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INFLUENCE OF AMIDE AND ESTER LINKAGES ON THE WATER RESISTANCE OF TEREPHTHALIC ACID–DERIVED POLY(ESTER-AMIDE) COPOLYMERS

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ABSTRACT

The present study describes the simple fabrication and characterization of a poly(ester-amide) (PEA) copolymer based on terephthalic acid (TA), ethylene glycol (EG), and ethylene diamine (EDA) using the gel combustion technique. The resulting product is hereafter referred to as the TA-EG-EDA copolymer. Fourier Transform Infrared Spectroscopy (FTIR) confirms the presence of ester and amide groups in the copolymer backbone. The morphology of the copolymer is examined utilizing Scanning Electron Microscopy (SEM). The diffraction pattern and crystalline nature of the copolymer are analyzed by X-ray Diffraction (XRD). The λ max value is determined using UV–visible spectroscopy. Furthermore, the water resistance of the copolymer is evaluated in media of different pH values using the weight loss method. The results indicate that the TA-EG-EDA copolymer has potential applications in biomedical and industrial fields. Beyond representing a simple and cost-effective preparation approach, this study contributes to the improvement of functional poly(ester-amide) ingredients by providing insights into the association between chemical structure, stability, and material presentation. These outcomes provide a foundation for future research aimed at modifying PEA copolymers for progressive biomedical devices, defending coatings, and sustainable engineering uses, thereby improving both the scientific understanding and practical consumption of multifunctional polymeric constituents.

Keywords: Terephthalic Acid, Ethylene Glycol, Ethylene Diamine, Water Resistance, Copolymer, Gel Combustion.
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INTRODUCTION

In recent years, the commercial applications and utilization of copolymeric materials have increased in all fields. The wide variety of fibrous materials have been commercially produced, such as polyester, PET bottles, PET film, poultry vessels, dyes, perfumes, medicines, pesticides, drugs, transfer glass, and non-fibrous materials.[1] The development of many materials focused on TA. The main growth of copolymeric materials in the market is because of the utility of copolymers in all industrial and medical fields. The combination of TA and EG has widely provided the saturated polyester in the form of semicrystalline nature.[2] In recent years, recycling of PET bottles is the most focused way to produce valuable by-products and numerous applications in industrial and commercial materials. A lot of research work is going on the degradation of PET waste converted into effective catalysts.[3] These activities have been enhanced for their significance and commercial applications to protect the environment from pollution.[4] TA (1, 4-benzenedicarboxylic acid), also called aliphatic diols, acts as a co-monomer. It is a kind of dicarboxylic

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acid, which is widely utilized for the fabrication of copolymeric polyester. These TA-based polyester materials exhibit various properties like greater toughness, optical clarity and heat resistance.[5] PET-based waste bottles act as organic ligands, which showed exceptional reusability and magnetic detachable properties. The α-Fe/Fe_3C based copolymer exhibits enormous potential for antibiotic-dirtied wastewater intensified applications.[6] Similarly, the fabrication of TA-reformed chitosan–magnetic (Cs–Tp@Fe_3O_4) copolymer utilized for elimination of acid blue 25 dye (AB-25) was carried out in industrial wastewater via batch process.[7] EG (Ethylene glycol) is a dicarboxylic acid, which enhances the flexibility of the polymeric chain or network.[8] It is also used for the fabrication of polyester and hydrogels. It has shown excellent thermal stability. EG and graphene oxide (GO) solutions combined with polypyrrole (PPy) were used to make supercapacitor material. The developed EG-RGO/PPy electrode exhibits greater specific capacitance, excellent performance, and charge–discharge stability properties.[9] Cai and Kim also discuss the thermally stable bacterial cellulose (BC)/poly(ethylene glycol) (PEG) copolymer that has been fabricated by the freeze-drying process. It exhibits good biocompatibility characteristics, which have been utilized for medical applications such as wound healing and tissue engineering materials.[10] EDA (ethylenediamine) acts as a co-monomer and crosslinking agent. It is widely used for the fabrication of fibres and dendrimers. Hyper-branched polymers can also be prepared.[11] Yuan, L *et al*. also deal with the synthesis of self-healing polymeric copolymeric materials with EDA and epoxy resin. It has a focus for microcapsules encapsulating nature.[12] The development of poly(ethylene-*co*-acrylic acid) copolymer from cellulose nanoparticles with ethylene diamine to aminodeoxy cellulose nanoparticles. It has been used for various medical and biomedical applications due to an excellent biocompatibility property.[13]

In general, copolymeric materials were prepared using different techniques, which have been used for wide applications.[14] Based on the availability, characteristics and nature of surface morphology of developed copolymers. However, functional modifications are a significant factor to get a better quality copolymer.[15] Many polymerisation techniques such as conventional, bevelled, micro filled, hybrid filled and nano filled with modification of process were utilised for the fabrication of copolymer.

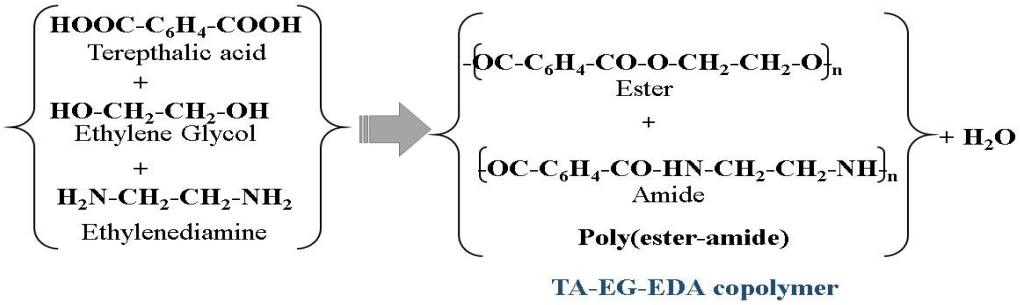
However, there are some limitations for producing suitable copolymeric materials, such as long preparation duration, difficult to separate the compound, usage of toxic solvents and toxic chemicals and a multi-step process. To overcome such setbacks, we need to reduce the above factors and suggest this gel combustion process. It is a simple, convenient and easy to develop of copolymeric material. Water resistance behaviour plays a significant role in the copolymeric substances' sustainability. Neelambaram et al. also reported increased water-resistance properties in siloxane-based solid acrylic latex coating polymer materials.[16] Park *et al.* demonstrated that the synthesised poly(methyl methacrylate) copolymers depicts decrease moisture absorption and enhanced heat resistance properties.[17] Fabrication of a copolyelectrolyte composite for greater water-resistant and flexible humidity sensor applications.[18] Likewise, Jin *et al.* examined the development of latex films from acrylate and poly(dimethylsiloxane) monomers for effective water-barrier applications.[19]

Hence, prepared copolymeric materials may be used for advanced water-resistant material applications. These kinds of materials need further attention and research focus. The objective of the present study is to synthesize TA-EG-EDA copolymer using diol (EG) with TA and EDA by a simple gel combustion technique. Furthermore, the structure of the TA-EG-EDA copolymer was confirmed with the help of the FTIR spectrum. The nature of the copolymer was analyzed by XRD. The surface morphology of the copolymer was observed using SEM, and the UV-Vis spectroscopy was utilized for the observation of broadband, which confirmed the formation of chain compounds. The water resistance behaviour was also observed in different pH media. The schematic representation of the formation of the TA-EG-EDA copolymer reaction (Scheme-1).

EXPERIMENTAL

Materials

Terephthalic acid (99.5% pure), Ethylene glycol (99.7% pure) and Ethylene diamine (99.5% pure) were procured from Sigma-Aldrich (Bangalore), India. Ethanol and double-distilled water were utilized throughout the preparation of solutions. NaOH and HCl chemicals were utilized for the measurement of water resistance analysis.



Scheme-1: Represents the Development of the TA-EG-EDA Copolymer

Synthesis of TA-EG-EDA Copolymer

A facile gel combustion technique was used for the fabrication of terephthalic acid (TA) based copolymers. Firstly, the adequate quantity of 1g of TA (0.60mol), 037g of EG (0.60mol) and 0.36g of EDA (0.60mol) was weighed and mixed in the beaker. Approximately 1.80 moles of total concentration was fixed during the preparation. An adequate amount of ethanol and deionised water was mixed to attain a clear solution, and then it was heated on a hot plate at 80 °C. The resultant product was further heated at 320 °C using the same hot plate, and the obtained gel substance was subjected to the gel combustion reaction. The combustion process was used to collect powder. A muffle furnace was used for the calcination process at 400 °C for 2 hr. The unreacted monomers were degraded after the above process. The sample code of the calcined product is TA-EG-EDA copolymer, washed and used for further studies. The possible schematic diagram is depicted in Fig.-1.

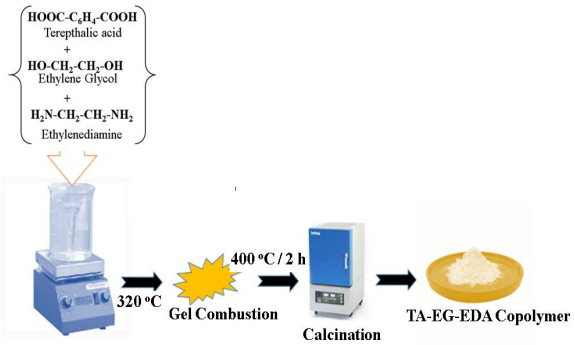


Fig.-1: Schematic Representation of Synthesised TA-EG-EDA Copolymer

Water Resistance Analysis

Water resistance analysis was conducted at three different temperatures, viz., 50 °C, 70 °C, and 90 °C. Three pH environments were nominated, such as neutral (pH ≈ 7), acidic (pH 2–4), and basic (pH 9–12).

Characterization

The resultant TA-EG-EDA copolymer was characterised as follows. FTIR spectra was done by a FT/IR300e (JASCO) spectrophotometer within the range 400- 4000 cm-1 (PerkinElmer 1600 series II spectrometer). UV-Vis spectrophotometer was used to measure the ƛ max value. Exterior morphology was observed by scanning electron microscope (U. K) (Leica Cambridge 440). The diffraction pattern was explored through the XRD method (Rigaku (Mini Flex Model, Japan). Water resistance analysis was done by a digital pH meter (Model SKY-468, Scispectrum, India).

RESULTS AND DISCUSSION

FTIR Analysis

The FT-IR spectrum of pure TA-EG-EDA copolymer is shown in Fig.-2. The stretching frequency of NH groups at 3099 cm^{-1} corresponds to the appearance of the CO-NH frequency of PEA copolymer. The C-H stretching frequencies of EG and EDA have been depicted as significant peaks at 2839 and 2679 $cm^{-1,}$ respectively. The peak appearing at 1740 cm^{-1} represents the COO- stretching (ester functional) group.[20]

The peaks appeared at 1573 and 1510 $cm^{-1,}$ which support the development of N-H bending of the amide group. The polymeric chain is present at 1429 cm^{-1}, depicted for CH_2 bending frequency of the chain compound. The peak observed at 1296 cm^{-1} relates to the C-O-C stretching frequency of the ester group. The peaks were depicted at 1138 and 1112 $cm^{-1,}$ which relate to -C-O-C stretching frequencies. Similarly, significant peaks were observed at 879 and 781 cm^{-1} resemble to Ar-C-H in plane and out-of-plane stretching.[21] The peak recorded at 732 cm^{-1}, which matches the combination of EG and EDA with the TA polymer matrix. This peak shows the appearance of CH_2 stretching within the TA-EG-EDA copolymer, which is due to the process of condensation or transesterification. The formation of the TA-EG-EDA copolymer matrix is well supported by the acquired FTIR data.

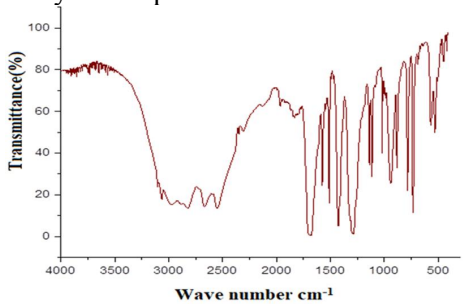


Fig.-2: FTIR Spectrum of Synthesised TA-EG-EDA Copolymer

XRD Analysis

The X-ray diffraction pattern of the TA-EG-EDA copolymer has been depicted in Fig.-3. The amorphous nature of the TA-EG-EDA copolymer has been noticed in the XRD analysis. More sharp and significant peaks have been exhibited, which indicate the nature of the copolymer. The TA-EG-EDA copolymer has been depicted with peaks at 2θ = 17.28°, 25.12 °, 27.84 °, 29.6 °, 32.54 °, 33.28 °, 35.04 °, 39. 54 ° and 41.02 ° respectively. A similar kind of discussion has been made in poly(ethylene terephthalate) (PET) copolymer.[22] Thus, the developed sharp peaks have been well supported by the TA-EG-EDA copolymer with the help of XRD study.

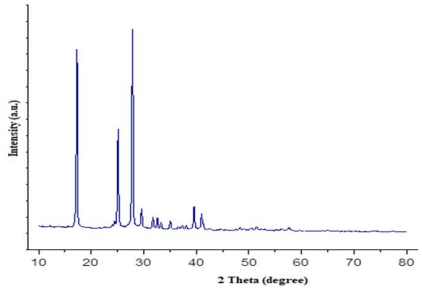


Fig.-3: XRD Pattern of Synthesised TA-EG-EDA Copolymer

UV-Visible Spectra

The recorded broad absorbance confirms the formation of a chain compound. Figure-4 depicts the UV-Visible spectrum of the synthesised TA-EG-EDA copolymer. The transition occurs in different vibrational levels, which is due to the large number of possible vibrational energies at each electronic energy level. This leads to a broad absorption peak. The synthesised TA-EG-EDA copolymer absorbs at higher energy and lower wavelengths such as 313nm, 318nm, 210nm, and 315nm. The interaction of molecules with high energy is more at shorter path lengths, which increases the absorbance.[23]

SEM Analysis

The morphology of the TA-EG-EDA copolymer has been displayed in Fig.-5 (i-ii). The appearance of rough surface morphology indicates the nature of the copolymer. The observation of porous and rough surfaces of the TA-EG-EDA copolymer may influence the swelling properties. In general, hydrophobic-based copolymers exhibit a lower swelling equilibrium ratio than hydrophilic copolymeric materials.[24] Based on the surface morphology and swelling studies, the copolymer may be used in different applications.

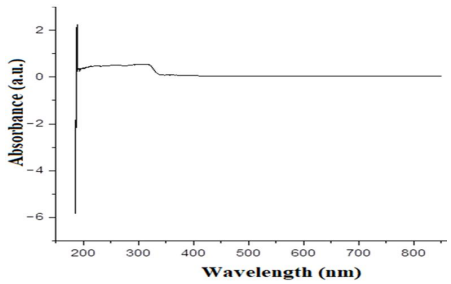


Fig.-4: UV-Visible Spectrum of Synthesised TA-EG-EDA Copolymer

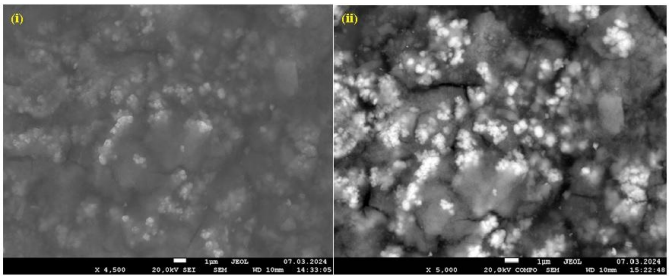


Fig.-5: SEM Picture of Synthesised TA-EG-EDA Copolymer

Water Resistance Analysis

The TA-EG-EDA copolymer samples were assessed after one week of exposure, and detailed observations are as follows:

Neutral Medium

In the neutral medium, the hydrolysis rate was perceived to be slow. This behaviour is mainly attributed to imperfect amide–water interactions, which slightly affect the swelling behaviour. Ester hydrolysis was negligible at temperatures below 100 °C. After one week, weight gain values of 0.1–0.2% at 50 °C, 0.3–0.5% at 70 °C, and 0.5–1% at 90 °C were noted. The slight increase in weight may be connected with water absorption eased by amide groups (–CONH–). Despite this, the ester groups remained relatively stable, and a marginal enhancement in mechanical strength was detected.

Acidic Medium

Similarly, the moderate hydrolysis was recorded in this case, which is due to the cleavage of amide bonds being greater than ester bonds at all temperature ranges. At 50°C slight surface whitening, at 70°C nearly 1–2% mass loss, and 3–5% of mass loss occur at 90°C respectively. The mechanical strength and surface change may vary after a few weeks.

Basic Medium

A significantly greater rate of hydrolysis was recorded in the basic medium due to the strong nucleophilic attack on ester bonds, leading to degradation of polyester sections. Weight loss values of 1–3% at 50 °C, 4–7% at 70 °C, and 10–15% at 90 °C were noticed. The substantial weight loss is ascribed to ester bond cleavage and polymer chain scission. Based on these interpretations, ester bonds reveal good stability in neutral and mildly acidic conditions, whereas amide bonds prove relatively better stability in basic environments. Generally, the material displays the highest resistance in neutral medium, moderate resistance in acidic medium, and the lowest resistance in basic medium. These kinds of polymeric materials may be widely used in industrial applications.

CONCLUSION

In the present work, TA-EG-EDA copolymer was effectively synthesised from terephthalic acid (TA), ethylene glycol (EG) and ethylene diamine (EDA) utilizing gel combustion method. The structure of TA-EG-EDA copolymer was confirmed by FTIR studies. The amorphous nature of the copolymer was observed using XRD analysis. Further, SEM analysis confirmed the surface morphology of the porous structure. The high-energy-level molecular interaction was examined using UV-visible spectroscopy.

Further, more resistance was observed in neutral medium, moderate resistance was noticed in acidic medium and in the basic medium, very low resistance occurred. The study contributes to the development of cost-effective and scalable synthesis strategies for poly(ester-amide) materials and provides a foundation for their potential application in biomedical and industrial fields. Future research should focus on optimising the copolymer composition to enhance its mechanical strength, chemical stability, and environmental durability. In addition, comprehensive investigations of its thermal behaviour, biodegradability, biocompatibility, and application-specific performance are recommended to validate its suitability for advanced biomedical devices, protective coatings, and other high-performance engineering applications.

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CONFLICT OF INTERESTS

The authors declare that there is no conflict of interest.

AUTHOR CONTRIBUTIONS

The present work is related to *SDG-9: Industry, Innovation, and Infrastructure*. All the authors contributed significantly to this manuscript, participated in reviewing/editing and approved the final draft for publication. The research profile of the authors can be verified from their ORCID ids, given below:

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