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Synthesis and Characterization of Copolymer 2-Aminothiophenol-Oxamide -Formaldehyde.

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ABSTRACT

Copolymer (2-ATOF) has been synthesized by the polycondensation of 2-aminothiophenol, oxamide with formaldehyde in 3:1:5 molar ratios of reacting monomers using 2M hydrochloric acid as a catalyst. Various spectral and physiochemical techniques like Ultraviolet-Visible, XRD diffraction, Scanning Electron Microscope (SEM), Fourier Transform Infrared (FTIR) techniques have been promoted to illuminate the composed copolymer. Elemental analysis and Number average molecular weight (Mn) has been determined by Gel Permission Chromatography. XRD pattern mark amorphous nature of copolymer Particle size, porosity, density of copolymer has been calculated by XRD data. The surface morphology of 2-ATOF was studied by Scanning Electron Microscope (SEM).

Keywords: Polycondensation, spectral, physiochemical, morphology, catalyst.

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INTRODUCTION

Most of the synthetic polymer are of relatively recent origin, the polymers have brought to the fore a scintillating range of irresistibly attractive choices. They have become so much a part of our society that it appears almost unthinkable that we could ever do without them. Copolymer nature may be amorphous, crystalline have different uses and special types of polymers. Thermal stability, morphology, synthesis, and application of transition metal organic coordination have been described numerous studies of polymer. In modern history of thermogravimetry, thermal stability of copolymer and their kinetics studies have been at the center of thermal analysis. At elevated temperature by changing the monomer composition, many researchers tried to improve the thermal stability. Gurnule and his coworkers prepared copolymers resulting from 8-hydroxyquinoline, 5- sulphonic acid, guanidine, and formaldehyde [1]. Various work on the synthesis, thermal degradation of copolymer has been undertaken [2-9].

A considerable attention has been paid in the past two decades to the synthesis of copolymer due to high thermality. Chakole et al synthesized and studied thermal properties of copolymer (2,2-dihydroxyphenyl, ethylenediamine and formaldehyde) [10]. Kohad et al studied kinetics of thermotropic, copolymer organic and photoluminescent properties [11-13]. Gupta et al observed thermal degradation studies of 2- amino-6-nitrobenzothiazole-oxamide-formadehyde copolymer [14]. Damini Shedmake and coworker studies antimicrobial activity of copolymer metal complexes [15]. Anti-inflammatory agent and molecular modeling of phenyl diamine, phenyl hydrazine-formaldehyde terpolymer has been studied by N. Mujafarkani et.al [16]. Nandekar and Mandavgade have synthesized copolymer by condensation of salicylic acid and thiosemicarbazone with formaldehyde and TGA method [17-20]. Bobde et al prepared a terpolymer by polycondensation of monomers 2, 2'-biphenol, ethylenediamine and formaldehyde in 1:1:2 molar ratios in the presence acid catalyst [21]. The terpolymer resin derived from o-toluidine, semicarbizide, formaldehyde and its composite with activated has been prepared [22]. Sorption studies of heavy metal ions by salicylic acid, formaldehyde and catechol have also been evaluated.[23] Several terpolymers were synthesized by condensation of 4- hydroxybenzaldehyde oxime, formaldehyde and chloro, bromo, methoxy or methyl substituted acetophenones and their thermal stability was investigated by using Thermogravimetric Analysis (TGA) and Differential Scanning Calorimetry (DSC) [24]. The present paper deals with the synthesis, characterization of copolymer derived from 2-aminothiophenol, oxamide and formaldehyde. The copolymer has been characterized by using spectral methods to know the structure of copolymer.

MATERIAL AND METHODS

Chemicals

Complete chemicals utilized were all analytical research grade (AR). 2-aminothiophenol, oxamide and formaldehyde (S.D. Fine Chemicals) was purchased having high purity (99%) from 2-aminothiophenol, oxamide and formaldehyde Central Scientific Company Nagpur.

Synthesis of 2-ATOF copolymer

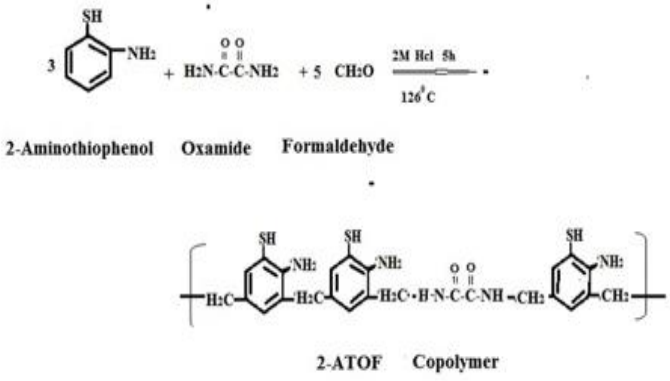

Figure:1 Reaction of 2-ATOF copolymer

Figure: 1 Synthesis of 2-aminothiophenol-oxamide—formaldehyde copolymer.

Copolymer 2-ATOF was synthesized by using polycondensation method with molar ratio of 3:1:5 taking monomers 2-aminothiophenol (0.3mol), oxamide (0.1mol) and formaldehyde (0.5mol) in round bottom flask 2M HCL (200ml) as a catalyst at 126^0 C for 5h. The reaction mixture was then cooled, new synthesized copolymer was precipitate by 8% NaOH solution by dropwise addition of 1:1 HCL with constant stirring, yellowish white solid was separated out and cleaned with distilled water and dried in the air. The proposed reaction appeared as in figure 1.

Characterisation of copolymer

The new synthesized copolymer is characterized by a variety of experimental approaches. Elemental analysis for Carbon, Hydrogen, sNitrogen and Sulphur on Elementar Vario EL-III Elememtal Anaylzer. The range of FTIR in KBr pellets inside the scope of 4000-5000 cm^{-1} has been examined. All the spectral characterisation out at Sophisticated Test and Instrumentation Center Cochin, University of Science and Techonology, Cochin.

Results and Discussion

The new synthesized 2-ATOF copolymer was yellowish white in color and amorphous in nature. It is dissolved in DMSO solvent and insoluble in common organic and inorganic solvents. The yields of 2-ATOF copolymer 80% were noted.

Elemental Analysis

The elemental analysis of carbon, hydrogen, nitrogen, and Sulphur in the 2-ATOF copolymer were calculated. The data given by analysis was a good link between the copolymer composition. This implies that $C_{25}H_{27}N_5O_2S_3.H_2O$ and 525 were discovered to be empirical formula and empirical weight of repeating unit, respectively.

FTIR spectral analysis

FTIR spectral results were used to gives a very informative data based on stretching, bending, twisting, wagging of the various types of bonding, linkages, and functional group present in 2-ATOF copolymer. The peak located at 3446.73 cm^{-1} presence of a -SH (phenolic thiol) group. The sharp band displayed at 1670.02 cm^{-1} may be due to the stretching vibration of carbonyl group of both Ketonic as well as moiety. The presence of weak bands within the range 1488.18-1060.84 cm^{-1} due to $-CH_2$ bridges in the polymeric chain. The 1,2,3,5 substitution of aromatic benzene ring recognized by the sharp, medium/weak absorption bands appeared at 1120.64 and 781.09cm^{-1}.

Table 1: FTIR Data of 2-ATOF Copolymer

Assignments	Observed frequency (cm^{-1})	Experimental frequencies (cm^{-1})
-SH (Phenolic Thiol)	2921.42	2550-2900
SH-C-NH	3431.70	3500-2800
>C=O (Ketonic and biuret moiety)	1671.51	1540-1870
Aromatic ring >CH_2(methylene bridges)	1060.49	1300-1000
1,2,3,4,5 substitution in benzene ring	1120.10	1200-800

UV-Visible spectra

UV visible spectrum of 2-ATOF copolymer in DMSO solvent recorded in the region of 200 to 800nm spectrophotometers, Shimadzu, model-1800 at the department of chemistry, Kamla Nehru Mahavidyalaya, Nagpur. The spectrum exhibits two absorption band maximums in the 280-300 and 300-330 nm. The more intense band can be account as π -π* transition, while weak band could be expected n-π* transition. The π-π*allowed transition of conjugation benzene group whereas the n-π*forbidden transition of -SH group. Both bathochromic and hyperchromic effects of conjugation are appear by chromophore due to auxo chromic substituents -SH and $-NH_2$ group in copolymer.

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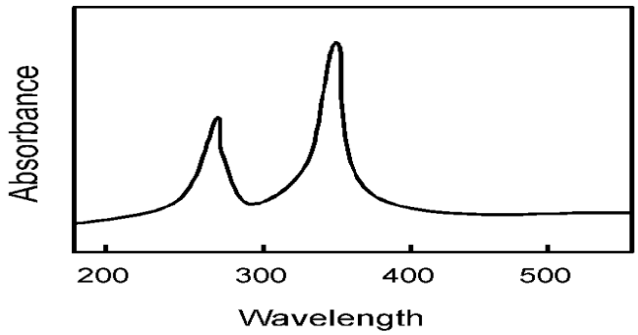

Figure 2: UV -Visible spectrum of 2-ATOF copolymer

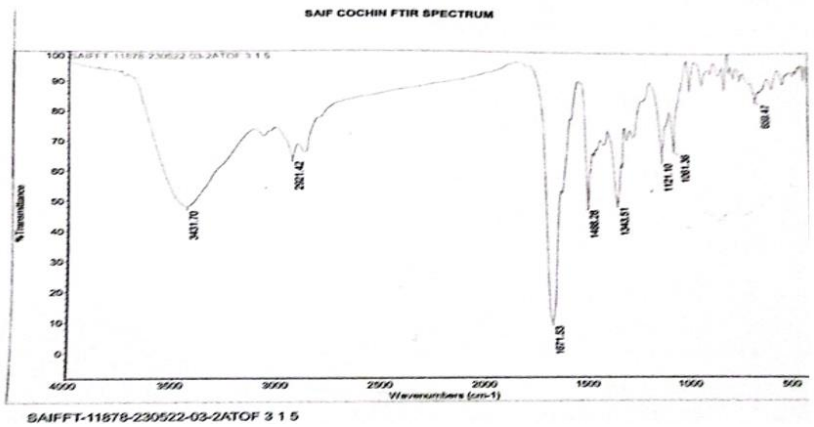

Figure 3: IR spectra of 2-ATOF copolymer

Scanning Electron Microscopy (SEM)

The scanning electron microscopy analysis has provided great application in characterization, recognizing surface properties and particle size of material. The surface morphology of all 2-ATOF copolymer was examined by scanning electron micrographs SEM Joe/6390 LV at STIC, Cochin at different magnification. The pictures of 2-ATOF copolymer shows a spherulites structure with a profound increase. The crystals are minor in the superficial zone being less closely packed with sponginess [37]. The spherules are polycrystalline formation smooth surface indicate amorphous- crystalline in nature.

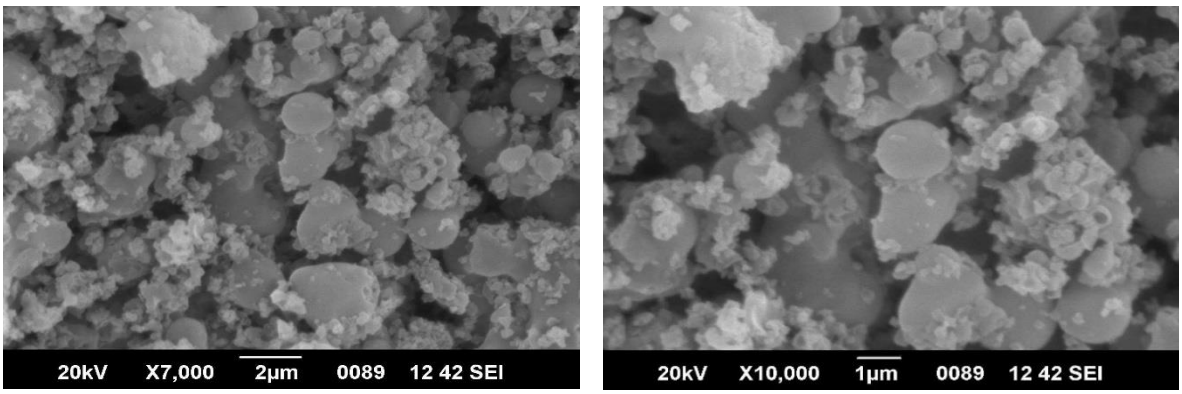

Figure 4: Scanning Electron Microscopy of 2-ATOF copolymer

CONCLUSION

New copolymer 2-ATOF has been synthesized by polycondensation method of 2aminothiophenol, oxamide and formaldehyde in the presence of 2M HCL as a catalyst. The proposed structure of copolymer was confirmed from the elemental analysis, UV Visible, FTIR Spectral studies. The 2-ATOF copolymers surface morphology was recognized the state of transition between crystalline and amorphous phases. SEM micrograph show the more active site and discrete pores present in the copolymers

REFERENCES

[1] Gurnule WB and Singru R N J. Therm Ana. Color 2010; 100; 1027.
[2] Rahangdale S, Das N, Vajpai K and Gurnule W. Int J Recent Sci Res 2019; 10: 31772.
[3] Gurnule WB, Das NC, Vajpai S, Rathod YU. Mater Today: Proc 2020;29 (4): 1071-1076.
[4] P Meng, Y Zhou, C Yaun, Q Li, J Liu, H Wang, J Hu, J He. IEEE Trans. Dielectrics Electr Insul 2019;26 (3):674–680.
[5] E Lee, W Shin, O Bae, FS Kim, Y-JHwang. Polymer 2019;184: 1–24.
[6] Sidharaj N, Rajaran G, Senthil. M. Mat Tod Proc 2020; 42: 1030-1036.
[7] Mahant R, Kongre N, Bhagat D and Gurnule WB. Alochana Chakra J 2020;9: 451.
[8] Sasikala T, Greeshma K, Gnana P, Muthulingam S and Nandhakumar C. GSC Advanced Research and Reviews 2020;05 :19.
[9] El-shazly R, Kamal R, Nassar A, Ahmed N and Sayed G. Appl Petro Res 2020; 10 :115
[10] YU Rathod, SB Zanje, WB Gurnule. J Phys.: Conf Ser 2021.
[11] Kohad CG, Gurnule WB. IJCESR 2019;6 (1):503–512.
[12] Gurnule WB, Kohad CG, Gurnule WB. Res J Pharma Biol Chem Sci 2018;9(5):393-402.
[13] Kohad C and Gurnule WB. Materials Today Proceedings 2019;15: 438.
[14] Gupta PG, Gupta RH, Gurnule WB. Alochana Chakra J 2020;9(5):106–117.
[15] Shedmake D, Khobragade JV, Gurnule WB. 2023; 11(2); 67-79.
[16] N Mujafarkani, AJ Ahamed. Materials Today Proc 2021; 47(9)1920–1928.
[17] Nandekar KA, Dontulwar JR, Gurnule WB. Rasa J Chem 2012;5(3):261–268.
[18] Mandavgade SK. Int J Innovat Eng Sci 2018; 3(5):137–141.
[19] Chakole SP, Nandekar KA, Gurnule WB. 2021; 1913: 012062.
[20] Barkade SS, Bansod PG, Doss VR, Mardikar SP. Adv Sci Engg Medic 2020; 12: 198-206.
[21] Rangari M, Rahangdale SS, Shrivastava S, Rathod YU, Waghe PU, Gurnule WB. Ind J Engg Mat Sci 2023; 30:816-822.
[22] Velmurugan G, Ahmed KR, Azarudeen SR. Iran Polym J 2015; 24(3):229-42.
[23] Bhatt RR, Shah AB. Arab J Chem 2013; 8; 414-26.
[24] Gupta PG, Gupta RH, Gurnule WB, Rao SN, Rathod YU. Ind J Engg Mat Sci 2023; 30:838-844.
[25] Gurnule WB, Gupta PG, Gupta RH, Rathod YU, Singh NB. IOP Conf. Series, Earth and Enviromental Sci., 203, 1281, 012026.
[26] Yaxinli H, Cheng B, Yishang C. Polym Sci 2020: 323-332.
[27] Mandavgade SK, Gurnule WB. Mat Tod Proc 2022; 60: 1814-1818.
[28] Gurnule WB, Rathod YU, Belsare AD, Das NC. Mat Tod Proc 2020; 29: 1044.
[29] Chakole SP, Rathod YU, Pandit VU and Gurnule WB. Mat Tod Proc 2022; 53:96-100.
[30] Gurnule WB, Rahangdale PK, Paliwal LJ, Kharat RB. React Func Polym 2003;55(3):255-265.
[31] Thakre M and Gurnule W. 2019 Materials Today Proceedings 15; 516.
[32] Das NC, Rathod YU, Pandit VU, Gurnule WB. Mat Tod Proc 2020; 53: 80-85.
[33] Morsch S, Liu Y, Lyon SB, Gibbon SR., Gabbriele B, Malanin M, Eichhorn K. Polym Degrad Stabil 2020; 176.
[34] YU. Rathod, SB Zanje, WB Gurnule. J Phys.: Conf. Ser 2021.
[35] Singru RN, Zade AB, Gurnule WB. J Appl Polym Sci 2008;109(2):859-868.
[36] Tarase, MV, Zade AB, Gurnule WB. J Appl Polym Sci 2008;108(2):738-746.
[37] Rahangdale SS, Gurnule WB, Zade AB. Ind J Chem 2009;48A:531-535.