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A Study on Thermal properties of AA copolymer-Silver metal particles complexes.

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ABSTRACT

Synthesis and characterization composites based on acrylic acid-acryl amide co polymer with silver metal particles have been reported in the present studies. Presence of silver metal particles in the AA copolymer network has been confirmed by spectrophotometric techniques. Due to interaction of silver metal particles, thermal stability of copolymer increased. However with the increase of metal particle content, thermal stability decreased. Correspondingly, SEM studies indicate that silver particles seems to bound to the copolymer matrix. With the increase of Ag content, agglomeration of Ag particles is observed.

Keywords: AA copolymer, AA-Ag composite, FTIR Spectra, DSC thermogram, SEM micrographs.

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INTRODUCTION

Composites consisting of polymers with metal particles have gained importance due to their potential applications in different branches of science and technology (1). In this context, composites based on Polyacrylic acid-Silver (PAA-Ag) (2), Acrylic acid-Acrylamide-Silver copolymer (AA-Ag) (3) and Polymethyl methacrylate-Silver (PMMA-Ag) (4) have been reported in literature. Effect of Ag content on spectroscopic, thermal properties of PAA-Ag composite has been reported by Srinivas et al (5). Due to increase in Ag concentration, thermal stability of complex is reported to increase. Though such studies are reported on PAA-Ag system, effect of Ag concentration on AA copolymer-Ag complex has not been reported. Therefore the authors have been made an attempt in this regard using Differential Scanning Calorimetry (DSC) and Scanning Electron Microscope (SEM) techniques.

EXPERIMENTAL

To synthesize AA copolymer-Ag composite chemical methods are used. 1 gram of AA copolymer is dissolved in 100ml of water and aqueous solution of 0.1 M AgNO_3 is added to it by drop by drop with constant stirring for several hours at 80°C . The resultant mixture is sonicated for one hour and annealed to get the powder of complex.

Optical absorption spectra is recorded on Shimadzu spectrometer in the wavelength region of 200-800nm with water as blank, FTIR spectra of complex are recorded on PERKIN ELMER spectrometer in pellet form. DSC thermo grams are recorded on TA Q-10 calorimeter. Few milligram of sample is sealed in aluminum pan and it is heated from RT to 300°C at a heating rate of $10^\circ\text{C}/\text{minute}$ while empty aluminum pan is used as reference SEM micrographs are recorded on Carl Zeiss electron microscope. The sample is fixed on the cylindrical stub which is covered with carbon strip and the strip is sputtered with gold coating to examine its surface morphology.

RESULTS AND DISCUSSION

Spectroscopic studies

AA copolymer has shown optical absorption bands centered around 280 nm corresponding to carboxyl groups of amide (CONH_2) and carboxyl (COOH) groups of copolymer. On complexation with silver, an absorption band around 350 nm and 400 nm were observed corresponding to filler metal particle or Plasmon resonance (6, 7). Upon increasing Ag concentration, intensity of these absorption bands decreased suggesting the agglomeration of silver nanoparticles and formation bulk silver.

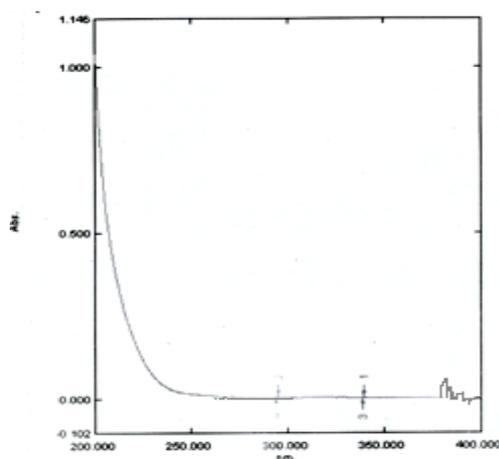


Fig 1: Optical absorption spectrum of AA-Ag copolymer complex

FTIR spectra of the copolymer and metal complex are recorded corresponding to the finger print region of copolymer i.e. amide (CONH_2) groups at $3500-3150\text{ cm}^{-1}$ and carboxyl groups centered around 1700 cm^{-1} are observed. Upon complexation, the intensity of 1720 cm^{-1} band decreased due to the interaction

of silver particles with the amide and carboxyl groups. These results suggest that the silver is reduced by the copolymeric system (8). Upon increasing Ag concentration, the FTIR absorption bands broadened suggesting that Ag-co polymer interaction increases.

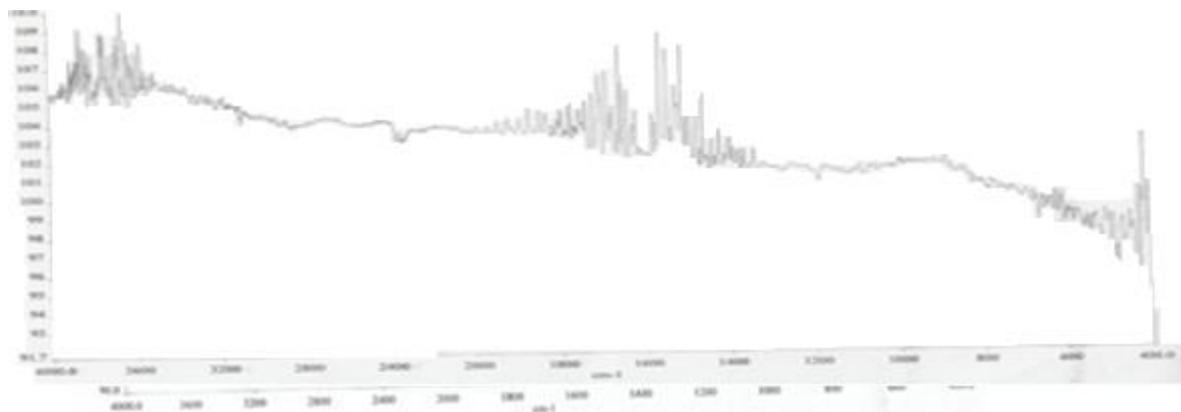


Fig 2: FTIR Spectrum of AA-Ag copolymer Complex

Thermal studies

DSC thermogram of AA Copolymer-Ag complex is shown in Fig 3. The thermogram consists of first order transition around 190^o C corresponding to glass transition temperature and a second order endothermic peak at 248^o C corresponding to the melting point. Melting point of AA copolymer is around 202^oC. Upon complexation, the T_m is found to shift towards higher temperatures, suggesting that thermal stability increases on complexation. The results also suggest that the complex is a two phase system with enhanced thermal stability than the copolymer. The higher thermal stability is attributed to the presence of silver nanoparticles which a part of incident thermal radiation causing an enhancement in thermal stability of copolymer.

On increasing Ag concentration, a change in DSC thermogram is observed as shown in fig (4). T_g of complex is remarked the same. However a broadened melting endotherm is found. Further T_m is also shifted towards lower temperature. Therefore increase in Ag concentration has an antagonistic effect on thermal stability of copolymer. Further melting thermo gram of low doped composite has well defined crystallize distribution but it is found to be due to the presence excess amount of silver present in the composite. Therefore highly doped composite with less crystallite size distribution is expected to exhibit less thermal stability as observed in the present studies. SEM micrographs also suggest the same.

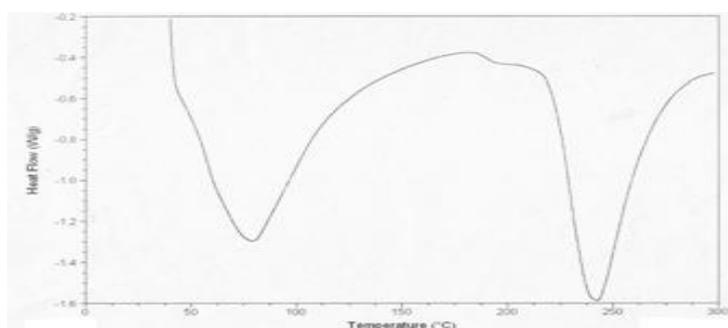


Fig 3: DSC thermogram AA copolymer -Ag complex with less Ag concentration.

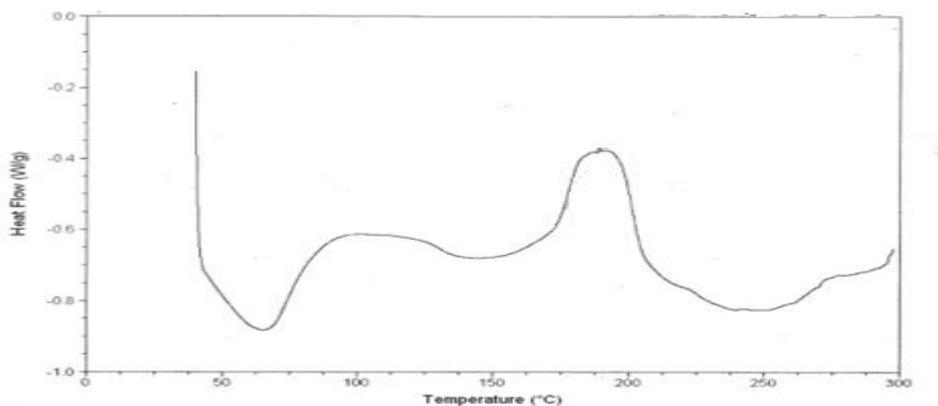
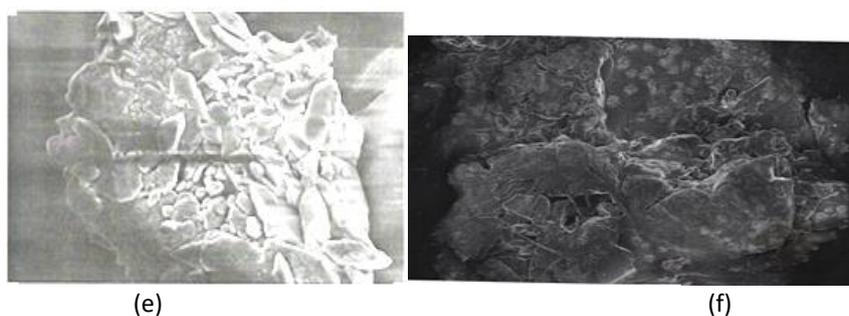
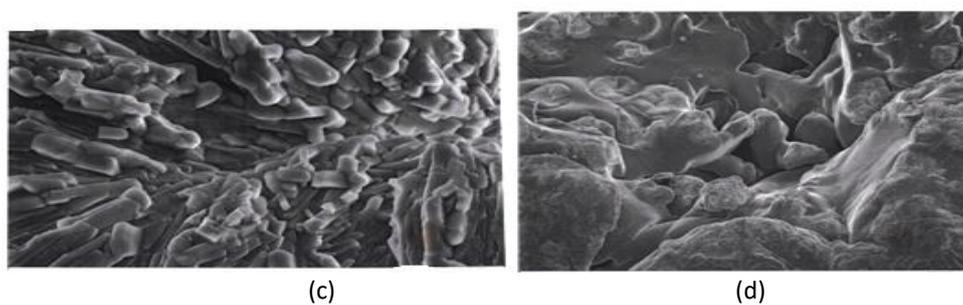
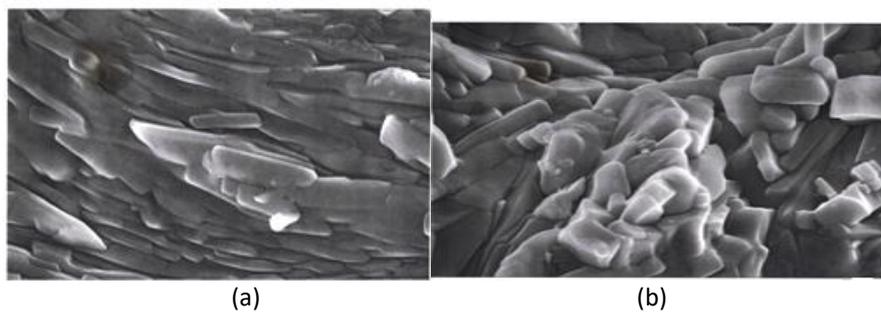


Fig 4: DSC thermo gram of AA copolymer -Ag Complex with more Ag concentration

SEM Studies

To examine surface morphology of copolymer and complex, SEM micrographs are recorded as shown in Fig 5. a, b, c, d, e, f, g, h and i represents the SEM micrographs of complex with increase of Ag concentration. The micrographs suggest that at low Ag concentration Ag particles are chemically bound to Copolymer network as shown in figure 5(a) to 5(f). However with the increase of Ag concentration agglomeration of Ag particles takes place forming bulk silver as shown in figure 5(g) to 5(i). As a result Ag-AA copolymer interaction is thought to be reduced decreasing the thermal stability of copolymer.



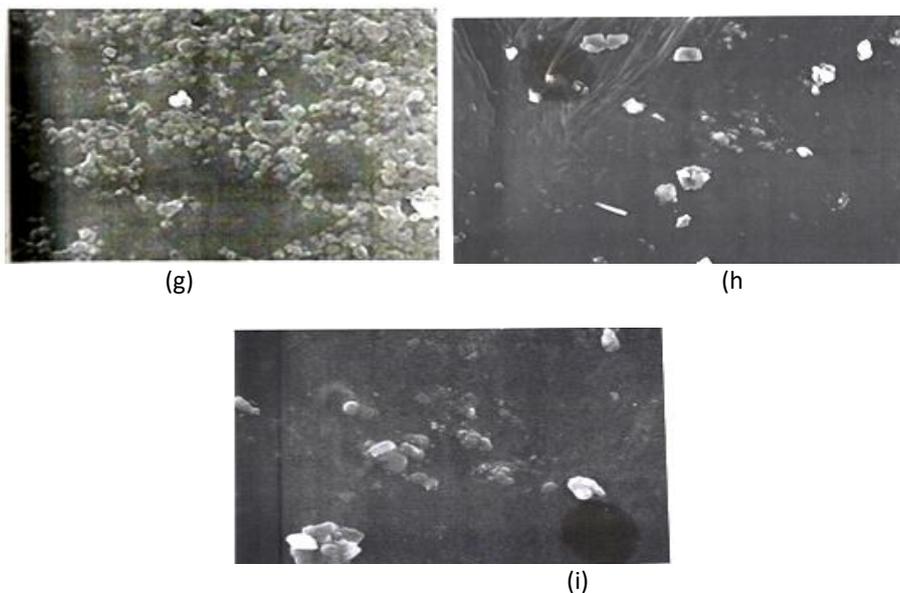


Fig 5: SEM micrographs of AA copolymer-Ag complex.

a-f: Complex with low Ag content. g-i: Complex with high Ag content.

CONCLUSION

Acrylamide- acrylic acid (AA) copolymer is used as a reducing agent to produce silver metal particles. Spectro photometry, FTIR studies confirm interaction of silver particles in the copolymer matrix. The resultant complex is thermally stable than the copolymer due to presence of silver metal particles. However with the increase of Ag concentration thermal stability decreases. SEM studies suggest that silver particles are bound to copolymer matrix when Ag concentration is less. However when Ag content is increased, agglomeration of metal particles is observed.

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