Synthesis And Characterization Of Terpolymer Derived From Salicylic Acid, Furfural And Catechol

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ABSTRACT

Terpolymer (SAFC) was synthesized by the polycondensation technique by using Salicylic acid, furfural and catechol monomers in 3:6:3 molar ratio using 2M HCl as catalyst and refluxing for 5h at 120^0C. The Terpolymer compositions have been determined on the basis of their elemental analysis. The Terpolymer have been further characterized by absorption spectra in non-aqueous medium, infrared (FTIR) spectra and nuclear magnetic resonance (^{1}H NMR) and ^{13}C NMR spectral studies. The physico-chemical and spectral methods were used to elucidate the structures of SAFCTerpolymer. The morphology of the Terpolymer was studied by scanning electron microscopy (SEM) showing semi crystalline nature of the Terpolymer.

Keywords: Synthesis, Polycondensation, Morphology, SEM, Semi crystalline

1. Introduction

The present age may be termed as an “Age of Polymer”. This is so because it assumed a foremost and revolutionary position in the modern technological human race. Polymer material at this time became a part of everyday life. In fact, our body is made up of lots of polymers, e.g. Proteins, enzymes, etc. Polymers were recognized as compounds with several repeating units connected to each other extending over a long range. The word polymer comes from Greek word “Poly” means many and “Mers” means parts of units. A large chain molecule which composed of large number of repeating units of monomer is known as polymer. The term polymer was first coined by J.J. Berzelius in 1833. The first-time synthetic polymer produced was Bakelite in 1909 and followed by Rayon which was developed in 1911. Plastics are the well-known example of polymers.

Terpolymer obtained from the copolymerization of two monomer species. Terpolymer can be characterized by a variety of technique such as Uv-Visible Spectroscopy, FTIR Spectroscopy, SEM Technique. Terpolymer are used for making a variety of materials with in Industrial, pharmaceutical and everyday use. The physical properties of copolymers are largely influenced by molecular weight and chemical structure. [1-3]. Some specific factors that affect copolymer properties include the length of the copolymer chain, the presence of any branching chain and presence of any cross linkages between chains and a crystalline or amorphous structure. [4-6]. Metal toxicity or metal poisoning is the toxic effect of certain metals in certain forms and doses on life. Some metals are toxic when they form poisonous soluble compounds. Certain metals have no biological role i.e. are not essential minerals or are toxic when in certain form. [7-9]. In case of lead, any measurable amount may have negative healtheffects. [10-12].

2. Materials and Method

Salicylic acid (Centre Scientific AR grade), Furfural (AR grade) and Catechol were purified by recrystallization method.Salicylic acid, Furfural and Catechol Copolymer Resin has been prepared by polycondensation technique in 3:6:3 mole ratio by using HCL as a catalyst refluxing on oil bath120^{o}c for 5 hours [13]. After the reaction time was over, the resultant mixture was cooled and filtered, repeatedly washed with warm water to remove the unreacted monomers and recrystallized from ethanol. The gel permeation chromatography (GPC) was used to determine the average molecular weight of the copolymer [14-15]. The sample has been purified and dried finely crushed and sieved to obtained uniform particles of 50-70 mesh and stored in polythene bottle. The resin has been then screened characterized using different instrumental analysis technique and was used for the entire experiment during the search period. Testing of solubility of resin in a different solvent were performed at room temperature and pressure.

Reaction of Terpolymer

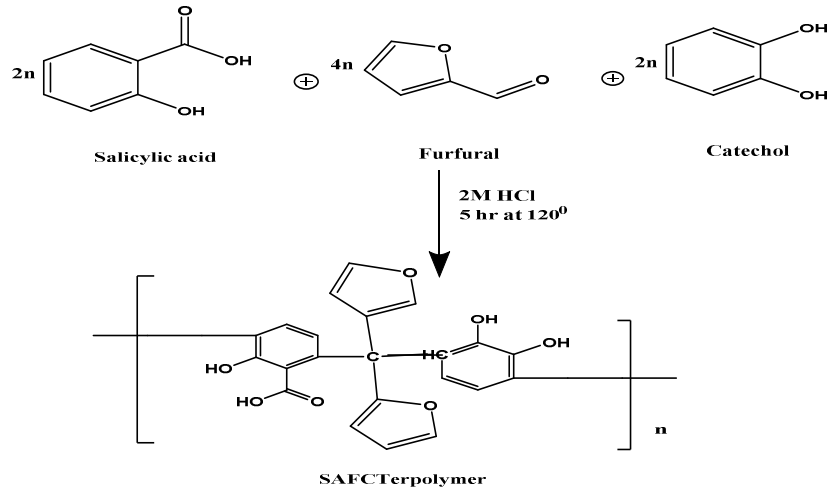

Figure 1: - Synthesis of SAFC Terpolymer

Properties of Terpolymer

The synthesized Terpolymer was in solid form and their particle size was 0.30-0.50mm. The SAFC Terpolymer was black in color and having semi-crystalline nature. The solubility test of Copolymer in different solvents was performed at room temperature and pressure.Importantly, the crystalline behaviors of conjugated polymers and their packing structures in thin films can be controlled using the terpolymer approach, which would be particularly important for enhancing the electron mobility of the polymer acceptor and producing all-PSCs with a high performance [16-17]. The incorporation of selenophile into terpolymers could significantly improve their intermolecular interactions and induce highly ordered polymer assembly due to better orbital overlap between the polymers resulting from the preferred formation of quinoidal structures of selenophiles rather than the formation of aromatic structures All polymers were synthesized on a laboratory scale and were analyzed using different characterization and property analysis methods. The investigation endeavors to reveal the influence of the monomer on the crystal structure as well as the effect of thermal

treatment. Also, the structural differences are correlated with macroscopic properties of terpolymers and copolymers, which determine their eventual commercial applications.

SAFC terpolymer was tested in various solvents.

Sr. No.	SAFC Copolymer	Solubility Behavior
1.	Tetrahydrofuran (THF)	Soluble
2.	Dimethylsulfoxamide (DMS)	Soluble
3.	Dimethylformamide (DMF)	Soluble
4.	Conc.H2SO4	Soluble
5.	Conc. HCL	Soluble
6.	Sodium hydroxide	Insoluble
7.	Toluene	Insoluble
8.	Chloroform	Insoluble

Table1. Solubility behavior of SAFC Terpolymer

3. Characterization

Fourier transform infrared (FTIR) spectroscopy is a form of vibrational spectroscopy that is useful in the study of a variety of soil chemical processes. In the mid-infrared (mid-IR) range, vibrations arise from many environmentally important molecules such as organic acids, soil organic matter, mineral phases, and oxyanions. It is possible to utilize FTIR spectroscopy as a quantitative analytical method and also as a tool to determine bonding mechanisms in solids and on surfaces. Molecular vibrations can be related directly to the symmetry of molecules, and so it is often possible to determine precisely how a molecule is bonding on surfaces or as a component in a solid phase from its infrared spectrum. Many experimental methods exist for probing samples of various states and at different spectral regions.
FTIR Spectral Characterization of copolymer is exhibited in fig 2. The band appearing at 3350cm.The medium band at 2900 cm is due to the C-H Stretching of the methylene group. The strong band at 1652cm may be assigned to c=o Stretching vibration of carbonyl group present in the copolymer. The weak band at 1372 cm is due to the C-O-H bending of the aromatic -OH group.

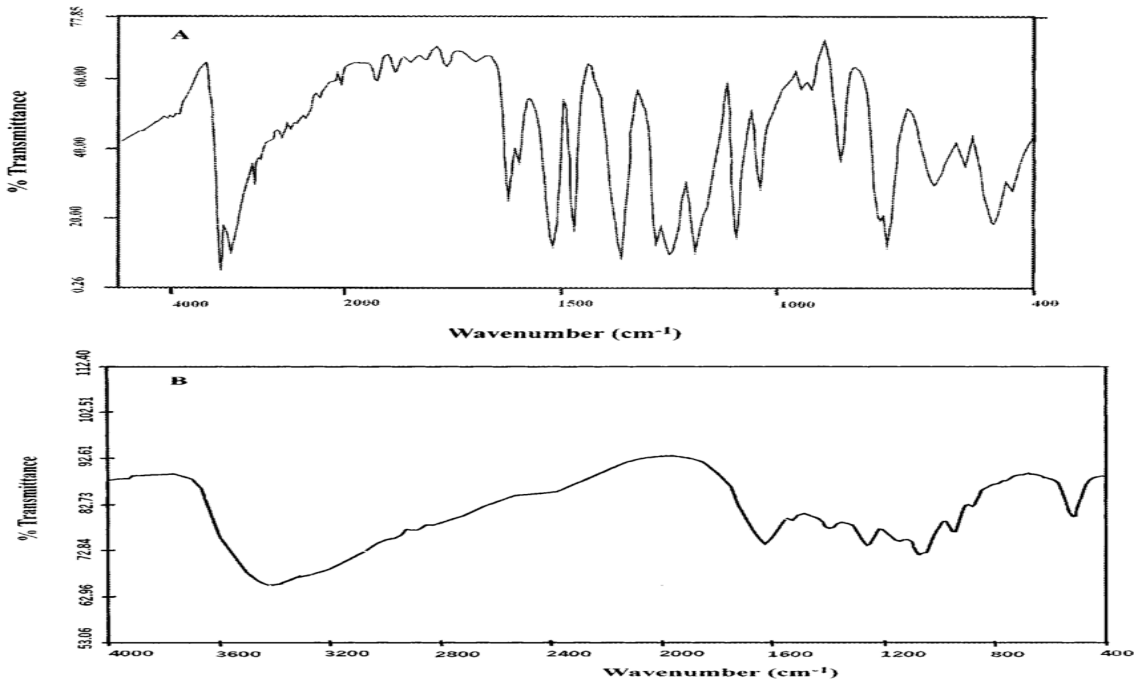

Figure 2: - IR Spectra of SAFC Terpolymer

Observed Frequency	Assignments
3440	-OH Bonded (phenolic)
2930,2820	C-H Stretches in aldehyde
1680	C=O Stretches (ester)
1650	C=O Band (an aldehyde)
1601	Aromatic ring
1480	CH2 bending
1420	-OH bending
1230	-OH bending (phenol)
1170	The O-C-C band stretches

Table 2 FT-IR spectrum data of SAFC Terpolymer

UV-Visible Spectroscopy refers to absorption spectroscopy in part of the ultra violet and the adjacent visible region of electromagnetic spectrum. The only requirement is that the sample absorb in the UV -Visible region i.e. be a chromophore. UV-Vis spectroscopy is an analytical technique that measures the number of discrete wavelengths of UV or visible light that are absorbed by or transmitted through a sample in comparison to a reference or blank sample. This property is influenced by the sample composition, potentially providing information on

what is in the sample and at what concentration. Since this spectroscopy technique relies on the use of light, Light has a certain amount of energy which is inversely proportional to its wavelength. Thus, shorter wavelengths of light carry more energy and longer wavelengths carry less energy. A specific amount of energy is needed to promote electrons in a substance to a higher energy state which we can detect as absorption.certain wavelengths of light suited to the sample type and analyte for detection must be selected for sample examination from the broad wavelengths emitted by the light source.

UV- Visible Spectral Characterization of Copolymer exhibited the maximum absorption position for furfural were at 276.78nm. Salicylic acid shows maximum absorbance at 301.2 nm.

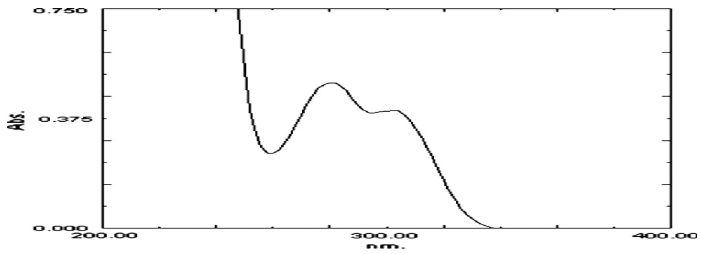

Figure 3: - UV – Visible Spectra of Terpolymer

Scanning electron microscopy (**SEM**) is the best known and most widely used of the surface analytical technique. High resolution images of surface topography, with excellent depth of field are produced using a highly focused, scanning electron beam.SEManalysis has been found to be of great use in understanding the surface features of the materials, imaging to surface feature of 10-10times magnification the white bar at the bottom of the SEM microscopy represents the scale. The SEM photographs exhibits irregular shape spheres and spherulites showing crystalline nature. The corrugations and having shallow pits on the surface show amorphous nature of Copolymer. Thus, SEM micrographs of salicylic acid and catechol Copolymer indicate the presence of transition structures between crystalline and amorphous. The crystalline nature shows semiconducting property.

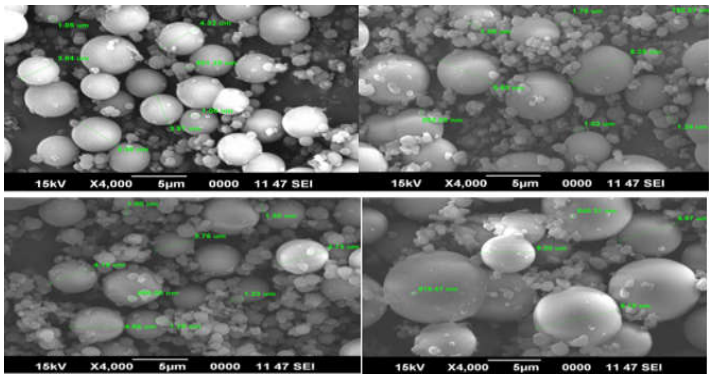

Figure 4: - Image of SAFC Terpolymer by SEM

Nuclear magnetic resonance (NMR) spectroscopy is the study of molecules by recording the interaction of radiofrequency (Rf) electromagnetic radiations with the nuclei of molecules placed in a strong magnetic field. Zeeman first observed the strange behavior of certain nuclei when subjected to a strong magnetic field at the end of the nineteenth century, but the practical use of the so-called ***"Zeeman effect"*** was only made in the 1950s when NMR

spectrometers became commercially available. For example, NMR can quantitatively analyze mixtures containing known compounds.
The **NMR** spectra of SAFC Copolymer shows that the carboxylic acid hydrogen downfield with a chemical shift of 11.75 ppm. The presence of this peak confirms that a carboxylic acid functional group is present in salicylic acid. The NMR for furfural obtained at 30.0 MHz The NMR spectra of Catechol was obtained at 23.4 MHz the following spectra shows the peaks of SAFC Copolymer.

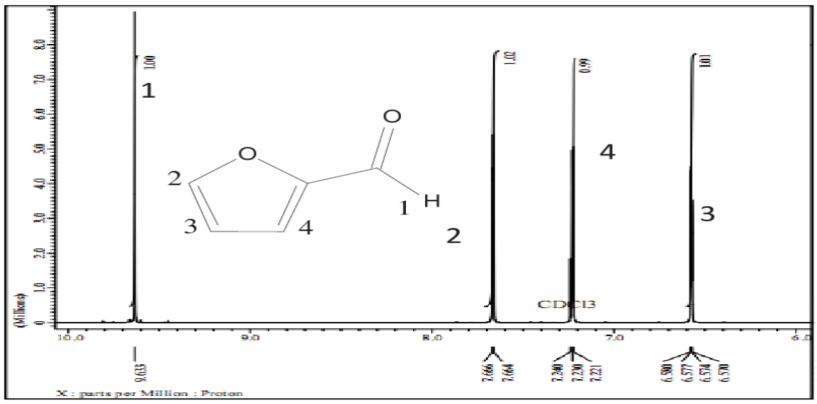

Figure 5: -NMR Spectra of SAFC Terpolymer

4. Result and Discussion

Terpolymer derived from salicylic acid, furfural and catechol with HCl were useful cation Exchangers for divalent metal ion. The FTIR spectral analysis confirmed the assumed structure of the Terpolymer. The melting point of Terpolymer was mostly high and it indicates that the polymer resin under study is thermally stable up to a high temperature. the synthesized resin can be used for the removal of heavy metals from aqueous medium and industrial waste water containing Cr3+, Mg2+, Ca2+ and Cd2+. The recovery of the metals from industrial effluents gives an indication of the utilized potential of the synthesized Terpolymer for wastewater treatment.

5. Conclusion

Terpolymer resin shows that under investigation exhibit better properties, making them better useful for various purposes. TGA and kinetic method study reveals that Terpolymer under study may stable at higher temperature. Therefore, may be used in aerospace, automotive industries and industries requiring polymers resistance to harsh environment and thermally stable polymer. The Terpolymer under study are semiconducting materials. Therefore, may have more significance in various electronics and optoelectronics industries, for making wide range of semiconducting electronic devices such as transistors, light emitting diodes, solar cells etc.

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