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Double metal cyanide complex as a heterogeneous catalyst for depolymerization of polyesters

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ABSTRACT

Plastic wastes have caused a series of environmental issues, and it has become imperative to promote the recycling of them. As an effective means to solve environmental problems and resource utilization, the chemical depolymerization of polyester via transesterification reaction was practical. Therefore, finding industrially viable transesterification catalysts is an effective strategy for establishing a recycling route for polyester plastics. Herein, we report double metal cyanide complex (DMC) directly catalyze polyester depolymerization to recover the corresponding monomers or chemicals. Poly(ethylene terephthalate) (PET) was used as representative polyester to investigate the influences of experimental conditions including the loading of the catalyst and the molar ratio of ethylene glycol (EG) to PET on the depolymerization of polyester and the selectivity of monomeric products. The kinetics of the glycolysis has been measured. PET could be completely depolymerized at 190 °C by 2 h with the molar ratio of EG/PET of 15 : 1 and the catalyst loading of 1 wt%. DMC also showed high catalytic activity on the depolymerizations of other commercial plastics of ester main-chains. DMC, a solid catalyst enjoyed inexpensive, robust, and water tolerant features in industrial process, showed prominent activity and selectivity in polyester depolymerizations and suggested scale commercial application scenario.

1. Introduction

Plastics play a significant role in our modern society due to their convenience and wide applications. Over the past few decades, the utilization of plastics has been growing rapidly. Global plastics production increased to 413.8 million tones in 2023 [1,2]; however, the wide range of applications will inevitably bring different levels of pollution. More than 80 % of plastic wastes are buried in garbage landfills or scattered in the natural environment [3,4]. Due to its inert molecular structures, it takes hundreds of years to degrade into small molecules completely or other nontoxic substances, thus leading to serious waste of resources and environmental burden. To address this issue, the development of depolymerization methods that can be used to convert end-life plastics into valuable monomers or chemicals may be a useful strategy [5–7].

Plastic recycling routes mainly implemented by physical, biological, and chemical methods [8–14]. Among them, chemical depolymerization has attracted a lot of attentions because of the ability to convert polymers into oligomers or monomeric fragments that can be used directly to produce new polymers. The chemical recycling of several common commercial plastics, such as poly(ethylene terephthalate) (PET), polylactide (PLA), and poly(bisphenol A carbonate) (BPA-PC) have been widely studied due to the presence of easily cleavable carbon−oxygen (ester) bonds. The chemical recycling of these polyesters by alcoholysis is essentially reversible transesterification reactions, the reaction rate can be increased in a controlled manner by using proper catalyst that can lower the activation energy [15].

Many transesterification catalysts have been developed for depolymerization of polyesters in recent years, such as metal salts [15–22], metal oxides [23–25], ionic liquids [11,26–37], and metal−organic frameworks [38,39]. However, these catalysts face some limitations, such as being able to be applied to only one type of polymer, or the catalyst being expensive and having no potential for industrialization. Therefore, there is still a need for the development of more versatile and

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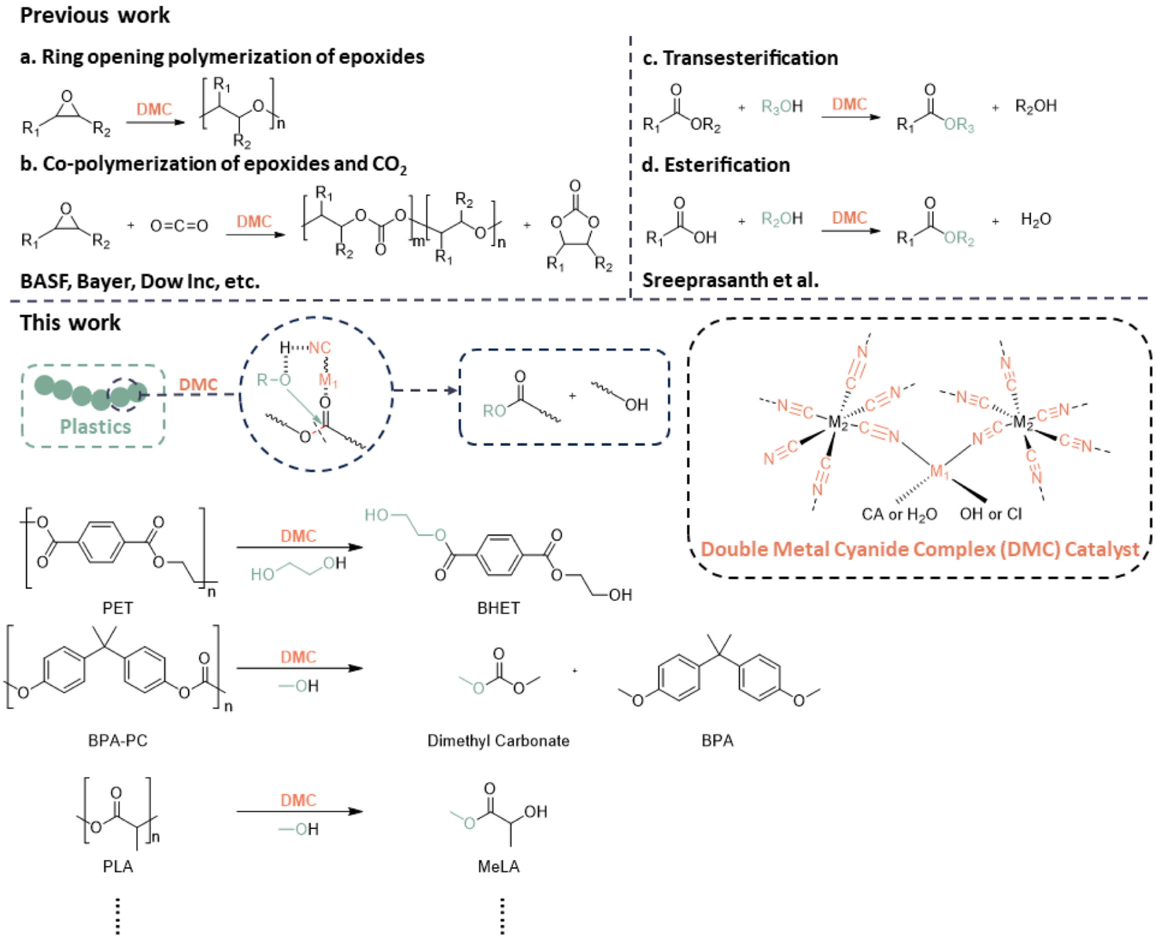


Scheme 1. Possible catalytic depolymerization of polyesters by double metal cyanide complex.

economic glycolysis catalysts and processes.

As an excellent transesterification catalyst [40], double metal cyanide complex (DMC) has attracted our attention. Over the past few decades, DMC has been widely used in industry for the ring-opening polymerizations of epoxides (Scheme 1, **a**) [41–44], and in the co-polymerizations of epoxides and CO_2 (Scheme 1, **b**) [45–49]. DMCs are well recognized metal complex with frameworks containing metal cyanide moieties. Chemically they are Prussian blue analogs. In previous studies, DMCs were characterized in cubic lattice structures (Scheme 1) [50,51]. In addition to polymerizations, other uses of DMC catalysts have been discovered [50,52–57]. The DMC catalyst was first used for esterification in 2006 [40]. Later, it was used in transesterifications such as in the manufacture of biodiesels [58,59], hydrolysis of oils and fats [60], and transesterifications of propylene carbonate with methanol [61]. Due to its high catalytic activity for transesterifications, DMC was potentially applicable in the depolymerizations of PET and other plastics containing ester groups in the main-chains (Scheme 1).

We report here, for the first time, the application of DMC catalysts for the depolymerization of plastics. Both the effect of the structures and compositions of the catalysts and the reaction conditoins on the depolymerization of PET was investigated. In addition, the depolymerizations of other commercial polyesters under the catalytic sovolysis by DMC were evaluated. The depolymerization products were characterized and analyzed, expecting that this kind of catalysts could be useful for the industrial recovery of plastics.

2. Experimental section

2.1. Materials

PET pellets were supplied by Sinopec Yizheng Chemical Fiber Co. Ltd. Before use in the glycolysis, PET pellets need to be crushed and sieved to ensure that their particle size is 60–80 mesh (180–125 μm). The post-consumer PET bottle was from Tingyi (Cayman Islands) Holding Corp. All the other chemicals and reagents were purchased from Aldrich Co. and used without further purification.

2.2. Characterizations

Specific surface area, pore volume, and the pore size of the catalyst were determined by the Brunauer−Emmett−Teller (BET) method from the N_2-adsorption-desorption isotherms using Quantachrome NovaWin Autosorb iQ-Chemisorption & Physisorption Gas Sorption Analyzer. The average pore diameter was determined by the Barrett− Joyner−Halenda (BJH) method. Scanning electron microscope (SEM) images were obtained on a FEI Scios 2 HiVac scanning electron microscope instrument. Energy Dispersive X-ray Spectroscopy (EDX) of elemental composition and distribution of metals were determined using a silicon drift EDX detector. X-ray powder diffraction (XRD) analysis was performed using a Bruker D8 Advance wide-angle goniometer 185 with Cu Kα radiation. XRF data was obtained on Axios wavelength dispersive X-fluorescence spectrometer. Thermogravimetric analysis (TGA) was done using a Q600 STD instrument with air flow (50 std cm^3 /min) at a heating rate of 10 °C/min in the 50 −600 °C range. Diffuse Reflection Infrared Fourier Transform Spectroscopy (DRIFTS) spectra for the catalyst samples were collected in the range of 1000 −4000 cm^{-1}, using a Bruker Tensor 27 FT-IR equipped with a Pike diffuse reflectance cell. ^1H and ^{13}C NMR spectra were recorded on a Bruker Avance 400 MHz NMR spectrometer in DMSO-d_6 as stated deuterated solvents. Chemical shifts δ are reported in parts per million (ppm) relative to a residual undeuterated solvent as an internal reference ($^1H\delta$ 2.50 for DMSO-d_6; ^{13}C δ 39.52 for DMSO-d_6).

2.3. General procedure for preparation of DMC

Typical preparation procedure of double metal cyanide complex described as follows: 0.01 mol of $K_4[Fe(CN)_6]·3\ H_2O$/ $K_4Co(CN)_6$ was dissolved in 40 mL of distilled water in beaker A. Targeted amount of $ZnCl_2/MnCl_2/NiCl_2$ was dissolved in 100 mL of distilled water and 20 mL of *tert*-butanol in beaker B. 15 g of poly(ethylene glycol)-block-poly(propylene glycol)-block-poly(ethylene glycol) ($EO_{20}PO_{70}EO_{20}$; average molecular weight, 5800) was dissolved in beaker C with 2 mL of distilled water and 40 mL of *tert*-butanol. The solution in beaker B was added to beaker A slowly over around 1 h at 323 K under vigorous stirring. Then solution in beaker C was added to the above mixture over 5–10 min. After this, the mixture was vigorously stirred for 1 h at 323 K. The solid formed was then washed and filtered to remove the uncomplexed ions. Then the solid was vacuum dried at 333 K for two hours. The final catalyst was obtained after activating the material at 453 K for 4 h.

2.4. Measurement of catalytic activity

A 10 mL Schlenck flask equipped with a magnetic stirrer was loaded with 0.5 g of PET pellets and a certain amount of EG and catalyst. Then seal the flask and immersion in an oil bath at a specific temperature for the required time. When each glycolysis reaction finished, the undegraded PET pellets were quickly separated from the liquid phase before the products cooled and precipitated. Then an excess amount of cold water was used to wash the undegraded PET pellets, and the water was then mixed with the product fraction. The undegraded PET was collected, dried, and weighed. The conversion of PET is defined by Eq. (1):

$$\text{conversion of PET}(\%) = \frac{W_{PET,i} - W_{PET,u}}{W_{PET,i}} \times 100 \quad (1)$$

where $W_{PET,\ i}$ represents the initial weight of PET and $W_{PET,u}$ represents the weight of undegraded PET.

The glycolysis product mixture was vigorously agitated (cold distilled water would dissolve the remaining ethylene glycol and the monomer) and then filtered. The collected filtrate was concentrated to about 20 mL. The concentrated filtrate was stored in a refrigerator at 0 °C for 24 h. White needle-like crystals were formed in the solution, which were then recovered by filtration before drying. ^1H NMR, ^{13}C NMR, and FTIR spectroscopic characterizations revealed the crystals to be highly pure bis(hydroxyethyl) terephthalate (BHET) monomer with characterizing data in accordance with commercially supplied BHET. ^1H NMR (400 MHz, DMSO-d_6) δ (ppm) 8.12, (*s*, 4 H, CH), 4.97 (*t*, 2 H, OH), 4.32 (*t*, 4 H, O–CH_2), 3.73 (*q*, 4 H, CH_2–OH). ^{13}C NMR: δ (ppm) 165.14 (2 C, C═O), 133.73 (2 C, –C–C═O) 129.50 (4 C, CH), 67.01 (2 C, O–CH_2), 58.96 (2 C, CH_2–OH). The selectivity of BHET is calculated by Eq. (2):

$$\text{selectivity of BHET}(\%) = \frac{\text{moles of BHET}}{\text{moles of depolymerized PET units}} \times 100 \quad (2)$$

Depolymerizations of other polyesters are described in the supporting information.

Table 1
PET glycolysis catalyzed by different composed DMC catalysts[a].

Entry	Catalyst	Conversion of PET (%)	Yield of BHET (%)
1[b]	-	3	< 1
2[c]	Zn-Co DMC	41	33
3	Zn-Co DMC	79	63
4[d]	Zn-Co DMC	77	62
5	Mn-Co DMC	18	< 1
6	Ni-Co DMC	5	< 1
7	Zn-Fe DMC	39	25
8	Mn-Fe DMC	46	31
9	Ni-Fe DMC	37	26

[a] Reaction conditions: PET (0.5 g), EG (2.17 mL), atmospheric pressure at 180 °C, 5 h. All of the DMCs were added to the PET glycolysis system with the same weight (2.5 mg).
[b] Glycolysis without DMC.
[c] Commercial catalyst.
[d] Depolymerization of post-consumer PET bottles.

3. Results and discussion

3.1. Preparation of DMCs

DMCs are Prussian blue analogs with a general formula of $M^A_m[M^B(CN)_6]_n·xH_2O$ consisting of a three-dimensional cubic M^A-NC-M^B framework with some $[M^B(CN)_6]$ vacancies. The metal M^A can be Zn^{2+}, Ni^{2+}, Mn^{2+}, and so on; and M^B can be Fe^{2+}, Fe^{3+}, Co^{3+}, and so on. For heterogeneous catalysts, the preparation method will affect the catalytic activity, so in addition to commercial catalysts, several catalysts were prepared under laboratory conditions. It is worth noting that DMCs are precipitated by the addition of an aqueous solution of metal halide to an aqueous solution of hexacyanometallate or vice versa, and most of the time the crystallization of the catalyst is performed in the presence of a kind of coordinating organic molecules (complexing agents, CA) to the improve the catalytic activity of DMCs. Among the complexing agents, acyclic ethers were superior to cyclic ethers and appeared to be the most effective and widely used complexing agent. In this study, the effect of the complexing agent will not be discussed, and *t*-BuOH will be selected as the complexing agent [62]. Moreover, surfactant category molecules (designated as co-complexing agents, co-CA) can be added during the synthesis. These agents can act as surfactants to disperse the crystal nucleus during DMC crystallization (protecting agents) resulting in the formation of smaller size catalyst particles. Commonly used co-complexing agents include poly(tetramethylene ether glycol) (PTG), polypropylene glycol (PPG), polyethylene glycol (PEG), poly(ethylene oxide)-poly(propylene oxide)-poly(ethylene oxide) triblock copolymer (P123) and so on. P123 is selected as the co-CA in this study. The characterization of catalysts is detailed in the supporting information.

3.2. Catalytic performance

The catalytic effect of the differently composed DMC on the glycolysis of PET was investigated, and the results are summarized in Table 1. Considering the uncertainty of the molecular weight of the DMC catalyst, the comparison of catalytic activity was performed by weight rather than by molar ratio. A minor degradation effect was observed for PET without the addition of catalyst (Table 1, entry 1). In contrast, the addition of DMC significantly improves the depolymerization efficiency (Table 1, entries 2–9). Compared with the newly prepared catalysts, commercial catalyst exhibits lower catalytic activity (Table 1, entries 2–3). Regarding the source of the depolymerization material, there is no significant difference in the depolymerization effect of commercial PET pullets and post-consumer PET bottles (Table 1, entries 3–4). Zn-Co-DMC, the most widely used DMC in literature [42,50,51,63], has significant catalytic efficiency compared with other DMCs, achieving 79 % PET transformation and 63 % BHET yield within 5 h at 180 °C (Table 1,

Table 2
Influence of the amount of the DMC catalyst on the glycolysis of PET[a].

Entry	Amount of Catalyst (wt%)	Conversion of PET (%)	Yield of BHET (%)
1	0	3	< 1
2	0.1	12	3
3	0.5	79	63
4	1	94	72
5	2	> 99	75
6	3	> 99	75

[a] Reaction conditions: PET : EG = 1 : 15; time, 5 h; temperature, 180 °C.

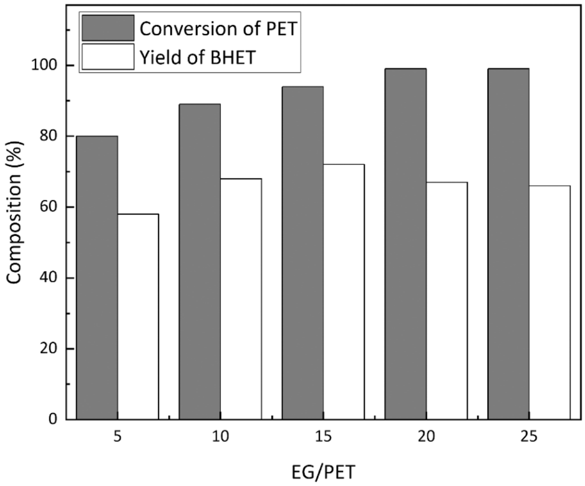


Fig. 1. Effects of the ratio of EG on the glycolysis of PET (Conditions: catalyst loading, 1 wt%; time, 5 h; temperature, 180 °C).

entry 3). DMC based on Zn-Co, Zn-Fe, and Mn-Fe have already been shown to be excellent heterogeneous catalysts for transesterifications [41,42,44,45,49,61]; they also show highly catalytic activity on glycolysis of PET (Table 1, entry 3, 7–8), which was corresponding with anticipation. The relationship between conversions and yields indicates that the long chain structure of the polymer was degraded into oligomers, while the oligomers are not completely degraded; thus the yield of BHET is always lower than the conversion of PET. Zn-Co DMC was chosen for further investigations.

3.3. *Influence of the amount of the catalyst*

The influence of the amount of catalyst on the conversion of PET and the yield of BHET is shown in Table 2. There is a clear correlation between the amount of catalyst and the effect of depolymerization. As mentioned earlier, no significant depolymerization effect was observed without the addition of catalyst. When the amount of Zn-Co DMC catalyst is 0.1 wt%, the conversion can be observed, which is 12 %. The conversion of PET and yield of BHET increases with the increase of the amount of Zn-Co DMC. When the amount of catalyst reaches 1 wt%, the conversion reaches 94 % and the yield reaches 72 %, the effect of DMC on the reaction appears to have reached saturation thus if the amount of catalyst increases to 2 %, the catalytic effect will not be significantly improved. From this perspective, 1 wt% of the amount of Zn-Co DMC seems to be a better option for further investigation.

3.4. *Influence of the ratio of EG on the glycolysis of PET*

EG is not only used as raw material to participate in the reaction but also as a reaction solvent to dissolve PET in the glycolysis of PET. Theoretically, an increase in the amount of EG within a certain range will promote depolymerization efficiency. However, excess EG will dilute the amount of catalyst and thus reduce the concentration of the reaction solution. Therefore, the effect of the amount of EG on the conversion of PET and the selectivity of BHET has been investigated (Fig. 1). Even reducing the equivalents of EG to 5 eq, the conversion still can reach 80 %. As the EG equivalents increases to 15, the conversion of PET also increases to 94 %, which is only 14 % higher than that at 5 equivalents. The reason that the amount of EG has little effect on the catalytic effect can be attributed to the fact that the catalytic efficiency of solid catalysts is mainly related to the surface area of the catalyst, the increase of EG ratio does not increase the surface area of the catalyst, thus its catalytic activity is constant within a certain range. This means that it may be possible to reduce the amount of EG in pursuit of higher economic efficiency.

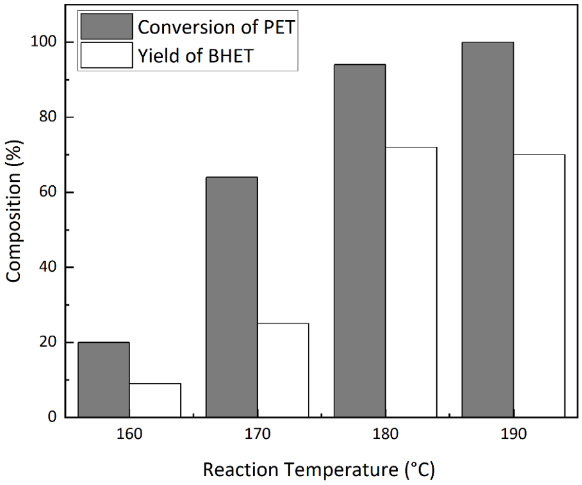


Fig. 2. Effects of reaction temperatures on the glycolysis of PET (Conditions: PET : EG= 1 : 15; catalyst loading, 1 wt%; time, 5 h).

3.5. *Effect of reaction temperatures*

Fig. 2 indicates the effects of reaction temperature on the conversion of PET and yield of BHET. Fig. 2 shows that the glycolysis of PET can be carried out when the reaction temperature is below 160 °C. This means that the Zn-Co DMC can catalyze the glycolysis reaction at a lower temperature. Subsequently, with increasing reaction temperature, the conversion of PET increases apparently; and it reaches 100 % when the reaction temperature is 190 °C. Fig. 2 shows the reaction result obtained at 5 h, and PET may be completely consumed at 190 °C in a very short time, so the kinetic experiments were investigated, and the results can be presented below.

3.6. *Kinetics analysis*

The depolymerization is assumed to be represented by a first-order reaction [64,65]. The depolymerization reaction rate should be proportional to the concentration of the PET repeating unit and EG. The equation for the rate of reaction can be shown as:

$$\frac{d[\mathrm{PET}]}{dt} = -k[\mathrm{EG}][\mathrm{PET}] \tag{1}$$

The mass of EG is much higher than that of PET, so the concentration of EG is considered constant. The $k[\mathrm{EG}]$ can be rewritten as k_1, Eq. (1) can be rewritten as:

$$\frac{d[\mathrm{PET}]}{dt} = -k_1[\mathrm{PET}] \tag{2}$$

[PET] is the concentration of PET repeating unit, which can be shown as:

$$[\mathrm{PET}] = [\mathrm{PET}]_0(1-x) \tag{3}$$

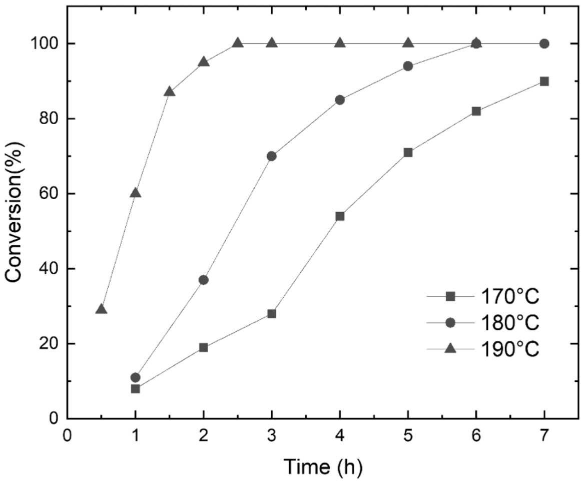


Fig. 3. Reaction temperature effect on conversion of PET (x).

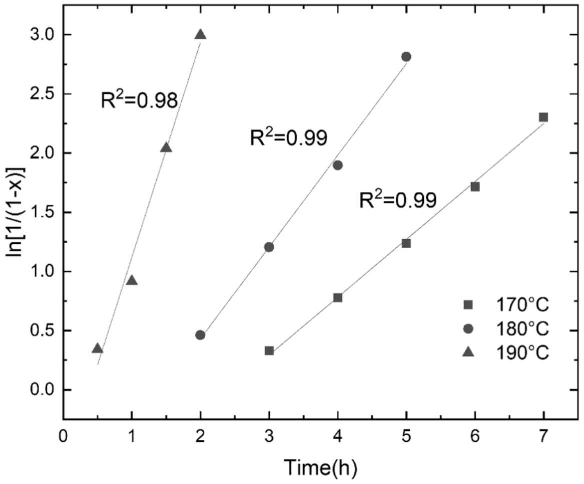


Fig. 4. Kinetics plots of the conversions of PET versus time.

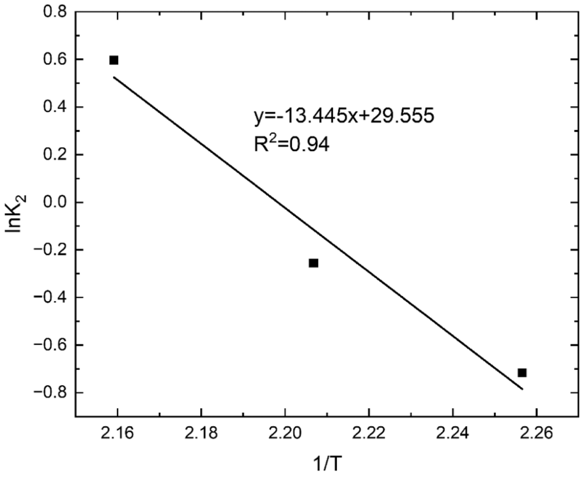


Fig. 5. The relationship between lnk_2 and 1/T.

where x is the conversion of PET. The $k_1[PET]_0$ can be rewritten as k_2, which is the pseudo first-order rate constant. Hence it can be obtained:

$$\ln\left(\frac{1}{1-x}\right) = k_2 t \tag{4}$$

Fig. 3 provides the relationship between the conversion of PET and the reaction temperature. At 190°C, the conversion of PET is close to 100 % after 2 h, and the yield of BHET was 73 %. This is the optimal reaction condition obtained by kinetics experiments. Applying Eq. (4), the plots of ln $1/(1-x)$] *vs.* times for glycolysis reactions at 170–190 °C are shown in Fig. 4. The linear correlative coefficients are all higher than 0.97, indicating that ln[1/(1 −x)] is proportional to reaction time, and the glycolysis process is a first-order reaction. The reaction rate constant k_2 can be determined from the slope of lines, the rate constants at 190°C, 180°C, and 170°C are 1.8167, 0.7747, and 0.4886 min^{-1}, respectively. Moreover, according to the relationship of rate constants with reaction temperatures, Arrhenius plots are shown in Fig. 5, and data fell on a straight line with linear correlative coefficients is 0.94. The apparent activation energy for glycolysis of PET is found to be 111.787 kJ/mol. This value is higher than those reported in the literature [66], which is due to the heterogeneous catalyst's requirements for specific surface area and mass transfer efficiency in the reaction.

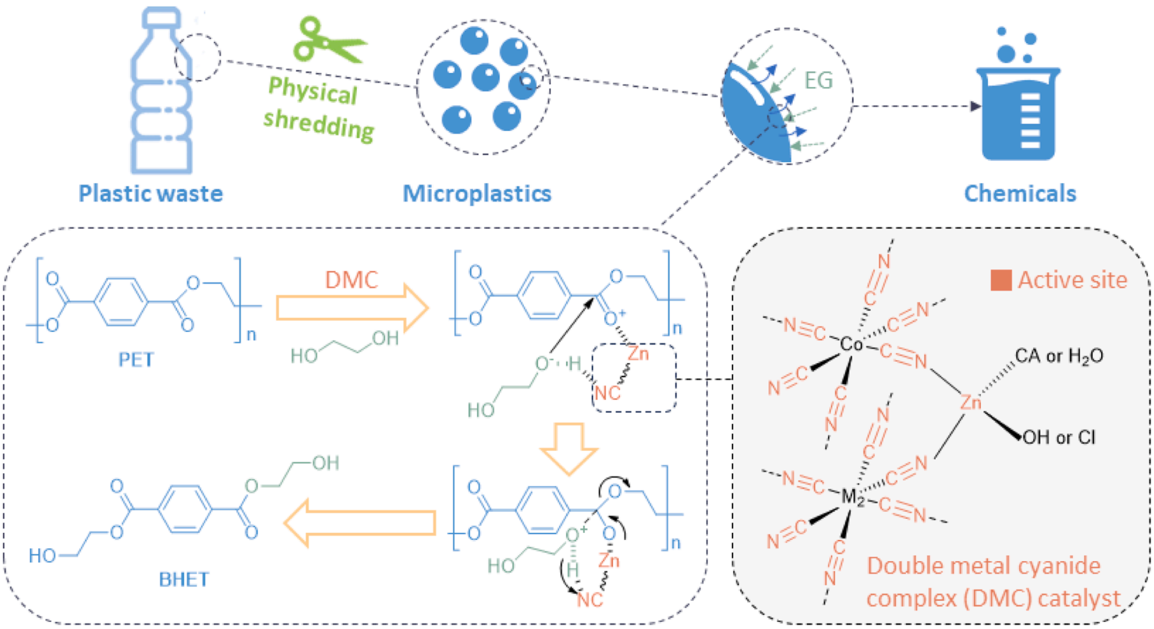
3.7. *Proposed reaction mechanism*

Zinc ions have been reported as the active species from the previous reports [40,50,58,67]. Otherwise, for glycolysis of PET, compounds containing zinc ions as the active species, such as zinc acetate have been widely used [36,37,68,69]. It is generally believed that the zinc ions attack the carbonyl groups on the ester group of PET. This leads to a nucleophilic reaction; the depolymerization reaction also happens as a result. Additionally, we suppose that the cyanide anion in DMC has the ability to activate the hydroxyl group and can participate in the reaction as a hydrogen bond acceptor [70]. This view will be examined in detail in future studies. Cobalt may just act simply as a metal-dispersing agent and a stabilizer of the cyano-bridged complex. With comprehensive consideration, the proposed reaction mechanism of PET glycolysis catalyzed by DMC is proposed in Scheme 2**.** The depolymerization takes place on the surface of the plastic fragments that were mechanically treated. The zinc ion and cyanide group of DMC collaborative catalyze glycolysis of PET, leading to the carbon atom on the carbonyl group being more electron-deficient, which makes it easier for the oxygen atom of EG to attack the carbonyl group. In addition, the surplus electrons of oxygen atoms on PET come back to form a stable carbon−oxygen double bond, and the carbon−oxygen single bond in PET is attacked by excess electrons. The dropped $-OCH_2CH_2OH$ fragments can combine with H^+ on the EG molecule. Finally, the product BHET is obtained.

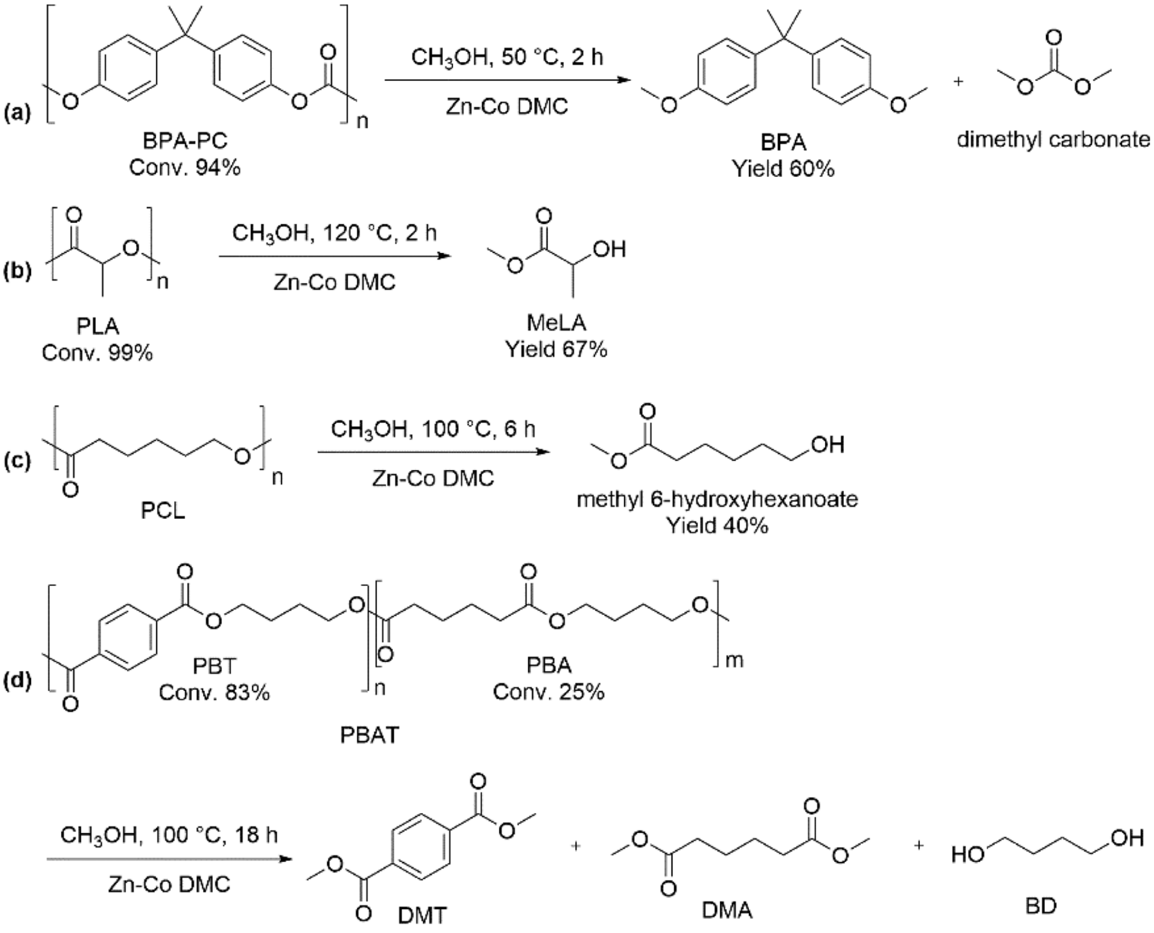
3.8. *Other polyesters depolymerized with alcohols to monomers promoted by DMC*

Inspired by the high efficiency of DMC catalysts in glycolysis of PET by transesterifications, the depolymerization strategy was generalized to other polymeric materials containing ester groups in the main-chains. The depolymerization of other polymers were carried out refer to the existing working conditions [71,72], employing the same amount of depolymerizing reagent and Zn-Co DMC catalyst.

Poly(bisphenol A carbonate) (BPA-PC) is a kind of commercial plastic with high strength and transparency. Its natural degradation product, BPA, is ecotoxic, thus the chemical recycling of BPA-PC seems to be particularly necessary. In this study, BPA-PC was depolymerized with dichloromethane (DCM) as a co-solvent. Zn-Co DMC showed the obvious catalytic effect on PC methanolysis (Scheme 3, **(a)**); the conversion of BPA-PC can reach 94 % and BPA yield can reach 60 % within



Scheme 2. Proposed mechanism for glycolysis of PET catalyzed by DMC.



Scheme 3. Commercial polycarbonate (BPA-PC) and polyesters PLA, PCL, and PBAT depolymerized by alcoholysis under the catalysis of DMC.

2 h. When the products were analyzed with ^{1}H NMR spectrum (Figure S5), BPA-dimethyl carbonate (BPA-DC) and BPA-monomethyl carbonate (BPA-MC) were also detected.

Polylactide (PLA), another commercial plastic that has been widely used, was subjected to methanolysis with the existence of Zn-Co DMC. PLA can be completely consumed within 2 h at 120 ℃. The final

Table 3
A comparison of DMC catalyst with reported mixed metal oxides catalysts in glycolysis of PET.[a]

Entry	Catalyst	Catalyst amount(wt %)	Glycolysis temperature (°C)	Time (min)	BHET Yield (%)	Synthesis condition	Ref.
1	Calcined Mg-Al hydrotalcites	1	196	50	81.3	One-step synthesis Calcined at 500°C	[73]
2	Magnetic Mg-Al-O@Fe3O$_4$micro particles	0.5	240	90	82	Multi-step Synthesis Calcined at 1000°C	[74]
3	Zn/Al mixed oxide	1	196	75	79	One-step synthesis Calcined at 500°C	[75]
4	(Mg–Zn)–Al layered double hydroxide	1	190	180	75	Multi-step synthesis No calcination required	[76]
5	S/Zn-Ti−300 °C	0.3	180	180	72	Multi-step synthesis Calcined at 500°C	[77]
6	sulfated cobalt oxide (SO_4^{2-}/Co_3O_4)	0.3	180	180	72	Multi-step synthesis Calcined at 300°C	[78]
7	$ZnMn_2O_4$	1	260	60	92.2	One-step synthesis Calcined at 300°C	[68]
8	$CoMn_2O_4$	1	260	60	89	One-step synthesis Calcined at 300°C	[68]
9	$ZnCo_2O_4$	1	260	60	91	One-step synthesis Calcined at 300°C	[68]
10	Magnetic nanoparticle $CoFe_2O_4$	2	195	150	95.2	Multi-step synthesis No calcination required	[79]
11	**Zn-Co-DMC**	**1**	**190**	**120**	**73**	**One-step synthesis** **No calcination required**	**This work**

[a] Reaction condition listed in Table 3 was the optimal condition of the reported literature.

methanolysis product methyl lactate (MeLA) was obtained with a yield of 67 % (Scheme 3, **(b)**). DMC also appears to have significant catalytic activity for the methanolysis of PLA.

Due to its good biocompatibility, poly(caprolactone) (PCL) has been widely used in the field of biomedicine. The depolymerization of PCL seems to be difficult due to its high crystallinity and hydrophilicity. With the toluene as a co-solvent, the methanolysis of PCL was attempted at 100°C. As a result of the facilitation of Zn-Co DMC, methyl 6-hydroxyhexanoate was obtained with a yield of 40 % (Scheme 3, **(c)**).

The methanolysis of poly(butylene adipate-co-terephthalate) (PBAT) has also been attempted (Scheme 3, **(d)**). Due to the overlap of characteristic peaks in the spectrogram, the yields of the methanolysis products were difficult to be determined; thus the degradation degree of the ester units was used to represent the reaction process. Analysis of the degradation experimental results showed that under the catalysis of Zn-Co DMC, the conversions of 83 % for PBT and 25 % for PBA were obtained at 100 °C.

Examples of these depolymerizations described above show that as a transesterification catalyst, DMC can indeed promote the depolymerizations of major plastics containing ester groups in the main-chains. This wide range of substrates also provides a new possibility for the depolymerization of mixed plastics.

3.9. *Catalyst evaluations*

The Zn-Co DMC catalyst developed in this study was compared with other catalysts. Since DMC was a unique class of bimetallic heterogeneous catalysts, our comparison did not include homogeneous catalysts. Even though DMC was a unique cyanide catalyst, its preparation method was similar to that of the ordinary metal oxides reported. We summarized the preparation methods of typical mixed metal catalysts, classified them according to the steps of their processing, and the results are shown in Table 3.

Previous research on heterogeneous catalysts for PET glycolysis has mainly focused on metal oxides and their derivatives. As heterogeneous catalysts, metal oxides, and their derivatives mostly require complex precursor synthesis steps or high-temperature calcination to form an oxidation state. In contrast, DMC shows its advantages due to its cheap raw materials, simple precipitation synthesis methods, and mild conditions without calcination.

4. Conclusions

The glycolysis of PET was catalyzed by commercial and lab-made DMC catalysts. Zn-Co DMC, Fe-Mn DMC, Fe-Zn DMC, and Fe-Ni DMC showed high catalytic activity. Zn-Co DMC was chosen as the optimal glycolysis catalyst in our investigation. The main product from the depolymerization of PET catalyzed by DMC is BHET monomer. A recovery yield of BHET (73 %) was achieved in the presence of Zn-Co DMC (1 wt%), and ethylene glycol (15 eq.) for 2 h at 190 °C. Glycolysis kinetics has also been studied. The apparent activation energy for glycolysis of PET is 111.787 kJ/mol. As a solid catalyst newly exploited in transesterification reactions, DMC shows high activity for depolymerization of PET, as well as alcoholysis of other plastics containing ester groups in the main-chains including BPA-PC, PLA, PCL, and PBAT. This wide range of substrates also provides a new possibility for the depolymerization of mixed plastics.

CRediT authorship contribution statement

Chang Tong: Writing – original draft, Investigation, Data curation. **Guo Kai:** Supervision, Project administration, Funding acquisition. **Li Ning:** Validation. **Xu Yue:** Investigation. **Li Chunyu:** Investigation. **Zou Xin:** Investigation. **Shi Na:** Methodology. **Hu Yongzhu:** Validation, Investigation. **LI Zhenjiang:** Writing – review & editing, Supervision. **Wang Yujia:** Software, Methodology, Investigation.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supporting information

Supplementary data associated with this article can be found in the online version at doi:10.1016/j.cattod.2025.115241.

Data availability

The authors confirm that the data supporting the findings of this study are available within the article and its supplementary materials. Further information concerning this study is available upon request from the corresponding authors.

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