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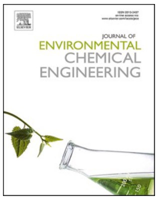

Chemical recycling of Polyethylene terephthalate: A mini-review

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ABSTRACT

Plastic waste management, particularly addressing the challenges of Polyethylene terephthalate (PET) waste, has become increasingly urgent in the face of escalating environmental concerns. This paper critically examines glycolysis-based PET recycling, focusing on its potential as a promising solution. Despite its lightweight properties and cost-effectiveness advantages, PET waste poses multifaceted challenges, including contaminant removal, scale-up hurdles, and economic viability. Catalyst optimization and maintaining end-product quality are crucial for enhancing recycling efficiency and market acceptance. Moreover, this review underscores the importance of environmental impact assessments and regulatory compliance in guiding sustainable waste management practices. To overcome infrastructure limitations and foster global collaboration, concerted efforts are needed to educate consumers and facilitate international cooperation. By addressing these challenges, glycolysis-based PET recycling holds promise in mitigating plastic pollution and promoting a circular economy. The paper highlights recent advancements in glycolysis-based PET recycling and examines potential applications for the resultant product, Bis HydroxyEthl Terephthalate (BHET).

1. Introduction

Proper management plays an essential role in reducing the production of plastic waste. If the current production trend continues, it is predicted that by 2050, approximately 12,000 million tons of plastic waste will be produced, and 150–200 million tons will accumulate in landfills or the natural environment [1–3]. Plastic recycling is being considered due to increasing environmental awareness and public demand for environmental sustainability. Plastic recycling is essential for many reasons, such as conserving oil, reducing greenhouse gas emissions, saving landfill space, saving energy, and reusing benefits [4,5]. High-density polyethylene (HDPE), low-density polyethylene (LDPE), polyvinyl chloride (PVC), polypropylene (PP), polystyrene (PS), and PET are considered recyclable polymers [1–10]. Three recycling methods are used to recycle polymers: primary recycling [7,11,12], mechanical recycling [13–15], and chemical recycling [5,16,17]. Chemical recycling methods include hydrogenation [18,19], glycolysis [20–22], gasification [20,23–25], hydrolysis [26,27], pyrolysis [28–30], methanolysis [31,32], aminolysis [33] chemical depolymerisation [34,35], degradation in a microwave reactor [6,7,36,37], and thermal cracking [38]. In recent years, one of the most widely used recyclable polymers for engineers has been PET, and this is due to cheap raw materials, lightness, temperature stability in a wide range (-60° to 220 °C), and low energy production. PET is one of the long-chain polymers

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Abbreviations: PET, Polyethylene terephthalate; PP, Polypropylene; PVC, Polyvinyl Chloride; PS, Polystyrene; LDPE, Low-density Polyethylene; HDPE, High-density Polyethylene; TPA, Terephthalic Acid; BHET, Bis hydroxyethylterephthalate; DMT, Dimethyl Terephthalate; HPLC, High Performance Liquid Chromatography; TBD, 1,5,7-triazabicyclo 4.4.0 dec-5-ene; NaY, Crystalline sodium Y zeolite; DMC, Dimethyl Carbonate; PILs, Poly Ionic Liquids; [VEIm]Ac, 1-Vinyl-3-Ethylimidazole Acetate; TGA, Thermogravimetric analysis; FTIR, Fourier transform infrared; XRD, X-ray powder Diffraction; ICP, Inductively Coupled Plasma; SCF, Supercritical Methanol Fluid; GVL, γ-valerolactone; DSC, Differential Scanning Calorimetry; PTSA, p-Toluenesulfonic Acid; CBMs, Carbohydrate-binding modules; MOFs, Metal–organic Frameworks; PLA, Polylactide; ^{1}H NMR, Proton nuclear magnetic resonance; [EMIm]2TPA, 1-Ethyl-3-methylimidazole terephthalate; DES, Deep Eutectic Solvent; 13C NMR, Carbon-13 nuclear magnetic resonance; DFT, Density Functional Theory; DMAP, Dimethyl Amino Pyridine; TBHDPB, Tributyl Hexadecyl Phosphonium Bromide; PTC, Phase Transfer Catalysts; CBMs, Carbohydrate-binding Modules; IL, Ionic Liquid; BHETA, Bis2-hydroxyethyl Terephthalamide; SEM, Scanning electron microscopy; BPA-PC, Poly Bisphenol A Carbonate; GCNC, Graphite Carbon Nitride Colloid; Zn-MNPs, Zinc ferrite nanoparticles; PU, Polyurethane; PUF, polyurethane foam; UPR, Unsaturated Polyester.

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belonging to the polyester family, a broad classification of polymers known for having ester functionalities in their macromolecular main chains [39]. PET, by having resin identification (recycling) #1, ranks as the third most used polymer in the packaging sector, dominating the beverage bottle market and accounting for nearly 16% of plastic consumption in the European packaging industry [40]. PET production materials include pure terephthalic acid and ethylene glycol (EG) derived from raw petroleum. The molecular formula is $(C_{10}H_8O_4)_n$ [8, 41]. The physical and mechanical properties of PET plastic are shown in Table 1.

The article's primary objective is to provide a comprehensive overview of different approaches to PET recycling, with a specific emphasis on chemical recycling and the catalyst used in this procedure. Through an analysis of recent studies carried out in this field over the last five years, the article seeks to investigate the efficiency and effectiveness of these recycling techniques. Furthermore, the report aims to assess how these methods could contribute to addressing the environmental concerns linked to PET waste.

2. PET recycling methods

Several factors are influential in the process of PET waste recycling. Factors such as solvents, catalysts (type and concentration), temperature and reaction time, surface wetting, shape, size, and thickness of PET particles each of these factors have a significant effect on the reaction process. PET, a frequently used plastic material for making various items such as bottles, packaging, and textiles, is crucial to recycle in waste management. This is because it helps to decrease environmental pollution and reduces the demand for new resources [43–46]. Primary, secondary (mechanical), and tertiary (chemical) are three methods of PET recycling (Fig. 1)

Primary recycling is known as re-extrusion. This type of recycling is highly dependent on the range of polymer grades in plastic waste [47]. Primary recycling is cheap and can be easily done in factories. This method cannot be used for PET plastics that contain other wastes. One of the disadvantages of this recycling is that the material's properties will be deteriorated over time. A limited number of cycles, lower value of recycled products, little market demand, and high energy consumption are the main disadvantages of primary PET recycling [48,49]. The industrial production of mechanical recycling was established in the 70 s. Mechanical recycling includes separating, cleaning, crushing waste, washing PET pieces, drying the cleaned particles, and finally extruding the PET particles. The presence of water or acid causes a decrease in molecular weight and leads to a reduction in the quality of products [50–53]. However, this process has some drawbacks that must be considered. Firstly, the process can contaminate the recycled material, which could reduce the quality of the material and necessitate further processing to remove impurities such as food residue, labels, or adhesives. Secondly, melting and reforming PET waste can result in material degradation, limiting the number of times PET can be recycled into similar-quality products. Thirdly, secondary PET recycling is energy-intensive, particularly during the melting and reforming stage, which can lead to increased greenhouse gas emissions, contributing to climate change. Lastly, secondary PET recycling requires significant capital investment, particularly for food-grade applications, necessitating strict quality control measures. This can limit asset availability for PET recycling and impede the growth of the industry [54–56].

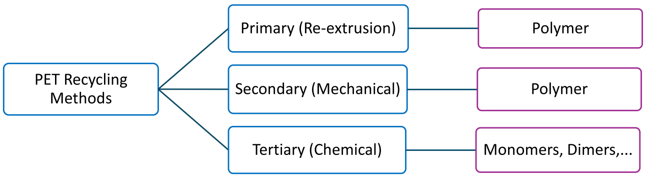

Fig. 1. PET Recycling Methods.

3. Chemical recycling methods

Chemical recycling, also called tertiary recycling, refers to the process of breaking down the PET polymer into its individual monomers that can be utilised to manufacture new PET products. In contrast to primary and secondary recycling approaches, chemical recycling presents numerous benefits. It increases sustainability by reducing greenhouse gas emissions by up to 50% compared to virgin PET production; it also can decrease greenhouse gas emissions by up to 80% by offering a carbon-neutral approach to PET production [57]. Also, contaminated or degraded PET waste, which is not suitable for mechanical recycling, can be recycled through chemical recycling. Besides energy efficiency, high-quality PET monomers can be produced by chemical recycling [57, 58]. The findings of the studies highlight the diverse benefits of chemical recycling in comparison to primary and secondary recycling methods, which supports the notion that chemical recycling holds potential as an effective strategy for promoting sustainability, recycling waste that is mixed or contaminated, producing superior quality products, reducing the consumption of energy and resources, and potentially creating a carbon-neutral means of PET production. In chemical recycling, PET's molecular and chemical structure is changed with the help of solvent, catalyst, microwave exposure, and temperature. Large molecular chains are broken down and converted into oligomers and monomers, which can be used to produce materials with high added value. This type of recycling is of interest to industrialists and researchers [58]. Chemical recycling of PET waste involves various methods, including glycolysis, methanolysis, hydrolysis, and aminolysis [59,60]. Glycolysis is a process that breaks down PET into its constituent monomers through a reaction with glycols. It produces bis(2-hydroxyethyl) terephthalate (BHET), which can be purified and used to make new PET. Glycolysis is advantageous as it can produce high-quality recycled PET with similar properties to virgin PET. It is also relatively low-cost and low energy compared to other recycling methods. On the other hand, methanolysis involves depolymerizing PET with methanol and a catalyst at high temperatures and pressures. It produces dimethyl terephthalate (DMT), which can be purified and used to make new PET. However, this method has disadvantages, such as creating impurities that can negatively affect

Table 1
Physical and mechanical properties of main plastics [42].

Material	Morphology	Density [g cm^{-3}]	Glass transition temperature [°C]	Melting Temperature [°C]	Initial degradation temperature [°C]	Tensile strength (MPa)	Tensile modulus (MPa)	Strain at break (%)
PET	Semi-crystalline	1.33–1.4	70–85	245–260	425–445	47	3100	50–300
LDPE	Semi-crystalline (40%-55%)	0.91–0.93	-130 to −100/-30 to −10	100–115	487–498	8–23	200–500	300–1000
HDPE	Semi-crystalline (60%-80%)	0.94–0.96	-130 to −100	125–135	480–498	18–35	700–1400	100–1000
PP	Semi-crystalline	0.90–0.91	-20–21	160–165	450–470	21–37	1100–1300	20–800
PVC-Plasticized	Amorphous	1.6–1.35	−50–80	—	290–315/ 460–475	10–25	—	170–400
PS	Amorphous or Semicrystalline	1.05	80–105	—	415–425	45–65	3200–3250	3–4

the quality of the recycled PET. It is also more expensive and energy-intensive than glycolysis [32]. Hydrolysis breaks down PET using water, a catalyst, and high temperatures and pressures. It produces terephthalic acid (TPA), which can be purified and used to make new PET. However, this method requires high energy consumption and cost. Aminolysis involves depolymerizing PET with an amine compound and a catalyst at high temperatures [61,62]. However, this method also requires high energy consumption and cost [58]. In summary, glycolysis is a favourable technique for researchers and industries due to its low cost and energy consumption, high-quality recycled PET, and accessibility. Glycolysis and methanolysis are the most widely used methods of PET waste recycling on an industrial scale, with glycolysis receiving more attention. It is important to note that each method has its advantages and disadvantages, and the choice of method depends on various factors such as cost, energy consumption, and quality of the recycled PET [16,63–65]. The main methods of chemical recycling of PET waste and the related products are shown in Fig. 2.

3.1. *Methanolysis*

Methanolysis is a chemical technique in which DMT and EG are produced by degrading PET at a high temperature (180–280°C) and pressure (2–4 MPa). The advantage of methanolysis is the short reaction time. The disadvantages of this method are high temperature and pressure and the high cost of separating and refining the product [66]. Hofmann et al. [67] reported a 95% DMT yield at 160°C, 20 minutes in a microwave heating catalysed by zinc acetate and an excess of methanol and dichloromethane as solvent. Besides, they converted synthesised EG into different types of diesters and diols through hydrogenation, making them useful starting materials for polyesters.

Pham et al. [32] illustrated the PET flakes conversion to its monomer DMT via methanolysis utilising potassium carbonate (K_2CO_3) as a non-toxic and low-cost catalyst through a selective reaction with low energy consumption (Fig. 3). A mixture of methanol and dichloromethane was used as reaction solvents. In contrast to current methanolysis techniques, the proposed system exhibited a consistent yet relatively slow reaction rate. However, it achieved complete decomposition of PET resins into monomers within a 24-hour timeframe, they reported, and high selectively. The DMT yield was 93.1% in mild conditions at 25 °C in 24 h.

Tollini et al. [66] used high-performance liquid chromatography (HPLC) to analyse the monomers produced during PET depolymerisation by methanolysis and ethanolysis with different catalyst systems. They compared PET conversion and yields in different components of bottle flakes and mixed fabrics. Potassium carbonate was the best-performing catalyst in terms of DMT selectivity, with 71% yield for PET flakes making it a good candidate for industrial scale-up due to easy monomer separation. Catalysts like potassium methoxide and 1,5,7-triazabicyclo 4.4.0 dec-5-ene had high DMT yield but were less attractive due to soluble catalysts leading to issues with monomer purification. Mixed fabrics had lower results (42%) due to higher moisture content and hydroxyl groups. Further research is needed to optimize particle size, solvent-to-PET ratio, and reaction time for the most promising catalyst and cosolvents for scaling up the process.

Tang et al. [68] conducted a study on using MgO/NaY (crystalline sodium Y zeolite) catalyst in the methanolysis of PET to address the plastic recycling crisis. They prepared catalysts with different MgO contents and characterized their properties using various techniques. The results showed that the PET conversion and DMT yield increased with the alkalinity of the catalyst. Under specific reaction conditions, the 21% MgO/NaY catalyst showed high catalytic activity, achieving 99% PET conversion and 91% DMT yield (Fig. 4) at 200 °C in 30 min. The study also investigated the catalyst's recyclability and found it effective even after six cycles.

Methanolysis involves a transesterification reaction. Common catalysts for this reaction include various metal salt forms, such as metal acetates (e.g., zinc acetate, lead acetate), metal oxides (e.g., aluminium isopropoxide, sodium silicate), and metal hydroxides. While these metal salt catalysts demonstrate effective catalytic capabilities in traditional methanolysis, a significantly lower reaction temperature than the melting point of PET (260°C) is inadequate to promote PET decomposition selectively and swiftly [69–71]. Tanaka et al. [72] catalysed the PET breakdown at ambient temperature by applying 1 wt% of LiOMe (an alkali metal alkoxide). The methanolysis reaction equilibrium moved towards more DMT by adding dimethyl carbonate (DMC) to capture EG and produce a side product of ethylene carbonate. The DMT yield was above 90% at 65 °C in 3 h. Metal ion catalysts are recognised for their high efficacy in PET methanolysis. However, they face the challenge of being challenging to retrieve, a concern that can be effectively resolved by capitalizing on the polymer's limited solubility. The approach of polymerizing one or multiple monomers can be explored to attain elevated catalyst activity and the capacity for reuse, all while preserving the catalyst's active functional groups. Jiang et al.[73] investigated methanolysis of PET using poly ionic liquids (PILs) that were synthesised employing 1-vinyl-3-ethylimidazole acetates ([VEIm]Ac) and metal salts of acrylic acid. The PILs' activities were assessed

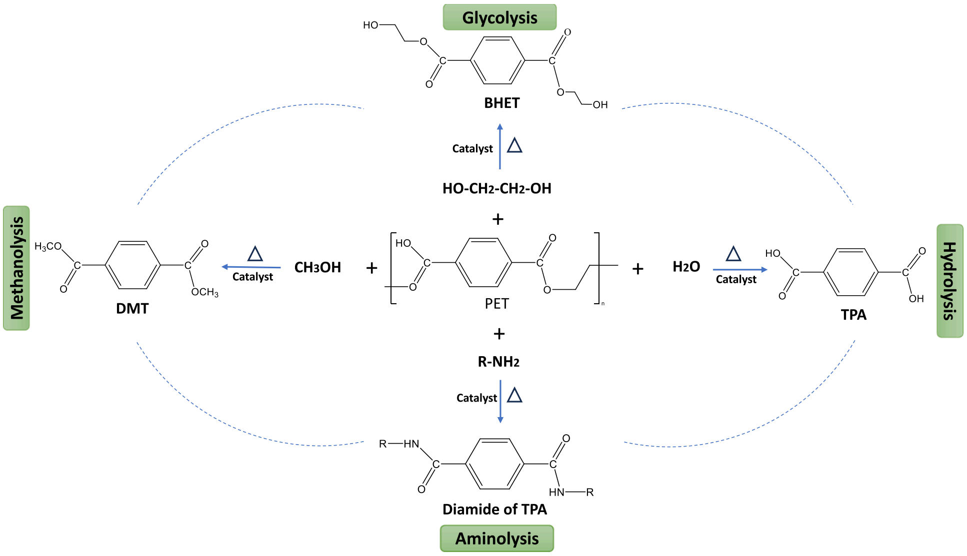

Fig. 2. Chemical recycling of PET and types of products.

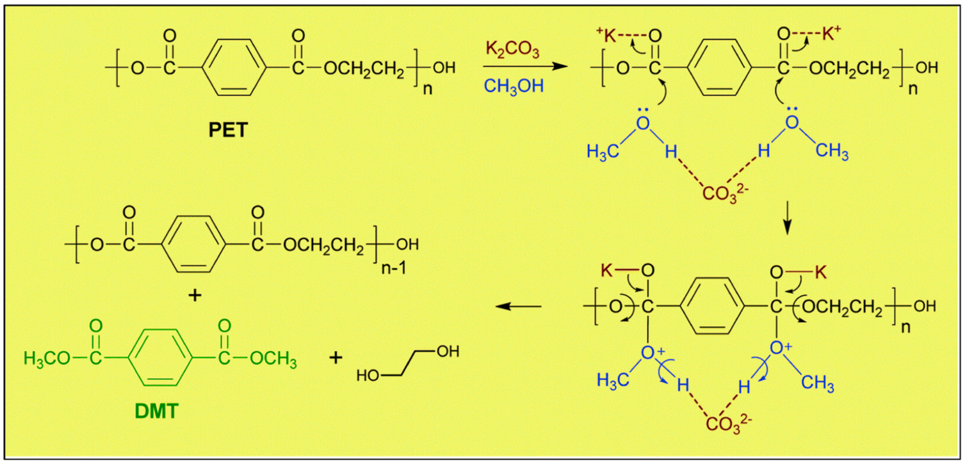

Fig. 3. Proposed mechanism for PET depolymerisation catalysed by K_2CO_3 [32].

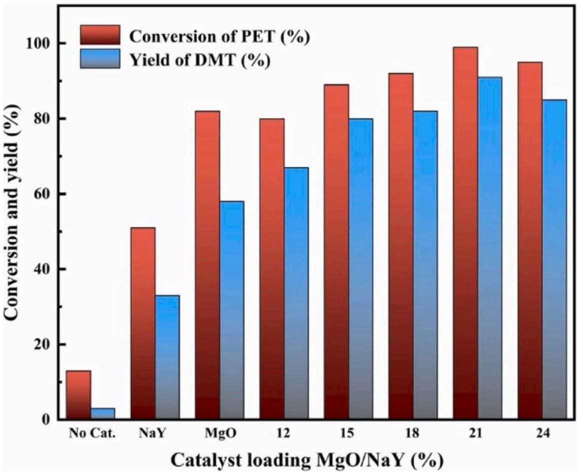

Fig. 4. The impact of varying MgO contents in MgO/NaY catalysts on the rate of PET depolymerisation and the resulting yield of DMT [68].

through screening. The PET conversion reached 100%, and the DMT yield achieved 89.1% under optimal conditions. Their proposed mechanism for the reaction is illustrated in Fig. 5. They reported that the PIL-Zn^{2+} catalyst exhibited recyclability via straightforward filtration after each reaction, retaining effectiveness through six cycles without significant reductions in PET conversion and DMT yield. Analysis of the catalysts through Thermogravimetric analysis (TGA), Fourier transform infrared (FTIR), X-ray powder diffraction (XRD), and Inductively Coupled Plasma (ICP) before and after recycling revealed an unchanged catalyst structure, indicating remarkable stability. The reaction kinetics of PIL-Zn^{2+} adhered to a first-order pattern characterized by an activation energy of 107.6 kJ/mol.

Limited research has focused on enhancing PET methanolysis performance in a supercritical methanol fluid (SCF). Operating under a supercritical methanol state enables PET to achieve substantial conversion and an enhanced DMT yield (reaching 95%) within just 1 hour under optimal reaction conditions (260–270°C, 9–11 MPa). Nevertheless, this method demands high-pressure conditions, resulting in notable drawbacks of elevated capital investments and operational expenses [32]. Liu and Yin [74] presented an experimental setup for supercritical methanol of PET, investigating the impact of various factors on the methanol-to-PET ratio, reaction time, and the influence of CO_2 on DMT yield. The optimal conditions were identified: 270°C temperature, 6:1 methanol to PET mass ratio, 40-minute reaction time, and 1000 rpm rotational speed. CO_2 addition enhanced the process, increasing DMT yield up to 95% and reducing reaction temperature, methanol usage, and energy consumption. The accelerated PET depolymerisation is attributed to CO_2 inserting between PET molecular chains, weakening interchain forces, enhancing molecular mobility, and gradually decreasing PET crystallinity.

Table 2 presents a summary of the examined catalysts and reaction conditions applied in the methanolysis of PET.

3.2. Hydrolysis

This chemical recycling method converts PET to TPA and EG. The traditional hydrolysis degradation method faces challenges due to harsh reaction conditions, which involve temperatures spanning 200°C to 250°C, pressures of 1.4–2 MPa, extended reaction durations, and the expense of purifying TPA from the reaction mixture. The hydrolysis method is performed in neutral, acidic, and alkaline environments in the presence of water [20]. Neutral hydrolysis is usually done by hot water or steam. This process is generally done in high-pressure autoclaves at temperatures of 200–300 °C and pressure of 1–4 MPa, and the products are TPA and EG. Unlike acid and alkaline hydrolysis, neutral hydrolysis eliminates unwanted inorganic salts, making it a more environmentally friendly hydrolytic method. However, due to the presence of PET impurities in the resulting TPA, the quality of the product is lower compared to acidic and alkaline methods, necessitating additional purification [75–77]. Acidic hydrolysis is usually done by concentrated sulfuric or nitric acid; it is fast and does not require high temperature and high pressure. However, the main drawbacks are the contamination and corrosion of the reaction equipment and significant quantities of highly acidic or alkaline generated wastewater within these reaction systems. Alkaline hydrolysis of PET is usually carried out in a concentrated solution of sodium hydroxide (NaOH) or potassium hydroxide (KOH). The products of PET alkaline hydrolysis are EG and disodium terephthalate salt [13,78]. Chen et al. [79] used γ-valerolactone (GVL) solvent in PET recycling via an alkaline hydrolysis method (Fig. 6). Hydrogen-bonding interactions with GVL significantly influence the dissolution of PET. Swelling differential scanning calorimetry (DSC) revealed that the processing of PET with GVL included swelling and dissolution stages.

According to optimized swelling DSC, waste PET bottle fragments achieved full swelling at 120 °C within an hour and complete dissolution at 170 °C within 2 minutes. PET’s maximum solubility in GVL was approximately 0.25 $g \cdot g^{-1}$. Water was introduced as an anti-solvent to prompt PET re-precipitation from the heated solution. Thanks to the effective pre-swelling and rapid dissolution, PET degradation during the dissolving phase was a mere 5.7%. GVL processing significantly increased PET’s surface area, facilitating subsequent thorough alkaline

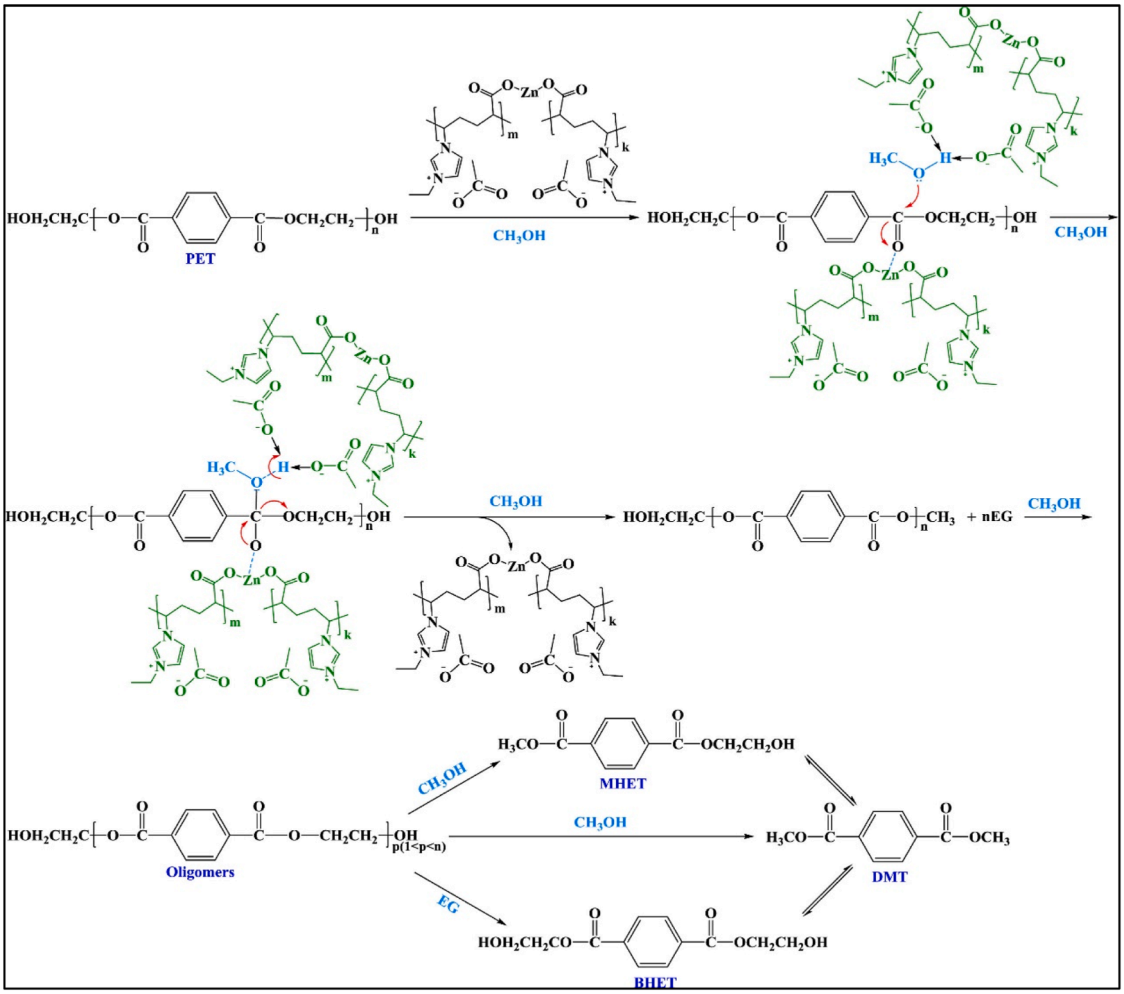

Fig. 5. Suggested mechanism for PET methanolysis catalysed by PIL-Zn^{2+}.

Table 2
Different studied reaction conditions and catalysts for PET methanolysis.

Catalyst	Solvent	T (°C)	Time (min)	PET conversion	DMT yield	Ref
Zinc Acetate	MeOH/ Dichloromethane	160/Microwave	20	-	95%	[67]
K_2CO_3	MeOH/Dichloromethane	25	1440	100	93.1%	[32]
K_2CO_3	MeOH/Dichloromethane	70	1200	100	71%	[66]
MgO/NaY	MeOH	30	200	99	91%	[68]
LiOMe	MeOH/Dimethyl carbonate	65	300	100	90%	[72]
PIL-Zn^{2+}	MeOH	170	60	100	89.1%	[73]
-	SC-MeOH/CO_2	270	40	100	95%	[74]

hydrolysis in approximately 8 minutes. In this research, a TPA yield of 99% was reported at 90 °C in 8 minutes.

Yang et al.[80] investigated using concentrated p-toluenesulfonic acid (PTSA) as a catalyst in PET hydrolysis, as shown in Fig. 7.

PTSA can be recovered through concentration and crystallisation techniques and used multiple times (Fig. 8a), while TPA yield was 96.4% at 150 °C in optimal conditions in 90 minutes. TPA can be easily extracted from the hydrolysis system by filtration (Fig. 8b). The reaction kinetics are confirmed to be first order with low apparent activation energy.

Li et al. [81] investigated an improved hydrolysis method with reactive processing aimed at simultaneously decomposing and decolorizing coloured PET fabrics to produce high purity terephthalic acid. The technique achieved comparable selectivity (98.35%) and yield (88.51%) to traditional processes. The process effectively decoloured PET fabrics coloured with either azo or anthraquinone dyes, resulting in average colour removal rates of 94.22% in solid products and 97.65% in a water solution. The recent methods of PET hydrolysis, as documented in recent studies, offer a practical approach to polyester treatment. While the recycling rates for PET bottles are elevated, PET trays and films experience notably lower recycling rates. This is attributed to their diverse array of colours, complex multilayer structures, and the challenge of inadequate collection systems. Ügdüler et al. [82] investigated different types of PET waste decomposition via alkaline hydrolysis. In optimum conditions, almost 95% mol of TPA was achieved at 80 °C, 60:40%vol of EtOH: H_2O, and 5 wt% of NaOH in less than 20 min. Besides, it was revealed that the maximum yield of monomer was obtained from the smallest particles of monolayer PET films and trays while the minimum amount was achieved from the multilayers (almost 40%), which was because of solvent diffusion limitation to the whole film layers. Barredo et al. [83] proposed using alkaline hydrolysis as a promising method for recycling PET tray waste. They studied the effect

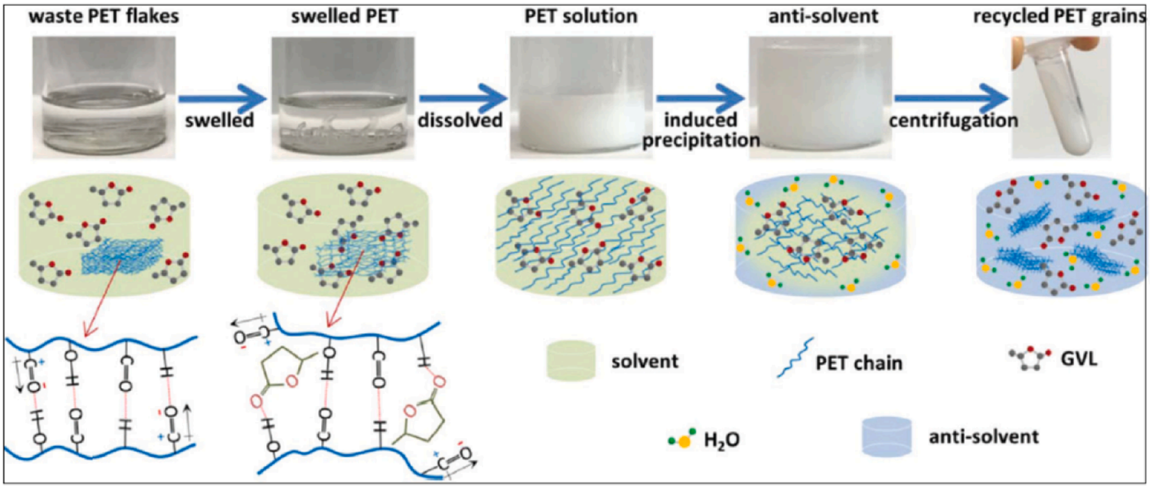

Fig. 6. Schematic of waste PET bottles recycling process using hydrolysis method [79].

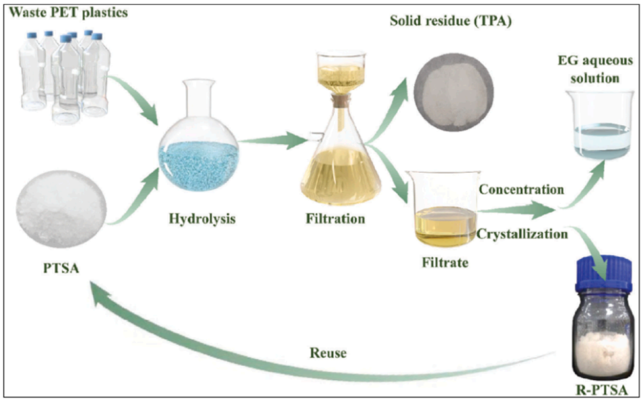

Fig. 7. Schematic of waste PET bottles recycling process using hydrolysis method [80].

of various operating variables on PET conversion and TPA yield, including using tributyl hexadecyl phosphonium bromide (TBHDPB) as a phase transfer catalyst (PTC). PTC has multiple uses, particularly in organic and fine chemical synthesis. The fundamental principle of PTC relies on specific 'phase-transfer agents' or catalysts, which can aid the movement of a reagent from one phase into another (typically immiscible) phase where the other reagent is present. In the alkaline hydrolysis of PET, an effective PTC should facilitate the transportation of the hydroxide anion from the aqueous phase to the organic phase and accelerate the reaction rate [84]. With no catalyst, the PET conversion over time was significantly lower than when various catalyst/PET ratios were employed. This difference can be explained by the fact that the hydrophobic nature of PET material hinders the attack by hydroxide ions present in the aqueous phase when no catalyst is present. The catalyst addresses external diffusion limitations by enhancing the interaction between PET and hydroxide ions, consequently augmenting PET conversion. The optimal conditions for the reaction are established as a catalyst mass ratio of 0.2, a temperature of 100 °C, a particle size of 1–1.4 mm, and a stirring rate of 525 rpm, which resulted 99.9% of PET conversion to 93.5% of TPA yield. The study concludes that TBHDPB can be an effective catalyst for alkaline hydrolysis and that this method can contribute to sustainable development in closed-loop applications [83]. Zhang et al. [85] presented a novel method for the hydrolysis of PET waste to TPA using EG and THF in the presence of KOH. This method achieves high efficiency and selectivity with 100% PET degradation and 97.5% TPA yield, at 60°C in 1 h. THF is utilised to promote mass transfer and auto-precipitation of the product, and THF activates the hydroxyl group of EG to synergize with KOH and cleave ester bonds (Fig. 9).

The mild non-aqueous method enables the possibility of degradation even at 30 °C. This approach showed potential for PET waste recovery and as a valuable supplement to the petrochemical industry. Yang et al. [86] catalysed the neutral hydrolysis of PET waste in water in the presence of TPA. This catalyst was easily recovered and reused again. At 220 °C, 0.1 g/mL of TPA, PET: H_2O of 1:8, and 180 min of reaction time, the high purity produced TPA yield was 95.5% mol. The more significant aspect of their study was that the effectiveness of hydrolysis remained consistent throughout eight successive rounds of reactions. Mahadevan

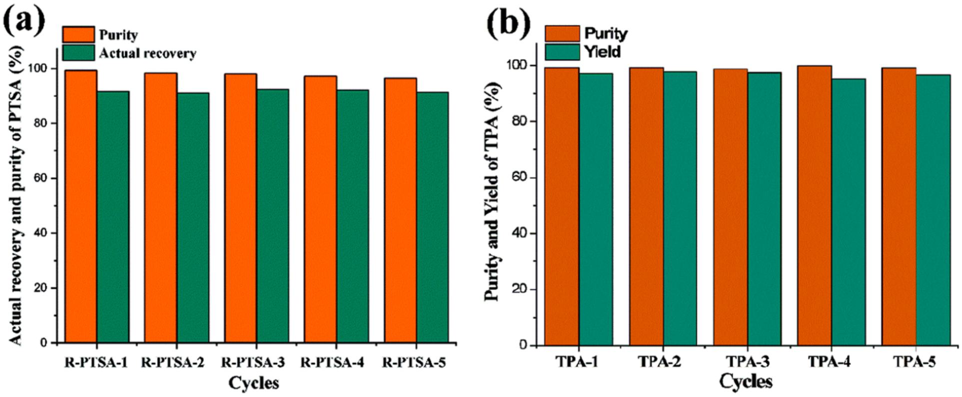

Fig. 8. (a) Recovery and the degree of purity of PTSA across various cycles. (b) The purity and amount of terephthalic acid (TPA) produced in each cycle[80].

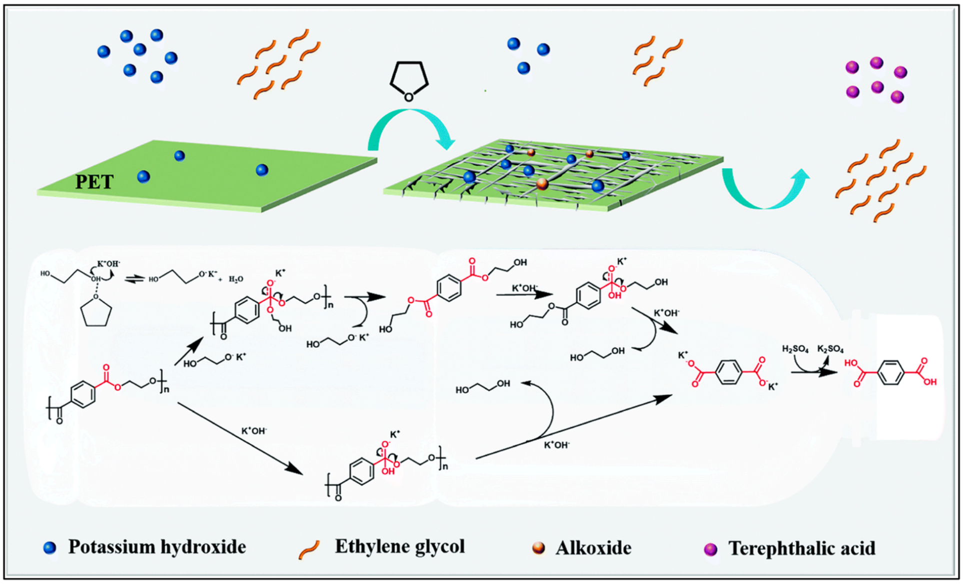

Fig. 9. Illustration of THF-facilitated PET etching and collaborative deterioration of PET involving alkoxide and potassium hydroxide, along with the suggested chemical reaction mechanism for the PET degradation process [85].

Subramanya et al. [87] found that adding polypropylene (PP) or cellulose to the reactor during the hydrolysis of PET at 250 °C for 30 minutes can increase the yield of TPA monomer recovered. The study showed that the TPA yield is affected by reaction time, reaction temperature, and PET type, and the presence of PP and cellulose did not significantly affect the TPA yield at 300°C and 30 minutes. The results of this study can contribute to the development of more efficient methods for recycling PET waste and reducing environmental pollution. Graham et al. [88] examined accessory binding modules' role in improving PET hydrolysis using engineered cutinase enzymes. They produced synthetic fusion constructs of a thermostable leaf compost cutinase variant with five type A carbohydrate-binding modules (CBMs). The CBMs were found to improve aromatic monomer yield from PET at solids loadings below 10 wt%, but conversion extends up to 97% were reached, with no added benefits from the presence of CBM fusions above this threshold. The study suggests that fusion constructs with the studied CBMs are not necessary for industrial enzymatic PET recycling.

Table 3 provides an overview of the investigated catalysts and reaction conditions utilized in the hydrolysis of PET.

Table 3
Different studied reaction conditions and catalysts for PET hydrolysis.

Hydrolysis	Catalyst or Solvent	T (°C)	Time (min)	PET conversion	TPA yield	Ref
Alkaline NaOH 15% wt	GVL	90	8	100	99%	[79]
Acid	PSTA	150	90	100	96.2%	[80]
Alkaline	H_2O	200	480	-	88.51%	[81]
Alkaline NaOH/ 5% wt60%v	H_2O/ EtOH	80	20	>90	95%	[82]
Alkaline NaOH	TBHDPB	100	240	99.9	93.5%	[83]
Alkaline KOH	EG /THF	60	60	100	97.5	[85, 86]
Acid	TPA	220	180	100	95.5	[86]
Neutral	PP / H_2O	250	30	-	75	[87]

3.3. Aminolysis

The corresponding diamides of TPA and EG are obtained from the aminolysis of PET. However, several obstacles need to be addressed for aminolysis to be economically viable, including the high cost of amines, high reaction temperatures and pressures, and the formation of byproducts. Aminolysis involves reacting PET with an amine and a catalyst to produce new polyester chains with different properties. The amine can be primary, secondary, or tertiary, and the catalyst can be acidic, basic, or enzymatic. The resulting product is a polyamide with properties that differ from the original PET, such as increased hydrophilicity, improved thermal stability, and reduced crystallinity. Catalysts like Lewis acids, Bronsted acids, bases, and enzymes can influence the reaction rate and selectivity, and recent advances have focused on developing new catalysts like Metal–organic frameworks (MOFs), zeolites, and solid acids. The products of aminolysis of PET can be used in applications like coatings, adhesives, fibres, and films, but challenges such as feedstock purity, byproduct formation, and conversion rates must be addressed to improve the efficiency and selectivity of the process. Research in this area should focus on developing new catalysts and reaction conditions to enhance efficiency and selectivity while reducing environmental impact [89–93]. Backstrom et al. [94] illustrated a simple approach for the selective recycling of post-consumer PET into a range of terephthalamides. Using microwave-assisted, catalyst-free aminolysis with hexylamine, furfurylamine, ethanolamine, or allylamine, they transformed PET flakes into terephthalamides (Fig. 10). The reaction times ranged from 10 to 60 minutes. The final product resulting from the interaction with allylamine during aminolysis underwent an additional step involving the radical thiol-ene reaction with a thiol, creating high-quality films with a glass transition temperature (Tg) exceeding room temperature. The other three terephthalamides were combined with polylactide (PLA) at a concentration of 10 wt% and assessed for their potential as plasticizers. The terephthalamide derived from aminolysis with furfurylamine drastically enhanced the strain at break by a factor of 20 compared to the strain at break observed in pure PLA.

Radadiya et al. [95] decomposed PET waste using ethanolamine as a depolymerizing agent in their study. Following the depolymerisation process using ethanolamine, N,N′-Bis(2-hydroxyethyl) terephthaldiamide (BHETA) was successfully produced, yielding a 77% output. By utilising a potent oxidizing agent such as $KMnO_4$, the monomer

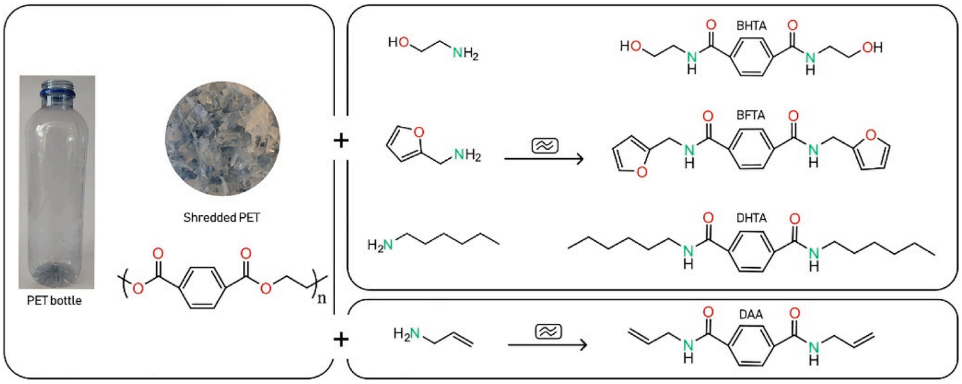

Fig. 10. Schematic illustration of chemical breakdown of PET waste using aminolysis with the assistance of microwaves [94].

BHETA was transformed into a key component of PET, TPA with a yield of 67% under mild reaction conditions at ambient temperature and pressure. The resulting TPA was characterized using FTIR and Proton nuclear magnetic resonance (1 H NMR) techniques. Furthermore, their research focused on predicting the progress of the aminolysis reaction of PET using easily accessible analytical tools like FTIR and TGA. Merkel et al. [96] carried out PET depolymerisation by a range of nucleophilic amines and synthesised a variety of terephthalic amides. By adding (18% wt.) these amides to the asphalt binder structure, they successfully enhanced the performance characteristics of asphalt, such as rutting and fatigue characteristics and thermomechanical and creep performance. Their study only used low-cost amine reactants and did not focus on creating specific chemical structures that could provide the best asphalt performance. Ghorbantabar et al. [97] considered PET waste depolymerisation using an extra amount of monoethanolamine. Their research mainly focused on tracking the aminolysis process using instrumental equipment such as FTIR, TGA, DSC, and CHN. They verified that both TGA and DSC were suitable techniques for tracing the aminolysis reaction, while FTIR, CHN, and TGA were not. Leng et al. [98] proposed a method of recycling waste plastic and rubber using them as additives in the asphalt structure. The study showed that applying waste PET derived additives and scrap tire rubber in asphalt can improve the engineering properties of asphalt pavement. Adding PET-based additives to crumb rubber-modified asphalt can improve storage stability, rutting, and fatigue resistance and increase the modified binders' rotational viscosity. The findings of this study can be helpful in the asphalt industry to develop sustainable and eco-friendly practices for waste management and road construction.

Chan and Zinchenko [33] proposed a method to convert waste PET bottles into cross-linked hydrogels through PET aminolysis using tri- and tetraamines. The resulting hydrogels show typical polyelectrolyte behaviour of polycations and have potential applications in fields such as environment, agriculture, and separation, among others. The absorption capacity of PET-derived hydrogels towards an anionic dye such as Congo Red was demonstrated to be approximately 500 mg/g. In another study, Chan et al. [99] introduced a new method for converting discarded PET plastic bottles into fluorescent nanomaterials using hydrothermal treatment. According to their findings, the most effective conversion method involves subjecting PET aminolysis products to hydrothermal treatment with an oxidizing agent (H_2O_2), which produces N-doped carbon dots with outstanding fluorescent properties. These PET-derived carbon dots can detect Fe^{3+} and Cu^{2+} ions in aqueous solutions, with sensitivities as low as ppb and ppm, respectively. The study suggested that combining hydrothermal treatment and a well-designed chemical conversion approach could be promising for creating new functional materials with significantly improved characteristics from waste PET bottles. The practical implication of this research is that it introduces a novel approach for upcycling waste PET bottles into valuable fluorescent nanomaterials that can be utilised for the sensitive detection of metal ions in aqueous solutions.

3.4. *Glycolysis*

In glycolysis, the degradation of PET can occur at a relatively lower temperature ($<190^0$ C) in the presence of glycols such as EG, diethylene glycol (DEG), propylene glycol, and dipropylen glycol and transesterification catalysts to achieve BHET and an oligomer of TPA monomer. In fact, it is the simplest and oldest method of PET depolymerisation, which is low-cost, and needs low energy, but is extremely slow in the absence of catalysts [100–102]. The glycolytic reagents and resulting products have low volatility and are non-toxic. Additionally, the glycolysis process does not generate any acid or alkali wastewater. Furthermore, the products obtained from glycolysis can be readily separated and purified using methods such as hot water extraction, cooling crystallisation, and adsorption. Besides, the oligomers produced can be used to produce recycled PET [103]. While the glycolysis process offers remarkable benefits and a considerable reaction time (1–8 h), the limited yield of BHET presents significant obstacles to the widespread commercial adoption of PET glycolysis, although as a commercial recycling method, it is practiced by world-renowned companies such as DuPont/DOW, Goodyear, Shell Polyester, Zimmer, and Eastman Kodak [13]. Therefore, the development of highly efficient novel catalysts is a critical area of focus for advancing PET glycolysis [58,104].

The glycolysis procedure can be categorized according to reaction conditions, types of solvents, heating methods, and the catalyst's characteristics or nature, which in this review is divided based on the catalyst's nature.

3.4.1. *Catalysts in glycolysis*

One of the main challenges in the industrialisation of glycolysis is catalyst development. Both homogeneous and heterogeneous catalysts have been utilised in PET glycolysis. Homogeneous catalysts have notable catalytic performance but have drawbacks such as separation challenges, recyclability issues, and product selectivity concerns. On the other hand, heterogeneous catalysts are used due to their suitability for commercial use in industrial PET glycolysis. They are easy to separate, whereas the drawbacks frequently involve restricted activity and selectivity [104–107]. Four groups of catalysts mostly used in glycolysis reactions are metal salt and metal oxide catalysts, nano catalysts, Ionic Liquids (ILs), and Deep Eutectic Solvents (DESs).

3.4.1.1. Metal salt and metal oxide catalysts. Metal salt and Metal oxide catalysts have been used for many years for the PET recycling process. Metal salts, such as zinc acetate, calcium acetate, and magnesium acetate, are frequently utilised as catalysts. Also, metal oxide catalysts like zinc oxide, titanium dioxide, and aluminium oxide have been studied. Nevertheless, using these catalysts has drawbacks, such as being expensive, inefficient, and environmentally harmful, prompting researchers to seek more sustainable and economical alternatives [108–111]. Krisbiantoro et al. [112] employed solvent-free mechanochemically synthesised MFe_2O_4 spinel catalysts (where M = Co, Ni, Cu, and Zn) for PET glycolysis. All catalysts demonstrated activity in converting PET to BHET, with $ZnFe_2O_4 > CuFe_2O_4 > CoFe_2O_4 > NiFe_2O_4$ in terms of PET conversion rate. Catalyst effectiveness correlated with the strength of the Lewis acid from M^{2+} in MFe_2O_4, indicating that higher Lewis acid strength led to greater catalytic activity. Despite $CoFe_2O_4$ ranking second in BHET yield, it possessed the highest saturation magnetization, facilitating magnetic catalyst separation. The reaction using $CoFe_2O_4$ had an apparent activation energy (Ea) of 188 kJ mol−1 and could be reused five times. Most PET glycolysis research utilises a standard method of separating PET from BHET, involving hot water addition, PET filtration, and BHET crystallisation. However, Jave and Vogt [59] believed this approach has limitations since water damages the catalyst and must be eliminated to recycle EG. They presented a novel environmentally friendly glycolysis method that eliminated the requirement for an anti-solvent by employing straightforward sodium alkoxides (MeONa and EtONa) as catalysts. The recycling of EG and the PET conversion remained consistent with the initial recycling run under the established optimal conditions. This optimal recipe was then utilised to assess the catalysts' (MeONa and EtONa) effectiveness in depolymerizing coloured PET waste and mixed PET waste. The outcomes revealed that both catalysts can efficiently break down both types of waste using environmentally conscious glycolysis conditions, with MeONa exhibiting higher PET conversion. This underscores the feasibility of achieving BHET precipitation without water usage, thereby reutilising EG and preserving catalyst performance for mixed PET waste. Mohammadi et al.[113] demonstrated PET conversion to its monomer in EG in the presence of antimony oxide (Sb_2O_3) as a catalyst. Sb_2O_3 has long been used in polyester production, particularly in PET synthesis. The achieved BHET was 97%, at 200 °C, 2.1 bar in 1 h by utilising 0.5 wt% of the catalyst and EG/PET weight ratio of 5.0. The resulting BHET was white, recoverable, and could be easily purified and polymerized using the same catalyst used in its production. They believed it might be possible to produce new PET in the same reactor by changing the reaction conditions to PET polymerisation settings, such as increasing the temperature to 270 °C and removing excess EG with a vacuum. In comparison, the activity of this catalyst was higher than zinc acetate, antimony (III) acetate, and antimony (V) oxide. While metal oxides have the potential to serve as effective components in developing strong and efficient heterogeneous catalysts for PET glycolysis, achieving substantial PET conversion and BHET yield using unsupported metal oxides demands more severe reaction conditions [108,114]. To address this issue, catalysts consisting of supported metal oxides on porous carbon allotropes and mixed metal oxide spinels as support materials were developed. Zhang et al. [115] focused on examining the effectiveness of a porous Mn3O4 catalyst supported by a $MgAl_2O_4$ spinel structure for PET glycolysis, with an emphasis on its ability to be reused and regenerated. A high surface area was achieved by creating a mesoporous structure and introducing oxygen defects in the MgAl2O4 spinel through a process involving MgAl–LDH calcination followed by targeted removal of MgO. This structure interacted effectively with Mn_3O_4, resulting in even distribution, stability at high temperatures, numerous active sites, and strong catalytic performance for PET glycolysis. The Mn_3O_4/p-spMgAl800 catalyst demonstrated outstanding potential for reusability and regeneration, maintaining consistent catalytic efficiency across multiple cycles and regeneration sequences. Under optimum conditions (190 °C for 3 hours with an EG/PET ratio of 20), the catalyst produced a BHET yield of 97.6%. The catalyst's capacity for multiple uses facilitated a cost-effective approach to scalability, enhancing glycolysis rates and energy efficiency. Xuan Yang et al.[116] achieved 81.7% yield of BHET at 180 °C for 4 h from glycolysis of post-consumer PET in the presence of EG and metal azolate framework-6 (MAF-6) as a catalyst. The catalyst was recovered easily and reused five times, preserving the same reaction activity. Their findings indicated that the pore size of MAF-6 could serve as a model for creating better-performing and more discerning catalysts. This insight could be valuable in developing PET waste conversion methods that can be sustained over time. Yao et al. [117] by synthesising mesoporous ZnO/SAB15 catalysts, obtained BHET with good chroma from PET depolymerisation through a glycolysis reaction. The BHET yield was 91% at 197°C in 1 h, the EG/PET ratio was 4:1, and the Cat/PET ratio was 5%. The catalyst was recycled and reused six times, but the catalyst activity decreased gradually. Calcium oxide (CaO) displays effective catalytic performance in transesterification processes used for biodiesel synthesis. Based on this observation, CaO holds the potential as a favourable catalyst for PET recycling. offering a sustainable avenue for PET recycling. Putisompon et al. [118] provided CaO from food waste and used it to depolymerize PET waste bottles. The BHET obtained yield was above 76% at 100°C in 2 h with a PET/Solvent ratio of 1:15. The study suggested that CaO derived from ostrich eggshell can be a promising catalyst for depolymerizing PET due to its low cost, environmentally friendly nature and high yield of the product.

3.4.1.2. Nano catalysts. Nano-based catalysts are patented for increasing efficiency in the recycling process and are known as environmentally friendly [119]. Nano catalysts have become a hopeful solution to address the issues at hand, surpassing traditional catalysts' benefits. Nano-based catalysts have higher surface areas, better dispersion, and more significant reactivity. Due to their catalytic performance in the PET glycolysis process, there is substantial research into metal oxide-based nano-catalysts, including MgO, Fe_2O_3, and ZnO. Apart from metal oxide-based catalysts, various types of nano-catalysts have also been investigated for their potential in the glycolysis of PET. Carbon-based materials like carbon nanotubes and graphene have shown significant catalytic activity and selectivity in PET glycolysis. Additionally, organic-based catalysts, including enzymes and ionic liquids, have demonstrated potential in PET glycolysis. In general, creating catalysts based on nanotechnology for the glycolysis process of PET presents a hopeful solution to the problems linked with traditional metal-based catalysts. However, additional investigation is required to refine the production process and enhance the efficiency of these catalysts, as well as assess their scalability and economic feasibility for recycling PET [120–122]. Jin et al. [123] synthesised 2D holey MnO_2/graphene oxide (GO) nanosheets. The nanosheets exhibited an ultrathin structure with pores that lay within the plane, yielding a specific surface area of 247 m^2 g^{-1} and nanopores measuring less than 20 nm in diameter. The utilisation of MnO_2/holey GO (HGO) as a catalyst in the glycolysis of PET revealed substantially improved catalytic effectiveness compared to MnO_2/GO. Achieving a complete glycolysis reaction and resulting in a BHET yield of 100% was possible under the conditions of 0.01 wt% catalyst, 50 mL of EG, and 3 g of PET in 10 minutes at 200°C using a round-flask reactor. Furthermore, in the fifth successive reaction (recycling test) conducted under the same conditions, both PET conversion and BHET yield reached 100%. Zishuai Wang et al. [124] catalysed the recyclable PET glycolysis and poly (bisphenol A carbonate) (BPA-PC) and polylactic acid (PLA) methanolysis by using the graphite carbon nitride colloid (GCNC). The use of GCNC led to a reduction in cost and environmental pollution. With optimized reaction conditions, they achieved complete conversion of PET within just half an hour. The solvent and catalyst both easily recycled, maintaining the same reaction activity for up to five cycles. BHET was obtained by chilling the solution without requiring water addition. Moreover, GCNC has also been shown to degrade PLA and BPA-PC into useful products effectively. Cao et al.[125] studied the synthesis of Co/ZnO and Mo/ZnO nanosheets as efficient catalysts in PET glycolysis. The yield rate of BHET production when using Mo/ZnO nanosheets compared to conventional $Zn(OAc)_2$ catalysts was reported to be 94.5% in this research. This increase in yield was due to two accelerations of C-O bond breaking in the PET structure due to synergistic catalytic action (Fig. 11.).

Nanosized heterogeneous catalysts are other types of catalysts studied in the glycolysis method. Guo et al. [126] used Mg-Al double oxides fused onto small magnetic Fe_3O_4 particles to create a hierarchical structure that provides a large active surface area to the resulting Mg-Al-O@Fe_3O_4 microparticles. These microparticles exhibited excellent catalytic performance in the glycolysis process of PET, and BHET yield was over 80% at 240 °C in 90 minutes in 0.5 wt% of the catalyst. Because of suitable magnetic properties, the catalyst was easily recovered and reused. They claimed this synthesised micro catalyst has the potential to facilitate a more sustainable and eco-friendly large-scale circular recycling of PET fibre waste. Veregue et al. [121] degraded PET waste using cobalt nanoparticles to catalyse glycolysis. The achieved BHET yield as the only product reaction was 77% at 180 °C in 3 h, and no dimer was detected. The nano-catalyst was recovered and reused five times. The process is environmentally friendly and economically sustainable since the catalyst doesn't need to be removed from the solvent, making it easy to reuse, and water is not required in the BHET precipitation process. Li et al. [127] checked the degradation of PET via glycolysis catalysed by zinc ferrite nanoparticles (Zn-MNPs). The BHET yield was 79.82% at 196 °C in 2 h with low EG consumption (EG/PET molar ratio = 7). The catalyst was recovered easily using magnetic decantation and reused with high catalyst activity five times. They declared that in contrast to other magnetic recoverable catalysts, the Zn-MNPs produced are more cost-effective, exhibit stable catalytic activity under mild conditions with minimum EG requirements, and can be used for eco-friendly upcycling of PET waste at an industrial level.

3.4.1.3. *Ionic liquids.*

Ionic liquids were first used in PET recycling by glycolysis in 2009. Ionic liquids have high solubility, thermal stability, non-volatility, low flammability, and high efficiency [128,129]. On the other hand, Ionic liquids, praised for stability and low volatility, contain organic cations and various anions, seemingly environmentally friendly. Yet, escalating usage in diverse industries raises contamination risks, necessitating an assessment of their ecological impact. Bioassays reveal their toxicity to different organisms, challenging their 'green' label. Urgent development of less toxic, biodegradable alternatives align with sustainability objectives. Balancing industrial needs with environmental consciousness remains imperative. Liu et al. [130] studied metal metal-free choline-based ionic liquids (ILs) effect as a catalyst in depolymerizing PET wastes through glycolysis reaction. Under the optimum condition, the BHET monomer yield was over 85% at 180^0 C, four h. The absence of metal in the structure of the catalyst makes it cheaper and environmentally friendly. The examination of the reaction kinetics revealed that it is the shrinking-core model. The apparent activation energy for this process is determined to be 131.31 kJ/mol, with a pre-exponential factor of 1.21×10^{13} $min^{-1.}$ The combination of the cation and anion in the ionic liquid (IL) facilitated the attack of oxygen in EG on the carbon of the carbonyl in PET, resulting in easier degradation. The research showed that this structure could degrade PET without using costly catalysts containing precious metals. As a result, it may guide the development of more efficient and economic IL catalysts for use in the PET recycling industry. Lei Wang et al. [131] employing 1,3-dimethylimidazolium-2-carboxylate (zwitterionic carbonate) as a metal-free organ catalyst, had an innovation in ILs application in the PET waste degradation in EG. The PET was completely degraded at 185 °C in 1 h, and the BHET recovered around 60% yield. Compared to the simple ionic liquid known as 1,3-dimethyl imidazolium acetate, the 1,3-dimethylimidazolium-2-carboxylate is considerably more efficient as a catalyst or pre-catalyst. Zhang et al. [132] synthesised a series of ionic liquids (ILs) based on terephthalic acid (TPA) for use in the glycolysis of PET. The interactions formed through hydrogen bonds between the IL's anions and cations effectively promoted the transesterification reaction. TPA's structural resemblance to PET can minimize the incorporation of heteroatoms in the reaction system, thereby enhancing the purity of the monomer product. Through optimization involving PET (5.0 g), EG (18.0 g), and 1-Ethyl-3-methylimidazole terephthalate ($[EMIm]_2TPA$) (0.24 g) at 197 °C for 106 minutes, both PET conversion and BHET yield reached 100% and 83.6%, respectively. Furthermore, $[EMIm]_2TPA$'s catalytic efficiency remained exceptional even after being reused 15 times, showcasing its potential for efficient recycling among reported IL catalysts. The resulting BHET from the catalytic depolymerisation by $[EMIm]_2TPA$ demonstrated enhanced purity. Moreover, the recycled PET generated through repolymerisation of the obtained BHET exhibited a relatively lighter colour The calculated values for the activation energy were 141.01 KJ/mol, and the pre-exponential factor 7.2×10^{13} min^{-1} was reported. Cot et al. [133] synthesised and characterized an oxalate-bridged binuclear iron (III) ionic liquid combined with an imidazolium based cation $(dimim)_2$ $[Fe_2Cl_4(\mu\text{-ox})]$ catalyst and investigated its effect under Microwave irradiation and conventional PET decomposition. Both methods demonstrated complete PET consumption and significant conversion to BHET, yet microwave-assisted heating exhibited substantially shorter reaction times and reduced energy consumption. Notably, the complex yielded higher BHET conversion under microwave conditions, achieving over 99% conversion in just 3 hours, compared to 80% conversion in 24 hours with conventional heating for post-consumer PET.

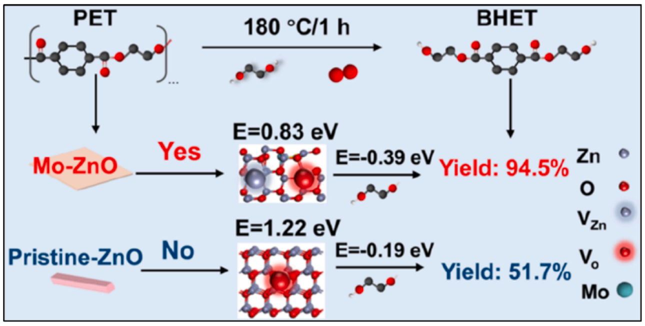

Fig. 11. Schematic of recycled PET for BHET production in the presence of nano catalyst [125].

3.4.1.4. *Deep eutectic solvents.*

Deep eutectic solvents (DESs) generally consist of two or three inexpensive and safe components that can interact through hydrogen bonding interactions to form a eutectic mixture. DES can be prepared with the mixing of two components: one of them is a hydrogen bond acceptor (HBA), and the other is a hydrogen bond donor (HBD). The lower melting point of each component characterizes the resulting DES. To qualify as a green solvent, these solvents must meet various criteria such as biodegradability, availability, non-toxicity, recyclability, flammability, and low cost [134–137]. DESs have demonstrated remarkable promise within PET glycolysis reactions. Bo Liu et al. [138] degraded PET in the presence of 1,3-dimethylurea (1,3-DMU) based DESs. The PET conversion was 100% and the maximum yields of obtained BHET was 82% in 20 min at 190° C. The temperature of the reaction is a critical factor in PET glycolysis. The kinetic analysis revealed that the glycolysis of PET using 1,3-DMU/Zn $(OAc)_2$ followed the shrink-core model and had an apparent activation energy of 148.89 kJ/mol. The catalytic efficacy of DES was attributed to the collaborative action of the acid and base formed between metal salts and 1,3-DMU. The proposed mechanism for the glycolysis reaction is illustrated in Fig. 12.

Li et al. [139] investigated the degradation of PET into BHET using DESs based on 1,8-diazabicyclo (5.4.0) undec-7-ene (DBU) because of its intense activity, economical cost, and feasible production characteristics (Fig. 13). To enhance the DBU catalytic activity, they added different molar ratios of nitrogenous compounds and synthesised several different DESs. Under optimum conditions (3.2 wt% DES, PET/EG ratio of 1:3, DBU/ 1,3-dimethylurea ratio 1:1, at 180 °C, and a reaction time of

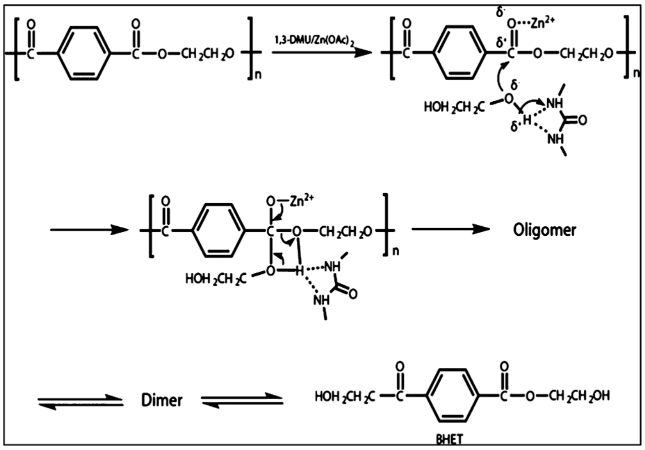

Fig. 12. Mechanism for the glycolysis reaction catalyzed by 1,3-DMU/Zn(OAc) 2 [138].

70 minutes), complete degradation of PET bottle flakes was achieved, yielding an 83% BHET output. They hypothesized that the interaction between EG and DES and between PET and DES likely facilitates glycolysis of PET, enabling the hydroxyl oxygen in EG to attack the carbonyl carbon in PET. The suggested mechanism, supported by 1 H NMR, Carbon-13 nuclear magnetic resonance (13 C NMR), and density functional theory (DFT) calculations, indicated that the creation of hydrogen bonds significantly enhances the PET glycolysis process.

Sert et al. [140] synthesised five types of different DESs, using various hydrogen bond donors and acceptors, and applied them as the catalyst in depolymerisation of PET material via glycolysis. The results proved that the most effective DESs were those composed of potassium carbonate and EG. The maximum amount of BHET was achieved as 88%, at 180°C, in 2 h, by EG/DES ratio of 15 and DES/PET ratio of 6. Nevertheless, DESs dissolve within the reaction system when they use homogeneous catalysts. A common approach to recycle the DES catalyst involves evaporating a substantial solvent volume under reduced pressure, leading to significant energy consumption. To address the challenge of liquid catalyst retrieval, a typical solution involves immobilizing the catalyst onto solid materials [141]. Wang et al. [142] used DES@ZIF-8 composite catalyst in the glycolysis method. To identify a suitable catalyst, four metal salts $ZnCl_2$, $NiCl_2·6\ H_2O$, $CoCl_2·6\ H_2O$, and $FeCl_3$, each with acetamide were used to create DESs and subsequently, were immobilized on ZIF-8, resulting in DES@ZIF-8. They compared their catalysts with some reported catalysts in Table 4.

The results showed that this catalyst had high performance and reduced reaction time. The catalyst was recovered simply through a filtration and reused for 6 times. They suggested the following mechanism for PET glycolysis (Fig. 14). The nitrogen atom of the acetamide's amino group creates a hydrogen bond with the hydroxyl group in ethylene glycol. This interaction enhances the electronegativity of the hydroxyl oxygen in ethylene glycol. Additionally, the Zn^{2+} ions in $ZnCl_2$ can coordinate with the carbonyl oxygen in PET. This coordination increases the positive charge on carbon, forming carbocations. Carbocations are notably more susceptible to attack by the hydroxyl oxygen in ethylene glycol, resulting in the breakage of the PET chain.

In a study, Zhu et al. [146] synthesised 1,5,7-triazabicyclo [4.4.0] decium acetate/zinc acetate (HTBD-OAc/$Zn(OAc)_2$) DES and examined it as a catalyst in the glycolysis process of PET waste. BHET was achieved by 88.5% yield at 190°C in 40 min. The catalyst recovered and was reused many times; still, the BHET yield was not less than 84.6%, even in the seventh cycle. Liu et al.[147] considered the separation of PET and cotton blend (PCBs) via selective glycolysis of PET in EG under betaine-based DESs. Under optimum conditions, 5 gr of PET was degraded entirely, highly purified BHET monomer was extracted with 85% yield within 45 minutes at 190 °C, and 95% of the cotton fibres were separated from PET without structural damage. The presence of betaine in the EG solvent significantly enhanced the solvent's activity, resulting in a highly effective and cost-efficient catalyst. The study's experimental design and characterization suggested that the interaction between betaine, Lewis acids, and hydrogen bonds forms a synergistic catalytic mechanism. In a similar study, Choi et al. [148] introduced a new, eco-friendly, and fast method to identify the composition of PET in blend fabrics. This technique offers an alternative to conventional identification methods that use harmful chemicals like halogenated organic solvents and strong inorganic acids. The process uses

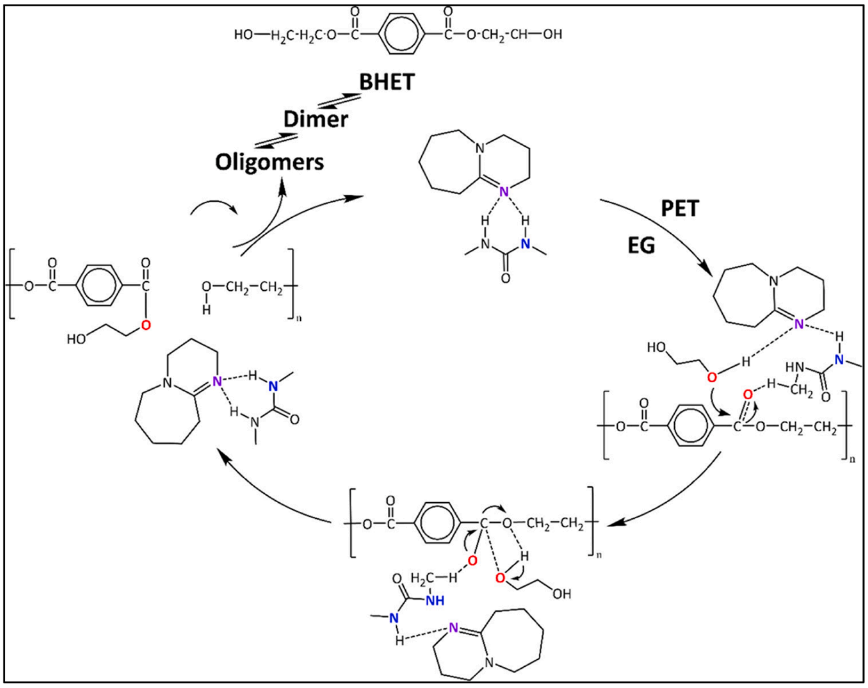

Fig. 13. Proposed mechanism for the PET degradation catalysed by DBU[139].

Table 4
Comparison of PET degradation with different catalysts [142].

Catalyst	Cat./PET	T (°C)	P (MPa)	Time (min)	PET conversion (%)	BHET selectivity (%)	BHET yield (%)	Ref.
Zinc acetate	1%	196	0.1	180	100	85.6	85.6	[143]
Sodium carbonate	0.56%	196	0.1	60	–	80	80	[144]
$ZnMn_2O_4$	1%	260	0.5	80	–	–	92.2	[108]
$CoMn_2O_4$	1%	260	0.5	80	–	–	88.8	
$ZnCo_2O_4$	1%	260	0.5	80	–	–	80	
Mn_3O_4	1%	260	0.5	80	–	–	70.1	
ZnO	1%	260	0.5	80	–	–	61	
Co_3O_4	1%	260	0.5	80	–	–	59.7	
β-Zeoilte	1%	196	0.1	480	–	–	66	[145]
γ-Zeoilte	1%	196	0.1	480	–	–	65	
γ-	5%	300	1.1	60	100	90	90	[114]
Fe_2O_3	10%	255	1.1	80	100	80	80	
DES_1@ZIF-8	**0.40%**	**195**	**0.1**	**25**	**100**	**83.2**	**83.2**	[142]
DES_1 @ZIF-8	0.40%	195	0.1	25	100	78.6	78.6	
DES_3 @ZIF-8	0.40%	195	0.1	25	86	75	65	
DES_4 @ZIF-8	0.40%	195	0.1	25	20	90	18	

Note: DES_1@ZIF-8: acetamide/$ZnCl_2$@ZIF-8; DES_2@ZIF-8: acetamide/ $CoCl_2 \cdot 6\ H_2O$ @ZIF-8; DES_3@ZIF-8: acetamide/ $NiCl_2 \cdot 6\ H_2O$ @ZIF-8; DES_4@ZIF-8: acetamide/ $FeCl_3$ @ZIF-8

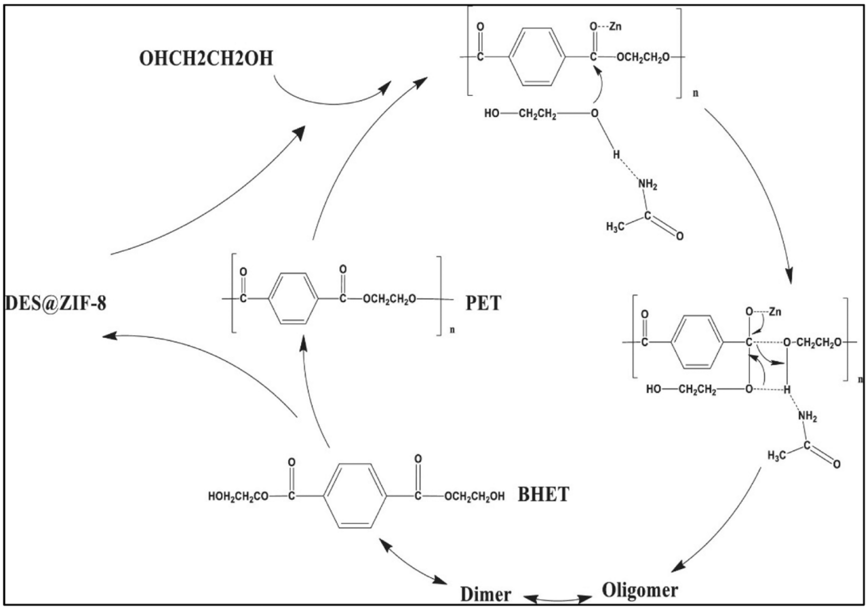

Fig. 14. Mechanism of PET glycolysis [142].

choline-based DESs, such as ethylene glycol choline chloride, as a treatment medium and microwave irradiation to remove the PET portion of blend fabrics, including 65/35 and 50/50 PET/cotton. The PET portion was entirely removed by DES containing 5% NaOH (w/v) after 100–140 s microwave irradiation. The method's effectiveness was confirmed through various instrumental analyses and practical applications on a commercial sample. In another study, they developed an eco-friendly and fast technique to depolymerize PET in blend fabric using a bio-based DES, glycerol-choline chloride (GC), and microwave irradiation. The method can replace conventional toxic solvents and remove PET from PET-cotton blends in less than 100 seconds. The depolymerisation mechanism was investigated, and a commercial sample was examined to verify the practical application [149]. Lee and Jung [150] proposed a process to recycle waste PET into a flame-retardant insulation material called PIR foam, using a special chemical catalyst known as a DES system. The method involves depolymerizing PET into BHET and its derivatives using a potassium-based DES system as a catalyst and then repolymerising the depolymerised polyols with isocyanate to produce PIR foam. The recycled PIR foam is more thermally stable than conventional PIR foam, making this an environmentally friendly and efficient method for upcycling waste PET into a useful product. Lee et al. [151] proposed a method for using DES as catalysts for the depolymerisation of waste PET to produce recycled monomers, which were then used to create polyurethane foam (PUF) via a one-pot reaction. This DES-based one-pot reaction was an environmentally friendly and efficient approach that required lower temperatures and reduced reaction times. The study evaluated the effectiveness of different DES catalysts and found that choline chloride/urea, zinc chloride/urea, and zinc acetate/urea effectively catalyse glycolysis. The PUF produced from unfiltered reaction mixtures exhibited higher thermal stability than PUF made from filtered polyols, suggesting that this process is economical for upcycling waste PET.

A summary of the studied catalysts and the corresponding reaction conditions employed in the glycolysis of PET is included in illustrated in Table 5.

4. Application of BHET

BHET obtained from glycolysis has various applications and is very important for researchers and industrialists. The utilisation of BHET

Table 5
Different studied reaction conditions for PET glycolysis.

Catalyst	EG/PET	T (°C)	Time (min)	PET conversion	BHET Yeild	Ref
Mn_3O_4/p-spMgAl800	20	190	180	100	97.6%	[115]
MAF-6 (with Zn^{+2})	6	180	240	92.4%	81.7%	[116]
ZnO/SAB15	4	197	60	100	91%	[117]
CaO from shells	15	192	120	100	76%	[118]
MnO2/HGO	18.5	200	10	100	100%	[123]
Mo/ZnO	4	180	60	100	94.5%	[125]
Mg-Al-O@Fe_3O_4	5	240	90	95	80%	[126]
CoNPs	-	180	180	96.7	77%	[121]
Zn-MNPs	7	196	120	100	79.82	[127]
Choline Acetate	4	180	240	~100	85.2%	[130]
1,3-dimethyl imidazolium-2-carboxylate	10	185	60	100	60%	[131]
$[EMIm]_2TPA$	3.5	197	106	100	83.6%	[132]
$(dimim)_2$ $[Fe_2Cl_4(\mu\text{-ox})]$	-	Microwave	180	>99	99%	[133]
		170	24 h	>99	80	
1,3-DMU/Zn (OAc)2	4	190	20 min	100	82%	[138]
DBU	3	180	70 min	100	83%	[139]
K2CO3/EG	15	180	120 min	100	88%	[140]
Acetamide/ZnCl2@ZIF-8	5	195	25 min	100	83.2%	[142]
HTBD-OAc/Zn(OAc)2	5	190	40 min	100	88.5%	[146]
[Betaine] Zn [OAc]2	5	190	45 min	100	85%	[147]

within the polymerisation process, alongside the existing raw material of EG/TPA, enables the production of PET [152,153]. On the other hand, the synthesis of materials such as PU (polyurethane), UPR (unsaturated polyester), vinyl esters resin, textile softeners, alkyd resin, epoxy resin, hydrophobic textile dyes, and waterborne coating was done using BHET. PU is a versatile material used in various applications such as adhesives, coatings, foams, and elastomers. BHET can be used as a precursor for synthesising polyurethane, improving the material's mechanical properties and thermal stability [154–160]. Besides, BHET can be used as a raw material to produce UPR and vinyl ester resins, improving the mechanical properties and reducing the environmental impact of composite materials. UPR and vinyl ester resins commonly produce composite materials such as fibreglass [161–164]. In a study, Cevher and Surdem [165] conducted PET bottle waste depolymerisation using EG and ethanolamine, yielding BHET and BHETA, respectively. These compounds were used as polyols to create PU adhesives, followed by mechanical testing. Introducing castor oil into the synthesis of PU adhesives with BHET and BHETA led to enhanced adhesive performance due to their role as crosslinking agents. However, the effect of castor oil addition on BHETA-based PU synthesis had a more pronounced impact on shear strength, possibly because BHETA achieved a more consistent mixture with castor oil than BHET. Paek and Im [166] created a range of biodegradable copolymers containing both aromatic and aliphatic components directly synthesised from BHET and aliphatic dicarboxylic acids, including succinic acid, adipic acid, sebacic acid, and dodecanedioic acid through a two-step melt polycondensation process. They adjusted the copolymer composition to optimize their crystalline structure, thermo-mechanical attributes, and biodegradability. Notably, the resulting copolymer films displayed remarkable elasticity with a tensile strength below 27 MPa and elongation at break approaching 700%. Additionally, these films exhibited impressive optical clarity (haze less than 10%), surpassing the performance of traditional petroleum-based and biodegradable polymers. The biodegradable aromatic-aliphatic copolymers, synthesised using BHET, showcased exceptional transparency, mechanical characteristics, and biodegradability, suggesting their significant potential for PET waste recycling and sustainable packaging applications.

Chandra et al. [167] demonstrated that incorporating BHET obtained from the PET waste glycolysis enhances sand-bentonite mixtures for landfill covers and liners. BHET polymer forms strong bonds with bentonite through electrostatic or ion-dipole interactions, resulting in a resilient microstructure within sand grains that resists compression and swelling. The hydrophilic properties of BHET create hydrocolloids, reducing water flow and hydraulic conductivity (*k*). The addition of BHET polymer forms a hydrogel network that interlocks sand grains and reinforces them with embedded bentonite, increasing resistance to compression (*qu*) and split tensile strength (*qt*), especially at higher dry densities. Furthermore, BHET's moisture-absorbing characteristics mitigate particle shrinkage and capillary forces, improving overall stability. The microstructural analysis supports the proposed binding mechanism. The study suggests that a 3% BHET content effectively enhances sand-bentonite mixtures, maintaining stability, low hydraulic conductivity, minimal shrinkage, and high strength, thus showing promise for landfill applications and waste management solutions.

MOFs possess desirable properties like substantial surface area and antimicrobial traits, making them suitable for controlled molecule release in biomedical uses. Nevertheless, their creation is costly and ecologically harmful due to organic ligands and solvents [168]. Cabrera-Munguia et al. [169] suggested a novel MOF synthesis method using the environmentally friendly approach of aluminium ions and BHET organic ligand recovered from PET glycolysis without applying any organic solvents. The structure and attributes of this new BHET-Al MOF were compared with the representative MIL53-Al MOF. Through XRD and Scanning electron microscopy (SEM) analyses, both materials were found to possess rhombohedral structures, with BHET-Al forming microporous clusters of smaller crystals. Nitrogen physisorption results support the microporous nature of the new MOF. TGA and FTIR characterizations indicated higher thermal stability in BHET-Al than MIL53-Al, attributed to stronger coordination bonds between aluminium ion and BHET ligand than with the BDC ligand. XPS analysis of binding energies indicated that the coordination bonds in this new material arise from electron sharing between Lewis bases and the 2p orbitals within the aluminium ion's coordination sphere. Besides, the chemical structure of BHET-Al MOF displayed no cytotoxicity to fibroblasts over 48 hours of culture, significantly improving cell viability compared to the control. This non-toxic nature makes it advantageous for potential biomedical applications. In summary, using BHET obtained from glycolysis offers a sustainable route to producing a range of high-value materials. By utilising BHET as a raw material, we can reduce our reliance on virgin petrochemicals and promote a more circular and sustainable economy.

5. Research gap and recommendation

a. The existing literature provides valuable insights into various aspects of recycling polyethylene terephthalate (PET), particularly focusing on decolorization and impurity removal processes within glycolysis-based recycling methods [82,127,170]. However, there remains a significant research gap regarding the specific challenges and opportunities associated with recycling coloured PET through glycolysis. To address this gap, it is essential to conduct comprehensive investigations that specifically explore the complexities of glycolysis in the context of coloured PET recycling. This research should encompass an in-depth analysis of the effects of different colorants and additives on the glycolysis process and evaluate the quality of the resulting recycled PET. By undertaking such a thorough inquiry, valuable insights can be gained into the obstacles and prospects associated with glycolysis-based recycling of coloured PET, ultimately enhancing the efficiency of recycling techniques tailored to coloured PET.

b. A research gap exists concerning comprehensive techno-economic analyses that compare different chemical methods for PET recycling within an industrial context. To address this gap, it is recommended to undertake thorough techno-economic evaluations encompassing diverse PET recycling methods. These evaluations should encompass mechanical recycling, chemical recycling, and depolymerisation techniques. A holistic approach should be taken, considering energy consumption, raw material costs, equipment investment, and environmental impact. By conducting such analyses, valuable insights can be generated to assist industries in selecting the most suitable, cost-effective, and sustainable PET recycling method.

c. Amidst the increasing interest in utilising DES for PET recycling [171], a research gap persists in comprehending the precise mechanisms and optimal conditions required for PET dissolution and separation. To address this gap, it is strongly advised to embark on comprehensive studies that delve into the intricate interactions between PET and a range of DESs. This endeavour should encompass a detailed exploration of the impacts of DES composition, temperature variations, and processing durations on PET depolymerisation and the efficiency of separation processes. Such endeavours will be pivotal in developing efficient PET recycling processes centring around deep eutectic solvents.

d. Limited research has been undertaken to comprehensively investigate the utilisation of BHET in polymer production, despite its considerable potential as a precursor monomer. It is imperative to initiate specialized research endeavours to delve into BHET's application in polymer synthesis. These endeavours should encompass an in-depth analysis of polymerisation kinetics, inherent properties, and plausible applications inherent to polymers synthesised from BHET. To illuminate the distinct advantages and challenges presented by BHET-based polymers, it is recommended to undertake comparative studies vis-à-vis conventional monomers. This multifaceted approach promises to unravel new horizons in polymer science and technology.

6. Conclusions and outlook

This review has delved into the critical realm of PET recycling, focusing primarily on glycolysis as a chemical recycling method. As the global production of plastic waste continues to rise, finding sustainable solutions to manage PET waste is paramount. This review has highlighted the significance of chemical recycling in addressing the environmental concerns linked to PET disposal. Primary recycling, though cost-effective, is limited in its applicability, and mechanical recycling presents challenges such as contamination and material degradation. In contrast, chemical recycling, particularly glycolysis, offers several advantages, including the potential for high PET conversion rates, reduced greenhouse gas emissions, and the ability to produce valuable monomers like BHET. The role of catalysts in glycolysis cannot be overstated, as they significantly influence reaction efficiency. Metal-based catalysts, nano catalysts, ionic liquids, and deep eutectic solvents have emerged as promising options, each with its unique advantages and challenges. These catalysts can potentially make glycolysis a more sustainable viable recycling method. Furthermore, the applications of BHET, the key product of glycolysis, in synthesising various materials like polyurethane, unsaturated polyester resins, and coatings underscore its importance as a valuable resource derived from PET recycling. While substantial progress has been made in PET recycling, ongoing research, and development are essential to optimise processes, improve catalyst efficiency, and enhance the scalability of chemical recycling methods. These advancements are crucial for achieving a more sustainable and eco-friendly approach to PET waste management, thereby contributing to a cleaner and greener environment. As the world grapples with the plastic waste crisis, chemical recycling, particularly glycolysis, is a promising avenue to turn the tide and pave the way for a more sustainable future.

Conflicts of interest

There are no conflicts to declare.

CRediT authorship contribution statement

Milad Jalilian: Writing – original draft, Resources, Investigation, Data curation, Conceptualization. **Mahsa Babaei:** Writing – original draft, Resources, Project administration, Investigation, Formal analysis, Data curation, Conceptualization. **Kaveh Shahbaz:** Writing – review & editing, Visualization, Validation, Supervision, Resources, Conceptualization.

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.

Data Availability

No data was used for the research described in the article.

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