

Synthesis and Characterization of Tercopolymer Derived From Vinyl Acetate, Maleic Anhydride and Acrylonitrile (VA-MA-AN)

Tusar Bajpai¹, Abhishek Verma², Abhinav Dewedi³

¹Department of Chemistry, Sai Nath University, Ranchi, Jharkhand, Uttar Pradesh, India

²Department of applied Chemistry BBAU, Lucknow, Uttar Pradesh, India

³Chemistry Department KNPG College, Gyanpur, Bhadohi, Uttar Pradesh, India

ABSTRACT

The present paper deals with the synthesis of tercopolymer derived from vinyl acetate, maleic anhydride and acrylonitrile using Azobis isobutyronitrile (AIBN) as an initiator. The sequence structure of the tercopolymer chain was characterized by Fourier transform infrared (FTIR). The results showed that sequence of the corresponding monomers still existed in the tercopolymer chain. The thermal properties of tercopolymer was identified by Thermogravimetric Analysis (TGA) and Differential Scanning Calorimetry (DSC). It has been good findings to ensure the optimum temperature at which the tercopolymer synthesized, reports maximum yield.

Keywords: Copolymerization, Poly(Maleic Anhydride), Poly (Vinyl Acetate)

I. INTRODUCTION

The tercopolymers offered, novelty and versatility, hence they occupy the pivotal position in the field of material science. The name 'ter' demands the union of three monomers, the tercopolymers are formed by three different monomers. They have good thermal and mechanical properties. Thermal behaviour and stability have been studied by Thermogravimetric analysis and Differential Scanning Calorimetry that dictate the use of the copolymer at high temperatures.

Various researchers have studied the synthesis of different ter-copolymers in different reaction conditions [1-12]. Sant work has been done for the synthesis and characterization of tercopolymer with acrylonitrile which prompted us to undertake synthesis, characterization and properties of tercopolymer derived from vinyl acetate, maleic anhydride and acrylonitrile.

The following objectives of present studies are:

- Synthesis of tercopolymer with acrylonitrile
- Solubility of tercopolymer with different solvent
- Characterization of tercopolymer
- Study of thermal properties of tercopolymer

II. METHODS AND MATERIAL

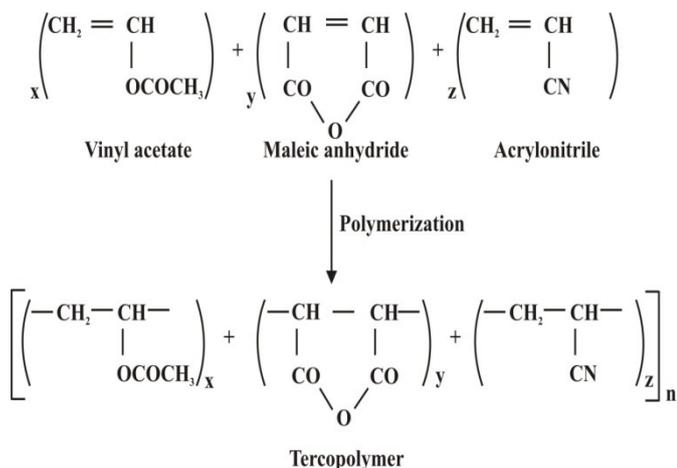
A. Material and Instruments

All of the substrates purchased were of good quality and they were purified again before use. Vinyl acetate was freshly distilled. All the solvents were distilled, dried and all monomers were purified.

B. Copolymerization

9 ml of vinyl acetate, 300 mg of maleic anhydride, 6 ml of acrylonitrile and 40 mg of Azobisisobutyronitrile were taken in a clean and dry small beaker. The beaker was swirled gently till the initiator dissolved in the monomers. Now each glass ampule was filled with 3ml of this solution. The ampules were sealed and put to heating on water bath at 60-70°C. After 15 minutes the solution of the mixture began to develop in light violet colour. It might be noticed that, the viscosity of the contents in ampules increased steadily with the passing of time as the liquid solution was converted into gum form. Reaction stopped after 3 hrs as the ampules attain a high viscosity and appeared in gummy form. The ampules were cooled and broken, then the tercopolymer was collected in petridish. The residual homopolymers

were washed away with benzene and the tercopolymer was dried under vacuum at 60°C. The yield of tercopolymer was 6.900 gm.



C. The solubility of tercopolymer in different solvents

S.No.	Solvent	Solubility	Remarks
1.	Acetone	Slightly Soluble	Turbidity
2.	Benzene	Insoluble
3.	Water	Insoluble
4.	Carbontetra chloride	Insoluble
5.	Chloroform	Insoluble

III. RESULTS AND DISCUSSION

A. FTIR curve for the tercopolymer VA-MA-AN

FTIR spectrum was recorded on FTIR spectrometers. The infrared spectrophotometric measurements showed bond formation processes occurring in the tercopolymer sample. There were only 5-6 peaks and many little humps in the spectrum. The presence of C-H stretching in $-\text{CH}_2$ was established from the bonds located at 2930.9 and 2832.4 cm^{-1} . A number of peaks namely at 2363.3, 2340.0 and 2245.3 cm^{-1} were probably of CN stretching of unsaturated aliphatic nitriles. An absorption band at 1739.3 cm^{-1} could be associated to $>\text{C}=\text{O}$ stretching of α, β - unsaturated ester. The peak which was seen at 1363.9 cm^{-1} , indicative of $-\text{CH}_2$ frequency and at 1248.1 cm^{-1} was assigned to C-O

stretching of vinyl acetate. A small peak at 1023.4 cm^{-1} was also observed for C-O vibrations of ester (figure 1).

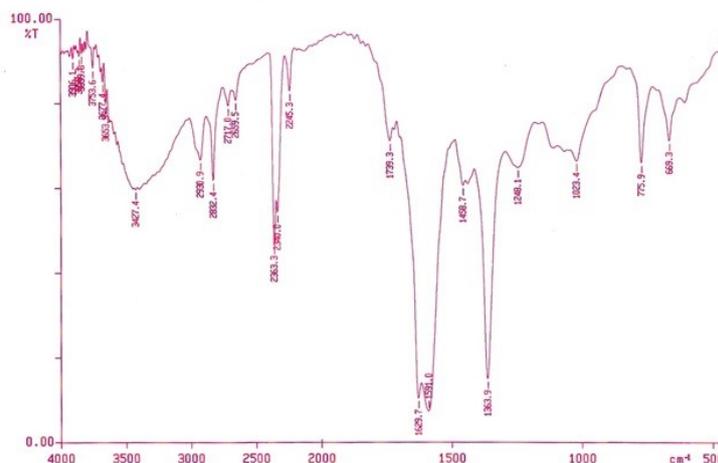


Figure 1: The FTIR curve of VA-MA-AN tercopolymer in KBr phase

B. Thermogravimetric analysis (TGA) for the tercopolymer VA-MA-AN

Thermogravimetric analysis of polymer was performed on the Universal V3.8B TA instrument. The TGA curve of tercopolymer revealed that the compound started to decompose at 25°C and with increased in temperature to 230°C, 5.399% loss in weight was observed. The loss in weight gradually increased on increasing the temperature to 500°C which was recorded to be 48.459%. Above this temperature the rate of loss in weight was observed to be slow and at a temperature of 800°C a total loss of 63.539% in weight was observed (figure 2).

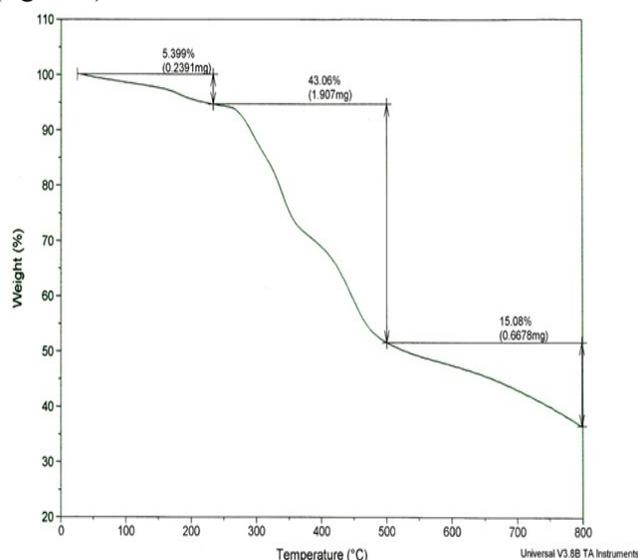


Figure 2: Thermo Gravimetric Analysis (TGA) curve of VA-MA-AN tercopolymer

C. Differential Scanning Calorimetry (DSC) for the tercopolymer VA-MA-AN

Many properties of the polymeric material undergo a change near the glass transition region. One such property is the heat capacity. By measuring this property as a function of temperature, we can determine the T_g of polymers. Differential Scanning Calorimeter directly gives a recording of heat flow rate (C_p) against temperature (T) as shown in Figure 3.

From this figure 3 it can be seen that the heat flow rate of the sample undergoes a change during transition. The T_g value can be computed from the plot by extrapolating the pre-transition and post-transition lines and by calculating the temperature where the heat flow rate is exactly at the middle of pre and post transition rate and this has been found to be 272.27°C.

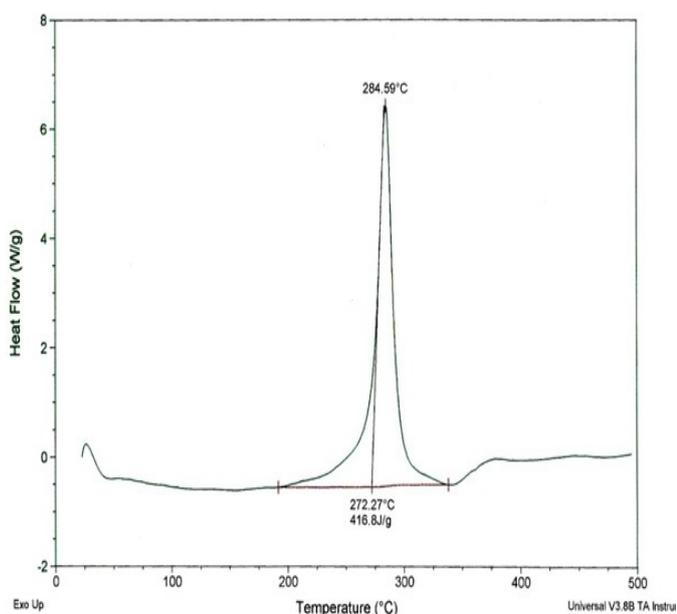


Figure 3 : Differential Scanning Calorimetry (DSC) curve of VA-MA-AN tercopolymer

IV. CONCLUSION

Tercopolymer of VA-MA-AN with different molar ratio were synthesized using AIBN as an indicator, with the reaction carried out at 60-70 °C. The structural analysis indicate that although the sequence of the corresponding monomer still existed in the tercopolymer chain, the random tercopolymer was ultimately obtained. The solubility of obtained tercopolymer also studied with different solvent. The molecular structure was characterized by Spectroscopic studies (i.e. FTIR). Thermal behaviour and stability also

studied by TGA indicate that decomposition of tercopolymer 63.539 % at 800°C. Heat capacity of tercopolymer also studied by Differential Scanning Calorimetry (DSC).

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