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Glycolysis-Mediated Depolymerization of Plastic Waste to Terephthalic Acid: Mechanisms and Technological Benchmarks

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ABSTRACT

This review systematically evaluates glycolysis-mediated depolymerization configurations to establish kinetic benchmarks for terephthalic acid (TPA) recovery, comparing conventional thermal heating against microwave-assisted routes utilizing Deep Eutectic Solvents (DES). Adhering to PRISMA guidelines, we analyze the operating parameters, degradation mechanisms, monomer yields, and carbon emission profiles to map the thermodynamic and mass transport boundaries of both pathways. The synthesized data reveals that microwave-assisted configurations achieve intense kinetic acceleration, enabling 100% polymer conversion within a highly compressed duration of 3.5 to 10 minutes by delivering uniform volumetric heating that bypasses traditional conductive resistance. Furthermore, integrating DES systems utilizing thiourea-based hydrogen bond donors optimizes charge density and coordination, pushing peak TPA yields up to 80.66%, which significantly outperforms conventional urea-based alternatives. However, from an environmental standpoint, the microwave-assisted system generates a higher carbon footprint of 0.364 tons of CO₂ per year compared to the 0.269 tons of CO₂ per year produced by simpler thermal techniques. While microwave-assisted DES glycolysis offers an expedited pathway for circular resource recovery, its practical industrial scale-up remains constrained by severe technical trade-offs, localized electrical arcing risks, thermal tracking lags, and elevated carbon loads.

Contribution to Sustainable Development Goals (SDGs):

SDG 9: Industry, Innovation, and Infrastructure

SDG 11: Sustainable Cities and Communities

SDG 12: Responsible Consumption and Production

SDG 13: Climate Action

1. INTRODUCTION

1.1. Research Background

The increase in Indonesia's population is driving a rise in annual waste generation. Polyethylene terephthalate (PET) dominates plastic waste streams because of its widespread use in packaging, beverage containers, and textiles. Its popularity is due to high

mechanical strength and flexibility [1][2][3]. This massive accumulation of PET waste poses a major challenge for waste management systems and threatens global ecosystem stability [4].

The national waste management effectiveness rate remains limited to 60.49%. Therefore, innovation in recycling strategies is now an urgent priority. Mechanical recycling approaches risk degrading polymer quality [5]. In contrast, chemical recycling breaks polymer chains and regenerates pure monomers without compromising product quality[6]. Beyond material advantages,



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this chemical approach may reduce greenhouse gas emissions by up to 50% compared with virgin PET manufacturing [7]. The glycolysis process uses ethylene glycol (EG) as a solvent, making it the most widely implemented method at industrial scale. It offers competitive operating costs and minimal solvent volatility, aligning with sustainable production principle [8][9][10]. The main depolymerization product, terephthalic acid (H2BDC), has high economic value as a polymer feedstock and as a precursor for Metal-Organic Frameworks (MOF) materials [11].

Conventional glycolysis has technical efficiency limits. Without a catalyst, reactions progress slowly and require high temperatures for long periods [7][12]. Such thermal conditions are suboptimal for energy efficiency and environmental sustainability [13]. As a volumetric heating technology, microwave irradiation provides a viable alternative to conventional heating by facilitating rapid reaction times and a more uniform heat distribution. Furthermore, this method can reduce solvent usage and lower specific energy consumption per batch. However, its overall sustainability profile must still be evaluated alongside its total carbon footprint [10][14][15].

This article provides a critical comparative review of terephthalic acid (TPA) synthesis from PET waste, focusing specifically on the synergy between microwave heating and Deep Eutectic Solvent (DES) catalytic systems. Unlike existing literature that merely describes individual recycling methods, this study establishes performance benchmarks by critically analyzing operational parameters, monomer yield, and energy consumption profiles for both microwave and conventional methods. By evaluating these technical parameters, this study aims to present an objective comparison between microwave and conventional heating technologies. It systematically assesses their respective impacts on reaction times, thermal energy utilization, and the overall efficiency of transforming PET waste into high-value products.

1.2. Literature Review

Inadequate management of post-consumer plastic waste presents a critical ecological challenge on a global scale. Conventional mitigation strategies predominantly rely on mechanical recycling pathways. These pathways encompass sequential stages of collection, sorting, washing, shredding, extrusion, and subsequent remanufacturing [9]. In the context of developing nations like Indonesia, data from the National Waste Management Information System (SIPSN) of the Ministry of Environment and Forestry (KLHK) indicated that total waste generation exceeded 33.67 million tons in 2023, with plastic waste constituting 19.4% of the total stream. A substantial portion of this municipal plastic fraction originates from single-use beverage containers fabricated from polyethylene terephthalate (PET) [16]. From a macromolecular perspective, PET is a semi-crystalline thermoplastic polyester synthesized via the polycondensation of terephthalic acid (TPA) and ethylene glycol (EG) [16] [17]. Attributed to its superior thermal stability, chemical inertia, and low carbon dioxide (CO₂) permeability, PET is extensively utilized in global packaging sectors. This widespread utility has driven a profound escalation in annual consumption [18] [19]. Although global production volumes have expanded significantly to approximately 24.2 million tons annually, the transient lifecycle of these single-use materials inevitably accelerates their accumulation within municipal solid waste streams [20]. This discrepancy between skyrocketing

consumption rates and suboptimal disposal infrastructures underscores the imperative for advanced chemical recycling methodologies. Among various upcycling paradigms, selective depolymerization to recover high-purity TPA is highly strategic. Because TPA constitutes the core aromatic backbone and the largest mass fraction within the PET polymer matrix, its direct synthesis from waste streams bypasses the structural degradation intrinsic to mechanical downcycling. This approach not only closes the material loop but also yields a high-value chemical platform, establishing recovered TPA as a vital objective for rigorous evaluation.

1.2.1. Polyethylene Terephthalate (PET)

Polymers are large macromolecules formed from long chains of repeating monomer units. Well-known polymer types include polyethylene terephthalate (PET), low- and high-density polyethylene (LDPE and HDPE), polypropylene (PP), polyvinyl chloride (PVC), and polystyrene (PS). The differences between these polymer types are determined by the substitution of one or more hydrogen atoms with specific other atoms [21]. Notably, PET is classified as a polyester because it contains ester functional groups in its main chain [17]. Its formation process involves a condensation polymerization reaction between terephthalic acid (H2BDC) and ethylene glycol (EG) [16]. PET is known for its excellent resistance to high temperatures and chemicals, as well as its low CO₂ permeability [19]. Owing to these superior characteristics, PET is widely used for beverage packaging and various other synthetic applications, thereby directly driving a surge in PET demand over time [18] This is reflected by global PET production, which reached 24.2 million tons in 2021. However, PET-based products generally have a very short lifespan due to single-use consumption patterns. The ongoing trend of rising consumption, coupled with the lack of an efficient waste management system, ultimately results in a massive annual accumulation of PET waste [20].

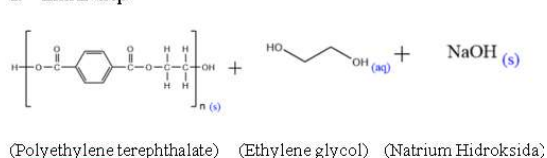
1.2.2. Depolymerization Mechanism: Glycolysis and Alkaline

Polyethylene terephthalate (PET) depolymerization involves breaking the polymer into its monomeric components. This is usually achieved using enzymatic or chemical pathways. Depolymerization is important for recycling and upcycling PET waste, converting it to the building block terephthalic acid (TPA) [22]. Glycolysis is one degradation pathway that breaks PET into monomers, mainly bis(2-hydroxyethyl) terephthalate (BHET) and TPA. This method uses a glycol solvent, such as ethylene glycol (EG), and a catalyst to help break the polymer chains [23]. As illustrated in **Fig. 1**, the complete depolymerization of waste PET into high-purity TPA follows a stepwise mechanism encompassing glycolysis, alkaline hydrolysis, and acidification. The initial stage involves the glycolysis of PET into BHET, driven by EG as the transesterification agent. The hydroxyl group of EG acts as a nucleophile, executing an attack on the ester linkages along the PET backbone. This nucleophilic interaction forms a transient tetrahedral intermediate. Subsequently, the C–O bond within the alkoxy group undergoes cleavage, thereby releasing smaller oligomers or monomers. This sequential transesterification recurs continuously to yield BHET as the primary product-characterized by free hydroxyl groups at both termini-while liberating EG as a byproduct.

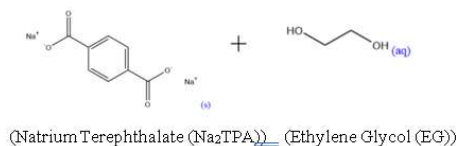
During the subsequent stage, the remaining ester linkages within the BHET intermediate undergo hydrolysis utilizing sodium hydroxide NaOH as a strong base. The hydroxide ion OH⁻ attacks the carbonyl carbon atom of the ester group, forming another tetrahedral intermediate. Following this, the O-CH₂-CH₂-OH moiety is displaced as a stable leaving group, resulting in the release of two EG molecules. The newly formed carboxylic acid intermediates are promptly deprotonated by NaOH, generating sodium terephthalate (Na₂-TPA), which is a water-soluble disodium salt of TPA.

Finally, the acidification of Na₂-TPA into TPA involves the reaction of the ionized disodium salt with a strong acid, such as hydrochloric acid (HCl). Protons H⁺ derived from HCl protonate the carboxylate ions (COO⁻), successfully regenerating the functional carboxylic acid groups (COOH). Due to its poor aqueous solubility, the regenerated TPA precipitates out of the solution as a solid phase, whereas sodium chloride (NaCl) remains dissolved as a byproduct. Overall, this controlled sequence of chemical transformations represents an efficient strategy for upcycling waste PET into its pristine monomeric precursor [24]. (Terephthalic Acid (TPA)) (Sodium Chloride) (Ethylene Glycol (EG))

a. Initial step



b. Glycolysis step



c. Acidification step

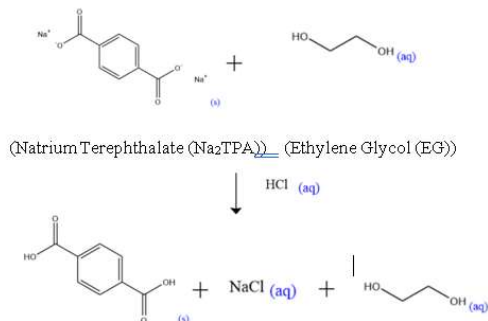


Fig. 1 PET Depolymerization Mechanism

1.2.3. Terephthalic Acid (TPA)

Terephthalic acid (TPA), or benzene-1,4-dicarboxylic acid (C₈H₆O₄), constitutes a critical industrial monomer predominantly utilized in the synthesis of polyethylene terephthalate (PET) [25]. Structurally characterized as a white crystalline powder, TPA serves as an essential precursor for manufacturing diverse polyester commodities, including synthetic fibers, films, and packaging bottles [5]. Commercially, TPA is manufactured via the liquid-phase catalytic oxidation of petroleum-derived *p*-xylene. This multi-stage process necessitates

stringent purification steps to achieve the high-purity grade required for subsequent polymerization [26]. To mitigate dependency on fossil resource extraction and address systemic plastic pollution, the selective chemical depolymerization of post-consumer PET bottles offers a highly sustainable alternative for TPA synthesis. Thermochemically cleaving the ester linkages within the waste PET matrix yields its two constituent building blocks: TPA and ethylene glycol (EG) [27]. These reclaimed monomers can be directly reintroduced into the polymerization loop to regenerate virgin-grade PET without losing structural fidelity. Given that synthetic fibers and polyester packaging materials account for a major fraction of global petrochemical demands, the closed-loop recovery of high-purity TPA from municipal plastic matrices presents a strategic pathway toward chemical sustainability and circular resource management.

1.2.4. Acidification Process

The objective of the acidification process is to efficiently convert terephthalate salt (Na₂TPA) into high-quality terephthalic acid (TPA) crystals. This is achieved by adding hydrochloric acid (HCl), chosen for its strong ability to dissociate H⁺ ions. The high concentration of H⁺ ions shifts the pH equilibrium of the filtrate and increases the system's protonation capacity. As HCl is added, there is a direct correlation between acid input and TPA precipitation rate; the resulting acidic environment triggers the conversion of terephthalate ions into water-insoluble free acid molecules that promptly form a solid phase. Accurate pH control by managing the rate of acid addition is the key operational variable to maximize yield and produce TPA crystals with the desired characteristics [28]

1.2.5. The Factors Influencing Depolymerization

The operational parameters governing the efficiency of polyethylene terephthalate (PET) depolymerization are detailed below:

a. Reaction Duration

The depolymerization of PET inherently involves the cleavage of ester linkages, which necessitates an adequate reaction duration to achieve comprehensive conversion. Insufficient reaction time restricts the complete transformation of PET into its constituent monomers, thereby resulting in suboptimal yields and incomplete depolymerization [29]. Conversely, the microwave-assisted glycolysis method has demonstrated the capacity to deliver high monomer yields within a rapid timeframe of 3.5 to 10 min, contingent upon the specific operating conditions.

b. Acid Catalyst Concentration

The concentration of hydrochloric acid (HCl) crucially influences the resultant yield of terephthalic acid (TPA) during PET depolymerization. At lower HCl concentrations, a diminished TPA yield is typically observed. This phenomenon is attributed to the competitive consumption of HCl by inherent impurities, such as stabilizers embedded within the PET polymer resin, which subsequently depletes the acid available to catalyze the acidification reaction. Consequently, utilizing higher HCl concentrations enhances the efficacy of the depolymerization process [18]. Based on safety parameters established in the HCl Material Safety Data Sheet (MSDS), concentrations ranging from 0.5 M to 2.5 M

are recommended for laboratory-scale research to minimize hazardous risks while maintaining reaction efficacy [30].

c. **Reaction Temperature and Microwave Power**

Reaction temperature exerts a profound impact on PET depolymerization by enhancing the segmental mobility within the amorphous regions of the polymer matrix. An elevated temperature accelerates molecular kinetic energy, thereby increasing the collision probability and reaction kinetics[31]. Previous literature indicates that the optimal acceleration of PET glycolysis occurs within a temperature range of 180°C to 240°C [32]. Furthermore, when performing this synthesis via the microwave-assisted glycolysis method, the applied microwave power typically ranges from 240 W to 440 W to facilitate efficient thermal distribution [33].

1.3. Research Objective

The objective of this study is to evaluate the performance of PET waste depolymerization methods through a comparative analysis of heating techniques, the glycolysis reaction mechanism, and operational parameter optimization to validate the superiority of the microwave-assisted route for the efficient production of terephthalic acid.

2. MATERIALS AND METHODS

This study employs a systematic review methodology to evaluate and consolidate contemporary literature regarding the efficacy of various heating methodologies in the glycolysis-mediated depolymerization of polyethylene terephthalate (PET) waste for terephthalic acid (TPA) synthesis. The objective of this review is to assess not only the reported monomer yields but also the operational limitations and structural consistency across current studies, thereby identifying existing knowledge gaps and providing strategic directions for future chemical recycling research. To ensure transparency and reproducibility, the multi-stage literature retrieval, screening, and selection workflow were executed in strict accordance with the Preferred Reporting Items for Systematic Reviews and Meta-Analyses (PRISMA) statement, as systematically outlined in Fig. 2.

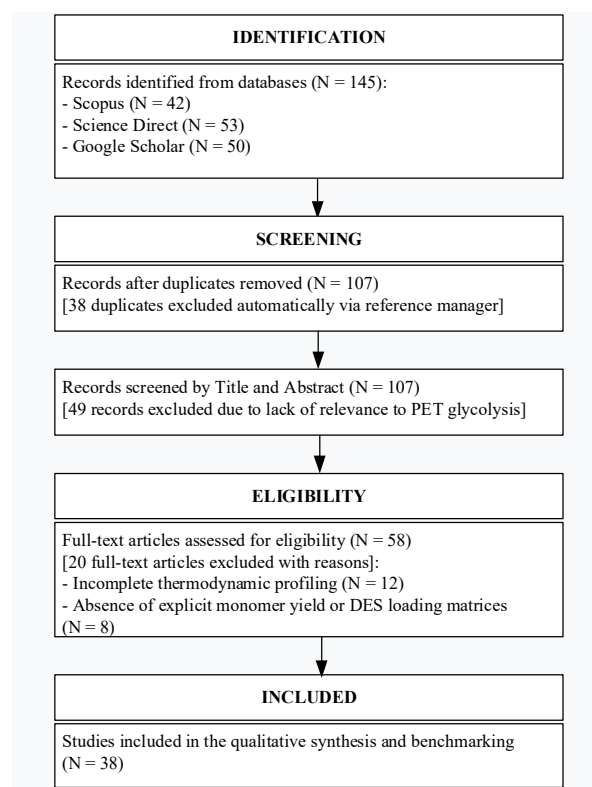


Fig. 2 PRISMA flow diagram for PET glycolysis and TPA recovery studies.

3. RESULT AND DISCUSSION

3.1. Comparison of Heating Methods and Operating Conditions in PET Depolymerization

A clear evaluation of operational parameters in various PET waste depolymerization routes is necessary to objectively assess their thermodynamic efficiency. **Table 1.** provides a comparative analysis of operating conditions-such as catalyst types, heating methods, operating temperature, reaction time, and final yield-from multiple sources.

Table 1. Comparative Evaluation of Operating and Performance Parameters Between Conventional Thermal Heating and Microwave Methods

Heating method	T _{operation} (°C)	Reaction Time (minute)	Yield (%)	Carbon emission (Ton CO ₂ /year)	Ref
Conventional Thermal	190	120	81	0,269	[34]
Reflux Heating	200	90	98	0,633	[24]
Microwave-Assisted	240	10	96,3	0,364	[35]

Table 1. shows the differences in reaction parameters and environmental impacts among conventional heating, reflux heating, and microwave irradiation for PET degradation. Conventional thermal methods generally exhibit slower heat transfer, requiring longer reaction times. For example, it takes 120 minutes at 190 °C to degrade PET into BHET [34]. However,

this method has the lowest environmental impact, generating only 0.269 tons of CO₂/year. The reflux heating method achieved a 98% yield, yet it still required 90 minutes due to kinetic limitations. While its subsequent TPA crystallization step did not require extra heat, the slow initial chain-breaking phase extended the processing time [20], contributing to the highest carbon emission among the three methods at 0.633 tons of CO₂/year. Alternatively, microwave irradiation significantly accelerates reaction kinetics by heating the solvent directly and bypassing conventional heat transfer resistance. A microwave-assisted method using 400 W of power at 240°C with a biocatalyst converted PET into BHET with a 96.3% yield in just 5 minutes [35]. This rapid reaction rate allows for immediate acidification for TPA formation. Nevertheless, despite its superior time efficiency, microwave heating produces 0.364 tons of CO₂/year. Therefore, selecting an optimal degradation method requires evaluating the trade-offs between kinetic efficiency, monomer yield, and carbon footprint.

3.2. The Mechanistic Insights into PET Glycolysis-Hydrolysis

An evaluation of the performance of various Deep Eutectic Solvent (DES) systems demonstrates that structural variations in the Hydrogen Bond Donor (HBD) components and depolymerization agents play a crucial role in determining both final product yield and selectivity.

Table 2. Performance Comparison of PET Depolymerization Using Various DES Compositions

DES Composition (HBA : HBD)	Depolymerization Agent	Reaction Condition	PET Conversion (%)	Monomer Yield (%)	Ref
1,3-dimethyl urea: Zn(Oac) ₂ (4:1)	Ethylene glycol	190°C, 20 min	100%	BHET (82%)	[36]
Choline chloride: Glycerol (1:2)	Glycerol, NaOH	Microwave assisted	38-61%	-	[37]
Choline chloride: Urea (1:2)	Na ₂ CO ₃ , Ethylene glycol	Microwave assisted, 3.5 min	98.62%	TPA (62.79%), MHET (34.79%), BHET (0.59%)	[38]
Choline chloride: Thiourea (1:2)	Na ₂ CO ₃ , Ethylene glycol	Microwave assisted, 3.5 min	99.31%	TPA (80.66%), MHET (17.22%), BHET (0.54%)	[38]

Based on the comparative data summarized in Table 2, the glycolysis approach using a synchronous Lewis acid-base catalyst composed of 1,3-dimethylurea: Zn(OAc)₂ (4:1) achieved complete PET conversion (100%) and a BHET yield of 82% under microwave-assisted heating within 20 min [36].

Conversely, the sequential glycolysis-hydrolysis system developed by Azeem et al. utilizing Na₂CO₃ in an ethylene glycol medium significantly accelerated the reaction kinetics, reducing the residence time to a mere 3.5 min while maintaining equivalent conversion rates [38]. This data demonstrates a clear kinetic advantage over traditional approaches. This rapid transesterification interaction is heavily influenced by the nucleophilic attack on the electrophilic carbonyl center within the PET chain [37]. In the choline chloride-based DES system with glycerol, the lower conversion efficiency (38–61%) was experimentally linked to the high intrinsic viscosity of glycerol, which severely restricts the mass transport of the nucleophile to the polymer surface [37].

A critical performance disparity was observed between the urea- and thiourea-based DES configurations, where the integrated glycolysis-hydrolysis system achieved PET conversions of 98.62% and 99.31%, respectively [38]. Mechanistically, the significant escalation in TPA yield from 62.79% (choline chloride:urea) to 80.66% (choline chloride:thiourea) represents a confirmed experimental trend [38]. To explain this divergence, it is hypothesized that the structural variations in the central atom of the HBD amine group—specifically, the oxygen atom (C=O) in urea compared to the sulfur atom (C=S) in thiourea—play a crucial role. The lower bond energy intrinsic to the C=S linkage in thiourea is proposed to offer higher coordination flexibility with the ethylene glycol solvent, thereby potentially facilitating the accelerated cleavage of the PET ester chains.

Furthermore, the enhanced TPA recovery in the thiourea system is inferred to be a function of its higher charge density and electrical conductivity, which are suggested to optimize the absorption rate of microwave energy. This electromagnetic synergy likely triggers localized micro-thermal effects that selectively destabilize the amorphous phase of the polymer, directing the reaction pathway toward complete hydrolysis into high-value TPA monomers. However, while these specific sub-molecular interactions align consistently with the empirical yields, further advanced spectroscopic validation or computational molecular dynamics simulations are required to confirm these proposed pathways definitively.

3.3. Evaluation of Chemical Kinetics, Catalytic Performance, and Monomer Quality

To provide a comprehensive evaluation of depolymerization methods, it is essential to assess not only heating efficiency but also key chemical kinetics parameters. Specifically, this includes examining how catalytic agents influence reaction selectivity.

Table 3 presents a comparison of catalysts and their impact on PET degradation efficiencies.

Table 3 illustrates the influence of different catalytic systems on PET conversion and product purity. In conventional thermal systems using a Zn/ZnO catalyst at a 2.0 wt% concentration, polymer conversion reached 99.0%, with a monomer yield of 81.0% [34]. Similarly, the reflux method utilizing a 2.0 wt% Y₂O₃ catalyst resulted in an 80.0% yield [25]. Despite these relatively high catalyst loads, the prolonged thermal contact required in these methods can lead to side reactions that degrade certain monomers into secondary oligomers. Conversely, microwave heating employing a zinc acetate (Zn(OAc)₂) catalyst at a significantly lower concentration of 0.04 wt% achieved a 100%

PET conversion and a 96.3% yield [35]. The rapid and efficient heat distribution in microwave systems facilitates prompt catalyst activation, which minimizes side reactions. Consequently, this approach helps maintain liquid-phase purity and aids in the isolation of terephthalic acid (TPA) crystals during acidification [35]

Table 3. Evaluation of Catalytic Systems and Conversion Efficiencies in PET Depolymerization

Heating method	Catalyst	Concentration of Catalyst (wt%)	Conversion PET (%)	Ref
Conventional Thermal	Zn/ZnO	2	99	[34]
Reflux Heating	Y ₂ O ₃	2	-	[24]
Microwave-Assisted	Zn(OAc) ₂	0.04	100	[35]

3.4. Safety Protocols and Risk Mitigation in Microwave-Assisted Pilot Systems

If high-power microwave systems are deployed in localised pilot projects, strict operational safety measures must be implemented to mitigate unique electromagnetic hazards. The critical technical risks and their corresponding engineering controls are detailed below [39]:

Electromagnetic Radiation Leakage: High-power industrial microwave generators (operating from kilowatts to megawatts) pose immediate biological risks. Uncontrolled electromagnetic field (EMF) leakage can cause deep tissue necrosis and cataracts due to the localized thermal heating of human cells. To prevent exposure, systems must incorporate heavy attenuation chokes and automated EMF sensors around all flanges and inspection ports.

Electrical Arcing and Vapor Ignition: The interaction between high-power microwave fields and metallic internals (such as stainless-steel impellers, thermowells, or baffles) creates high localized electrical field intensities. This interaction induces severe electrical arcing or plasma sparking. In the presence of pressurized, flammable glycol vapors, these sparks can trigger catastrophic internal explosions. Therefore, metallic internals must be substituted with microwave-transparent, non-conductive dielectric materials, such as specialized polytetrafluoroethylene (PTFE), industrial quartz, or high-purity silicon carbide (SiC).

Thermal Runaway Tracking: Rapid volumetric microwave heating often induces electromagnetic interference (EMI) in traditional thermocouple probes, leading to false or delayed temperature readings. This tracking lag can mask critical thermal runaway events within the reactor core. To ensure precise real-time thermodynamic profiling, pilot operations must utilize EMI-immune monitoring technologies, specifically non-invasive fiber-optic temperature sensors or infrared thermography.

4. CONCLUSION

This comparative review establishes that microwave-assisted glycolysis synchronized with Deep Eutectic Solvent (DES) catalytic systems provides a rapid, kinetically accelerated alternative to conventional thermal recycling for converting post-consumer PET waste into value-added terephthalic acid (TPA) monomers. By delivering uniform volumetric heating, this

integrated configuration successfully bypasses internal heat and mass transport limitations, compressing reaction times from several hours down to just 3.5 to 10 minutes while achieving a peak TPA recovery of 80.66% through the use of highly coordinated thiourea-based hydrogen bond donors. Despite these kinetic advantages, the process faces severe scaling boundaries and environmental trade-offs, notably generating a carbon footprint of 0.364 tons of CO₂ per year, which is substantially higher than the 0.269 tons of CO₂ per year emitted by conventional thermal methods. Operationally, scaling this system to pilot levels introduces critical hazards, including extreme localized electrical arcing at metallic boundaries, tracking lags in standard thermal sensors during runaway events due to electromagnetic interference, and severe mass transport resistance in highly viscous media like glycerol. To transition this technology from laboratory success to industrial viability, future research must shift toward developing low-viscosity, regenerable bio-based DES formulations, engineering microwave-transparent reactor internals such as silicon carbide or quartz to eliminate arcing, integrating electromagnetic-immune fiber-optic or infrared diagnostics for real-time thermal profiling, and conducting comprehensive life-cycle assessments under realistic, multi-component post-consumer plastic waste streams.

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