Effect of different catalysts on recovery and reuse of waste polyurethane rigid foam

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Abstract
The sharp increase of waste polyurethane rigid foam has caused great harm to the natural environment. Ethylene glycol (EG) and propylene glycol (PG) was the alcoholysis agents, and KOH and Zn3[Co(CN)6]2(DMC) were used as catalysts for alcoholysis of waste polyurethane rigid foam. Regenerated polyols were prepared. The effects of different catalysts on the infrared, viscosity and hydroxyl content of alcoholysis products were discussed. Regenerated polyurethane rigid foam was prepared from alcoholysis products and analyzed by compression strength, thermal conductivity, water absorption, scanning and thermogravimetry. The effects of different catalysts on the recycling of waste polyurethane rigid foam were studied. The experimental results show that DMC catalyst is more effective than KOH catalyst under the same conditions. The alcoholysis product of DMC system has lower viscosity and higher hydroxyl content, which is more suitable for the preparation of regenerated polyurethane rigid foam. The compressive strength of the prepared regenerated polyurethane rigid foam is 254 Kpa, and the thermal conductivity is 0.0213 W m⁻¹·K⁻¹ when the amount of DMC added is 0.04%. Meanwhile, the regenerated polyurethane rigid foam has good hydrophobicity, excellent heat preservation performance, complete cell morphology and good thermal stability. It can replace the traditional polyols to prepare the regenerated polyurethane material which meets the national standard ‘rigid polyurethane foam for building insulation’ (GB/T21558-2005).

1. Introduction

The polyurethane industry has been developing for about 90 years, and the Germans successfully synthesized polyol polyurethane products in 1930s [1]. After World War II, the United States and Britain introduced this technology from Germany one after another. Especially, the United States took the lead in synthesizing soft foam polyurethane with propylene oxide and ethylene oxide copolyether and diisocyanate, which made the United States a major country in polyurethane production and consumption. Germany and Japan have also carried out small-scale recycling of waste polyurethane [2]. After the reform and opening up, domestic industrial departments have purchased a lot of advanced production equipment from abroad, but also imported a large number of polyurethane materials [3]. As a result, the research, development and application of polyurethane materials have been accelerated. Especially since 1986 [2, 4, 5], the polyurethane industry has entered a period of rapid development. In recent years [6], the production technology of polyurethane has been greatly improved. Polyurethane materials are related to all aspects of people’s production and life, and have become indispensable materials in our daily life. However, the application of polyurethane materials is facing great challenges. With the expansion of the global production scale of polyurethane, the consumption of polyurethane also increases. A large number of polyurethane wastes will be produced in the process of production and use of polyurethane, which can only be degraded in 500–1000 years [7]. This will cause a huge burden on the natural environment. Therefore, the recycling of waste polyurethane is an important issue related to environment and resources.
The research on the degradation of waste polyurethane at home and abroad seldom mentions the influence of catalyst [8–10]. The degradation efficiency of traditional catalyst is low, so it is very important to study new and efficient catalysts for the degradation of waste polyurethane [10]. In this paper, the degradation catalyst for recycling polyurethane waste was discussed. The alcoholysis products were obtained by alcoholysis of waste polyurethane rigid foam with different catalytic systems. The infrared spectrum, viscosity and hydroxyl content of the alcoholysis products were tested in order to obtain an efficient catalytic degradation catalyst, effectively degrading waste polyurethane and optimize the best degradation method. The alcoholysis products are used to replace polyether polyls to prepare polyurethane materials [11, 12]. It can not only solve the problem of waste treatment, but also reduce the consumption of resources and realize environmental protection and circular economy [13]. In this paper, waste polyurethane was degraded, recycled and reused under KOH and DMC catalysis system, the alcoholysis product regenerated polyether polyl was obtained, and the regenerated polyurethane rigid foam were synthesized by alcoholysis products, additives, diphenylmethane diisocyanate (MDI) and other raw materials. The degradation efficiency and mechanism were studied. The effects of catalyst types and addition amount on the mechanical properties, water absorption capacity, thermal conductivity, thermal stability, molecular structure and micro-morphology of regenerated polyurethane rigid foam were also studied. This research has great practical application value, which is helpful to promote the industrialization of waste polyurethane recycling.

2. Experiment

2.1. Material
Waste polyurethane rigid foam waste was from Zibo, Shandong, China. Ethylene glycol content of 98% was from Shandong lianhaoyao new material Co., Ltd, China. Propylene glycol content of 98% was from Guangzhou Feirui Chemical Co., Ltd, China. KOH content of 98% was from Tianjin Ruian Technology Co., Ltd, China. DMC content of 99% was from Changzhou Hongyu Chemical Co., Ltd, China. Silicone oil stabilizer content of was from Shanghai Kono Chemical Co., Ltd, China. Tin solution content of 98% was from Shanghai Jieer Technology Co., Ltd, China. Industrial foaming agent 141b was from Shandong Vanke New Materials Co., Ltd, China. Industrial isocyanate (MDI) was from Yantai Fulong Chemical Co., Ltd, China.

2.2. Alcoholysis of waste polyurethane rigid foam
According to the previous experimental study, EG: PG = 6:4 (mass ratio) is the best ratio for alcoholysis. Therefore, 60g EG and 40g PG were mixed and added into the reactor with KOH or DMC catalysts in different proportions (the addition amount was 0.02%, 0.04%, 0.06%, 0.1%, 0.5%, 1%, respectively). Placing the 500ml spherical glass reaction kettle in an electric heating jacket, stirring at 400r/min and heating to 100 °C, and then adding 100g of waste polyurethane rigid foam which is crushed and processed to 10–15 mm, heating to 200 °C for 4h to alcoholize the waste polyurethane rigid foam. Cooling to room temperature, the alcoholysis product is filtered, impurities are removed and poured into a cup to obtain the regenerated polyol, and the yield can reach 95%, which is named DM, viscosity and infrared test were carried out. The reaction mechanism is mainly that the urethane bond in polyurethane is broken and replaced by small molecule alcohol chain under the joint action of alcoholysis agent and catalyst so as to obtain recycled regenerated polyol.

2.3. Preparation of regenerated polyurethane rigid foam
In order to explore the influence of DM prepared by alcoholysis under different catalysts on the properties of recycled polyurethane hard foaming, DM prepared under the above different conditions was used as the raw material to prepare regenerated polyurethane hard foaming. The preparation process is as follows:

(1) Weighing 10 g of alcoholysis product DM and 20 g of polyether polyl (4110), vacuum drying and dehydrating at 80 °C for 1.5 h. Then adding the alcoholysis product and polyether polyl (4110) into a disposable plastic cup, stirring for 5 min by an electric stirrer, and placing in a vacuum drying oven for secondary dehydration for 1 h, get the mixture.

(2) Weighing 50 g of diphenylmethane diisocyanate (MDI), vacuum drying and dehydrating at 80 °C for 1.5 h. After cooling to room temperature, the mixture is added and stirred at a constant temperature of 65 °C for 20 min minutes to perform a prepolymerization stage.

(3) Adding the catalyst (amine and tin), foaming agent (141b and H2O) and surfactant (silicone oil) into the disposable plastic cup, stirring and mixing evenly, and then let it stand for 2 min When the mixed solution is obviously layered, it is mixed with the prepolymer and stirred until the filament of 1 cm can be pulled out with a glass rod, and then stopped, waiting for the foam to grow continuously until the foaming reaction is...
finished. The prepared porous regenerated polyurethane rigid foam is designated as PUDM. The corresponding synthetic formula is shown in table 1.

2.4. Performance testing and structural characterization

The viscosity was measured by rotational viscometer DSW-4. The alcoholysis products were placed in a designated container and the viscosity was tested at 25 °C. According to GB/T12008.3-2009 standard, a proper amount of regenerated polyol was put into a 10ml conical flask, and the hydroxyl content was determined by pyridine with ester anhydride method. According to GB 8813-2008 test standard, sample size is 50 mm × 50 mm × 50 mm, and compression strength is tested by EFS-24RE universal testing machine.

According to GB/T8810-88 test standard, the sample size is 50 mm × 10 mm × 10 mm, and the water absorption rate is measured by distilled water, and the quality of the sample before and after soaking is weighed to calculate its water absorption rate.

According to QB/T 3806-1999, the sample size is 200 mm × 200 mm × 20 mm, and the instrument adopts FEHC-5 thermal conductivity tester of Changzhou Huaoa Instrument Manufacturing Co., Ltd. The foam sample was cut into thin slices, and then the microstructure of the foam was observed by scanning microscope, magnified 20 times. The area with clear and uniform cell structure was observed and the observation results were recorded.

The structure of foam sample was analyzed by the GR-285 infrared spectrometer produced by Dalian Precision Scientific Instrument Co., Ltd. The sample was prepared by KBr pressing method and the test wavelength range was 400–4000 cm⁻¹.

The thermogravimetric analysis was carried out with the heating from room temperature 25 °C to 500 °C at a rate of 30 °C/min in a nitrogen atmosphere and the gas flow rate is 50 ml/min. Alumina is taken as the reference object.

3. Results and discussion

3.1. Reaction Mechanism of Alcoholysis and DM foaming of waste PU hard foam

The alcoholysis mechanism of waste polyurethane is:

\[
R_1 - \text{NHCOO} - R_2 + \text{HO} - R_3 - \text{OH} \rightarrow R_1 - \text{NHCOO} - R_3 - \text{OH} + R_2 - \text{OH}
\]  (1)

Many side reactions will occur because there are many groups participating in the reaction in the degradation process. The main side reaction is that the urea group breaks to form amine and polyol under the action of alcoholysis agent. The reaction process is:

\[
R_1 - \text{NHCONH} - R_2 + \text{HO} - R_3 - \text{OH} \rightarrow R_1 - \text{NHCOO} - R_3 - \text{OH} + R_2 - \text{NH}_2
\]  (2)

Figure 1 shows the foaming mechanism of regenerated polyurethane rigid foam. The reaction mechanism is mainly that isocyanate reacts with water to generate carbon dioxide, and bubbles generated by carbon dioxide are foamed. At the same time, the reaction is exothermic reaction. The reaction will release a lot of heat, and the foaming agent is volatile when heated, volatilization into gas to complete the auxiliary foaming.
3.2. Chemical structure analysis of waste polyurethane rigid foam DM

It is known that waste polyurethane can be alcoholyzed into DM under the action of DMC and KOH catalysts, and the DM can be used to prepare regenerated polyurethane rigid foam. Therefore, taking polyether polyol (4110) as the contrast sample, the DM prepared by different catalysts was characterized by infrared spectroscopy. The results are shown in figure 2.

As can be seen from the figure 2, the alcoholyis products under the two catalyst systems are basically similar to polyether polyol 4110 in characteristic peak type. The absorption peak near 3200–3500 cm$^{-1}$ is the absorption peak of the stretching vibration peak of alcohol hydroxyl groups [14]. The absorption peak at 1735 cm$^{-1}$ is the absorption peak of benzene [15], and the clear absorption peak at 1070 cm$^{-1}$ is the absorption peak of polyether polyurethane ether group (C-O-C). The results show that the waste polyurethane is successfully alcoholyzed into the mixed product of polyether polyol and aromatic polyol under the two catalyst systems. Its chemical properties are similar to polyether polyol 4110, which can replace 4110 to prepare polyurethane materials. The mechanical properties of regenerated polyurethane rigid foam are better than those of polyurethane rigid foam prepared by 4110 because there are benzene rings with rigid structure in aromatic polyols.

3.3. Viscosity analysis of DM in different catalytic systems

In the production process, the viscosity of DM is an important factor affecting the properties of the product. It can be seen from figure 3 that the viscosity of DM changes with the type and content of catalyst in the process of alcoholyis foaming of waste polyurethane rigid foam. Only when the fluidity of DM reaches the standard in the
foaming process can be fully filled, so the fluidity of DM is very important. The viscosity standard range of polyether is 4000–8000 MPa·s (25 °C) \[16\], and the lower the viscosity in the standard range, is better. It can be clearly seen that the viscosity of DM prepared by DMC catalyst system is basically lower than that of KOH system. The viscosity of DM decreases with the increase of KOH content and is generally higher when the amount of KOH is small. The viscosity decreases obviously and the value of the viscosity is 4408 mpa.s when the addition amount increases to 0.5%. When the addition amount is 1%, the viscosity is 4320 mpa.s. So, the viscosity change is not obvious when the addition amount is greater than 0.5. In the DMC system, the viscosity of DM decreases first and then increases with the increase of DMC content. The viscosity changes greatly when the amount of DMC is small, and the viscosity value is also low. The minimum viscosity is 2340 MPa.s when the amount of DMC is 0.04%. Therefore, it can be seen that DMC catalyst is a highly efficient catalyst with high activity and can play a good catalytic effect at a low dosage. However, when the addition of DMC is too high, the viscosity of DM increases mainly because the DMC catalyst is a nanometer catalyst. It will lead to the agglomeration of the catalyst and fail to achieve the original catalytic activity. So that the alcoholysis reaction is not thorough enough and cannot be completely alcoholized into low molecular weight polyols, resulting in the increase of viscosity of the system.

### 3.4. Hydroxyl content analysis of DM in different catalytic systems

Under the same experimental conditions, the alcoholysis of waste polyurethane rigid foam was carried out with different amounts of KOH or DMC catalyst as variables, and the hydroxyl content of DM was tested. The results are shown in Table 2.

| Addition of catalyst (%) | Hydroxyl content (mgKOH·g⁻¹) |
|-------------------------|-------------------------------|
|                         | DM of DMC | DM of KOH |
| 0.02                    | 421       | 135       |
| 0.04                    | 437       | 153       |
| 0.06                    | 415       | 192       |
| 0.1                     | 372       | 248       |
| 0.5                     | 321       | 385       |
| 1                       | 254       | 350       |

It can be seen from Table 2 that the maximum content of DM hydroxyl content in DMC system is greater than that in KOH system. In DMC system, the hydroxyl content is the maximum 437 mgKOH·g⁻¹ when the addition of DMC is 0.04%. In KOH system, the hydroxyl content is the maximum 385 mgKOH·g⁻¹ when the KOH addition is 0.5. This shows that the catalytic activity of DMC is much higher than that of KOH, and it can achieve higher catalytic effect with lower addition amount. Therefore, DMC catalyst has the characteristics of low addition and high activity, which can fully crack the urethane bond in waste polyurethane into polyol in a short time and prepare DM with larger hydroxyl number. In the two catalytic systems, the hydroxyl content of DM first increases and then decreases with the increase of catalyst addition, and the hydroxyl content of DMC system decreases more obviously. On the one hand, the reason is that the DMC catalyst has high activity and more obvious side reactions will occur after excessive amount, which will lead to urea group cleavage to generate amine instead of being substituted by short alcohol chain to generate polyol. On the other hand, the DMC catalyst is a nano-scale catalyst. If the addition amount is too high, it will cause the catalyst to agglomerate and fail to achieve the original catalytic activity, making the alcoholysis not complete enough, and cannot be completely alcoholized into low-molecular-weight polyols. The two factors together cause the DM hydroxyl content to drop more significantly.

### 3.5. Analysis of mechanical properties of different PUDM

The prepared PUDM was subjected to a pressure test (percentage deformation is 20%). The influence of different catalyst additions on the compressive strength of PUDM was known through the test, as shown in Figure 4.

It can be seen in Figure 4 that in DMC and KOH catalytic system, the compressive strength of PUDM first increases and then decreases with the increase of catalyst addition. In DMC system, the main factor is that agglomeration occurs after excessive catalyst, which leads to incomplete alcoholysis of DM and further affects the preparation of PUDM. In KOH catalytic system, the hydroxyl content of DM decreases due to side reaction after excessive catalyst, which further affects the preparation of PUDM. Overall, the compressive strength of
PUDM prepared by DM in DMC catalytic system is higher than that of PUDM prepared by DM in KOH catalytic system. The difference is more obvious at low addition level. It indicates that the DMC catalyst has the high catalytic activity. The reason is that the lattice type of DMC catalyst is a face-centered cubic structure and has good structural stability. It improves the mechanical properties of PUDM and increases its compressive strength. At the same time, the DMC catalyst is nano-sized that can be well dispersed in DM and can induce the formation of hydrogen bonds between PUDM molecules in the foaming process. It also enhance the intermolecular force and further improving the compressive strength of PUDM. The compressive strength (254 Kpa) of PUDM prepared by 0.04% DMC system is 35.1% higher than that of 1% KOH system (188 Kpa) when the optimum amount of catalyst is added to the two systems respectively.

3.6. Analysis of thermal conductivity of different PUDM

The DM was foamed with different adding amounts of DMC and KOH. The thermal conductivity of the prepared PUDM is tested and the influence of different catalyst addition amount on the thermal conductivity of PUDM can be shown in table 3.

PUDM prepared by DM in DMC catalytic system is higher than that of PUDM prepared by DM in KOH catalytic system. The difference is more obvious at low addition level. It indicates that the DMC catalyst has the high catalytic activity. The reason is that the lattice type of DMC catalyst is a face-centered cubic structure and has good structural stability. It improves the mechanical properties of PUDM and increases its compressive strength. At the same time, the DMC catalyst is nano-sized that can be well dispersed in DM and can induce the formation of hydrogen bonds between PUDM molecules in the foaming process. It also enhance the intermolecular force and further improving the compressive strength of PUDM. The compressive strength (254 Kpa) of PUDM prepared by 0.04% DMC system is 35.1% higher than that of 1% KOH system (188 Kpa) when the optimum amount of catalyst is added to the two systems respectively.

### Table 3. Effect of different catalyst addition amount on thermal conductivity of PUDM.

| Addition of catalyst (%) | Thermal Conductivity (W/m·K) |
|-------------------------|-----------------------------|
|                         | PUDM (DMC system) | PUDM (KOH system) |
| 0.02                    | 0.0236             | 0.0284             |
| 0.04                    | 0.0213             | 0.0275             |
| 0.06                    | 0.0232             | 0.0265             |
| 0.1                     | 0.0238             | 0.0254             |
| 0.5                     | 0.0243             | 0.0238             |
| 1                       | 0.0249             | 0.0242             |

It can be seen from the table 3 that the thermal conductivity of PUDM prepared by two different catalyst systems DM decreases at first and then increases with the increase of catalyst addition. There is little difference in thermal conductivity. However, the thermal conductivity of PUDM prepared by DMC system is relatively lower. The minimum thermal conductivity of PUDM is 0.0213 W m⁻¹·K⁻¹ when the addition of DMC is 0.04%, which is lower than that of 0.03 W/(m·K). It meets the requirements of 0.025–0.036 W/(m·K) in the national standard ‘Rigid Polyurethane Foam for Thermal Insulation of Buildings’ (GB/T44248—2012) [17]. It indicates that its thermal insulation performance is relatively better.
3.7. Analysis of water absorption performance of different PUDM

The effect of different catalyst addition amount on water absorption of PUDM was studied by the measuring water absorption. At first, PUDM is made into a cubic sample with a volume of 1 cm$^3$, and then placed in deionized water weighed after waiting for free water absorption. Finally, put the sample in an oven for drying, dewatering and weighing. The water absorption of PUDM with different amount of catalyst is calculated by the formula, and the result is shown in Figure 5.

It can be seen from the Figure 5 that the water absorption of PUDM prepared by two different catalyst systems DM is quite different. The water absorption of PUDM increases first and then decreases with the increase of catalyst addition. In general, the water absorption rate of PUDM prepared by DMC system is lower. The water absorption rate of foam is closely related to the integrity of cells. The lower the water absorption rate, the more complete the cell structure and the higher the closed cell ratio of PUDM, which can lock more gas and make PUDM have better heat insulation performance and heat preservation performance. In addition, the bubble will burst under the pressure of water when the foam enters the water. It will improve the water absorption of PUDM. So, the compressive strength of PUDM will also affect its water absorption. The possibility of bubble rupture is smaller and the water absorption is lower when the compressive strength of PUDM is greater.

3.8. Morphology analysis of different PUDM

The PUDM prepared by the two groups of schemes with better comprehensive properties in DMC and KOH systems were selected and cut into thin slice samples. The areas with clear field of vision and well-organized bubbles were selected by the scanning electron microscope shown in Figure 6.

It can be seen that, the molecular chain of waste polyurethane is not completely broken and the alcoholyis reaction is not complete when the catalyst is added too little in DMC system. It leads to the incomplete cell structure of PUDM prepared when the amount of DMC is 0.02%. In the KOH system, the by-products will be produced in the alcoholyis reaction when too much catalyst is added. It will affect the performance of PUDM and result in the incomplete cell structure of PUDM when the amount of KOH is 1%. Therefore, it can improve the integrity of the bubble hole and make its shape more regular only throught appropriate amount of catalyst. The prepared PUDM cells are relatively intact and regular and the cell size is relatively uniform in the whole field of vision when the DMC addition is 0.04% or the KOH addition is 0.5%. It indicates that the foam is relatively stable in the initiation process. Among them, the cell wall of the foam is thicker and more uniform and the geometric configuration of the skeleton is better when the addition of DMC is 0.04%. It indicates that the cross-linking degree of the foam is very good and can provide a good compressive strength for the foam. This is because the lattice type of DMC catalyst is face-centered cubic structure and has good structural stability. It will increase the compressive strength of PUDM and further verifies that PUDM has the best mechanical properties when the addition of DMC is 0.04%. At the same time, it can be found in the visual field that the cell integrity rate is very high, the film formation is relatively good, and the cell closure degree is also high. So the closed cell rate of foam is relatively high and the closed cell rate is of great significance to the heat preservation and heat insulation.
performance of PU foam. Therefore, it can be seen that foam with good thermal insulation and insulation performance can be prepared by adding proper amount of catalyst.

3.9. Thermal stability analysis of different PUDM

Two groups of PUDM with better comprehensive properties in DMC and KOH system were selected for thermogravimetric analysis. Figure 7 shows the thermal weight loss spectra of different PUDM. The thermogravimetric curves in the figure are the thermogravimetric rate of PUDM at different degradation temperatures that is used to study the thermal stability of PUDM.

It can be seen in figure 7 that the thermogravimetry curve mainly includes two stages of thermogravimetry (155 °C–290 °C) and (320 °C–410 °C) due to the soft segment and hard segment of PUDM. The quality loss in the first stage is mainly water loss and hard segment decomposition in PUDM. The quality loss in the second stage is mainly the decomposition of soft segment in PUDM.

In the first stage (155 °C–290 °C), it can be seen that the initial decomposition temperatures of PUDM prepared with 0.5% and 1% KOH are 152 °C and 201 °C and those with 0.02% and 0.04% DMC are 212 °C and 252 °C. This indicates that the thermal stability of PUDM prepared with DMC system will be improved. It can be seen from the second stage (320 °C–410 °C) that the thermal weight loss temperature of PUDM prepared by DMC system is obviously increased and the rate of thermal degradation is obviously decreased compared with the KOH system. It indicates that the thermal stability of PUDM is improved. On the one hand, the reason is that the hydroxyl content in the DM prepared by DMC system is larger and it makes the hard segment of the prepared PUDM larger. It further increases the cross-linking density of the space in PUDM. The thermal weight loss rate of PUDM increases with the increase of the amount of KOH. It indicates that the increase of KOH catalyst will reduce the thermal stability of PUDM [18]. Therefore, DMC system can enhance the thermal stability of PUDM to some extent.
4. Conclusion

Waste polyurethane rigid foam was alcoholyzed into regenerated polyol and PUDM was successfully prepared using EG and PG as alcoholysis agents. The infrared spectrum shows that the spectrum of the recycled polyol is similar to that of the traditional polyether polyol with hydroxyl and ether bonds. It indicates that the waste polyurethane rigid foam has been degraded successfully and can replace the traditional polyether to prepare PUDM. Viscosity and hydroxyl content tests show that DMC catalyst can make the alcoholyis of waste polyurethane more thorough at low dosage and the prepared regenerated polyol has lower viscosity and higher hydroxyl content. It is more suitable for the preparation of PUDM. The mechanical properties test shows that it has the best reinforcement and toughening effect on PUDM by adding 0.04% DMC. The test of thermal conductivity shows that the thermal conductivity is the lowest and its thermal insulation performance is better when DMC content is 0.04%. The water absorption test shows that the hydrophobic performance of PUDM is the best when the amount of DMC is 0.04%. It indicates that the thermal insulation performance of PUDM is better at this time. The characterization of scanning electron microscope shows that when the amount of DMC was 0.04% the cell wall of the prepared PUDM foam was thicker and uniform, the skeleton geometry was better and the foam had high compressive strength and thermal insulation properties. The thermogravimetric characterization spectra show that the DMC system can enhance the thermal stability of PUDM to some extent. Through the test, it can be seen that the alcoholysis effect of DMC catalyst system is better than that of KOH catalyst system in the alcoholyis process of waste polyurethane. The alcoholyis is more thorough and the prepared PUDM also has better comprehensive properties. At the same time, the added amount of DMC catalyst can achieve high catalytic efficiency when it is very low.

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

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