

Microwave Assisted Synthesis of Smf-4 Tercopolymer Resin

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Abstract:

Literature survey reveals that no tercopolymer resins from 2, 4-Dihydroxyacetophenone/2, 4-Dihydroxipropiophenone/Salicylaldehyde, Melamine/Biurate and Formaldehyde have been investigated so far. The polymer Scientists are trying to synthesize a polymeric resin which shows many potential uses as functional materials with desirable chemical and physical properties. Melamine polymers are quite hard and are used in making plastic crockery. Cups, plates and other articles made from melamine polymers are unbreakable. Most of these polymers are also used as ion exchangers for separation of various metal ions and catalyst for various chemical reactions. Depending upon the nature of high polymers, these can be used as thermally stable semiconductors in electronic industries.

Keywords: Potential, desirable, crockery, properties, exchangers and semiconductors.

Introduction:

Terpolymer resins are found to be amorphous powder, crystalline or resinous in nature and form special class of polymers which are widely known for their uses as an ion exchangers^{1, 2}. Simple copolymer resins obtained by condensation of mixture of phenols or hydrobenzoic acid various amines are also known^{3, 4}

Research on polymers or giant molecules has been carried out due to their importance, currently attached to the problem of producing material with high temperature resistance. Thermal stable materials are used as high temperature lubricant, surface coating, adhesives, fibers, elastomers, construction material required in high speed aircrafts and space vehicle constructions. These tercopolymers can also be used as abrasives, binders, insulators, retardants, dyes, rectifiers in electronic industries, fungicides in plants and living tissue

Ion-exchangers are widely used in analytical chemistry, hydrometallurgy, antibiotics, purification, separation, of radio isotopes and find large scale application in water treatment as well as in pollution control^{5, 6}. Resceptophenone has been used as an analytical reagent in the determination of metal ion because of its chelation ability⁷.

Polymers are the chief product of modern chemical industry. The materials made up of polymers find multifarious uses in all walks our life. The polymers are high molecular weight compounds. Available literature shows that, different kinds of polymers using different organic compounds were synthesized and their physico-chemical properties with applications were studied by various workers.

Material and Methods:

The Experimental work has been carried out by using all chemicals and the solvents were of analytical reagent grade with carming glass apparatus. Double distilled water was obtained by distilled water containing potassium permanganate



and alkali in glass apparatus. Condensation method is used for synthesis of tercopolymer resin.

Experimental:

Synthesis of SMF-4 Tercopolymer Resin

A mixture of Salicylaldehyde (4mole, 488ml), Melamine (1mole, 126gm), and Formaldehyde (3mole, 90ml) in (4 : 1 : 3) molar proportions and 2M hydrochloric acid (200ml) was taken in a round bottom flask fitted with water condenser and heated in an oil bath at $137 \pm 2^\circ\text{C}$ for 7 hours with occasional shaking³⁻⁹. The temperature of electrically heated oil bath was controlled with the help of dimmerstat. The resinous solid product obtained was immediately removed from the flask as soon as the reaction period was over and then purified.

The resinous product so obtained was repeatedly washed with cold distilled water dried in air and powdered with the help of agate mortar and pestle. The powder was washed many times with hot water to remove unreacted monomers. The air dried powder then extracted with diethyl ether and then petroleum ether to remove salicylic acid-melamine copolymer which might be present along with SMF tercopolymer. It was further purified by dissolving in 8% sodium hydroxide solution, filtered and reprecipitated by gradual dropwise addition of ice cold 1:1 (v/v) concentrated hydrochloric acid / distilled water with constant and rapid stirring to avoid lump formation. The process of reprecipitation was repeated thrice. The SMF-4 tercopolymer resin so obtained was filtered, washed several times with hot water, dried in air, powdered and kept in vacuum desiccator over silica gel³⁻⁹.

Result and Discussion:

The purity of newly synthesized tercopolymer resin has been tested and confirmed by thin layer chromatography technique¹⁰. The resin sample showing any sign of impurity was again purified by known standard methods like extraction, reprecipitation etc and used for further studies only after the confirmation of purity.

The melting temperature of synthesized tercopolymer resin was determined by usual technique employed for the organic compounds¹³. Accurate melting temperature of the tercopolymer resin has been noted by using a melting point apparatus.

The tercopolymer resin obtained above experimental methods was yellowish white, white or chocolate in colour. The solubility behavior of the resin sample was examined in various organic solvents, mineral acids and alkalies¹⁰⁻¹³. The elemental analysis of carbon, hydrogen and nitrogen of SMF-4 tercopolymer resins was done by microestimation techniques.

UV-Visible spectra of SMF-4 tercopolymers was recorded at room temperature in dimethyl formamide (spectroscopic grade) in 190 to 700 nm range using UV 240 double beam spectrophotometer fitted with an automatic pen chart recorder. The Infrared spectra of the above newly synthesized tercopolymer resin was scanned in KBr pellet on Perkin Elmer Model 577 IR spectrometer in the region 4000 to 200 cm^{-1} while NMR spectra was scanned on 90MHz for ^1H using Perkin Elmer Model R-32 NMR spectrometer in deuterated dimethyl sulphoxide (DMSO- d_6).

The molecular weight of the above synthesized tercopolymer resin has been determined by two methods, Conductometric titration in non-aqueous medium and



Vapour pressure osmometry methods. The information about molecular weight is also supported by the results of viscometric study of the polymer.

The thermogravimetric analysis (TGA) of the synthesized tercopolymer sample has been carried out using Perkin Elmer TGS-II thermal analyser at heating rate of 15°C per minute and in nitrogen atmosphere upto 850°C. The thermal stability of the tercopolymer resins can be known from kinetic parameters and activation energy.

The electrical conductivity of SMF-4 tercopolymer resins has been measured by BPL-India Million Megohmmeter Model RM 160 MK III A.

Conclusion:

The tercopolymer of salicylaldehyde, melamine and formaldehyde has been synthesized by the above discussed condensation method is very simple. The purity of newly synthesized SMF-4 tercopolymer resin has been experienced and established by special physico-chemical and experimental techniques.

Acknowledgement:

I take this opportunity to express my deepest sense of gratitude towards Dr. I. Z. Jadhav and Dr. A.B. Kalambe, Institute of Science Nagpur for providing necessary laboratory facilities for carry out this work.

A sincere acknowledgement is made to Dr. R. D. Raut , J. B. College of Science Wardha and Dr. W. B. Gurnule, Kamla Nehru Mahavidhaya Nagpur for suggesting the problem, encouragement and better co-operation during the course of work.

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