Research Article

Synthesis of 5-Indanyl Methacrylate Using Methacryloyl Chloride and Its Characterisation Studies.

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ABSTRACT

A monomer of 5-indanyl Methacrylate (5-IMA), has been synthesized from the precursor viz., 5-indanol and characterized by Fourier transform infrared (FT-IR), Nuclear Magnetic Resonance Spectroscopic Techniques ^{1}H-NMR and ^{13}C-NMR.

Keywords: 5-indanyl Methacrylate,5-Indanol, synthesis, NMR, FTIR.

INTRODUCTION

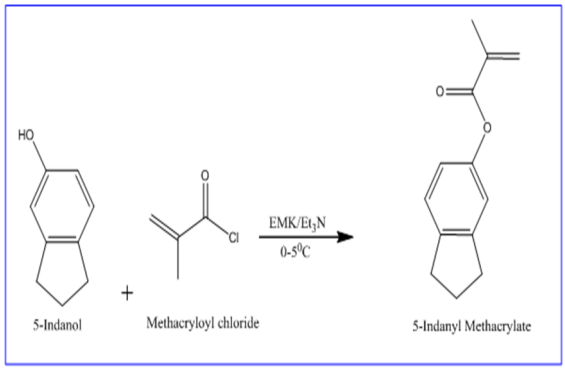
Methacrylates based polymers are a type of important materials and wide applications drive efforts to prepare materials with highly improved properties. The advantage of methacrylate based polymers is its high thermal, chemical and mechanical stability, which makes them best candidates for applications that require adhesion to various substrates, abrasion resistance, flexibility, toughness and excellent resistance to chemicals, solvents, and water. The degradation temperature of such Polymers could have high temperature as 500^{o}C. Incorporation of activated acrylates or methacrylate into polymers provides one of the most versatile routes for the preparation of reactive polymers. For example, copolymers of activated (meth) acrylates have been utilized to synthesize potentially electroactive polymers [I], macromolecular drug carriers[2] and polymeric reagents for peptide synthesis[3]. Epoxy group has a unique reaction capability. They undergo ring opening when reacting with substances possess hydroxyl, amine or activated methylene groups. For these reasons, polymers with epoxide groups offer numerous functionalization possibilities in mild reaction conditions[4]. Addition reactions of alcohols, phenols, carboxylic acids and amines to pendant epoxide groups in poly(GMA) and its copolymers are well known[5,6]. The epoxide opening reaction with nucleophiles is generally performed with acidic or basic catalysis and in the absence of such catalysts, the reaction is moderately slow[7,8]. Methacrylate monomers consisting of an alkyl group, an acrylate ester group, and a functional carboxyl group can react with a wide range of monomers and functionalized molecules providing flexible polymer chains. Alkyl methacrylates are clear and volatile liquids that are slightly soluble in water and highly soluble in alcohols, ethers, and organic solvents (Wright, 1981; Braden; Parker). Aromatic acrylates and methacrylate are highly reactive monomers due to the presence of the aromatic ring and thus form an interesting class of polymers. In continuation of our effort to synthesize new polymeric materials for industrial application, we have synthesized and characterized a new monomer, 5-Indanyl Methacrylate (5-IMA), which is further is characterised by using IR, ^{1}H-NMR, and ^{13}C-NMR.

MATERIALS AND METHODS

Experimental Section

5-indanol (Aldrich) was used as received. Benzoyl peroxide was recrystallized from methanol at 0-5°C. Benzene and Ethyl Methyl Ketone (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.

Monomer Synthesis



Scheme 1: Synthesis of 5-Indanyl Methacrylate

5-Indanol (27 g, 0.2 mol) dissolved in Ethyl methyl ketone was placed along with trimethylamine(31 ml,0.22 mol) in a two-necked 500 ml flask. With continuous stirring of the reaction mixture at 0-5°C, the freshly distilled reagent Methacryloyl chloride (23 ml, 0.28 mol) was added slowly in drops from the addition funnel. After completion of addition, the contents were washed with water to

remove the quaternary ammonium salt formed and the un reacted 5-indanol was then removed by washing with 5% sodium hydroxide solution. The filtrate was then dried with anhydrous sodium sulphate and the monomer 5-indanyl methacrylate was recovered (33 g, 88% yield) after EthylmethylKetone evaporation. The reaction scheme for the synthesis of 5-Indanyl Methacrylate is shown in **Scheme 1**.

RESULTS AND DISCUSSION

Characterization of 5-Indanyl Methacrylate

FT-IR Spectrum of the 5-Indanyl Methacrylate

The FT-IR spectrum of 5-IMA is shown in Fig.1. The C-H absorption of asymmetric and symmetric stretching vibrations are appeared at 2955.13 cm^{-1}. The =C-H out-of-plane bending in the range 1037.73-648.59 cm^{-1}. The Peak due to –CH bending and –C=C- vinyl stretching appeared at 1292.85 and 1609.58cm^{-1}. The ring stretching vibration often occurs at 1484.24 cm^{-1}. The main evidence of the monomer is the appearance of ester carbonyl group C=O stretching frequency at 1735.25 cm^{-1}.

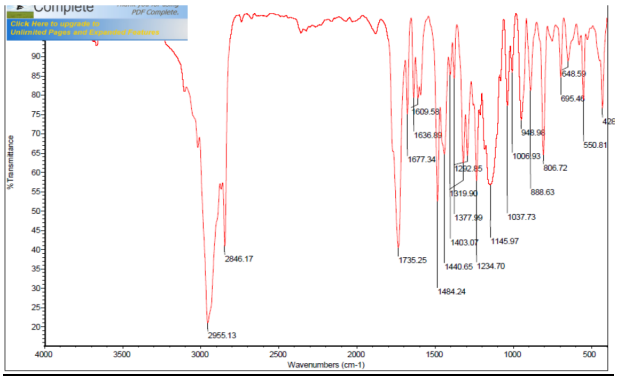


Figure 1: FT-IR spectrum of 5-Indanyl Methacrylate

^{1}H-NMR Spectrum Of the 5-Indanyl Methacrylate

The ^{1}H-NMR spectrum of the 5-IMA is shown in Fig. 2. The signals at 7.127 to 7.148 ppm for aromatic protons and δ5.66ppm (2H) for olefinic protons of the methacryloxy group. The α-methyl group protons are observed at δ 2.009 ppm. The methylene proton were observed at 6.18-6.43 ppm.

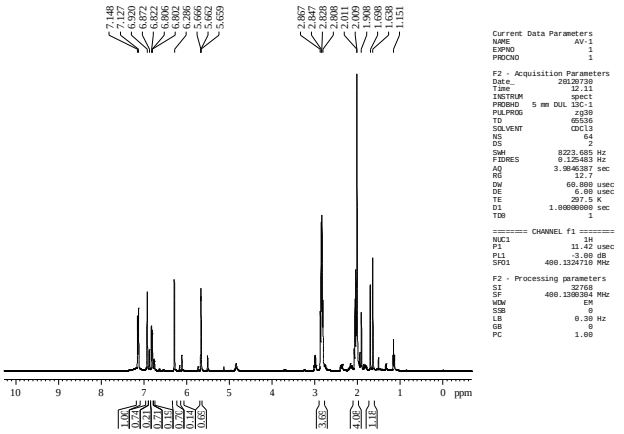


Figure 2: ^{1}H NMR Spectrum of 5-Indanyl Methacrylate

^{13}C-NMR Spectrum of the 5-Indanyl Methacrylate

The ^{13}C-NMR spectrum of the5-IMA is shown in Fig. 3. The signal at 18.03 ppm is due to the presence of α-CH_3 carbon of methacryloxy unit. The signals at 122.94 to 148.7ppm for aromatic ring carbons and 128.0 ppm for olefinic carbon peak (=CH_2) of the methacryloxy group. The ester carbonyl carbon is appeared at 166.0ppm. The peak at17.97ppm shows the presence of alpha methyl carbon.

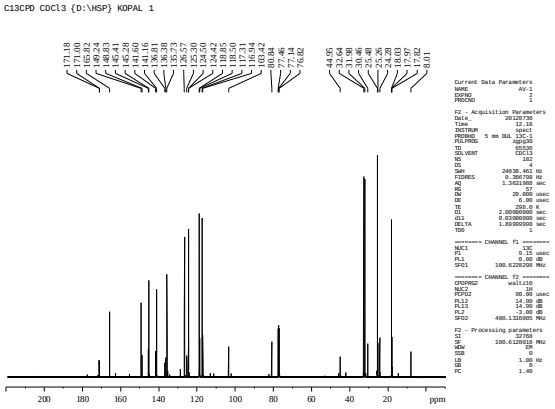


Figure 3: ^{13}C NMR Spectrum of 5-Indanyl Methacrylate

CONCLUSION

An attempt has been made to synthesize the 5-IndanylMethacrylate and its Characterizations were performed by FT-IR, ^{1}H NMR and ^{13}C-NMR spectroscopic techniques. The Copolymerization of different composition is in Progress.

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