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OPTICAL AND MICROSTRUCTURAL STUDIES ON $COSO_4.7H_2O$ FILLED EPOXY

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ABSTRACT:

I. Background:
Hybrid materials belong to a class of modern novel materials, which are mixtures of materials at a molecular or nanoscale level. An organic component in the form of polymer and an inorganic component together make a hybrid material. The best properties of both the components can be brought together by the process of hybridization. In the present work, the organic component is chosen to be an epoxy polymer and inorganic component is cobaltous sulfate heptahydrate ($CoSO_4.7H_2O$). Epoxy is mainly known for its adhesive and mechanical properties. Incorporation of $CoSO_4.7H_2O$ can induce various properties which are solely not possible by epoxy polymer.

II. Results:
Epoxy-$CoSO_4.7H_2O$ hybrid polymer films, with various weight percentages (wt. %) of $CoSO_4.7H_2O$ (from 0.28 wt. % up to 5.00 wt. %) were prepared by physical blending method. Optical characterization of the prepared epoxy-$CoSO_4.7H_2O$ hybrids were carried out by UV-Vis spectroscopy and Fourier Transform Infrared (FTIR) spectroscopy. Microstructural studies were carried out by Scanning Electron Microscopy (SEM). At a filler level (FL) of 0.56 wt. %, the band gap of the material was found to decrease drastically compared to unfilled epoxy, showing the better complexation of inorganic component with epoxy matrix. FTIR studies confirmed the crosslinking of resin and hardener to form epoxy polymer and also the shift of certain peaks, broadening of OH peak could possibly confirmed the formation of hydrogen bonding between epoxy and inorganic filler. SEM images showed the smoother surfaces indicating better compatibility and complexation between epoxy matrix and $CoSO_4.7H_2O$. Self-grown nanostructures were also seen for certain FLs, indicating possible change in the microstructure of epoxy due to incorporation of $CoSO_4.7H_2O$.

III. Conclusions:
Epoxy-$CoSO_4.7H_2O$ polymer hybrid films were prepared with easy synthesis method such as physical blending with low cost of synthesis. The prepared hybrids with better optical properties and lower band gaps can be possibly used in optical applications. Due to better mechanical properties and induced optical properties, the prepared hybrids can find use in various other applications.

Key words: Epoxy; Cobaltous sulfate heptahydrate; UV-Vis spectroscopy; FTIR;SEM

INTRODUCTION:

Epoxies are important thermosetting polymers, which are formed by the crosslinking reaction between resin and hardener. Epoxy polymers are used as matrix materials in many polymer composites due to their appreciable mechanical, thermal, adhesive and electrical properties [1]. Physical properties of pristine polymers can be effectively tuned by the addition of various fillers such as transition metal salts, semiconducting particles, which may be in the form of particles, fibers, whiskers. Reducing the size of inorganic constituent to that of organic building block leads to the formation of hybrid materials, which are homogeneous mixtures (unlike that of composites). Hybrid materials are also broadly defined as nanocomposites with intimately mixed organic and inorganic components [2]. In this work, hybrid organic inorganic material is formed by using epoxy as an organic component. Cobaltous sulfate heptahydrate ($CoSO_4.7H_2O$), a hydrated transition metal salt,is incorporated as an inorganic component (filler) in the epoxy polymer. The influence of inorganic component on optical and microstructural properties of pristine epoxy polymer is studied by various characterization techniques.

MATERIALS AND METHODS:

Epoxy polymer used in this work, is a commercial available two component (resin and hardener; separately packed) adhesive, which cures at room temperature. The epoxy resin is Diglycidyl ether of bisphenol A (DGEBA) based and the hardener used is amine based. The inorganic filler, Cobaltous sulfate heptahydrate pure with molecular weight 281.1 g/mol was purchased from Himedia laboratories Mumbai. Hybrid organic inorganic material using epoxy and $CoSO_4.7H_2O$, was prepared in the laboratory by a simple and low cost technique such as physical blending. Various quantities of filler (0.28 wt. % to 5.00 wt. %) were added to a specified amount of resin and the mixture was grinded in agate mortar. A measured amount of hardener (resin and hardener ratio of 5:4) was then added and thoroughly grinded. The prepared mixture of epoxy polymer and $CoSO_4.7H_2O$ was then casted in the form of thin film on a Teflon sheet. The casted film was allowed to cure for one day at room temperature, in order to get a crosslinked epoxy polymer hybrids. The prepared epoxy hybrid materials were characterized by UV-Vis absorption spectroscopy, Fourier transform infrared spectroscopy (FTIR) and Scanning electron microscopy (SEM).

RESULTS, DISCUSSION:

UV-Vis absorption:

JASCO V-670 UV-Vis-NIR spectrophotometer was used to study the UV-Vis absorption properties of the epoxy hybrid materials. Absorption studies were carried out in the wavelength range of 200-800 nm. Electronic transition from highest occupied molecular orbital (HOMO) to lowest unoccupied molecular orbital (LUMO) on absorption of photon of suitable frequency, corresponds to an absorption edge in the absorption spectrum.

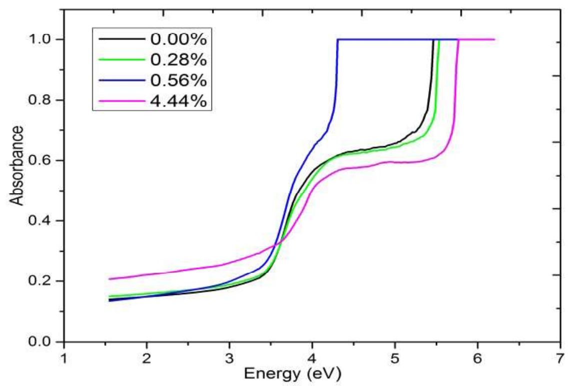

Fig.1. Absorbance versus Energy plots, for different FLs of $CoSO_4.7H_2O$ in epoxy hybrid films (expressed in weight percentage)

Fig. 1 represents absorbance versus energy plots for pure epoxy and epoxy hybrids with various filler levels (FLs). It can be seen that for FL of 0.56 wt. %, the absorbance has shifted to lower energy (higher wavelength) region, indicating a possible decrease in the optical band gap of the material. This drastic decrease in the band gap can be attributed to better interaction between polymer matrix and filler and also to possible change in the microstructure of pristine epoxy polymer. Band gap (E_g) values were calculated by famous Mott-Davis equation. Direct forbidden energy gap was found to decrease from 5.23 eV (for pure epoxy) to 4.16 eV (FL of 0.56 wt. %) and energy gap for indirect allowed transitions decreased from 5.05 eV (pure epoxy) to 4.12 eV (FL of 0.56 wt. %). Multiple absorptions can also be seen for higher FL of 4.44 wt. %.

Fourier transform infrared spectroscopy (FTIR):

FTIR technique is very efficient, non-destructive technique to study the crosslinking reactions in epoxies and also to identify the molecular groups present in the composites and polymer hybrid materials. Thermoscientific's Nicolet 6700 FTIR instrument was used for FTIR studies. Fig. 2 represents FTIR spectra of epoxy hybrids.

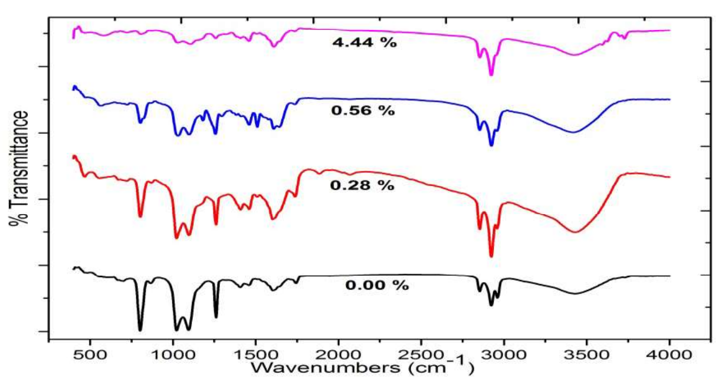

Fig.2. FTIR spectra of pure and filled epoxy hybrids with mentioned FLs of $CoSO_4.7H_2O$ in epoxy hybrid films (expressed in weight percentage).

DGEBA based epoxy resin is characterized by the presence of FTIR peak due to oxirane ring (the main feature of epoxy monomer) stretching at 3057 cm^{-1} and peak at 915 cm^{-1} corresponds to C-O stretch of oxirane ring. The consumption of these two bands in FTIR spectra indicates the cure or crosslink reaction between resin and hardener. Broadening of OH peak at wavenumber of 3500 cm^{-1} , and shift of carbonyl (C=O) peak to lower wavenumber indicates interaction between epoxy and $CoSO_4.7H_2O$ through C=O group, with possible formation of hydrogen bonds [3].

Scanning electron microscopy (SEM):

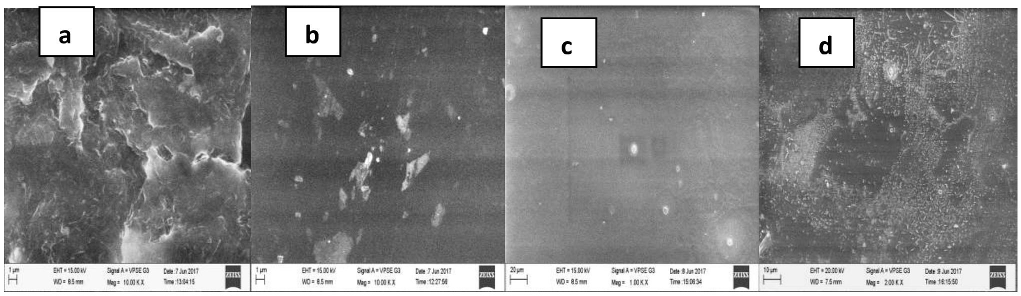

Fig.3. SEM images for a) pure epoxy b)0.28 wt. % FL c) 0.56 wt.% FL d) 4.44 wt. % FL

Microstructural characterization of prepared epoxy hybrid materials was carried out by EVO MA18 scanning electron microscope. Fig. 3 represents SEM images of pure epoxy and epoxy hybrid materials. It can be seen that surface roughness has decreased considerably with increase in FL. At higher FL of 4.44 wt. %, formation of nano-needles is seen. This indicates better interaction and influence of $CoSO_4.7H_2O$ on epoxy polymer.

CONCLUSION: Epoxy polymer, incorporated by $CoSO_4.7H_2O$ at various FLs, showed better optical and microstructural properties. These properties suggest the possible use of material for various optical and optoelectronic applications.

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