


Mechanical Recycling of PET– Current Situation and Perspectives

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Abstract:

The exponential rise in global petroleum-based polymer production and consumption presents a significant environmental challenge, with projections indicating an alarming growth in annual plastic waste to 460 million tons by 2030. Despite the widespread use of poly(ethylene terephthalate) (PET) for its economic appeal and versatility, the accumulation of PET waste calls for urgent and effective recycling strategies. The critical importance of PET recycling to reduce environmental impact is emphasized by current worldwide efforts, such as restrictive laws imposed by the EU and the specific material limits implemented in countries like South Korea and Japan. While several mechanisms are employed for PET recycling, mechanical recycling emerges as a standout approach due to its industry applicability. However, challenges arise from PET degradation during reprocessing, necessitating innovative solutions. In this context, diverse chain extenders, including oxazolines, isocyanates, anhydrides, epoxides, and the promising Meldrum's acid derivatives, have been explored. These agents exhibit significant potential in improving the characteristics of PET throughout the recycling process, providing an avenue to reduce the environmental impact of plastic waste. This comprehensive perspective explores the current status and future potential of PET recycling. The combination of global initiatives, mechanical recycling innovations, and advanced chain extender strategies pave the way for a more sustainable and environmentally conscious future in polymer recycling.

Keywords: PET recycling, mechanical recycling, chain extenders, PET

Poly(ethylene terephthalate) (PET) Mekanik Geri Dönüşümü – Güncel Durum ve Perspektifler

Özet:

Küresel ölçekte petrol kökenli polimer üretiminde ve tüketiminde yaşanan büyüme, yıllık plastik atık miktarının 2030'a kadar endişe verici şekilde 460 milyon tonu aşmasını öngörerek ciddi bir çevresel zorluk ortaya koymaktadır. Üretim maliyetinin ekonomikliği ve çok yönlülüğü nedeniyle poli(etilen tereftalat) (PET) yaygın bir şekilde kullanılmaktadır, ancak PET atığının birikimi, acil ve etkili geri dönüşüm stratejileri gerektirmektedir. Bu bağlamda, PET geri dönüşümünün çevresel etkileri azaltma konusundaki kritik önemi, AB'nin getirdiği kısıtlamalar ve Güney Kore ile Japonya gibi ülkelerde uygulanan özel malzeme sınırlamaları gibi küresel çapta yapılan çabalar da vurgulanmaktadır. PET geri dönüşümünde kullanılan çeşitli mekanizmalar arasında, endüstri uygulanabilirliği nedeniyle mekanik geri dönüşüm ön plana çıkmaktadır. Ancak, PET'in geri işleme sırasında yaşadığı bozunma sorunları, yenilikçi çözümleri zorunlu kılmaktadır. Bu kapsamda, oksazolinler, izosiyanatlar, anhidritler, epoksitler ve umut vaadeden Meldrum asidi türevleri gibi çeşitli zincir uzatıcılar incelenmiştir. Bu ajanlar, plastik atığının çevresel etkisini azaltma hedefine yönelik PET özelliklerini geliştirmek için önemli bir potansiyele sahiptir. Bu kapsamlı perspektif, PET geri dönüşümünün mevcut durumunu ve gelecekteki potansiyelini araştırmakta ve küresel çapta yapılan girişimler, mekanik geri dönüşümdeki yenilikler ve gelişmiş zincir uzatıcı stratejilerin entegrasyonu ile polimer geri dönüşümü alanında daha sürdürülebilir ve çevre dostu bir geleceğin yolunu açmaktadır.

Anahtar Kelimeler: PET geri dönüşümü, mekanik geri dönüşüm, zincir uzatıcı ajanlar, PET

REVIEW PAPER

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Citation: Alkan Göksu, Y., (2024), Mechanical Recycling of PET– Current Situation and Perspectives, ITU ARI Bulletin of the İstanbul Technical University 55(2) 45–52.

Submission Date: 14 January 2024
Online Acceptance : 13 March 2024
Online Publishing : 1 April 2024

1.Introduction

The escalating global production and consumption of petroleum-based polymer materials are heightening environmental concerns. Projections indicate that the annual global plastic waste, which stood at 260 million tons in 2016, is expected to surge to 460 million tons by the year 2030 (Hundertmark et al., 2018). Notably, as of 2016, a mere 16% of plastic, constituting 40% of recyclable polymer waste, has undergone recycling. In contrast, 40% has been directed to landfills, and 25% has been incinerated (Hundertmark et al., 2018). These numbers highlight the severity of the plastic pollution issue. In addition to this problem, the COVID-19 pandemic has further complicated the issue of plastic pollution in the natural environment by increasing the demand for single-use plastics derived from petroleum-based sources (Irfan et al., 2022; Jang et al., 2022; Prata et al., 2020).

On the other hand, poly(ethylene terephthalate) (PET) is renowned for its favorable economic cost, lightweight nature, durability, high strength-to-weight ratio, recycling potential, reusability, and extensive range of applications. Consequently, PET is widely utilized in the food and non-food packaging sectors. However, as a consequence of this extensive use, the accumulated amount of PET waste in the environment is rapidly increasing, causing harm to the natural ecosystem. Worldwide measures are being taken to reduce the use of virgin PET. For instance, in accordance with European Union regulations, all plastic packaging waste must be reused and recycled by the year 2030. Additionally, the South Korean government has prohibited the use of plastic materials involving challenging recycling processes, such as polyvinyl chloride (PVC) and colored PET (Neo, 2020). The PET Recycling Council in Japan recommends the use of only pure colorless PET for beverage bottles (Yamamoto & Eva, 2022). These examples illustrate that serious international steps are being taken in the field of PET recycling. Furthermore, a search using the keyword "PET recycling" on Web of Science reveals a significant increasing trend in academic studies on PET recycling between 2016 and 2022.

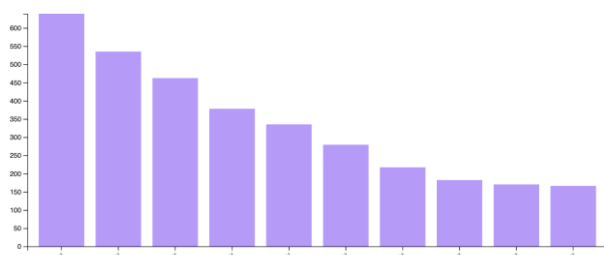


Figure1. The evolution of publications on PET recycling between the years 2016-2022.

PET recycling methods can be categorized into three main groups: (i) incineration for energy generation, (ii) chemical recycling, and (iii) mechanical recycling (Figure 2). Burning plastic waste in appropriate facilities is one of the thermal conversion methods that converts waste into heat energy. However, this process leads to a significant amount of greenhouse gas emissions, posing environmental issues, and is not considered an effective recycling method (Gurgul et al., 2018). In chemical recycling, PET polymers are converted into monomers using chemical methods such as hydrolysis, methanolysis, or glycolysis. These monomers are then polymerized again to obtain PET. However, chemical recycling processes are not yet fully developed, requiring implementation and optimization by personnel with specialized training. Additionally, due to the use of specific catalysts, this method is relatively expensive (Grigore, 2017; Thiounn & Smith, 2020). Furthermore, chemical recycling demands substantial amounts

of chemical resources during the recovery process, posing a risk of causing other environmental problems. For all these reasons, the chemical recycling rate for PET is around 4% (Raheem et al., 2019; Takenaka et al., 2017).

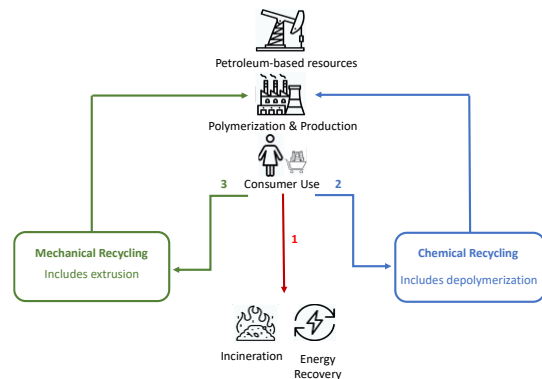


Figure 2. PET Recycling Mechanisms.

In mechanical recycling, PET is melted in an extruder or molding machine and then reshaped. Mechanical recycling is highly advantageous in terms of industry applicability since it utilizes the same infrastructure as virgin PET production. Additionally, personnel implementing this method do not require extra knowledge or experience, making it a cost-effective approach compared to chemical recycling methods. In mechanical recycling, PET waste is first collected, and colored and colorless materials are separated. Subsequently, plastics are ground into flakes, and they are washed using 2% NaOH or tetrachloroethylene (Al-Sabagh et al., 2016). Impurities, including PVC and other polymers, are separated using density-based flotation methods to obtain pure PET flakes and avoid any impurities in recycled PET (rPET) (Wang et al., 2020). Next, PET flakes are dried to minimize water content and prevent molecular weight reduction through hydrolysis (S. Muthu, 2020). Subsequently, these dried PET flakes can be recycled into various rPET-based materials such as bottles, threads, or food packaging materials through production systems.

However, the reprocessing of PET in a molten state can lead to several types of degradation, including chemical, mechanical, thermal, and oxidative, which result in a reduction in the molecular weight, viscosity, and processability. Additionally, the physical and mechanical properties of PET show deterioration as a consequence of mechanical recycling. The thermal stability of PET during processing is crucial for obtaining desirable properties in the final product. The degradation of PET has been extensively researched, with numerous studies focusing on the thermal degradation products of PET. These studies suggest that the thermal degradation of PET is initiated by the chain scission of the ester-linkage, yielding carboxyl and vinyl ester groups (Kang et al., 2011; Karayannidis & Psalida, 2000). In a study conducted by Wu et al. (2019), virgin PET (vPET) and recycled PET (rPET) were processed in a twin-screw extruder, and the resulting changes in thermal and mechanical properties were examined. According to their results, with an increasing number of recycling cycles, both the molecular weight and viscosity of vPET and rPET polymers exhibit a significant decrease. The degradation of PET during recycling processes poses challenges, as the material's properties can be negatively affected by the recycling process itself.

2. Chain Extension of PET

Chain extension reactions emerge as a promising option to counteract the degradation challenges encountered during the recycling process of PET. In the recycling journey of PET, especially during extrusion or molding stages, exposure to elevated temperatures above its melting point induces thermal and oxidative degradation reactions. These reactions

adversely affect the molecular weight, viscosity, and overall processability of PET, leading to a decline in its ultimate properties. To mitigate these adverse effects and enhance the recycled PET's characteristics, the incorporation of chain extension reactions becomes significant. By introducing various functional groups such as oxazolines, isocyanates, anhydrides, epoxides, and innovative agents like Meldrum's acid derivatives, it becomes possible to reconnect and strengthen the broken PET chains. This process not only prevents the degradation of PET but also increases its viscosity and molecular weight, contributing to the overall improvement of recycled PET properties and promoting a more sustainable approach to plastic waste management.

2.1 Oxazolines

Oxazolines are functional materials with low toxicity, and due to their reactivity, they can be used as chain extenders in PET recycling. The oxazoline ring, under extrusion conditions of 250–280 °C, can undergo ring-opening reactions with the carboxylic acid chain ends of PET (Figure 3), connecting two PET chains and thereby increasing molecular weight and viscosity (Karayannidis & Psalida, 2000; Liu & Xu, 2013).

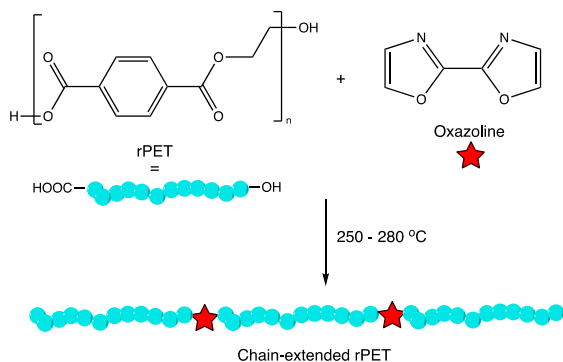


Figure 3. Chain extension of r-PET in the presence of oxazolines.

In a recent study, Berg et al. (2019) utilized the 1,3-phenyl-bisoxazoline structure as a chain extender in PET recycling. They observed an increase in molecular weight and viscosity of PET during melt recycling in the presence of 1,3-phenyl-bisoxazoline. Despite the promising positive results obtained, the number of studies using oxazolines as chain extenders is limited, possibly due to the relatively high cost of oxazolines. Additionally, only the structure of poly(2-ethyl-2-oxazoline) has been approved by the FDA for use in food packaging.

2.2. Isocyanates

Isocyanate is a functional group that exhibits high reactivity due to its strong electrophilic nature in reactions with various compounds such as alcohols, amines, and water (Figure 4) (Li et al., 2020). However, chain extenders containing isocyanate cannot be used for materials in contact with food, as isocyanates can pose health issues such as carcinogenicity, asthma, and other pulmonary problems (Mehta, 1990). Despite the limited use of isocyanate-containing chain extenders in materials in contact with food due to potential health concerns, studies in the literature show that isocyanate-based agents effectively enhance the melt properties and viscosity of rPET (Tang et al., 2007; Torres et al., 2001; Zhang et al., 2010). In a study conducted by Torres et al. (2001), the effects of chain extenders containing isocyanate, oxazoline, and epoxy structures on rPET were investigated. Among these functional groups, isocyanate showed the most significant increase in the molecular weight and viscosity of rPET. This could be attributed to the highly reactive isocyanate group effectively reacting with both hydroxyl (-OH) and carboxyl (-COOH) groups of PET chains.

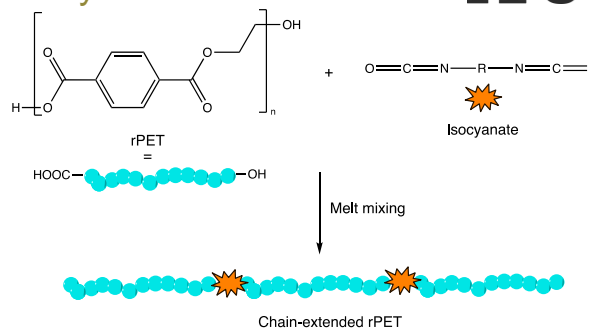


Figure 4. Chain extension of r-PET in the presence of isocyanates.

2.3 Anhydrides

Anhydrides, due to their functionality, tend to react with the carboxyl groups at the chain ends of PET, making them suitable for improving the performance of rPET. Among anhydrides, pyromellitic dianhydride (PMDA) is one of the most investigated anhydride-based chain extender agents in PET mechanical recycling. As seen in Figure 5, using PMDA in PET recycling allows the formation of 4-branched structures (Arayesh et al., 2020). However, as research has revealed the superior effectiveness of chain extender agents containing epoxy over anhydrides, studies in reactive chain extender agents have predominantly focused on structures containing epoxy functional groups. For example, Arayesh et al. (2020) reported that the crystallinity of rPET modified with PMDA is 26.85% lower than that of rPET modified with an epoxy-containing chain extender agent. Similar results were also obtained by Yang and colleagues (Yang et al., 2018).

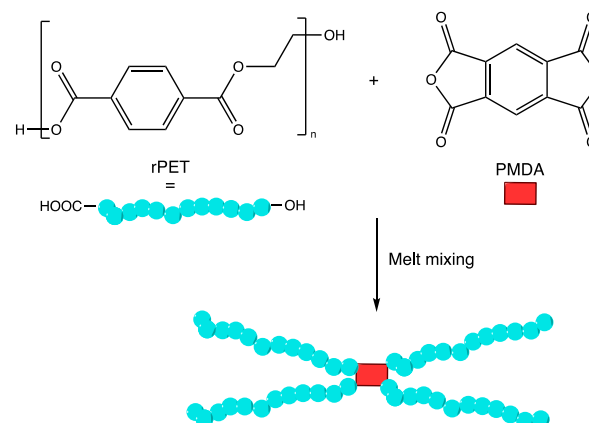


Figure 5. Chain extension and branching of r-PET in the presence of PMDA.

2.4. Epoxides

Epoxy, widely used in various applications such as coatings and adhesives, owing to its toughness, chemical resistance, and hardness, features an oxirane ring that readily undergoes ring-opening reactions with several other functional groups (May, 2018). Consequently, numerous studies have explored the use of epoxy-based materials as chain extender agents. The ring-opening reaction between epoxy and PET primarily follows two pathways. The first involves the reaction between the oxirane ring and the carboxyl groups of the PET chain. The second pathway involves the interaction between the oxirane ring and the hydroxyl groups of the PET chain. Incorporating chain extender agents with epoxy functional groups in reactive extrusion offers a promising avenue to enhance the properties of recycled PET (rPET) through these chain extension mechanisms.

Among these chain extender agents, epoxy-containing oligomers known under the commercial name Joncryl® ADR (Figure 6) are widely used to enhance the melt properties of PET. In a pioneering study, various recycled polymeric structures were modified with Joncryl 4368 chain extender,

revealing the broad process window of this epoxy-based chain extender agent (Villalobos et al., 2006). Another study conducted by Raffa et al. (2012) added Joncryl 4368 at a mass ratio of 1.5% during the extrusion stage, resulting in a significant increase in the melt viscosity values of rPET.

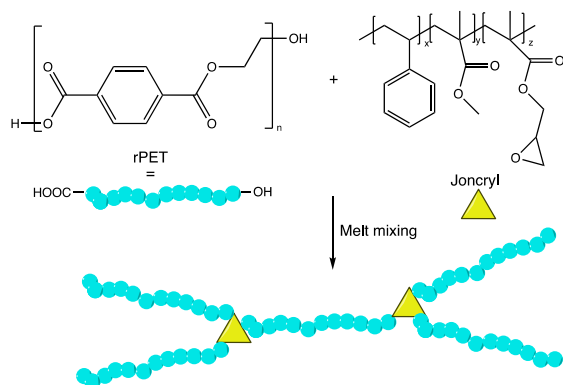


Figure 6. Chain extension and branching of r-PET in the presence of Joncryl.

In a recent study by Härth et al. (2021) PMDA and Joncryl 4368 were added to PET, and the molecular structure and rheological properties of the obtained PET were examined. While PET structures modified with PMDA exhibited linear characteristics, Joncryl-modified PET formed a highly branched network structure. Moreover, in a recent investigation carried out by our research group, the incorporation of Joncryl 4468 into rPET at mass ratios of 0.2% and 0.4% yielded a network structure leading to significant enhancements in the viscosity of rPET (Guclu et al., 2022).

2.5 Meldrum's Acid Derivatives

The incorporation of Meldrum's acid derivatives as chain extenders in PET recycling represents a promising strategy, particularly leveraging highly reactive ketene intermediates. These intermediates can be generated in situ through the thermolysis of Meldrum's acids (Figure 7) (Alkan Goksu et al., 2019). In the absence of nucleophiles, these ketenes undergo [2+2] cycloaddition reactions to form cyclobutane-1,3-dione rings. However, in the presence of nucleophiles, ketenes readily engage in reactions, yielding the corresponding esters or amines (de Lucas et al., 2001; Leibfarth et al., 2010a). Considering that PET possesses both carboxylic and hydroxyl terminal groups as nucleophiles, the in situ-formed ketenes can effectively react with them without undergoing dimerization. The emphasis on ketenes as a target functional group is substantiated by its well-established history in organic chemistry and its versatile reactivity towards a majority of nucleophiles. Despite being discovered by Staudinger (1905) and extensively employed in sophisticated small-molecule syntheses, the full potential of ketenes as a versatile functional group in polymer and materials science has remained largely unexplored even after more than a century (Tidwell, 2006). Pioneering studies in polymer science by Hawker and Leibfarth have demonstrated that thermally generated ketenes derived from Meldrum's acid derivatives serve as valuable precursors for diverse macromolecular architectures (Burke et al., 2012; Jung et al., 2013; Leibfarth et al., 2010a, 2010b, 2012; Leibfarth & Hawker, 2013).

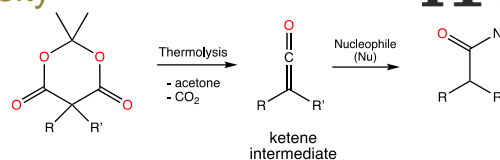


Figure 7. Formation of ketene intermediate and its subsequent reaction with nucleophiles

As environmental concerns and regulatory structures progress, the exploration of inventive solutions, like chain extenders containing Meldrum's acid, has the potential to help alleviate the environmental consequences of polymer waste. For this purpose, in a study conducted by Alkan Goksu, (2024) 2,2,5-trimethyl-5-(4-vinylbenzyl)-1,3-dioxane-4,6-dione (St-MA) was synthesized (Figure 8) and its homopolymers were produced through conventional free radical polymerization.

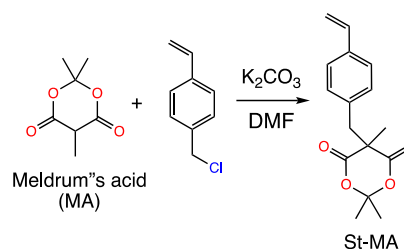


Figure 8. Synthesis of Meldrum's acid bearing monomer (St-MA).

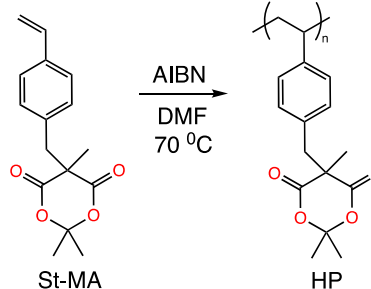


Figure 9. Synthesis of Meldrum's acid bearing polymers (HP).

Following the characterization of the Meldrum's acid-based chain extender (HP) and the thermal behavior assessment of recycled PET (rPET) and modified rPET* (rPET*), the impact of the ketene-based reaction between rPET and HP was examined. A blend of pre-mixed rPET and HP with weight percentages of 0.5 wt% and 1.5 wt% was introduced into a micro compounder. The compounder operated at a temperature of 280 °C and a rotating speed of 100 rpm for 10 minutes. Figure 10 depicts force-versus-time graphs obtained during the compounding process. The noticeable increase in force values during extrusion is often associated with a substantial enhancement in mechanical strength and resistance, signifying structural modifications in the polymer. This increasing strength demonstrates the material's ability to withstand and adapt to the mechanical strains and shear pressures inherent in the extrusion process. Ketene formation and its rapid reaction with the chain ends of rPET are evident from the observed force value increase within 30 seconds of introducing rPET and HP into the compounder. Despite fluctuations in the force value over 600 seconds, no significant alterations occur, indicating the rapid formation and reaction of all ketenes. Furthermore, the introduction of 0.5 wt% of HP led to a more pronounced increase in force values compared to the addition of 1.5 wt%. This phenomenon could be attributed to the rapid reaction of ketenes with the terminal groups of rPET. A higher level of HP might have caused a substantial increase in viscosity, potentially hindering subsequent reactions. Continued research in the use of Meldrum's acid precursors in PET recycling not only has the potential to

deepen our fundamental understanding of the chain extension chemistry behind but also to mold the future of sustainable polymer engineering.

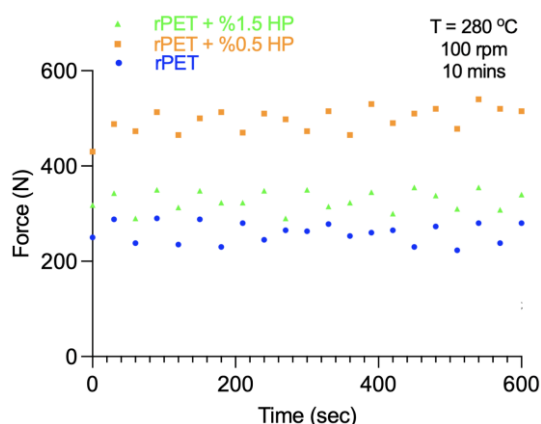


Figure 10. Graphs depicting the relationship between time and force for rPET, rPET + 0.5 HP, and rPET + 1.5 HP.

3. Conclusion

Ultimately, the increasing worldwide manufacturing and use of petroleum-derived polymers provide a significant challenge, as the amount of plastic waste is estimated to reach 460 million tons per year by 2030. In the middle of this ecological disaster, poly(ethylene terephthalate) (PET) has become a commonly used material that poses serious threats to the environment. As a result, there is an immediate need for effective and prompt recycling methods. The significance of PET recycling is emphasized by global initiatives, such as the strict laws enforced by the EU and the unique material restrictions in countries like South Korea and Japan. Mechanical recycling, known for its suitability in industrial settings, is a prominent method for recycling PET. However, it has difficulties due to the degradation of PET throughout the reprocessing process. The continuous investigation of chain extenders, such as oxazolines, isocyanates, anhydrides, epoxides, and the promising Meldrum's acid derivatives, provides a viable solution to tackle the issues associated with PET degradation. These novel compounds demonstrate substantial potential in improving the thermal and mechanical properties of PET throughout the recycling process.

In the future, the significance of PET recycling is expected to increase as regulatory frameworks become stricter and worldwide consciousness of plastic pollution grows stronger. The combination of worldwide efforts, inventive methods for recycling materials, and enhanced techniques for strengthening polymer chains signifies a future in polymer recycling that is more sustainable and environmentally conscious. In order to address the environmental consequences of PET waste and convert it into a valuable asset for future generations, it is essential to maintain ongoing collaboration, conduct research, and make technological progress in the pursuit of a circular economy. The combination of worldwide awareness, advancements in technology, and the adoption of sustainable methods has the potential to transform PET recycling into a fundamental aspect of environmental responsibility, guaranteeing a resilient and environmentally friendly future.

Acknowledgements

This study is dedicated to the memory and immortal legacy of Prof. Yusuf Yagci, whose leadership and contributions to the field of polymer chemistry have served as an inspiration to many scientists.

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