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A Study on Characterization of AA-AMPS-Pb Composite by Spectroscopic and Thermal Methods

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Abstract
Spectroscopic and thermal properties of Acrylamide-acrylamide 2- methyl propane sulphonic acid (AA-AMPS) copolymer have been investigated by spectrophotometric, Fourier transform infrared (FTIR), differential scanning calorimetry (DSC) and scanning electron microscope (SEM) techniques. The UV absorption studies indicate the presence of lead nano particles and FTIR results suggest that complexation has been taken place between reactive groups of copolymer and lead ion. Due to the presence of lead nano particles thermal stability of copolymer is increased.

Keywords: *FTIR spectra, UV-VIS spectra, DSC thermo gram, AAAMPS copolymer and Pb nano particles*

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INTRODUCTION

PbO particles have applications as luminescent materials, gas sensors, storage devices [1, 2]. Arulmozh and Mythily [3] have reported synthesis and characterization of PbO nano particles with different organic capping agents. In order to avoid aggregation of nano particles capping agents are used. In this study these authors have used oleic acid, ethelene diamine tetra acetic acid (EDTA) as capping agents. The generated PbO nano particles have been characterized by X-ray diffraction (XRD), Fourier transform infrared spectra (FTIR), Photolumenecense (PL) and Field effect silicon electron microscope (FE-SEM) techniques. The PbO nano particles produced by this method are reported to have well defined crystalline structure and have a size of 25–30 nm. Pavani et al. [4] have synthesized lead nano particles by biological methods and characterized them by different experimental techniques. The nano particles are reported to have a size of 5–20 nm. Harish et al. [5] have studied gamma ray shielding applications of lead oxide filled isopthalic polymer resin. They have used different filler concentrations of 0.5,10,20,30,40 and 50 in the polymer laminate. The performance of polymer composite is observed to be excellent which is assigned to be due to the presence of filler lead nano particles. Structure and properties of palladium metal particles in polycarbonate matrix are found to depend on synthetic procedure [6, 7]. Aluminum nano particles embedded in poly ethylene matrix has been reported by Hang et al. [8]. When Palladium nano particles are incorporated into polyanilene matrix, the resultant materials is used as sensor to detect methanol. Copper-polyanilene nano composites are used to detect chloroform [9]. In the present studies the authors attempts synthesis of polymer composite consisting of AA copolymer with lead nano particles. Spectroscopic and thermal properties of the composite are investigated by different experimental techniques.

EXPERIMENTAL DESIGN

Acrylamide-AMPS copolymer in powder form is dissolved in water and aqueous solution of 0.1 M lead nitrate is added to it drop by drop followed by stirring for 1 hour. The mixture is sonicated for 1 hour and annealed 80^{0}C to get the AA-Pb complex in the powder form. Optical absorption spectra are taken for dilute solutions of complex in the wave length region of 200–800 nm on Shimadzu spectrophotometer with water as blank. Fourier transform infrared spectra are recorded on Perkin Elmer spectrometer in pellet form. DSC thermo grams are recorded on TA Q-10 model Calorimeter. For this purpose, about 2–3 mg of complex was sealed in aluminum pan and heated from room temperature (RT) to 350^0C with a heating rate of 10^0C/min. Empty aluminum pan is kept as reference and nitrogen gas is flushed throughout the experiment to avoid oxidation of the sample.

To record scanning electron micrographs of the complex the sample is fixed on cylindrical stub covered with carbon strip. The surface of the sample was gold coated with sputtering unit and morphology was examined with a Carl Zeiss Scanning electron microscope.

RESULTS AND DISCUSSION

UV-VIS Studies

UV-VIS spectra of AA copolymer and AA–Pb composite are shown in Figures 1 and 2. Optical absorption of spectra possess absorption band around 220–250 nm, followed by humps at 275 and 350 nm. Li et al. [9, 10] have observed optical absorption bands at 274, 261, 254 nm position corresponding to capped PbO particles while 278 nm absorption bond is due to uncapped PbO particles. PbS nano particles also exhibited optical absorption band around 330 nm (3). Therefore, presence of absorption bands around 220–250, 277 and 350 nm confirm the presence of lead nano particles.

FTIR STUDIES

FTIR spectra of AA-Pb composites under different conditions are recorded as shown in Figures 3, 4 and 5. The spectra possess characteristic absorption band corresponding to both AA copolymer and Pb. These bands and assignments are as listed in Table 1. The FTIR absorption bands are consequent to chemical structure of copolymer i.e., to acryl amide (N-H,C=O,CH_2/CH_3) groups and AMPS(C=O,SO_3H,CH_3) groups. On complexation, the absorption bands corresponding to Pb-O are observed at 652–440 cm^{-1}, suggesting the formation of AA-Pb complex. On increasing the concentration of Pb, the absorption bands are found to diffuse and finally for high doped complex, diffused FTIR spectra are observed as shown in Figures 3, 4 and 5. The results suggest that there is strong interaction between Pb and reactive groups of AA copolymer.

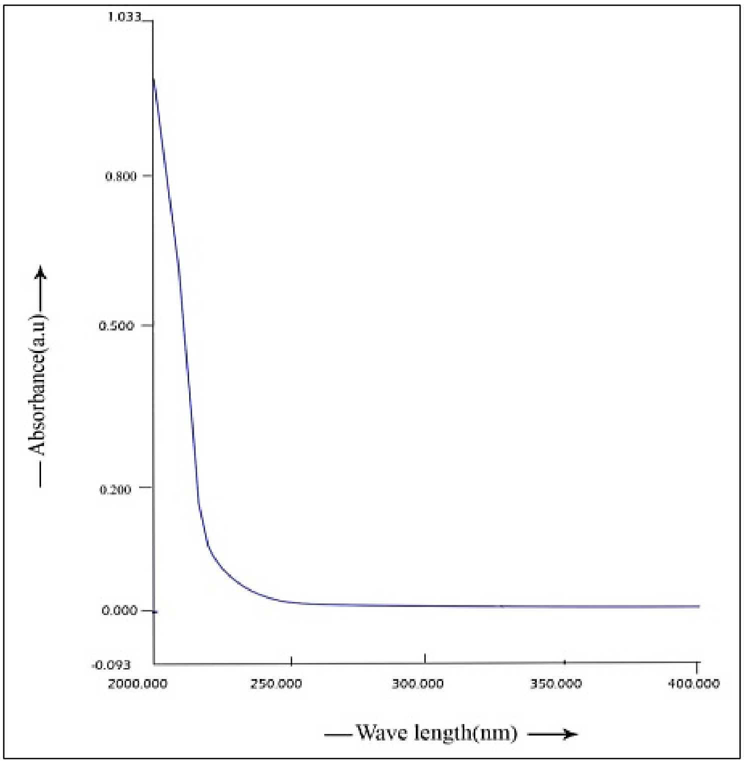

***Fig. 1:** UV-VIS Spectra of AA Copolymer.*

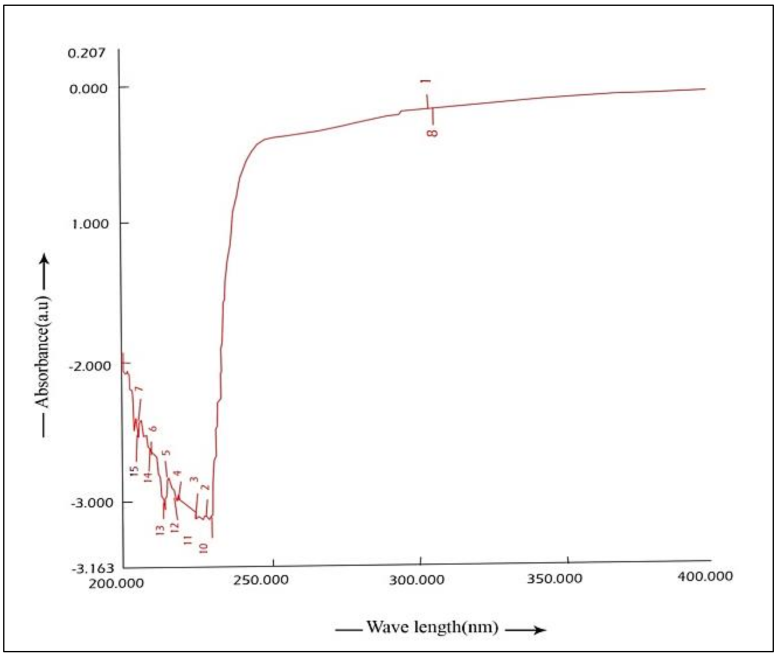

Fig. 2: *UV-VIS Spectra of AA-Pb Composite.*

Table 1: *FTIR Absorption Bands of AA- Pb Complexes.*

S. No.	Band position	Intensity	Assignment
1	3368,3212	Medium	SO_3H and NH groups
2	2920,2860	Shoulder	CH_3/CH_2 groups
3	1763,1693,1635,1593	Intense and medium	C=O/C-O
4	1493	Medium	CH_3
5	1265,1245	Medium	$CH_2/CH/SO_3H$
6	1206,1145	Medium	C-O-C/CH
7	1097,	Medium	SO_3H groups
8	990		
9	880		
10	624,502,440	Weak	Pb-O

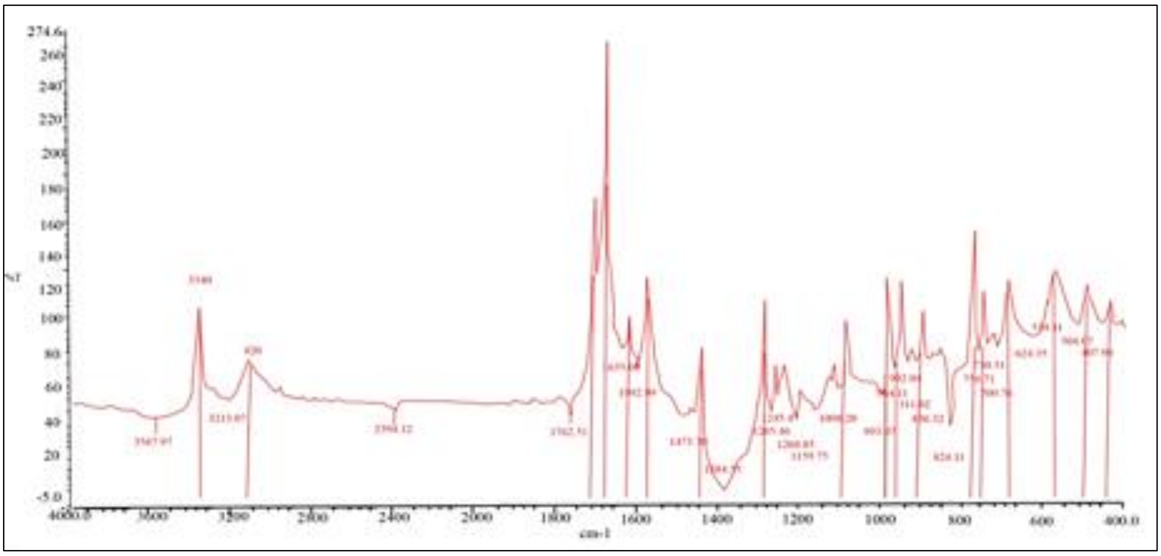

Fig. 3: *FTIR Spectra of AA Copolymer-Pb Composites (less doped).*

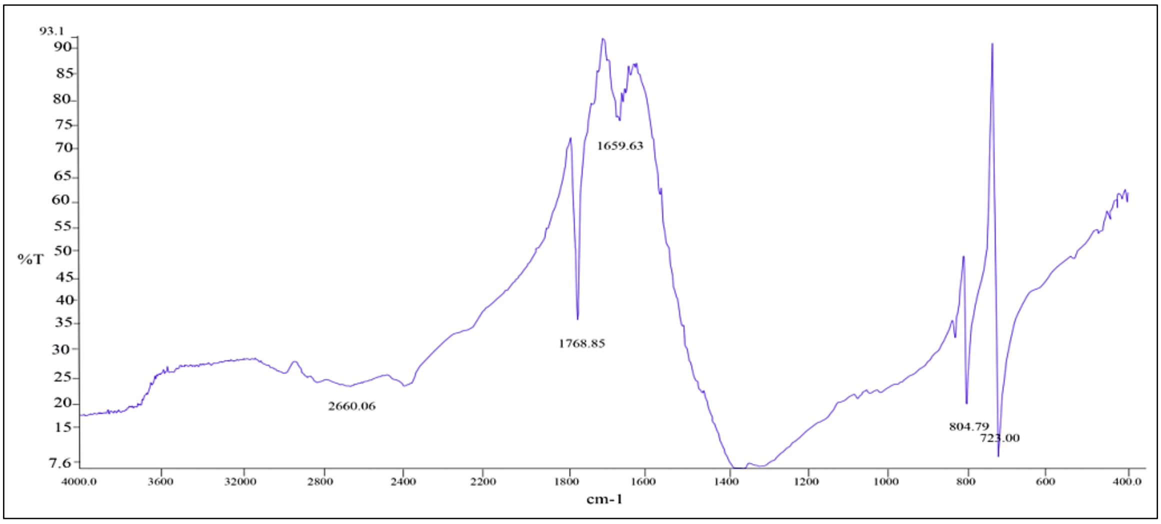

Fig. 4: *FTIR Spectra of AA-Pb Composites (medium doped).*

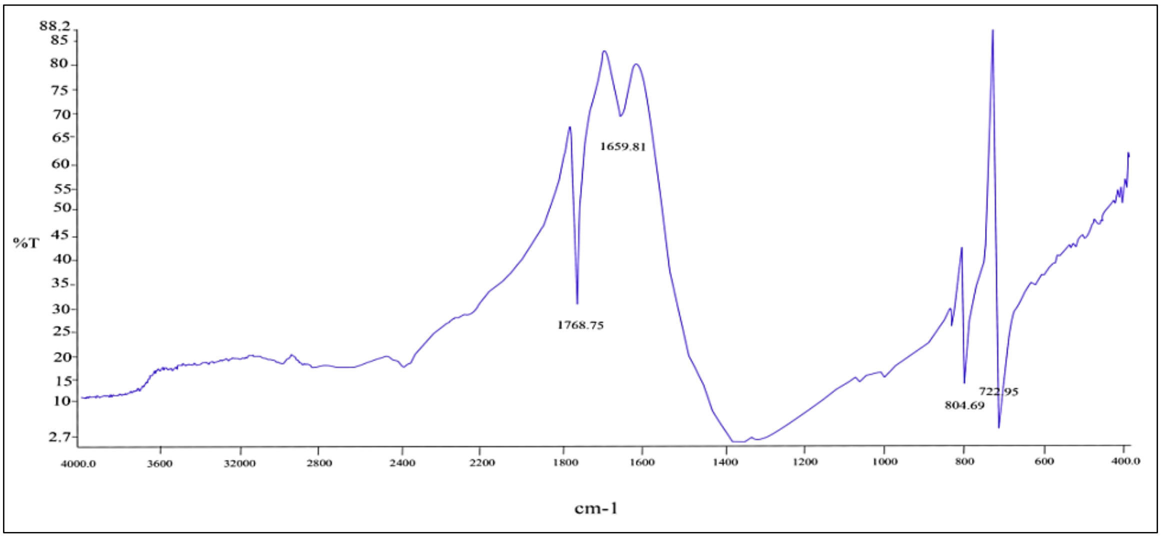

Fig. 5: *FTIR Spectra of AA-Pb Composites (high doped).*

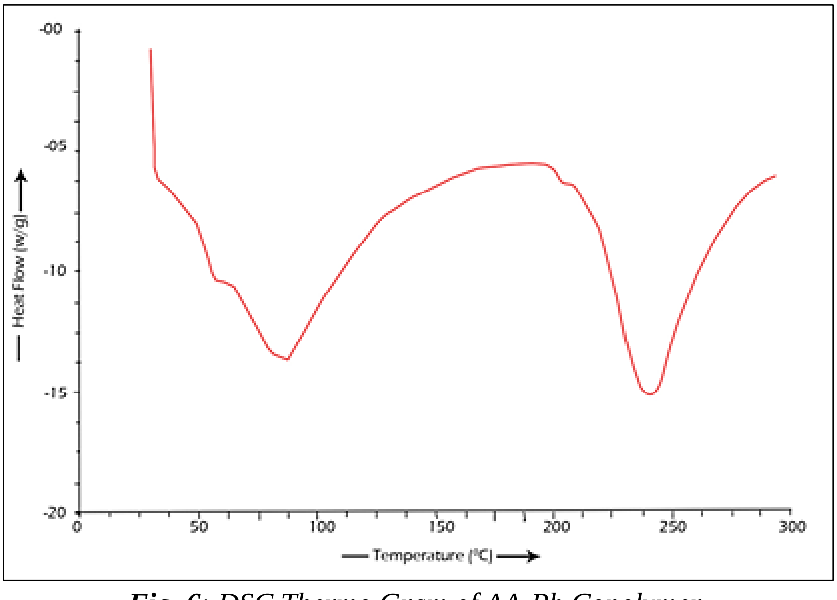

Fig. 6: *DSC Thermo Gram of AA-Pb Copolymer.*

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THERMAL STUDIES

DSC thermo gram of AA copolymer is as shown in Figure 6. The thermo grams consist of endothermic peak centered around 90^0C and 240^0C. Endothermic peaks at 90^0C corresponding to dehydration of copolymer while second endothermic peak at 240^0C is due to chemical transformations occurring in copolymer due to thermal treatment. DSC thermo gram of AA-Pb composite is shown in Figure 7. It consists of a base line shift at 70^0C corresponding to T_g followed by endothermic peak at 240^0C. Additionally two more endothermic peaks are observed at 304^0C. The results suggest that due to incorporation of Pb particles, the thermal and chemical transformations are delayed. Therefore, AA-Pb complex is thought to be thermally more stable than copolymer.

SEM STUDIES

Morphology of AA and AA-Pb composites is examined by SEM technique. The morphology under different conditions is as shown in Figure 8. The studies indicate that lead nano particles lay on the surface of AA matrix while some of them are appeared to chemically bound to the copolymer matrix.

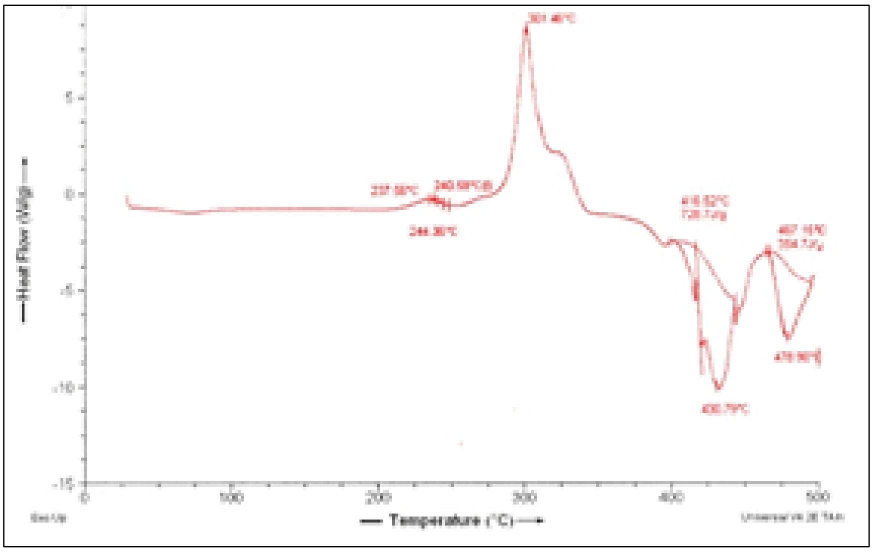
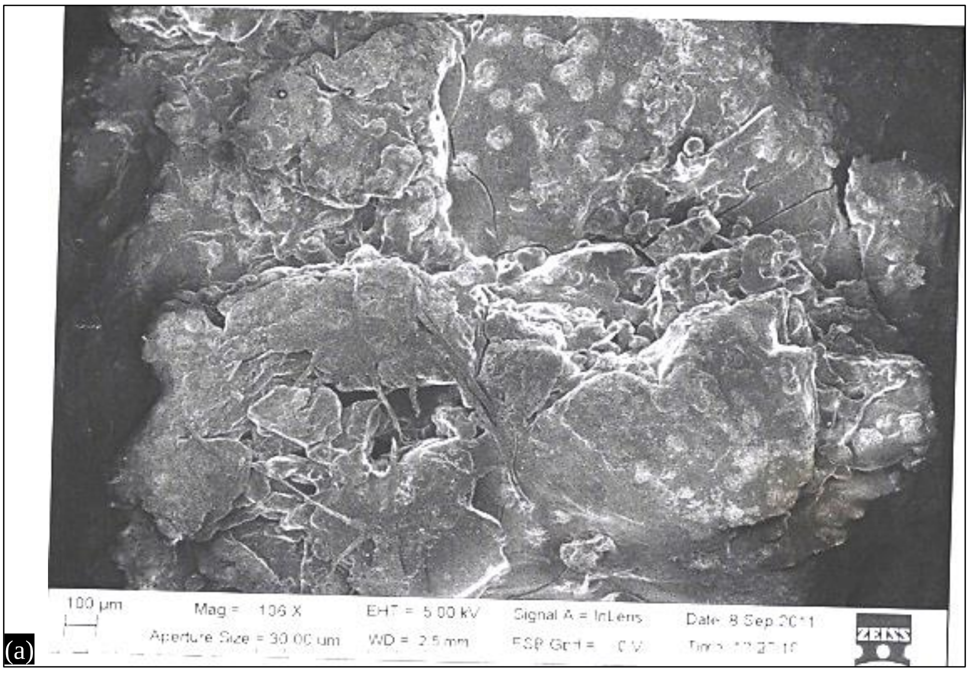


Fig. 7: *DSC Thermo Gram of AA-Pb Composite.*



(a)

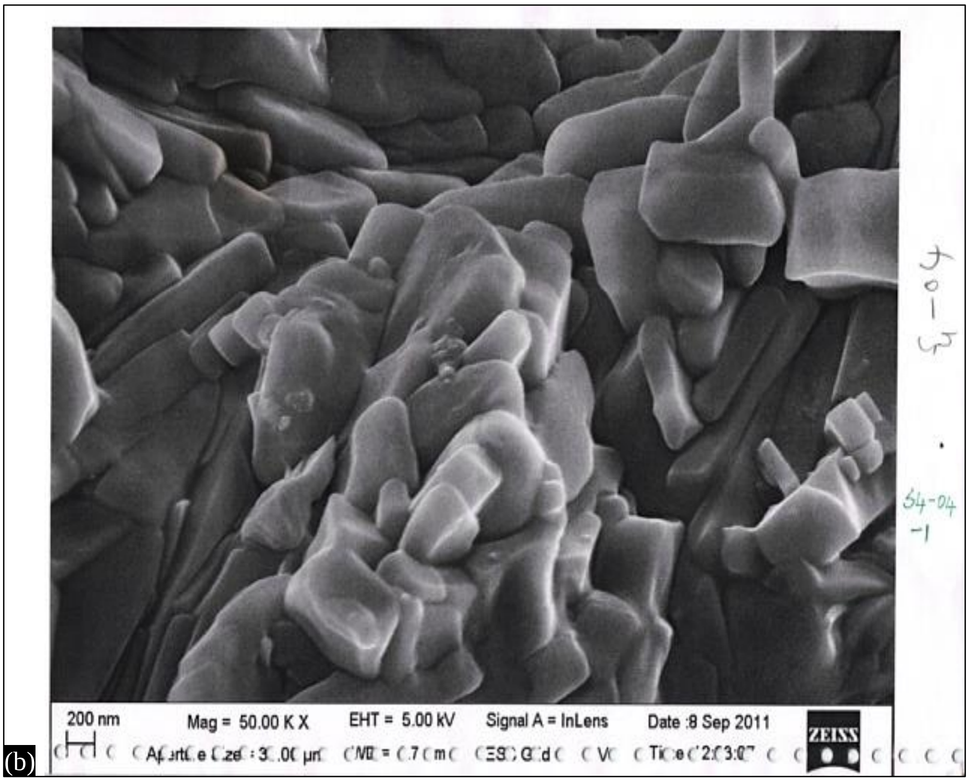


Fig. 8: *SEM Micrographs of (a) Copolymer, (b) Complex.*

CONCLUSION

In conclusion, we have successfully synthesized the AA-Pb complex with more thermal stability. Spectroscopic studies confirm the presence of lead nano particles are embedded in AA copolymer matrix. Due to their presence, thermal stability of copolymer is found to increase.

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