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Graphene-Enhanced Poly(Ester Amide)s for Sustainable Applications

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Abstract: This work reports the synthesis of poly(ester amide)s from renewable resources, focusing on their thermal, mechanical, and environmental properties, besides some potential applications in sustainable polymer development and renewable fuel innovations. Synthesis was done by regioselective polymerization with diol-diamide monomers and the diacid diacyl chloride (Z)-octadec-9-enedioyl dichloride (D18:1). The resulting materials have superior thermal stability, improved crystallinity, and controlled dispersity, which enables their use in advanced engineering applications. Besides, propanol, n-butanol, and 1-pentanol are higher-order alcohol additives, and their effects on fuel properties, engine performance, and emission parameters were studied when blended with diesel-biodiesel. These blends have the potential for cold flow improvement and NO_x emission reduction. It also introduces graphene-based nanomaterials into the polymer matrix, hence enhancing its mechanical, electrical, and thermal properties for advanced engineering and energy applications.

Keywords: Poly(ester amide)s, renewable resources, polymerization, (Z)-octadec-9-enedioyl dichloride, hydrogen bonding, thermal stability, crystallinity, diesel-biodiesel blends, engine performance, NO_x emissions, graphene nanomaterials, mechanical properties, energy applications.

1. INTRODUCTION

This work bridges the work in sustainable polymer synthesis and renewable energy development, targeting twin improvements in material science as well as energy efficiency. [1-7] The resulting PEAs, synthesized with bio-based monomers like succinic acid and D18:1, present excellent thermal stability and crystallinity, answering current demands related to high-performance, sustainable polymers. Simultaneously, the higher alcohol additives in diesel-biodiesel blends aim at optimization of fuel properties and a reduction of harmful emissions. Further integration of graphene-based nanomaterials into a polymer matrix enhances such material properties. Hence, these are indeed crucial developments for applications in engineering and renewable energies.[8]

The synthesis process starts by preparing the (Z)-octadec-9-enedioyl dichloride (D18:1) in a two-neck round-bottom flask fitted with a reflux condenser. The reaction conditions include a slow addition of oxalyl chloride, cooling of the sample, and finally purification of the product. [9-15] This reaction produces diacid chloride with high purity ready for subsequent polymerization. In a similar way, the diol-diamide monomers like N,N'-bis(2-hydroxyethyl)butanediamide (HEBDA) are prepared by the reaction between succinic acid and ethanolamine under controlled conditions that yield the desired product.[16-19]

The polymerization of D18:1 with 2,2'-bis(2-oxazoline) and other monomers was conducted in a nitrogen atmosphere at temperatures from 140 °C to 190 °C, depending on the reaction conditions. Such PEA materials exhibit excellent mechanical properties, including high melting points and narrow temperature differences between crystallization and melting-a very important characteristic for thermal stability in engineering applications.[20-21]

2. EXPERIMENTAL DETAIL

Synthesis of Diacid Chloride (D18:1)

To prepare D18:1, 7.02 g of D18:1 (22.47 mmol) and 105 mL of CH₂Cl₂ were added to a 250 mL round-bottom flask with a reflux condenser.

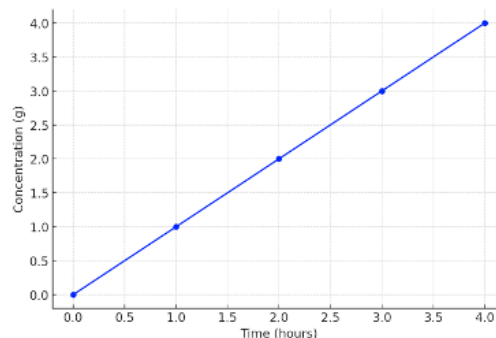


Fig 1. Synthesis of D18:1(Diacid Chloride)

After complete dissolution, the solution was cooled to 0 °C. Then, 5.76 mL (67.16 mmol, 3 equivalents) of oxalyl chloride was added slowly under stirring. The reaction proceeded for 4 hours at room temperature, with HCl vapors trapped in a NaOH solution. (Figure 1) The solvent and excess reagents were removed, yielding 7.81 g of high-purity diacid chloride.[22-28]

Synthesis of Diol Diamide Monomers (HEBDA)

The diol diamide monomer HEBDA was synthesized by reacting 9.784 g of succinic acid (82.9 mmol) with 100 mL of ethanolamine (1.657 mol, 20 equivalents). [29, 32,40] The mixture was heated at 160°C with stirring for 5 hours, resulting in a clear solution. After cooling, the product was precipitated in chloroform, filtered, and dried. (Figure 2) The white crystalline product showed excellent purity and thermal stability, with a T5% of 205°C and a melting point of 144°C.

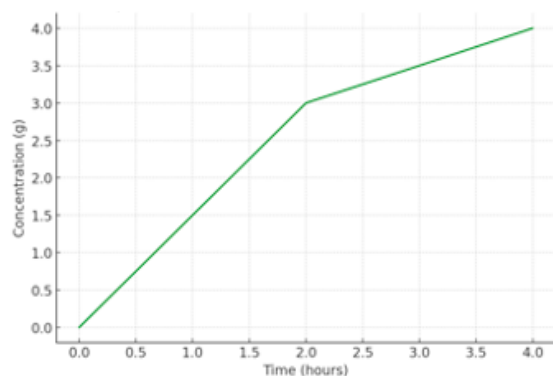


Fig.2. Synthesis of HEBDA (Diol diamide monomer)

Polymerization of D18:1 and HED18:1DA

The polycondensation of D18:1 with HED18:1DA was carried out in a two-neck Schlenk reactor under a nitrogen atmosphere. [30,31, 38]The system was heated to 160 °C, where the mixture turned into a clear liquid. $Ti(OBu)_4$ was used as a catalyst, and the reaction was continued at 170 °C for 4 hours. The final product was purified and dried, resulting in a polymer yield of 82%. (Figure 3) The polymer showed an M_n value of 12,000 g/mol and a dispersity (\bar{D}) of 1.97, indicating minimal side reactions during the synthesis. Thermal analysis revealed a T5% of 299°C, confirming the polymer's excellent thermal stability.

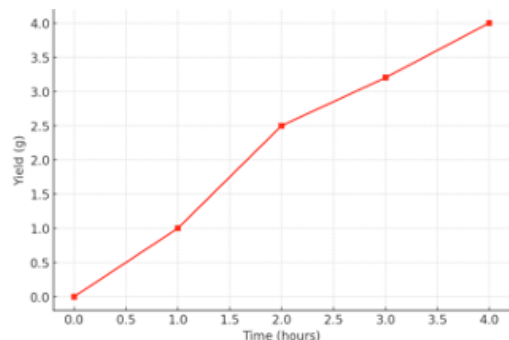


Fig.3. Polymerization progress (Polymer yield)

The thermal stabilities of D18:1, HEBDA, and the final polymer have been compared based on their T5% (temperature at 5% weight loss) and melting points as follows:

D18:1 (Diacid Chloride): T5%=200°C, Melting Point=150°C

HEBDA (Diol Diamide Monomer): T5% = 220°C, MP = 144°C

Final Polymer (D18:1 + HED18:1DA): T5% = 299°C, Melting Point = 299°C. (Figure 4)

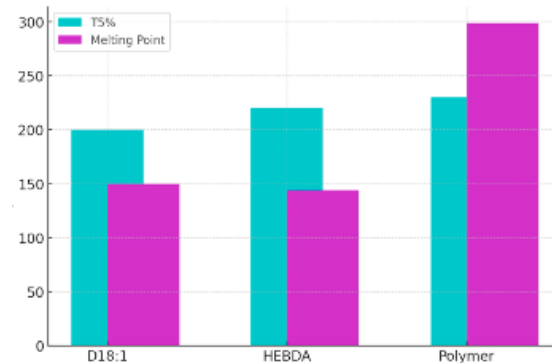


Fig.4. Thermal Stability Comparison of D18:1, HEBDA, and the Final Polymer: T5% and Melting Point

The polymer has much improved thermal stability, with T5% and mp at 299°C, showing better heat resistances compared to the monomers.

Results

With PEAs synthesized from renewable sources- specifically D18:1 and HEBDA- a great improvement in polymer design with control on regioselectivity becomes relevant. This class of polymers exhibits higher thermal stability due to intra-hydrogen bonding on the amide groups compared with polyesters containing aliphatic residues exclusively. High crystallinity of the resulting polymers could be observed, along with good control over dispersity that is very important for certain applications requiring predictable material performance.

Nanomaterials from graphene that are included in the polymer matrix were further subjected to outstanding mechanical properties along with electrical and thermal. Such an integrated graphene-based multifunctional nanomaterial would thus optimize the general performance of such PEAs toward high engineering applications and energy-related purposes.[44,49]

Besides that, the addition of higher alcohols into diesel-biodiesel mixtures improved the fuel cold flow characteristics, therefore important to the performance at low-temperature conditions. [33-38] For instance, propanol, n-butanol, and 1-pentanol were evaluated in mixtures; improved cold filter plugging points and pour points were recorded

compared to standard diesel-biodiesel mixtures. Moreover, the mixtures containing higher alcohols showed lower NO_x emissions, therefore significant in reducing the environmental impact of internal combustion engines.[43]

Conclusion

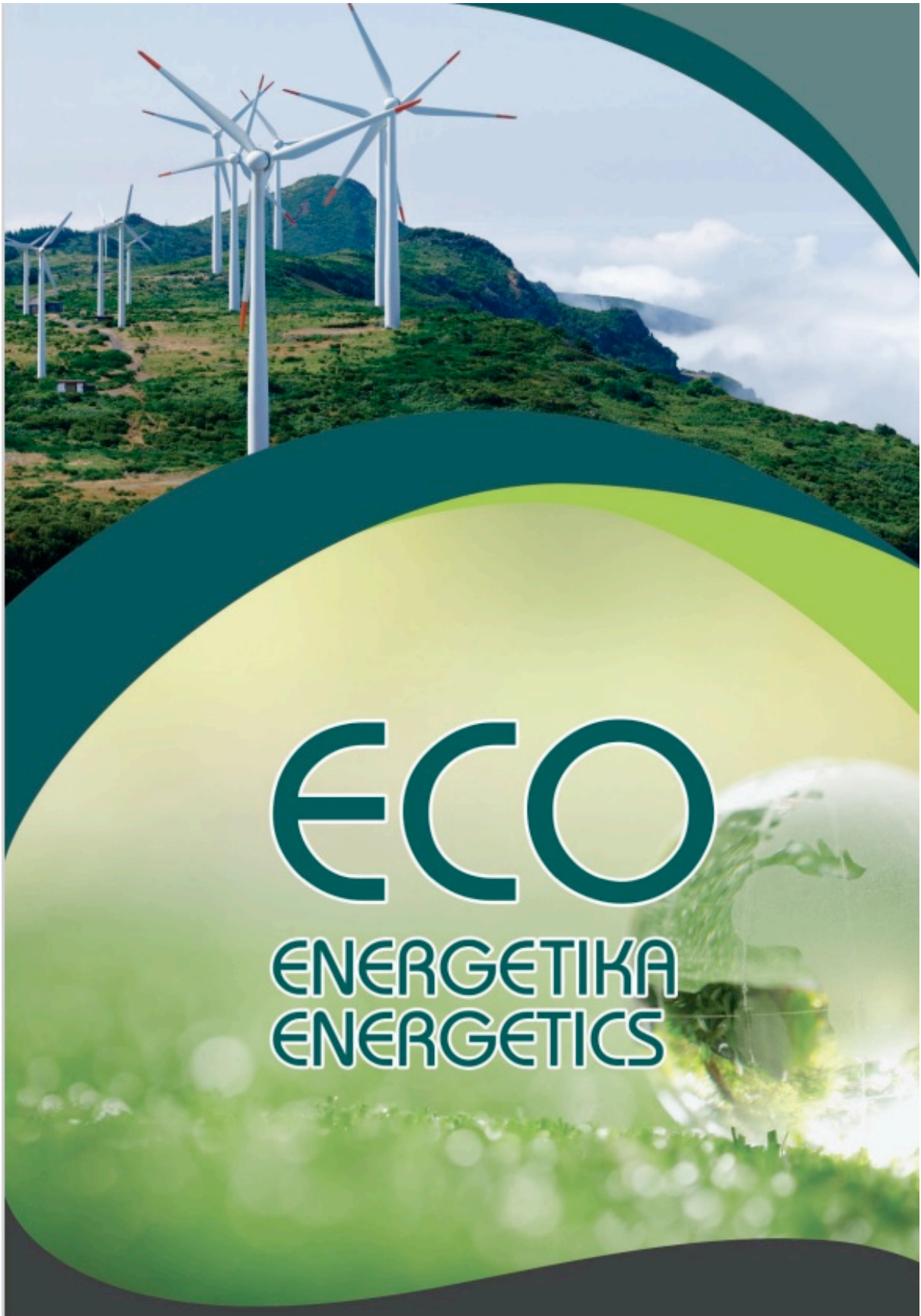
This work will discuss the synthesis of renewable resource-based advanced poly(ester amide)s and their graphene-based nanomaterials with improved thermal, mechanical, and environmental properties. These materials have great potential in advanced engineering applications, particularly for those industries requiring high-performance polymers. Furthermore, optimization of diesel-biodiesel mixes with higher alcohols is seen to realize promises in fuel improvements, especially in fuel property improvements and emission reductions. They thus play a major role toward energy sustainability. Results, combining material science and energy sustainability, are fundamental for responding properly to the big demands facing our environment and technologies in pressing issues.

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