



Biodiesel production from waste cooking oil using a waste diaper derived heterogeneous magnetic catalyst

HongXu Chen¹ · Wei Xia² · Song Wang¹

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Abstract

A series of waste diaper-derived heterogeneous magnetic catalysts for the synthesis of biodiesel was developed through the two-step method, i.e., first wet-impregnation in nickel nitrate solution and then calcination at 700 °C. The structure and catalytic ability of the catalyst were characterized by SEM, FTIR, XRD, BET, VSM, and acid–base titration techniques. The investigation results indicated that WDHMCs were mainly composed of Ni, Na₂CO₃, and carbon. With the increase in the ratio of nickel nitrate to waste diapers, the magnetization of the prepared catalysts increased, while the catalytic activity decreased. When the ratio of nickel nitrate to waste diaper was 2 mmol/g, the obtained catalyst exhibited relatively high catalytic activity with the biodiesel yield of 96.4% and high magnetic separation property in the transesterification reaction of waste cooking oil with methanol. Moreover, the prepared waste diaper-derived heterogeneous magnetic catalyst could be easily reused by simple magnetic separation and maintain high catalytic activity after being reused four times.

Keywords Waste diaper · Biodiesel · Transesterification · Catalyst

Introduction

As a green renewable energy source, biodiesel has attracted more and more attention under the social background of the increasing demand for green energy (Ma et al. 2020; Liu et al. 2021; Vahid et al. 2017). Global biodiesel production is forecast to increase by 14% every year and the production capacity of biodiesel can reach 33 billion liters by 2021, which can greatly benefit the optimization of energy structure (Liu et al. 2021; Hajjari et al. 2017). As a mixture of fatty acid methyl esters (FAME), biodiesel can be obtained by transesterification of vegetable oil, animal oil, waste oil, or microbial oil with methanol. Waste cooking oil (WCO) is produced in food processing. Using WCO as the raw material to prepare biodiesel can reduce the preparation

cost of biodiesel and benefit the waste disposal (Kulkarni and Dalai 2006).

During the preparation process of biodiesel, catalyst plays an important role. In the traditional biodiesel preparation process, homogeneous catalysts such as KOH and NaOH were used and they exhibited high catalytic activity. However, these homogeneous catalysts exposed the disadvantage of being difficult to be reused (Abdullah et al. 2017; Kawashima et al. 2009; Souza et al. 2009; Zabeti et al. 2009; Liu et al. 2016). Compared with the traditional homogeneous catalysts, heterogeneous catalysts showed much better reusability. Many different types of heterogeneous catalysts have been developed in the past years, including ion exchange resin, zeolite, metal oxide, basic metal, and so on (Thushari and Babel 2018; Umar et al. 2019; Ashok et al. 2019). Most reported heterogeneous catalysts exhibited advantages with the respect to catalytic activity and reusability.

Nevertheless, the application of heterogeneous catalysts still has some limitations such as the high time and energy consumption in the separation and recovery process of catalyst (Lu et al. 2007). It is important to develop heterogeneous catalyst which can be easily separated from the reaction product with low time and energy consumption and high catalyst recovery percentage (Zhang et al. 2014).

✉ Song Wang
wangsong@sut.edu.cn

¹ Key Laboratory of Polymer and Catalyst Synthesis Technology of Liaoning Province, School of Environmental and Chemical Engineering, Shenyang University of Technology, Shenyang 110870, China

² Nonmetallic Mineral Industry Association of Liaoning Province, Shenyang 110870, China

Thus, the heterogeneous magnetic catalysts were developed in the past years (Wang et al. 2019). For example, when ZnO/BiFeO₃ nanomagnetic catalyst was used in the synthesis of biodiesel, the yield of biodiesel can be higher than 92% and the catalyst can be recycled easily (Salimi and Hosseini 2019). The high recovery efficiency of magnetic catalyst attracted more and more attention to the development of magnetic catalysts. However, the complex preparation procedure and high preparation cost made the industrial application of the reported heterogeneous magnetic catalyst difficult. It is an inevitable trend to develop high-efficiency and low-cost heterogeneous magnetic catalyst.

Heterogeneous catalysts prepared by low-cost waste have attracted wide attention in the field of transesterification reactions these years (Yang and Xie 2007; Yu et al. 2011; Viola et al. 2012; Semwal et al. 2011; Laskar et al. 2018; Kumar et al. 2018). Maneerung et al. found that CaO obtained from chicken manure can be converted into an active catalyst for the synthesis of biodiesel after the calcination at 850 °C (Maneerung et al. 2016). Rawat et al. used metal oxide mixed CaO, which was derived from chicken eggshell, as the catalyst in the transesterification of jatropha oil and karanja oil with methanol to produce biodiesel (Rawat et al. 2018). This waste-derived catalyst provided a new way to obtain low-cost and high-performance heterogeneous catalysts.

Waste diaper is a typical municipal solid waste. In some countries, the amount of waste diapers even accounts for 6% of total municipal waste (Espinosa-Valdemar et al. 2014). Since the main components of the waste diaper are non-biodegradable polymers such as sodium polyacrylate and polypropylene, the careless disposal of the waste diaper can lead to environmental problems (Cordella et al. 2015). To solve the problem in the field of waste diaper disposal, several methods have been applied to the reuse of waste diapers. For example, after blending with the yard waste, the waste diaper can be made into high-quality compost (Colón et al. 2013). Using the sodium polyacrylate in the waste diaper, porous material can be prepared by pyrolysis method and the prepared porous material can be successfully used as the anode material for lithium-ion batteries (Wei et al. 2018). Developing more ways to utilize waste diaper is conducive to environmental protection (Correia et al. 2014). Before that, our team published the research results of using waste diapers and NiNO₃ to prepare catalysts for the synthesis of glycerol carbonate. However, there is no research on the use of waste diapers for biodiesel (Wang et al. 2020).

In this study, waste diaper was used as the raw material to prepare the heterogeneous magnetic catalyst for the synthesis of biodiesel by wet-impregnation and calcination method. Compared with other catalysts, the heterogeneous magnetic catalyst prepared from waste diapers and nickel nitrate solution is more environmentally friendly, highly efficient, and

low-cost. The structure and morphology of the catalyst were studied by various techniques. Then the prepared catalysts were used in the transesterification of WCO with methanol, and their catalytic activity was compared. The catalyst with the best performance was selected to study the effect of reaction conditions on biodiesel yield. Besides, the stability of the selected catalyst was evaluated by the reuse experiment.

Experimental section

Materials

Analytical grade reagents, including methanol and nickel nitrate, were bought from Damao Chemical Reagent Co., Tianjin, China. All the reagents were used directly without a purification process. Waste diapers were collected from the local municipal waste. Waste cooking oil (WCO) was obtained from our university canteen. The composition and properties of the WCO were presented in Table 1.

Catalyst preparation

Catalysts based on the waste diaper were prepared by the wet-impregnation and calcination method as reported in literature (Wang et al. 2018). Waste diapers were dried up in an oven at 120 °C to remove the absorbed water. The dried waste diapers (10 g) were immersed in 0.25 mol/L, 0.5 mol/L, 0.75 mol/L, and 1 mol/L of NiNO₃ solution (40 ml). After being dipped for 6 h at room temperature, the fully impregnated waste diapers were dried at 120 °C for 12 h to remove the adsorbed water. The dried waste diapers were carbonized in a furnace at 700 °C for 2 h under nitrogen gas with a flow rate of 60 mL/min. The residual carbon material was ground and passed through the sieve of 125 μm. The obtained catalyst was named as WDHMC-X, where WDHMC presented the waste diaper derived heterogeneous magnetic catalyst and X indicated the ratio of NiNO₃ to waste diaper. For example, when the ratio of NiNO₃ to waste weight was 2 mmol/g (20 mmol NiNO₃ blended with 10 g waste diaper), the obtained heterogeneous magnetic catalyst was named as WDHMC-2.

Table 1 The composition and properties of the WCO

Composition and properties of the WCO	Value
Linoleic acid (%)	58.4
Oleic acid (%)	36.7
Palmitic acid (%)	4.9
Acid value (mg KOH g ⁻¹)	2.1
Density (g·cm ⁻³)	0.9
Viscosity (Cst at 40 °C)	38.1

Characterization of catalysts

Functional groups of the catalysts were characterized by Prestige-21 FT-IR spectrometer (Shimadzu, Japan). The scan range was recorded between 400 and 4000 cm^{-1} . The crystalline structure of the catalysts was characterized by the PW3040 X-ray diffraction (XRD) with a scanning speed of $4^\circ/\text{min}$ in the range of $5\text{--}80^\circ$. The surface morphology of the catalyst was observed by a Pro X (Phenom, Netherlands) scanning electron microscope (SEM). The elemental composition of the catalysts was recorded by energy-dispersive X-ray spectrum (EDS). The magnetic property of the catalysts was measured by a vibrating sample magnetometer (VSM, Lake Shore 7410, USA). Brunauer-Emmet-Teller surface area measurement (BET) of the catalysts was carried out by using a JW-BK122W instrument (JWGB, China). The basicity of the catalyst is determined according to the method in the literature (Li and Wang 2011).

Experimental procedure for transesterification reaction

A three-necked round-bottomed flask was used as the reactor. The flask was equipped with a spherical condenser, a thermometer, and an oil bath. In a typical experiment, 10 g of the filtered waste cooking oil, 3.2 g of methanol, and a certain amount of catalyst were charged into the flask. Then the reaction mixture was heated to the selected temperature and reacted for a certain time. After the reaction, the catalyst was separated using a $3 \times 1 \times 6 \text{ cm}^3$ rectangular magnet. The residual methanol in the reaction product was evaporated by vacuum distillation. 0.1 g of the distilled product and 0.01 g internal standard, methyl laurate, were dissolved in 10 ml n-hexane to prepare the sample for the gas chromatography analysis. The composition of the product was determined by an Agilent GC-7890A gas chromatography (HP, USA). The biodiesel yield was calculated according to the method reported in the literature (Teo et al. 2019).

Results and discussion

Catalyst characterization

Figure 1 shows the FT-IR results of the WDHMC catalysts. The FT-IR spectrum of the WDHMC catalysts is similar. Five characteristic absorption peaks were observed at 3416, 1627, 1438, 875, and 700 cm^{-1} . The peak at 3416 cm^{-1} can be related to O–H stretching vibrations (Shikhaliyev et al. 2018). The peaks at 1627, 1438, 875, and 700 cm^{-1} can be assigned to the vibration of carbonate (Viriya-empikul et al. 2010; Song et al. 2017). Thus, FT-IR investigation indicated that carbonate existed in the WDHMC catalysts.

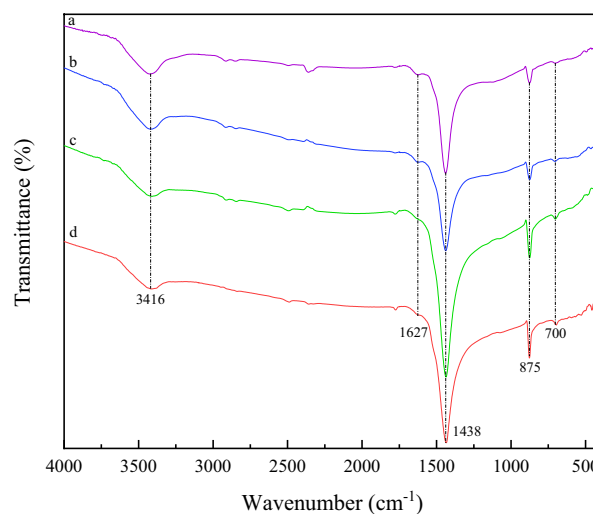


Fig. 1 FTIR spectra of the (a) WDHMC-1, (b) WDHMC-2, (c) WDHMC-3, (d) WDHMC-4

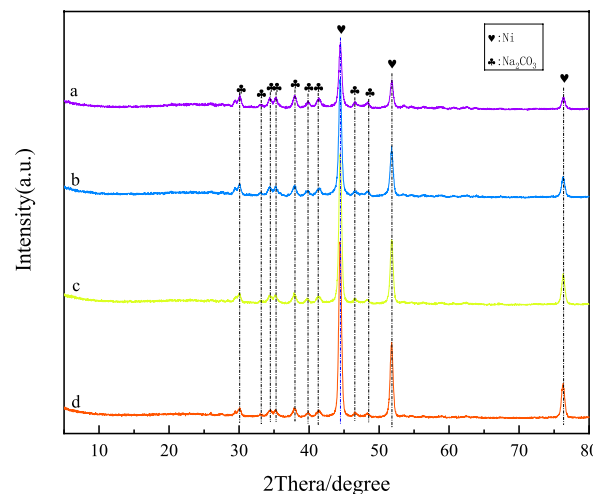


Fig. 2 XRD of the (a) WDHMC-1, (b) WDHMC-2, (c) WDHMC-3, (d) WDHMC-4

To further study the structural differences of the WDHMC catalysts, XRD studies were performed, which can effectively detect crystalline structure (Liu et al. 2022). Figure 2 shows the XRD patterns of the WDHMC catalysts. Strong diffraction peaks were observed in the prepared catalysts, indicating that these catalysts contained crystalline materials. Similar XRD diffraction peaks were found for the WDHMC catalysts. The weak diffraction peaks appeared at 30.2° , 33.2° , 34.5° , 35.3° , 38.1° , 39.9° , 41.4° , 46.6° , and 48.4° can be assigned to the diffraction of Na_2CO_3 (JCPDS 77-2082). The strong diffraction peaks observed at 44.2° , 51.8° , and 76.4° demonstrated the existence of Ni (JCPDS87-0712). Meanwhile, there is a broad peak that can be observed around 20° , which proved

the existence of carbon. XRD examination results illustrated that the prepared WDHMC catalysts were composites of Na_2CO_3 , nickel, and carbon. Na_2CO_3 can provide sufficient basicity for the catalyst, nickel can provide magnetism for the catalyst, and carbon can stabilize Na_2CO_3 and Ni particles, indicating that the prepared catalyst is a potential heterogeneous basic magnetic catalyst (Wang et al. 2019).

Due to the decomposition of the waste materials during the carbonization process, their morphology can be changed considerably (Chen et al. 2022). Figure 3 shows the changes in the surface morphology of the catalysts prepared with different NiNO_3 ratios. It can be observed that all the prepared catalysts exhibited similar morphology. Irregular

holes and small particles can be observed on the catalyst surface. Since the NiNO_3 ratio used during the catalyst preparation process was different, the element content of the catalyst must be different.

EDS data of the WDHMC catalysts is presented in Table 2. EDS analysis was performed to verify the elemental composition of the WDHMC catalysts. Na, Ni, C, and O are the main element of the WDHMC catalysts. Meanwhile, with the NiNO_3 ratio in the WDHMC catalysts increased, the weight percentage of Ni element in the catalyst increased, and the weight percentage of Na element decreased. Usually, the content of Na element is related to the catalyst basicity and the content of Ni element is related to the catalyst

Fig. 3 Surface images of the (a) WDHMC-1, (b) WDHMC-2, (c) WDHMC-3, (d) WDHMC-4

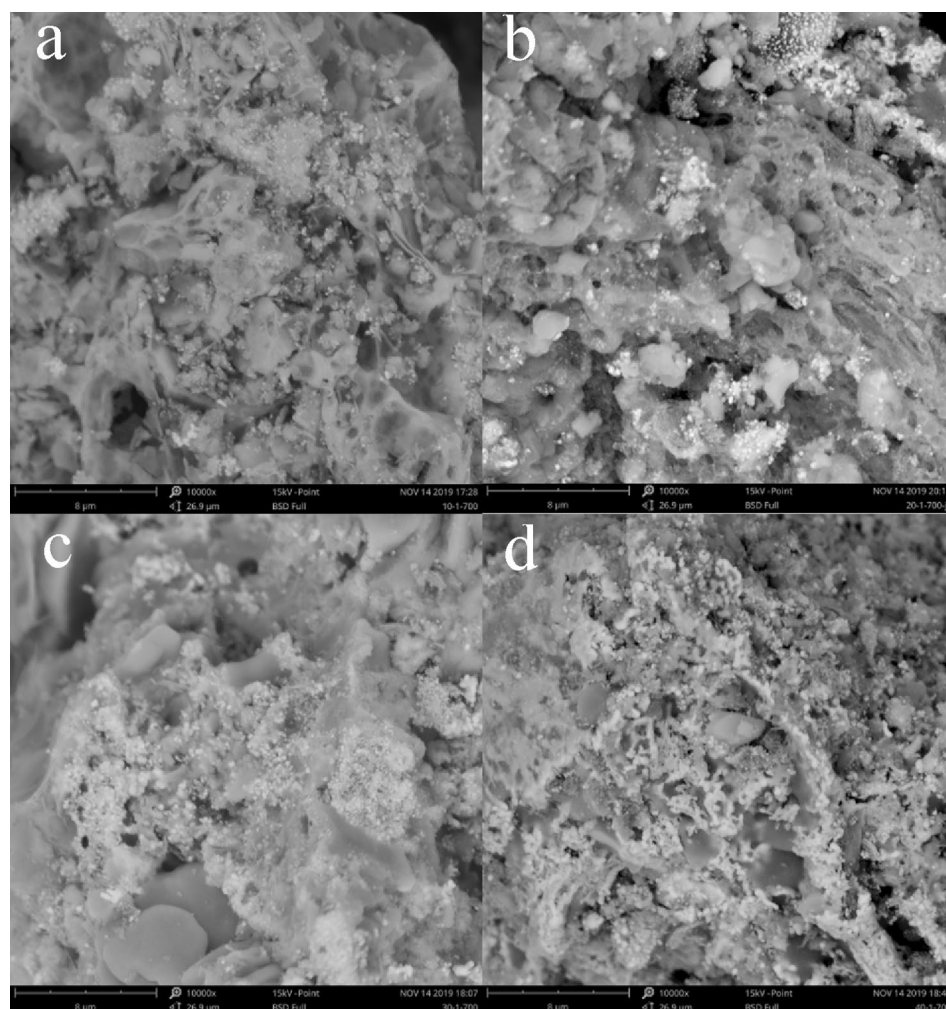


Table 2 Elemental composition of the WDHMC catalysts

Sample	Na (%)	Ni (%)	C (%)	O (%)	Cl (%)	K (%)	N (%)
WDHMC-1	31.9	21.8	13.2	30.3	0.2	0.5	2.1
WDHMC-2	28.4	25.5	12.8	29.9	0.2	0.8	2.4
WDHMC-3	19.5	35.4	14.5	28.3	0.4	0.6	1.3
WDHMC-4	14.1	45.7	11.6	25.8	0.7	0.3	1.8

magnetic property. The different content of Na and Ni in the catalyst can affect the catalytic and separation properties of the WDHMC catalysts.

The magnetic properties of the WDHMC catalysts were studied at room temperature, and the results are shown in Fig. 4. The shapes of the four catalysts hysteresis loop are similar. The magnetic strengths of WDHMC-1, WDHMC-2, WDHMC-3, and WDHMC-4 are 4.2, 7.9, 9.7, and 12.1 Am^2/kg , respectively. Since the magnetic strength can affect the separation property of the WDHMC catalysts, the magnetic separation experiment using a $3 \times 1 \times 6 \text{ cm}^3$ rectangular magnet was carried out and the result is shown in Fig. 5. When WDHMC-1 was used, the magnetic separation of the catalyst was not good. The used catalyst cannot be totally absorbed by a magnet (Fig. 5a). WDHMC-2, WDHMC-3, and WDHMC-4 exhibited good magnetic separation properties because they can be totally absorbed by magnet (Fig. 5b–d). The investigation on the magnetic properties of the WDHMC catalysts indicated that, when the magnetic strength of the catalyst was higher than 7.9

Am^2/kg , the prepared WDHMC catalyst showed good magnetic separation property. WDHMC-2, WDHMC-3, and WDHMC-4 can be used as magnetic catalyst for the synthesis of biodiesel.

Textural and basic properties of the WDHMC catalysts are presented in Table 2. WDHMC1 showed the highest BET surface area and total basicity among the prepared catalysts. However, the magnetic separation property of WDHMC1 is poor (Fig. 5a), making it unsuitable for the application as a magnetic catalyst. The basicity of the catalyst can directly affect its catalytic activity in the reaction between methanol and waste cooking oil. To illustrate the catalytic activity of the WDHMC catalysts, the prepared catalysts were applied in the synthesis of biodiesel. The comparison experiment was carried out under the reaction condition with the methanol to WCO molar ratio of 9:1 and 4 wt% of catalyst at 65°C for 6 h. It can be observed from Fig. 6 that only WDHMC-1

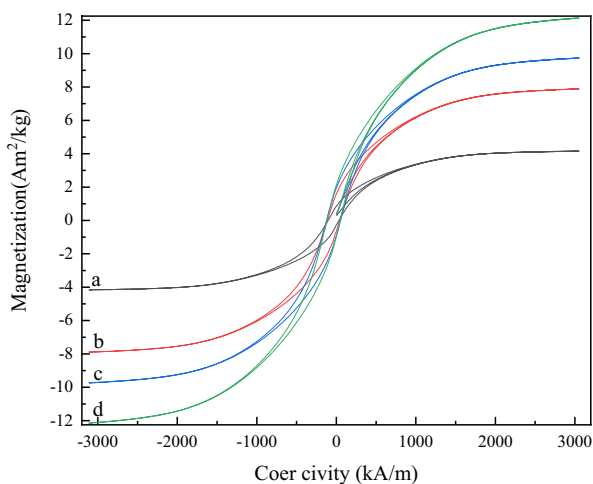


Fig. 4 Hysteresis loops of the (a) WDHMC-1, (b) WDHMC-2, (c) WDHMC-3, (d) WDHMC-4

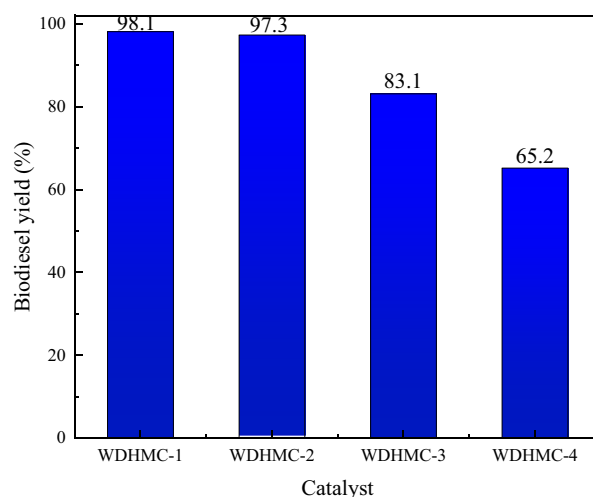
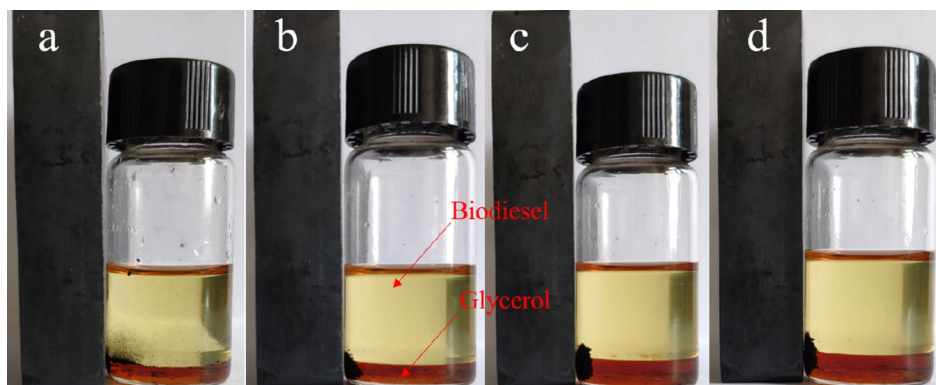


Fig. 6 Comparison of the catalytic activity of the WDHMC catalysts

Fig. 5 Magnetic separation of the (a) WDHMC-1, (b) WDHMC-2, (c) WDHMC-3, (d) WDHMC-4



and WDHMC-2 showed high catalytic activity with the biodiesel yield higher than 95%. Therefore, in terms of the consideration of catalytic activity and magnetic separation performance, WDHMC-2 was selected as the suitable catalyst for the synthesis of biodiesel and applied in the following studies on the effect of the reaction parameters on the biodiesel yield (Table 3).

Table 3 BET, basicity, and magnetization of the WDHMC catalysts

Catalyst	BET surface area (m ² /g)	Total basicity (mmol/g)	Magnetization (Am ² /kg)
WDHMC-1	128.6	7.4	4.2
WDHMC-2	59.9	4.7	7.9
WDHMC-3	38.9	3.1	9.7
WDHMC-4	23.8	2.3	12.1

Effect of the transesterification reaction parameters on biodiesel yield

Sufficient reaction time is an important factor to achieve a high biodiesel yield (Amani et al. 2014b, a). Figure 7a shows the effect of the reaction time on biodiesel yield. The result indicated that the reaction time has an obvious effect on biodiesel yield. When the reaction time was 1 h, the biodiesel yield only reached 26.9%. With the increase of the reaction time, the biodiesel yield increased significantly. Biodiesel yield reached 96.4% when the reaction time was 4 h. When the reaction time was further prolonged, the biodiesel yield did not change obviously.

Lots of comparative investigation on the heterogeneous base catalysts in the synthesis of biodiesel proved that the biodiesel yield is greatly affected by the content of catalyst amount (Ngamcharussrivichai et al. 2010). Figure 7b shows the effect of catalyst amount on biodiesel yield. When the catalyst amount was only 1 wt%, the biodiesel yield reached

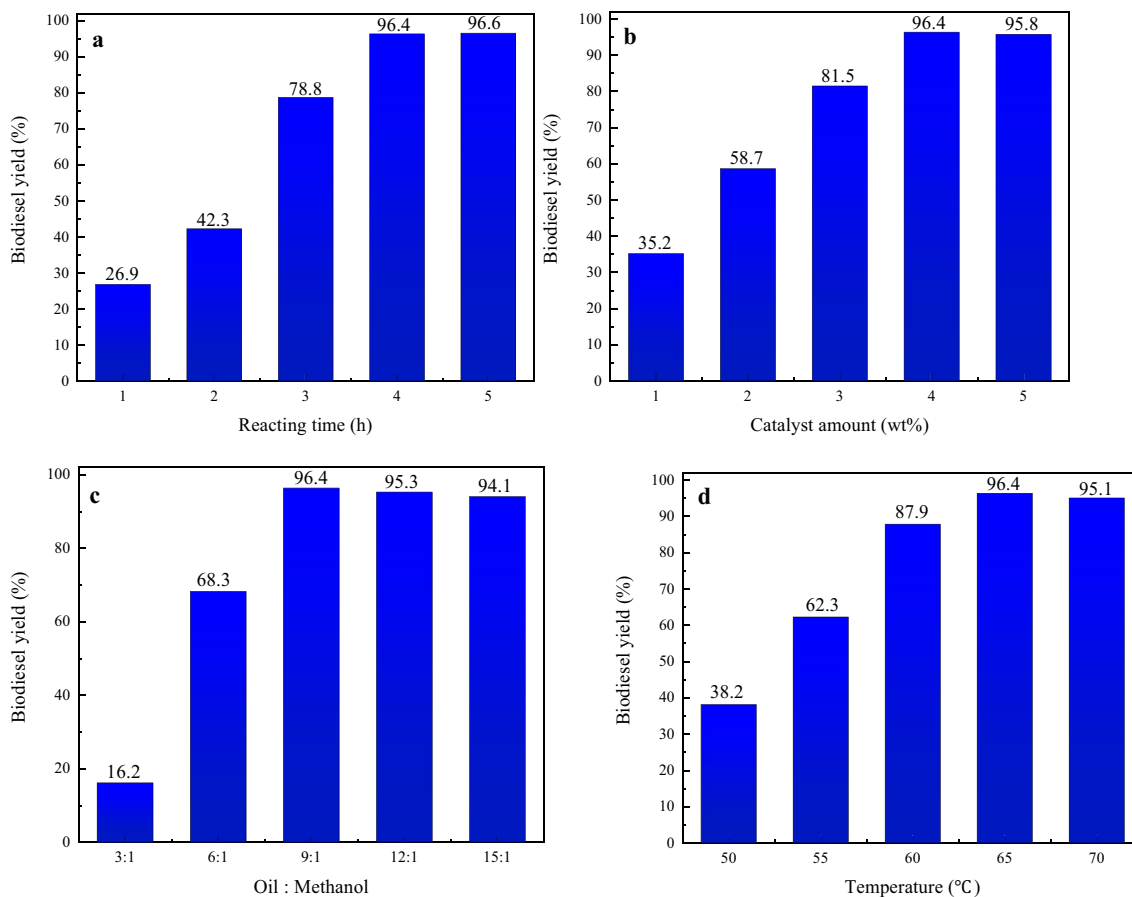


Fig. 7 (a) Biodiesel yield as a function of reaction at different times, 65 °C, 9:1 oil to methanol molar ratio, and 4 wt% of catalyst; (b) Biodiesel yield as a function of reaction at 4 h, 65 °C, 9:1 oil to methanol molar ratio, and different catalyst amount; (c) Biodiesel

yield as a function of reaction at 4 h, 65 °C, different oil to methanol molar ratio, and 4 wt% of catalyst; (d) Biodiesel yield as a function of reaction at 4 h, different temperature, 9:1 oil to methanol molar ratio, and 4 wt% of catalyst

35.2%. As the catalyst amount increased, the biodiesel yield increased simultaneously. When the catalyst amount was 4 wt%, the biodiesel yield was up to 96.4%. A high catalyst amount can lead to the increase of biodiesel yield due to the increase of the active site in the reaction system. However, the biodiesel yield declined slightly when the catalyst amount exceeded 4 wt%. This was due to the poor diffusion between the reagent and catalyst in the case of a high catalyst amount (Nizah et al. 2012).

Since the transesterification of methanol with WCO is a reversible reaction, methanol must be used excessively to achieve a high biodiesel yield (Wong et al. 2015). With the increase of the methanol to WCO molar ratio from 3:1 to 9:1, the biodiesel yield increased from 16.2 to 96.4% as illustrated in Fig. 7c. However, when the methanol to WCO molar ratio exceeded 9:1, the biodiesel yield decreased. This is because that excessive methanol can promote the dissolution of glycerol in the WCO and subsequently lead to a decrease of biodiesel yield (Jo et al. 2013).

Transesterification of WCO with methanol is an endothermic reaction. The increase of reaction temperature is beneficial to the reaction. Figure 7d shows the effect of reaction temperature on biodiesel yield. When the temperature was 50 °C, the biodiesel yield reached 38.2%. As the temperature increased, the biodiesel yield increased. When the reaction temperature was 65 °C, the biodiesel yield was up to 96.4%. With the further increase of reaction temperature, biodiesel yield decreased slightly, which was caused by the evaporation of methanol and the decrease of the actual methanol to WCO molar ratio (Tang et al. 2020).

Reusability of the WDHMC catalyst

Compared with homogeneous catalysts, heterogeneous catalysts can be reused, which can decrease the preparation cost and simplify the purification process of the product. To examine the reusability of the prepared WDHMC catalyst, the magnetically separated catalyst was washed with methanol, dried in an oven at 110 °C for 2 h, and reused in the next cycle. Figure 8 shows the reusability of the WDHMC catalyst. It can be seen from Fig. 8 that the biodiesel yield decreased to 62.4% after the WDHMC-2 was used for four cycles. To find out the reason for the reduction of catalytic activity of the WDHMC catalyst, XRD analysis was used to compare the structure difference between the fresh WDHMC catalyst and the reused WDHMC catalyst. Figure 9 shows the XRD analysis result. It can be observed that, after the WDHMC-2 was used four cycles, the peak intensity of Na_2CO_3 decreased, while the peak intensity of Ni changed little. This result demonstrated that the content of Na_2CO_3 in the WDHMC-2 decreased and the content of Ni unchanged. Since Na_2CO_3 provides sufficient basicity for

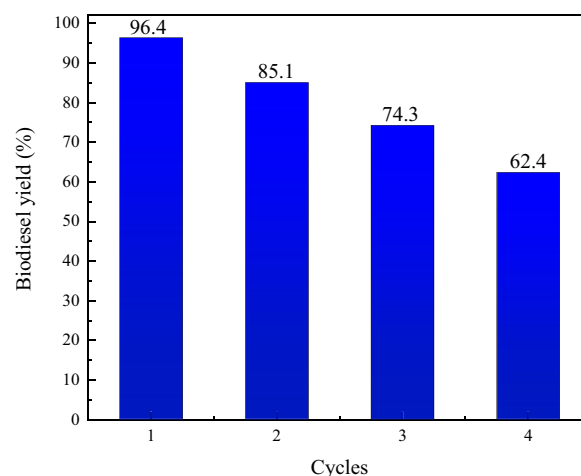


Fig. 8 Biodiesel yield with different cycles

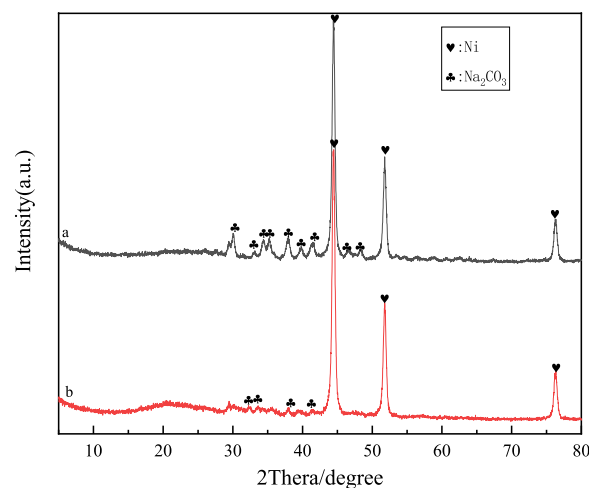


Fig. 9 XRD of the (a) fresh WDHMC-2, (b) reused WDHMC-2

the catalyst, the leaching of Na_2CO_3 can lead to the reduction of the catalyst activity.

Comparison of WDHMC-2 with the reported catalysts

Finding suitable material and preparation methods to prepare heterogeneous catalysts is an important research topic in the field of biodiesel synthesis. Due to the high separation efficiency, the heterogeneous magnetic catalyst has drawn more and more attention. A lot of heterogeneous magnetic catalysts have been prepared in the past years. As a heterogeneous magnetic catalyst, to evaluate the quality of the WDHMC-2, a comparison between the WDHMC-2 and some reported heterogeneous magnetic catalysts was carried out. The comparison result is shown in Table 4. All these catalysts presented high catalytic activity with the biodiesel

Table 4 Comparison of the WDHMC-2 with the reported catalysts

Entry	Catalyst	Materials	Yield ^f (%)	Cycles	Yield ^r (%)	Reference
1	CCC _{opt}	Corncob and papaya seed	84.5	5	47.5	Tang et al. (2020)
2	CBPA	Musa balbisiana colla peels	100.0	5	50.0	Gohain et al. (2017)
3	CaO	Waste mollusk shell	94.0	3	53.8	Hadiyanto et al. (2016)
4	Na ₂ SiO ₃ @Ni/C	Ni(NO ₃) ₂ , solid urea, bamboo powders, Na ₂ SiO ₃	98.1	5	80.9	Zhang et al. (2016)
5	Fe ₃ O ₄ /ZnMg(Al)O	FeCl ₃ , FeCl ₂ , Zn(NO ₃) ₃ , CuSO ₄ , Al(NO ₃) ₃ , Mg(NO ₃) ₃	94.0	7	82.0	Chen et al. (2018)
6	WDHMC-2	Waste diaper and NiNO ₃	96.4	4	62.4	This work

Yield^f: biodiesel yield when the fresh catalyst was used

Yield^r: biodiesel yield when the recycled catalyst was used

yield higher than 80%. However, their catalytic stability, preparation cost, and separation efficiency were different. In terms of catalyst stability, most of these catalysts exposed the problem of the decrease of the catalytic activity. The biodiesel yield decreased to 53.8% when CaO was used for three cycles. In terms of catalyst preparation cost, CBPA, CaO, and WDHMC-2 showed an obvious advantage because these catalysts were prepared by waste materials with negligible cost. In terms of catalyst separation efficiency, Fe₃O₄/ZnMg(Al)O, Na₂SiO₃@Ni/C, and WDHMC-2 can be separated from the reaction media by a magnet, indicating the high separation efficiency of these catalysts. Thus, from a comprehensive perspective, WDHMC-2 is a low preparation cost, high separation efficiency, and good catalytic activity catalyst, which has potential application in the future.

Conclusion

A series of waste diaper-derived heterogeneous magnetic catalysts were prepared using nickel nitrate and waste diaper as raw material. The level of Ni in the catalysts can greatly affect the magnetization and catalytic activity of the prepared catalysts. As the level of Ni increases, the magnetization increases, but the yield of biodiesel reduces probably caused by the alkalinity reduction. When the catalyst with the ratio of nickel nitrate to waste diaper at 2 mmol/g was applied in the biodiesel synthesis, under the reaction condition with the methanol to WCO molar ratio of 9:1 and the reaction temperature of 65 °C, the biodiesel yield reached 96.4% within 4 h in the presence of 4 wt% of the catalyst. Compared with the reported heterogeneous catalyst, the prepared waste diaper-derived heterogeneous magnetic showed advantages in the respects of catalytic stability, preparation cost, and separation efficiency. Therefore, with the improvement of the law on the recovery and reuse of biological contaminated materials, the waste diaper derived heterogeneous magnetic catalyst has great potential

application in the transesterification reaction of WCO with methanol. This study not only provided a new method to prepare heterogeneous catalyst used in biodiesel synthesis, but also explored a new way to utilize diaper waste, which is beneficial to waste management and resource conservation.

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Declarations

Conflict of interest On behalf of all authors, the corresponding author states that there is no conflict of interest.

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