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Resource Utilization of Waste Cooking Oil Catalyzed by Na_2CO_3/ZSM-5

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ABSTRACT: A catalyst with a simple synthetic process and good catalytic performance was prepared using Na_2CO_3 as the active component and ZSM-5 as the carrier for the resource utilization of waste cooking oil. The structure of Na_2CO_3/ZSM-5 was characterized by Fourier transform infrared spectroscopy, X-ray diffraction, and scanning electron microscopy, and the effects of parameters such as Na_2CO_3 loading, catalyst percentage, and reaction time on the yield of fatty acid methyl esters were investigated. The results showed that the conversion of waste cooking oil to fatty acid methyl esters yielded up to 96.89% when the Na_2CO_3 loading was 35%, the reaction temperature was 65 °C, the reaction time was 2 h, and the catalyst percentage was 1 wt %. The Na_2CO_3/ZSM-5 catalyst could be used to replace H_2SO_4 or $NaOCH_3$ in the industrial treatment of waste cooking oil for its resource utilization.

1. INTRODUCTION

With the declining supply of fossil fuels and growing environmental concerns, searching for sustainable and green alternative energy sources is essential.[1−5] Biodiesel is a renewable, nontoxic, and biodegradable green fuel with great performance, lubricating qualities, and clean combustion.[6−9] Its main component is fatty acid methyl ester, which is considered as an excellent substitute to petrochemical diesel,[10,11] and it may be produced by combining fats and oils (vegetable oils, animal fat, microalgae, edible oil wastes, etc.) with methanol under the action of a specific catalyst.[12−14]

Waste cooking oil (WCO), a nonreusable animal and vegetable oil created during food processing,[15] having complicated composition and contains hazardous elements like heavy metals and mold that must be carefully disposed of to prevent threats to the environment and human health.[16−18] Therefore, how to "turn waste into treasure" by recycling catering waste oil has become a hot topic in the field of environmental protection and resource utilization both domestically and internationally. The transesterification reaction is a widely used technique for dealing with used oil. Most industrial transesterification processes use homogeneous acid/base catalysts, such as concentrated H_2SO_4, $NaOCH_3$, NaOH, KOH, and so forth. However, there are many problems such as high difficulty in recycling, large production of three wastes (water, gas, solid), being environment unfriendly, and so on.[19−22] For this reason, researchers have begun to explore heterogeneous catalyst systems to improve the esterification process.

It has been found that Na_2CO_3, a catalyst used for transesterification, exhibited a high conversion rate and good storage stability in the transesterification of sunflower seed oil and rapeseed oil.[23,24] In the esterification reaction of WCO, the reaction rate was slower and the reaction time was longer due to the small specific surface area of Na_2CO_3 and its low solubility in methanol. To solve this problem, Na_2CO_3 could be introduced into other components to minimize its leaching behavior, thereby improving the stability of the catalyst and shortening the reaction time. As a result, some researchers have loaded Na_2CO_3 on waste carbide residue, foam carbon, electric furnace dust, and so on to prepare solid base catalysts. The catalysts had high specific surface area and a large number of micropores, which effectively improved the contact area between the catalyst and the alcohol oil system, thus improving the conversion rate of transesterification.[25−27] The previous research indicated that solid alkali catalysts had enormous potentiality in the treatment and resource utilization of WCO efficiently.

ZSM-5 zeolite is a novel type of molecular sieve that contains organic amine cations synthesized by Mobil in the late 1960s.[28−31] Its exceptional catalytic performance was attributed to its unique pore structure, adjustable acidic sites, and hydrothermal stability, which made it a promising catalyst in the petrochemical industry with increasingly diverse applications.[32−34,49,50] Meanwhile, it exhibited excellent catalytic performance in the esterification process.[35] Therefore, ZSM-5 molecular sieves were commonly utilized as either carrier materials or direct catalysts in catalysis studies. However, the small pore structure of ZSM-5 made it difficult for reactant molecules to enter the catalyst, limiting the efficiency of the catalytic reactions. At the same time, its high acidity could

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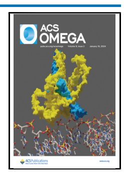
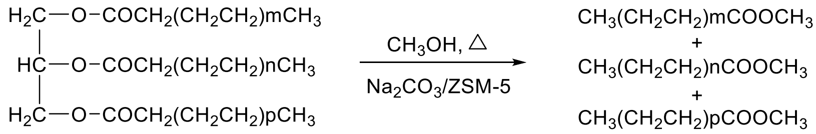


Scheme 1. WCO Was Converted into Fatty Acid Methyl Esters Catalyzed by Na_2CO_3/ZSM-5



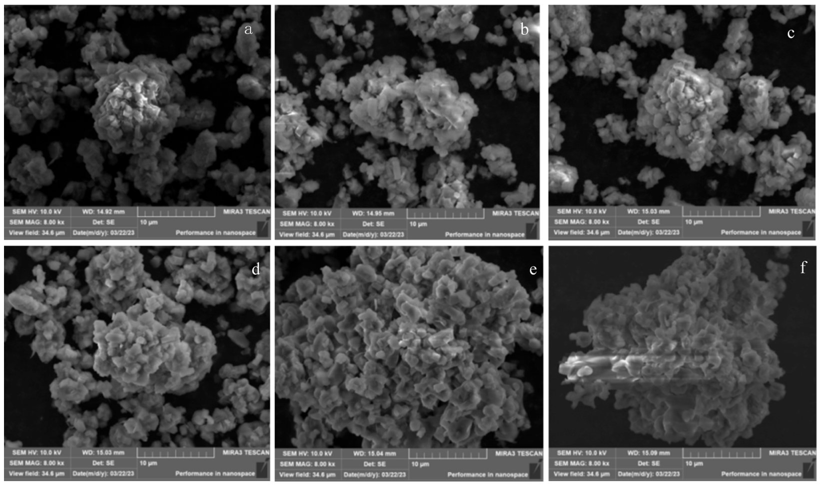


Figure 1. SEM images. (a) ZSM-5; (b) 5% Na_2CO_3/ZSM-5; (c) 10% Na_2CO_3/ZSM-5; (d) 15% Na_2CO_3/ZSM-5; (e) 25% Na_2CO_3/ZSM-5; and (f) 35% Na_2CO_3/ZSM-5.

easily cause other side reactions during the reaction process, which ultimately affected the purity of the reaction products.

The development of heterogeneous catalysts aimed at finding a structurally simple and relatively inexpensive catalyst to replace concentrated sulfuric acid or sodium methanol so as to improve the industrial production efficiency and economic value of resource utilization of WCO. We imagined that loading Na_2CO_3 on ZSM-5 could further improve the activity and stability of the catalyst while retaining their original respective advantages (the chemical reaction equation is shown in Scheme 1) based on our previous research.[36,37] In addition, the recycling of catalysts could also save a large amount of energy and production costs, improving the practical application values of resource utilization of WCO.

2. MATERIALS AND METHODS

2.1. Instruments, Materials, and Reagents. WCO was collected from homes, schools, and food and beverage establishments. The reagents were analytically pure and purchased from Sino Pharm Chemical Reagent Co. The instruments involved in this experiment include a Model 78-1 magnetic heating stirrer (Ronghua Instrument Manufacturing Co., Ltd.), a temperature-regulating electric heating sleeve (Shanghai Meitian Electric Appliance Co., Ltd.), SHZ-D (III) circulating water vacuum pump (Gongyi IYUHUA Instrument Co., Ltd.), a DZ-2BC vacuum drying oven (Tianjin Taishi Instrument Co., Ltd.), a Nicolet 6700 Fourier transform infrared spectrometer (Thermo Fisher Scientific, Inc.), a TESCAN-MIRA3 field emission scanning microscope analyzer (Czech TESCAN Co. Ltd.), a TESCAN-MIRA3 field emission scanning electron microscope analyzer (TESCAN, Czech Republic), and an Empyrean powder X-ray diffractometer (Panacor, the Netherlands).

2.2. Preparation of the Catalyst. A certain amount of Na_2CO_3 was dissolved in 50 mL of distilled water to obtain 5, 10, 15, 25, and 35% solutions, respectively. 2.5 g of ZSM-5 was slowly added to the aforesaid solution and impregnated with stirring at 80 °C for 2 h. After that, the mixture was poured into a beaker and cooled for 3 h to crystallize. The solution was filtered and rinsed with distilled water two to threetimes. The dried solid powder was laid flat in an alumina crucible and roasted at 600 °C with a heating rate of 5.0 °C/min for 3 h. The solid was taken out and ground to make the supported solid catalyst, named 5, 10, 15, 25, and 35% Na_2CO_3/ZSM-5 catalysts, respectively.

2.3. Characterization of the Catalyst. *2.3.1. Scanning Electron Microscopy.* After ZSM-5 and Na_2CO_3/ZSM-5 with different loading capacities of Na_2CO_3 were fully ground into powder, a small amount of the samples were fixed with conductive tape. The topography characteristics of the catalyst were compared and observed by a high-resolution field emission scanning electron microscope for analysis at 10 μm and a voltage of 10.0 kV.

2.3.2. Fourier Transform Infrared Spectroscopy. Fourier transform infrared spectroscopy (FTIR) was used to analyze Na_2CO_3, ZSM-5, and catalysts loaded with a certain amount of Na_2CO_3, with a resolution range of 4000−600 cm^{-1} at 4 cm^{-1}. Each spectrum was the overall average of 16 scans.

2.3.3. X-ray Diffraction. One g portion of dry and uniformly sized catalyst was weighed, then placed on the surface of a quartz plate, flattened with a glass plate, and measured using a powder X-ray diffractometer (Cu-K_α radiation, diffraction wavelength λ = 1.54 Å, working current of 30 mA, working voltage of 35 kV, and scanning speed of 3°/min).

2.4. Catalytic Performance. After preheating the collected WCO at 60 °C for 10 min, the Na_2CO_3/ZSM-5 catalyst was added. Under the ratio of methanol to oil (12:1), the effects of Na_2CO_3 loading, catalyst proportion (0.4, 0.6, 0.8, 1.0, 1.2, and 1.4 wt %), and reaction time (1, 2, 3, 4, 5, and 6 h) on the yield of fatty acid methyl esters were investigated. The reusability of the catalyst was investigated by washing

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repeatedly with methanol, drying at room temperature and then putting it into the next reaction.

3. RESULTS AND DISCUSSION

3.1. Characterization. *3.1.1. SEM Analysis.* Scanning electron microscopy (SEM) images of ZSM-5 and Na_2CO_3/ZSM-5 with different Na_2CO_3 loadings (5, 10, 15, 25, and 35%) are shown in Figure 1. It could be seen that ZSM-5 was a spherical particle with a smooth surface. As the amount of Na_2CO_3 increased, the catalyst particles gradually connected together to form an ant-nest structure (Figure 1b−f). In particular, the surface of the catalyst containing 35% Na_2CO_3 became rough (Figure 1f), which indicated that the active substance Na_2CO_3 was uniformly dispersed on the surface of ZSM-5.

3.1.2. FTIR Analysis. FTIR spectra of Na_2CO_3 and catalysts with different Na_2CO_3 loadings are shown in Figure 2. It was obvious that the asymmetric stretching vibrational peaks of Si−O−Si or Si−O−Al in ZSM-5 were at 1080 cm^{-1}.[38] The appearance of CO_3^{2-} absorption peaks were at 1496 and 768 cm^{-1} with the increase of Na_2CO_3 loadings, which indicated that Na_2CO_3 had been successfully loaded onto the surface of ZSM-5.

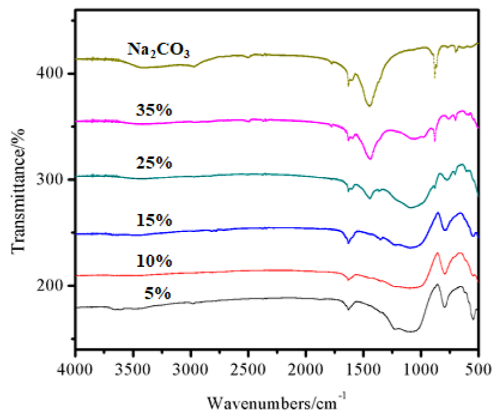

Figure 2. FTIR plots of Na_2CO_3 and catalysts with different Na_2CO_3 loadings.

3.1.3. XRD Analysis. The X-ray diffraction (XRD) plots of Na_2CO_3, ZSM-5, and catalysts with different Na_2CO_3 loadings are shown in Figure 3. The peak intensities of Na_2CO_3/ZSM-5 decreased with the increase of Na_2CO_3 loading as compared to ZSM-5. This clearly demonstrated that the crystallinity of ZSM-5 was reduced after impregnation with Na_2CO_3. At the same time, the remarkable characteristic diffraction peaks of ZSM-5 were present in the profiles of 5, 10, 15, and 25% Na_2CO_3/ZSM-5. In addition, different diffraction peaks corresponding to Na_2CO_3 were observed in Na_2CO_3/ZSM-5, and the intensities of the peaks gradually increased with the increase of Na_2CO_3 loading, indicating that the size of Na_2CO_3 microcrystals gradually increased.

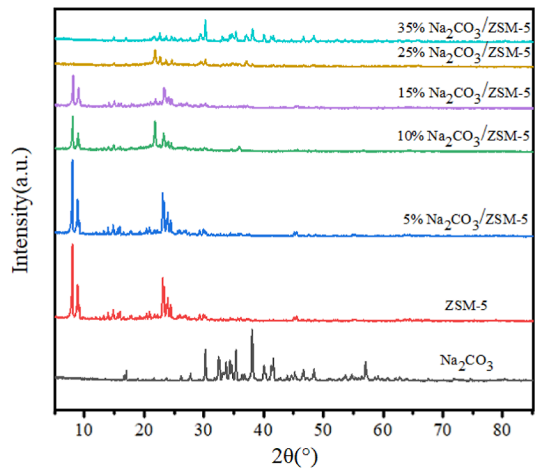

Figure 3. XRD plots of Na_2CO_3 and catalysts with different Na_2CO_3 loadings.

3.2. Catalytic Activity. *3.2.1. Effect of Na_2CO_3 Loading.* The yields of fatty acid methyl esters catalyzed by Na_2CO_3/ZSM-5 with different Na_2CO_3 loadings are shown in Figure 4. Obviously, when ZSM-5 was solely used as a catalyst, the yield of fatty acid methyl ester was low (1.33%), which indicated that the catalytic activity of ZSM-5 was weak. With the increase of Na_2CO_3 loading, the catalytic conversion efficiency of fatty acid methyl esters increased significantly from 1.33 to 96.23% when the amount of Na_2CO_3 loading was 35%. The reason might lie in the fact that the number of alkaline active sites on the surface of ZSM-5 gradually increased with the increase in Na_2CO_3 loading in the catalyst. Therefore, subsequent testing was based on an optimal Na_2CO_3 loading (35%).

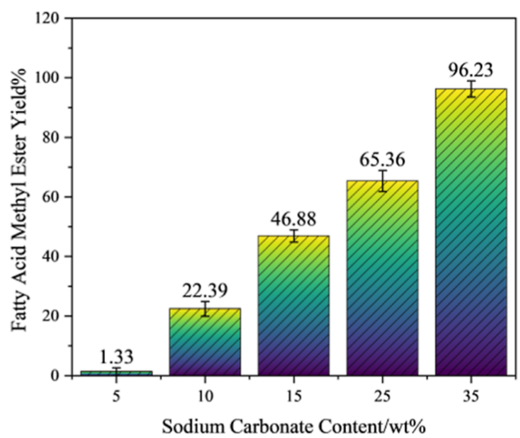

Figure 4. Yields of fatty acid methyl esters catalyzed by Na_2CO_3/ZSM-5 with different Na_2CO_3 loadings.

3.2.2. Effects of Catalyst Dosage. The effects of catalyst dosage (weight ratio of the catalyst to WCO, wt %) on the chemical reaction yield are shown in Figure 5. With the increase of the weight ratio of 35% Na_2CO_3/ZSM-5 to WCO, the yield of the fatty acid methyl ester increased significantly. Specifically, the yield of fatty acid methyl ester reached the maximum (91.23%) when the weight ratio was 1.0%. However, the yield actually declined to 79.36% with further increase of the weight ratio. The reason for these results was that the viscosity of the reaction mixture increased accompanied by the increase in the proportion of the catalyst. Consequently, catalyst activity of Na_2CO_3/ZSM-5 decreased, resulting in decrease in reaction yield. Therefore, the optimized catalyst dosage was 1.0 wt %.

3.2.3. Reaction Time. As shown in Figure 6, the yield of fatty acid methyl ester was only 8.43% when the reaction time was 1 h under the condition of 35% Na_2CO_3/ZSM-5 and 1.0 wt %. This might be attributed to the insufficient reaction progress and short reaction time. When the reaction reached 2 h, the yield increased rapidly to 96.89%. If the reaction time

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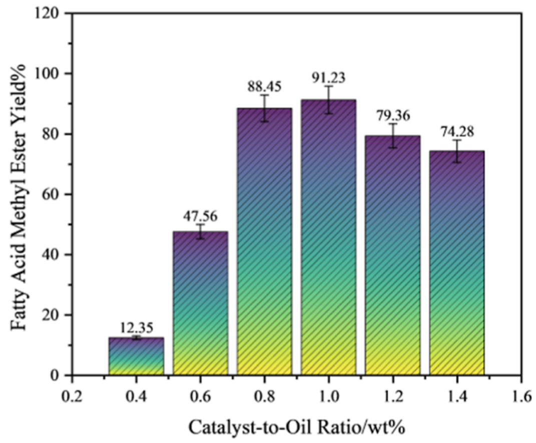

Figure 5. Effects of the catalyst dosage on the yield of fatty acid methyl ester.

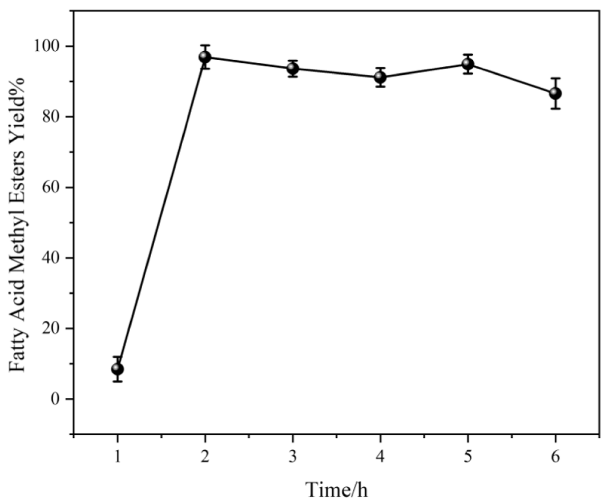

Figure 6. Effects of the reaction time on the yield of fatty acid methyl ester.

was further extended to 3, 4, 5, and 6 h, the yields showed slight fluctuations and maintained above 82%. Based on the experiments, the catalytic reaction between WCO and methanol could be fully completed in 2 h, and the yield of fatty acid methyl ester was as high as 96.89%. Nevertheless, the continued prolongation of the reaction time did not significantly enhance the overall yield.

3.2.4. Reusability. The reusability of Na_2CO_3/ZSM-5 was investigated as follows: the reaction solution was filtered to obtain the catalyst; then, the solid powder was washed with methanol three times and dried in an oven under reduced pressure at room temperature for 2 h; and the recovered catalyst could be used for the next reaction. The impacts of reuse cycle times on the catalytic activity are shown in Figure 7. It could be seen that Na_2CO_3/ZSM-5 maintained good catalytic activity after eight cycles of use, and the yield of fatty acid methyl ester still remained above 80%. The recycling of catalysts could offer substantial benefits in terms of energy conservation and cost reduction and greatly improve the practical application value of the resource utilization of WCO.

3.2.5. Comparison with Other Catalysts. The catalytic activity of the 35% Na_2CO_3/ZSM-5 was compared with other classic catalysts such as concentrated H_2SO_4 and $NaOCH_3$ as shown in Figure 8. Under the optimal catalytic conditions of

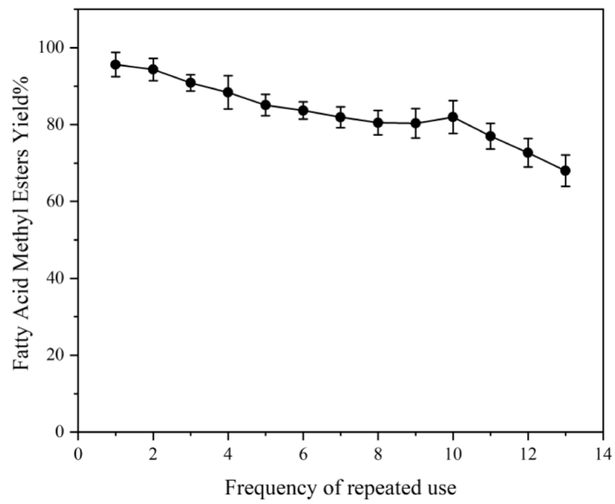

Figure 7. Effects of reuse cycle times on the yields of fatty acid methyl ester.

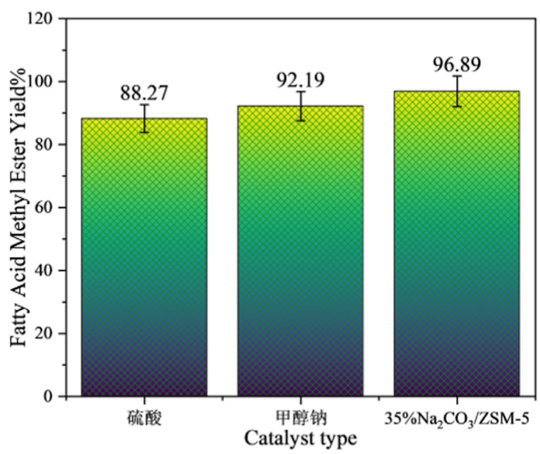

Figure 8. Yields of fatty acid methyl esters catalyzed by concentrated H_2SO_4, $NaOCH_3$, and 35% Na_2CO_3/ZSM-5.

each catalyst, the yield of fatty acid methyl esters was 88.27% (1.2 wt %, 110 °C, 3 h, H_2SO_4) and 92.19% (1.0 wt %, 65 °C, 1.5 h, $NaOCH_3$), respectively. Evidently, the above reaction's relatively harsh conditions, corrosiveness, and recycling challenges resulted in an increase in energy consumption and decrease in reaction efficiency and economic feasibility, despite the catalytic activities being demonstrated by H_2SO_4 and $NaOCH_3$. In addition, the other catalysts for the resource utilization of WCO in the literature are summarized and compared in Table 1. The results indicated that the Na_2CO_3/ZSM-5 catalyst reported by our group had superior performance under relatively low load. Based on these findings, Na_2CO_3/ZSM-5 had great potential in replacing the traditional concentrated H_2SO_4 and $NaOCH_3$ for the resource utilization of WCO, which had significant industrial application value.

4. CONCLUSIONS

In conclusion, an efficient catalyst based on Na_2CO_3/ZSM-5 for the conversion of WCO to fatty acid methyl esters was prepared. The study found that Na_2CO_3/ZSM-5 exhibited excellent performance (yield: 96.89%; reusability: eight times) when Na_2CO_3 loading was 35% and the weight ratio of the catalyst to WCO was 1%. More importantly, Na_2CO_3/ZSM-5

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Table 1. Comparison of the Catalytic Activity of Na_2CO_3/ZSM-5 with Previously Reported Catalysts

name of the catalyst	reaction time (h)	reaction temperature(°C)	WCO/methanol molar ratio	load of the catalyst (wt %)	yield of FAMs (%)	reference no
APS impregnated catalyst	3.5	75	10:1	2.5	93	39
biochar-Fe_2O_3/$Fe_2K_6O_5$	3	60	7:1	4	89	40
$Ca_2Fe_2O_5$−$CaFeO_3$	2	60	12:1	5	90	41
42% HPA/TiO_2	3	60	9:1	0.25	74	42
clinoptilolite/CaO	2	55	16:1	8	85	43
K/KOH/Al_2O_3	1	60	9:1	4	85	44
KOH/Al_2O_3	9	60	12:1	3	89	45
xKOH/Al_2O_3 (x = 25 wt %)	3	65	15:1	4	76	46
xKOH/Al_2O_3 (x = 35 wt %)	5	60	8:1	10	90	47
KF/Al_2O_3 (0.33 wt %)	3	65	12:1	4	90	48
Na_2CO_3/ZSM-5	2	65	12:1	1	97	this paper

demonstrated many advantages over traditional homogeneous catalysts, including mild reaction conditions, environmental friendliness, and so forth.

To the best of our knowledge, Na_2CO_3/ZSM-5 was the first catalyst specifically designed for the resource utilization of WCO, though there was significant room for improvement of catalytic performance. Anyway, this paper provided a new catalyst for resource utilization of WCO, which revealed a potent industrial application value in the future.

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Notes

The authors declare no competing financial interest.

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