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Synthesis and characterization poly (5-indanyl methacrylate-co-ethyl methacrylate)

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ABSTRACT

The copolymer (5-IndanylMethacrylate-co-Ethylmethacrylate) is synthesised by the reaction of free radical Solution polymerisation at 70°C using Benzoyl Peroxide. The copolymer is characterised by NMR and IR Spectroscopy. The solubility of the polymers was tested in various polar and non-polar solvents. The Thermal Properties of the copolymer has been determined by means of Differential Scanning Calorimetry Analysis (DSC) and Thermogravimetric analysis (TGA). Thus, the thermal stability of copolymer increases with a decrease of Ethylmethacrylate content in the copolymer.

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INTRODUCTION

The Methacrylates Polymers and its derivatives are very important materials, and they have wide application to prepare the materials with highly improved properties. The advantage of methacrylate polymers and copolymers are highly thermal stability and mechanical stability. The copolymer of activated methacrylates has been used to synthesis the electroactive polymers, Macromolecular drug carries and Polymeric reagents for peptide synthesis. Aromatic acrylates and methacrylates are highly reactive monomers because of the aromatic ring, and they form an interesting class of polymers. The advantage of copolymers especially designed with functional active groups as a lateral substituent of the main chain is a topic of increasing activity and interest in polymer science.

Copolymerization is the most successful method adopted for the preparation of materials with tailor-made properties (Vijayaraghavan *et al.,* 1996; Soykan *et al.,* 2000; Vijayanand *et al.,* 2002; Balasubramanian *et al.,* 1996; Pitchumani *et al.,* 1982; Bankova *et al.,* 1983). The introduction of ester or amide linkages in the polymer backbone renders the resulting the aim of our research to study the synthesised and characterisation of Copolymer of poly (5-Indanyl Methacrylate (5-IMA)-co-Ethylmethacrylate). The Copolymer was characterised by Fourier transform infrared (FT-IR), Nuclear Magnetic Resonance Spectroscopic Techniques (^{1}H-NMR) spectroscopy. (Senthilnathan *et al.,* 2016) Analysis of the thermal properties of the Poly (5-Indanyl Methacrylate –co- EthylMethacrylate) by Thermogravimetric analysis (TGA) and Differential Scanning Calorimetry Analysis (DSC) are also reported.

MATERIALS AND METHODS

Ethyl methacrylate (Aldrich), 5-indanol (Aldrich) was used as received. Benzoyl peroxide was recrystallised from methanol at 0-10°C. Benzene and diethyl ether (AR) and Methanol of LR grades were used without further treatment. Methacryloyl chloride was prepared by distilling a mixture of acrylic acid and benzoyl chloride. (Senthilnathan *et al.,* 2016)

Synthesis of poly (5-indanylmethacrylate–co-ethylmethacrylate)

Required quantities of the monomer (5-Indanyl Methacrylate) and Ethyl methacrylate along with BPO, are dissolved in 25 ml of Benzene placed in a standard reaction tube to obtain a homogeneous solution. The mixtures are flushed with oxygen-free dry Nitrogen gas. The inlet and outlet of the reaction tube were closed by means of rubber tubing and pinch cork. The reaction vessel is then immersed in a thermostatic water bath maintained at 70 ± 1°C. The copolymerization reaction was allowed to proceed for an appropriate duration. Then the solution was poured in ice-cold excess hexane to precipitate the copolymer. The copolymers were purified by repeated precipitation by hexane from solution in chloroform. It was then dried in a vacuum oven at 45°C for 24 h. (senthilnathan *et al.,* 2016). The structure of the monomeric units of the poly (5-IMA-co-EMA) is shown in Scheme 1.

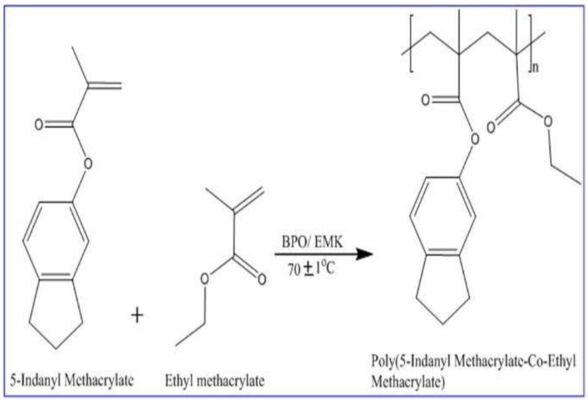

Figure 1: Synthesis of poly (5-IMA-Co-EMA)

Characterisation Of Poly (5-IMA-co-EMA)

The characterisation of a copolymer of Poly (5-IMA-co-EMA) by two methods, such as spectral analysis, thermal analysis**.**

RESULTS AND DISCUSSION

Solubility test

The solubility of the copolymers was tested in various polar and non-polar solvents. About 5–10 mg of the copolymer is added to about 2 ml of different solvents in a test tube and kept overnight with the test tube tightly closed. The solubility of the copolymers was noted after 24 h.

FT-IR spectrum of the poly (5-IMA-Co-EMA)

The FT-IR spectrum of the poly(5-IMA-Co-EMA) is shown Figure 4. The ring stretching absorption often occurs in pairs at 1589.03 cm^{-1}. The main evidence for the formation of the copolymer is an appearance of broad ester carbonyl group C=O stretching frequency of the copolymer at 1738.73cm^{-1}. The C-O stretching frequency of ester group appears at 1147.2cm^{-1}. The alkanes =C-H stretching frequency is disappeared at 3057.16cm^{-1}confirms the copolymer formation.

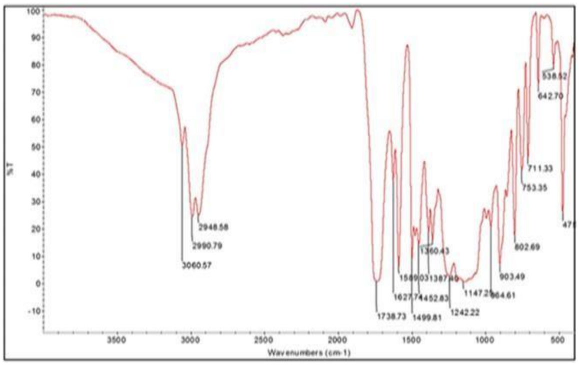

Figure 2: FT-IR spectrum of poly (IMA-Co-EMA)

^{1}H-NMR spectrum of the Poly (5-IMA-Co-EMA)

The ^{1}H-NMR spectrum of the Poly (5-IMA-Co-EMA) is shown in Figure 5. The signals at δ 6.96-7.26ppm are of aromatic protons. The CH_3 proton of EMA group appears at δ 1.33ppm. The peak at δ1.19ppm (3H) and δ1.25ppm (3H) corresponds to -CH_3 proton of5-Indaylmethacrylate and ethyl methacrylate groups. The signal at δ 3.24-3.56ppm is due to –CH_2 proton of EMA group and δ1.64ppm (6H) corresponding to the proton of the 2-methyl group between two phenyl rings. The main evidence for the formation of the copolymer (5-IMA-Co-EMA) is the disappearance of vinyl protons peaks at δ5.7ppm(2H) and appearance of methylene groups in copolymer backbone at δ2.06ppm.

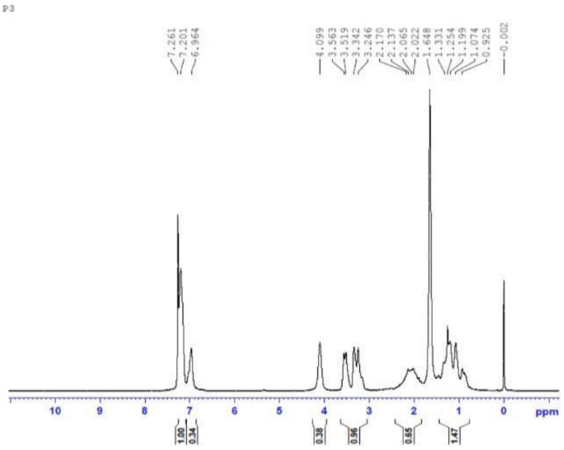

Figure 3: 1H NMR spectrum of POLY (5-IMA-co-EMA)

Thermogravimetric analysis

Thermogravimetric analysis is used to estimate the percent weight loss of the copolymer which undergoes decomposition. The initial decomposition temperature (IDT) increases with the decrease of EMA content in the copolymer shown in Table: 1.

Table 1: Thermal stability of developed Copolymers

Polymer	Stable up to temperature (°c)
5-IMA: EMA (20:80) PBI 1	160
5-IMA: EMA (50:50) PBI 2	194
5-IMA: EMA (80:20) PBI 3	242

Table 2:

Solvent	Solubility
Dimethyl Sulfoxide	Partially soluble
Chloroform	Completely soluble
Acetone	Completely soluble
Toluene	Completely soluble
Benzene	Completely soluble

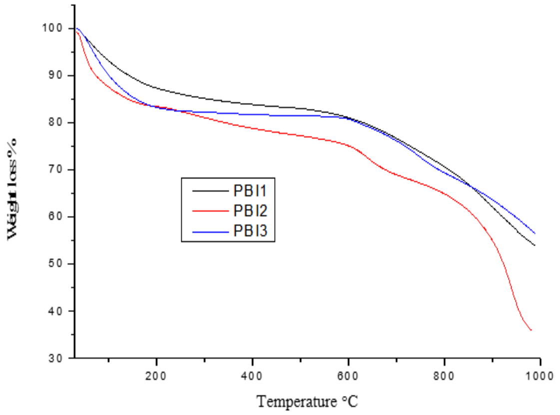

Figure 4: Differential scanning calorimetry analysis (DSC)

The DSC Spectrum of poly 5-IMA-CO-EMA is shown figure 6. DSC a standard tool for measuring the melting and freezing points of polymers. The 'Tg' value for copoly (5-IMA-Co-EMA) is 84°C. Actually, by the incorporation of 5-IMA unit in the copolymer, there is a visible increase in the 'Tg' value.

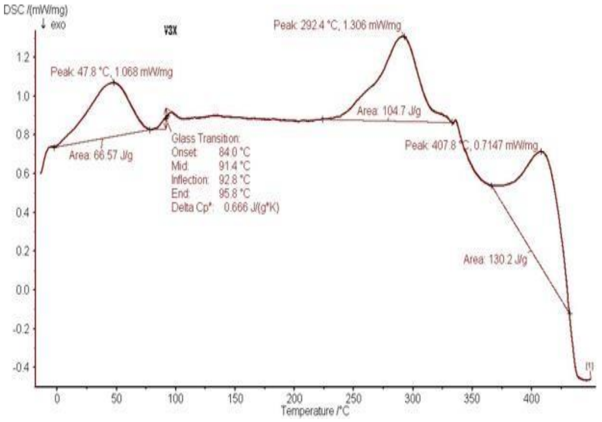

Figure 5: DSC of poly (5-IMA-co-EMA)

Solubility test

The solubility of copolymers is carried out by various solvents of different polarity. The polymers were easily soluble in various solvents, namely toluene, benzene, chloroform and acetone.

CONCLUSION

The copolymers of (5-Indanyl Methacrylate-Co-EthylMethacrylate) having different compositions were synthesised by free radical solution polymerisation mechanism. The Characterizations of the copolymer were performed by FT-IR, ^{1}H-NMR, spectroscopic techniques. The copolymers are soluble in acetone, toluene and benzene. The thermal stability of the Copolymers increases with the decrease of EMA content in the copolymer. The glass transition temperature of the copolymer increases with EMA content.

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