1.07		
							1-Propene, 2-chloro-	2.04		
							Hexahydroindole	1.36		
							cis-1,4-Dimethyl-2-methylenecyclohexane	0.71		
							3-Nitrostyrene	1.03		
							1,2-Dihexylcyclopropene	2.10		
							Hexadecanoic acid, methyl ester	3.05		
							1,2-Benzenedicarboxylic acid, mono(2-ethylhexyl) ester	1.54		
							6,7-Dimethyl-triazolo(3,4-c)(1,2,4)-triazine	0.56		
							Cyclooctyl alcohol	0.98		
							9,12-Octadecadienoic acid, methyl ester	9.25		
							9-Octadecenoic acid (Z)-, methyl ester	16.62		
							Octadecanoic acid, methyl ester	1.71		
							Hexanoic acid, hexadecyl ester	14.31		
							3-Methyl-2,5-oxazolidine-dione	0.87		
							Benzaldehyde, 2-hydroxy-3-nitro-	0.60		
							9(10H)-Acridinone, 1-hydroxy-2,3,4-trimethoxy-10-methyl-	2.83		
							2,2,4-Trimethyl-3-(3,8,12,16-tetramethyl-heptadeca-3,7,11,15-tetraenyl)-cyclohexanol	1.47		
Rape straw	20-40 mesh	700	140	15	Methanol	H_2SO_4	Furan	1.16		[67]
							3-Acetoxy-3-hydroxypropionic acid, methyl ester	3.02		
							5-Amino-3H-[1,2,3]triazole-4-carbo	1.03		
							Hexadecanoic acid, methyl ester	1.13		
							Citric acid, trimethyl ester	1.92		
							α-D-Ribopyranoside, methyl	24.84		
							2H-Pyran-3,4,5-triol, tetrahydro-2-methoxy-6-methyl-	3.41		
							α-D-Lyxofuranoside, methyl	1.55		
							α-D-Ribopyranoside, methyl	9.77		
							Methyl(methyl4-O-methyl-α-D-mannopyranoside)uronate	7.44		
							Benzoic acid, 4-hydroxy-3,5-dimethoxy-, hydrazide	1.00		
							Methyl(methyl4-O-methyl-α-D-mannopyranoside)uronate	2.04		
							3-Methylmannoside	6.69		
							α-D-Galactopyranosiduronic acid, methyl, methyl ester	3.84		
							α-D-Galactopyranoside, methyl	2.31		

Table 2: Continued.

Feedstock	Particle size	Microwave power (W)	Temperature (°C)	Time (min)	Solvent	Catalyst	Main compositions	Area (%)	Yield of bio-oil (%)	Ref.
Lignin residue from black liquor		600	120	30	Isopropanol		Vanillin		45.35	[70]
							Acetovanillone			
							Homovanillic acid			
							Syringaldehyde			
							Acetosyringone			
Corn straw	60 mesh	600	180	22	Phenol	H_2SO_4	2,6-Dimethoxyphenol			[92]
							Phenol			
							p-Hexaphenol			
							2-Methoxyphenol			
							2-Methoxy-4-vinylphenol			
							4-Allyl-2,6-methoxyphenol			
							2,4-Dimethylphenol			
							o-Methoxyphenol			
Nannochloropsis oceanica			60	30	Chloroform	H_2SO_4	9-Hexadecenoic acid, methyl ester (C16:1)	34.51		[93]
							Hexadecanoic acid, methyl ester (C16:0)	15.46		
							9-Hexadecenoic acid, methyl ester, (Z)-(C16:1)	1.24		
							9,12-Octadecadienoic acid (Z,Z)-, methyl ester (C18:2)	3.17		
							9-Octadecenoic acid (Z)-, methyl ester (C18:1)	3.95		
							Phytol	2.48		
							5,8,11,14,17-Eicosapentaenoic acid, methyl ester (C20:5)	20.12		
							5,8,11,14-Eicosatetraenoic acid, methyl ester (C20:4)-	4.00		
							7,10,13-Eicosatrienoic acid, methyl ester (C20:3)	1.01		
							Octaethylene glycol monododecyl ether	1.23		
							1,2,3,4-Cyclopentanetetrol	0.40		
							2(3H)-Furanone, 5-heptyldihydro	0.39		
							Methyl Z-11-tetradecenoate (C14:1)	0.41		
							Methyl tetradecanoate	0.55		
							9-Hexadecenoic acid	0.78		
							n-Hexadecanoic acid	0.48		
							1,3,12-Nonadecatriene	0.39		
							Eicosen-1-ol, cis-9-	0.59		
							Ethanol,2-(9-octadecenyloxy)-,(Z)-	0.71		
							(Z)-14-Tricosenyl formate	0.55		

[*] HY (Si/Al ratio 5.0). The *X* stands for the weight ratios of the $Fe(NO_3)_3 \cdot 9H_2O$ to the parent HY catalyst.

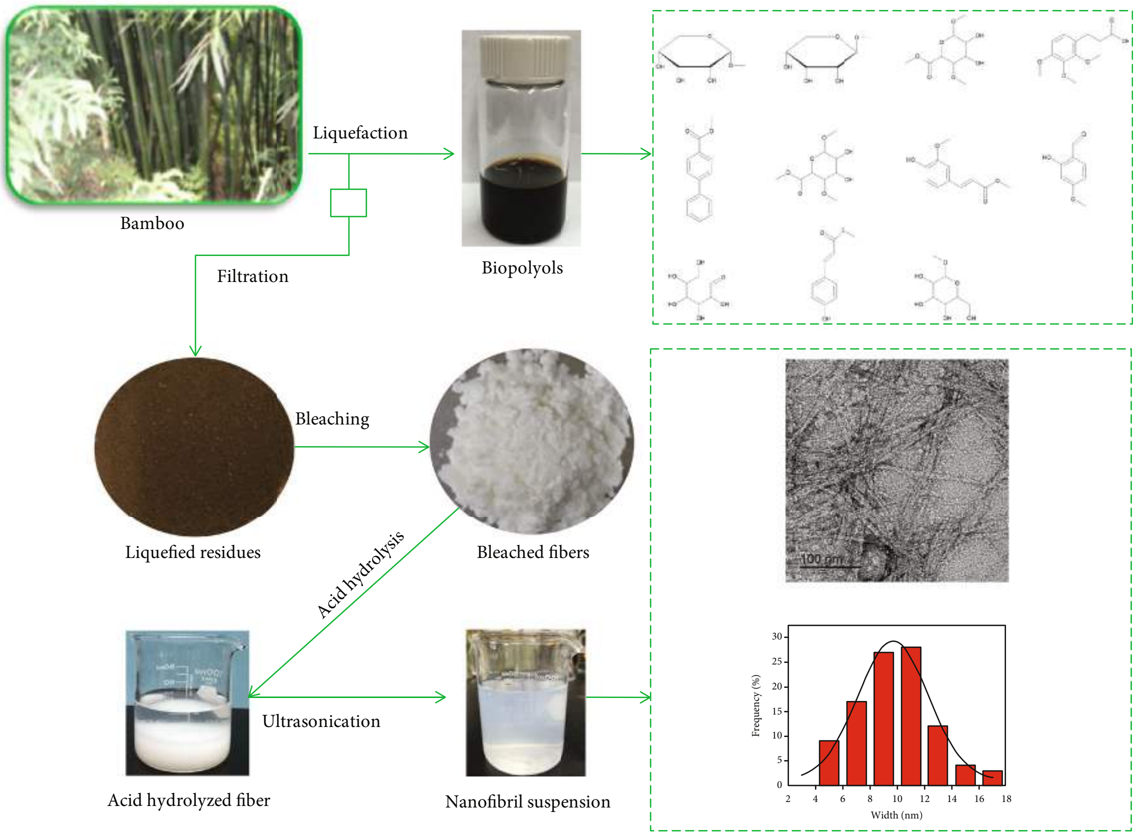

Figure 3: Potential application of microwave-liquefied bamboo residues for the extraction of cellulose nanofibers based on the residue characterization [50].

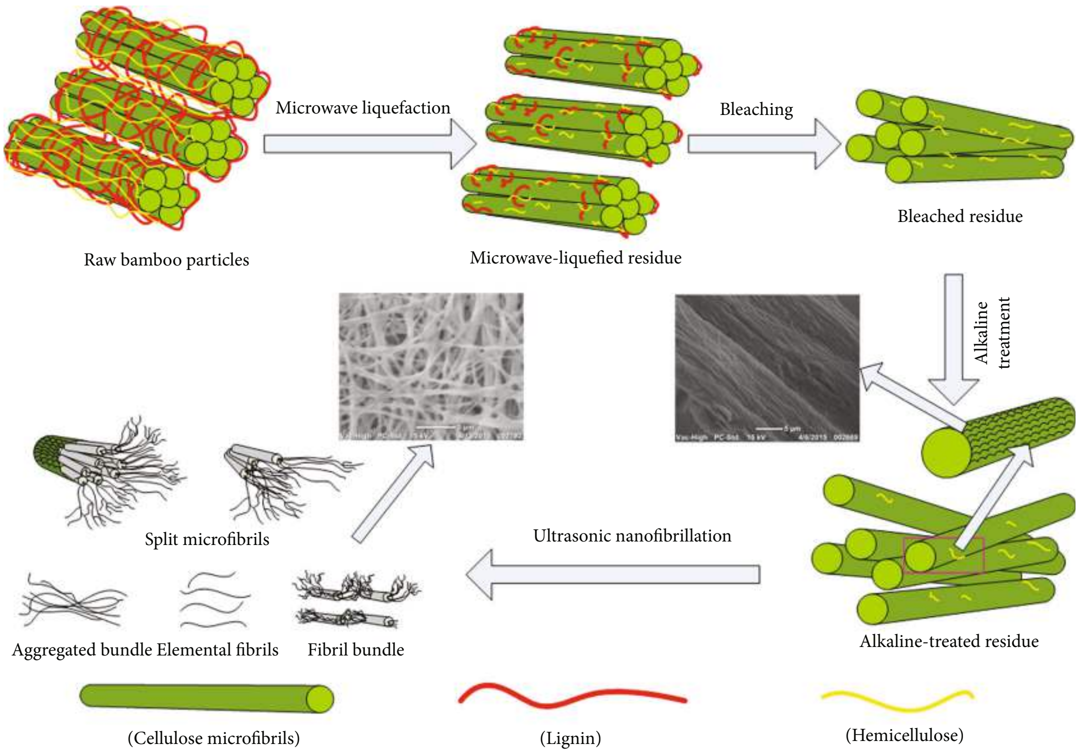

Figure 4: Proposed mechanism on the preparation of nanocellulose using microwave selective liquefaction assisted with chemical treatments [89].

liquefaction of lignocellulosic biomass was proven to be an efficient and environmentally friendly method to produce biochemicals and biomaterials. The obtained products exhibited comparable properties to commercially available biochemicals and bioproducts and thus could likely be applied in the bioproduct industries. Possible further work is still needed regarding scalable production and affecting factors of microwave liquefaction.

(1) All the reviewed previous research data on microwave liquefaction of lignocellulosic biomass was collected using small laboratory-scale microwave reactors. Pilot-scale experiments on the microwave liquefaction and the product utilizations should be considered and conducted in the near further

(2) Microwave dielectric heating depends on the ability of a material to absorb microwave energy and convert it into heat. A hypothesis may be that the absorbance of the components of lignocellulosic biomass may differ from each other, which may decide the decomposition order of each component and influence the chemical reactions of the components during the liquefaction process. However, further research is needed in this regard

Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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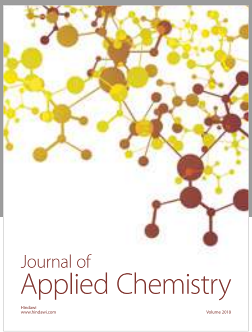
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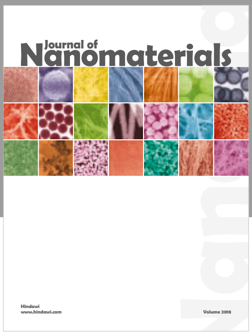
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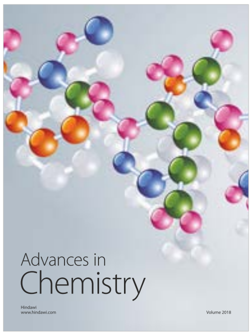
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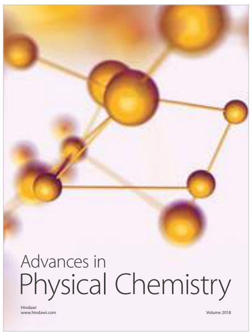
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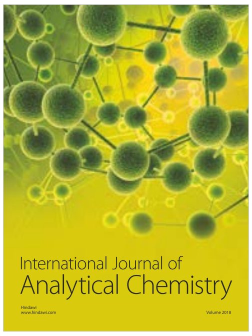
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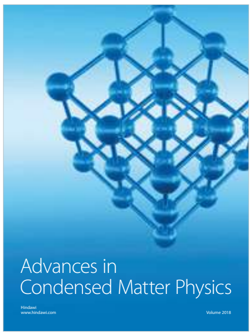
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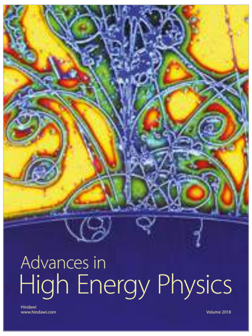
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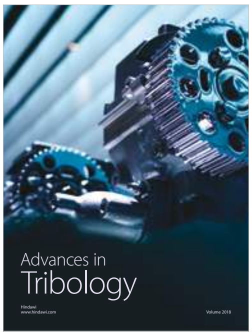
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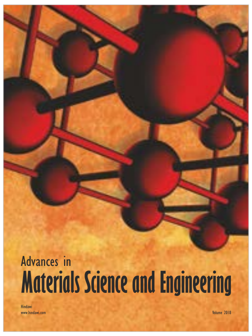
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