

Analysis of Microstructure of Fumed Silica Reinforced Polyester Composites

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Abstract

Fumed silica reinforced unsaturated polyester composites are developed by simple mechanical stirring and compression molding technique respectively. Investigation on morphological characteristics of fumed silica reinforced polyester composite is presented. The effect of fumed silica taken as different weight fractions and their interactive influences on the morphological characteristics of these composites has been studied. The results showed that amorphosity is inversely proportional to crystallinity with increasing the filler loading. The study revealed that fumed silica particle addition in polyester composite has dramatic effect on the microstructure of polyester fumed silica composites.

Keywords: Fumed silica, unsaturated polyester, amorphosity, filler etc.

“1. INTRODUCTION.”

Modern structural composites, mostly referred to as ‘Advanced Composites’, are a blend of two or more components, one of which is made up of stiff, long fibers, and the other, a binder or ‘matrix’ or ‘resin’ which holds the fibers in place. The fibers are relatively strong and stiff compared to the matrix and are generally orthotropic (having different properties in two different directions). The fiber used for advanced structural composites is long, having length to diameter ratios of over 100. Unsaturated Polyesters (UPR) are thermosetting resins. They are used for making double or triple bonds. They are used as casting materials, fibreglass laminating resins and non metallic auto-body fillers. These are based on dibasic acids and dihydric alcohols. The polyester unit formed must be capable of copolymerizing with a vinyl-type monomer, thereby yielding a vinyl-polyester copolymer or simply cured polyester having a thermo set structure. Fiberglass-reinforced unsaturated polyesters find wide application in bodies of yachts and as body parts of cars. Fillers are used to extend a material and to reduce its cost.

Filler will always modify the mechanical properties of the final filled products, or composites.

“2. EXPERIMENTAL.”

2.1 Materials Used

Unsaturated Polyester Resin procured from Crest Composites & Plastics Pvt. Ltd., Mumbai and Fumed Silica Powder procured from Amorphous Chemicals Pvt. Ltd., Haryana

2.2 Preparation of Composite Samples

Different sheets of polyester resin reinforced with varying amounts of fumed silica filler i.e. 2%, 4%, 6%, 8% (by weight of resin) used MEKP (1-1.5 % by weight of resin) and Cobalt Octoate (0.5% by weight of resin) were prepared and tested. Filler amount was limited to 8% in composite sheet because as the filler concentration increased to 8%, the gelation occurred rapidly and mixture became thick and losing fluidity. The compositions of composite sheets were given in table 1.

“Table 1: Composition of various polyester fumed silica filler samples”:

Sample identification	Amount of resin used	Amount of fumed silica used
S ₁	82	0
S ₂	80	2
S ₃	78	4
S ₄	76	6
S ₅	74	8

“3. SAMPLES CUTTING FOR TESTING.”

Sample for X-Ray Diffraction: The samples were sectioned in machine shop and cut it into the rectangular shape with dimensions of 30mm x 25mm. After cutting the samples, the edges were cleaned properly with the help of fine grit paper of 400 grades. The test was carried out in X-Ray Diffractometer.

Sample for FTIR Testing: FT-IR spectra were recorded on a Fourier Transform Infrared Spectroscopy (MAGNA 506 Nicolet USA). The mean size and the zeta potential of the NFS and MNFS-2 were measured with Zetarsizer Nano series (MAL VERN).

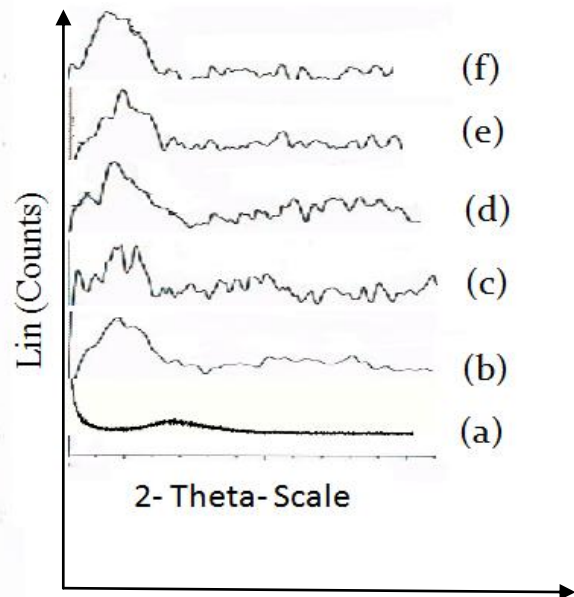
“4. CHARACTERIZATION OF COMPOSITE SAMPLES.”

The composite samples were tested for their morphological behaviour using X-Ray Diffractometer and Fourier transform infrared spectroscopy (FTIR).

“5. RESULTS.”

X-RAY DIFFRACTION

X-ray diffraction was widely used technique for polymer characterization provided information about both crystalline and amorphous states. X-rays were high-energy photons having short wavelength ($h=0.05-0.25\text{nm}$) that interact with electrons. When X-ray beams were focused on material, some electrons would be absorbed, some would transmitted unmodified. The XRD pattern of fumed silica powder, unsaturated polyester and unsaturated polyester-silica composites contained 2%, 4%, 6%, 8% filler contents by weight of resin were shown in figure 1.



FTIR SPECTROSCOPY:

Fourier transform infrared (FTIR) spectroscopy is a measurement technique that

It was observed that the composite was crystalline in nature as lowest peaks appears at an angles 4.879° , 4.779° , 4.734° and 2.653° corresponding by addition of filler 2%, 4%, 6% and 8% (total weight of the resin). On further addition of fumed silica, there was decrease in crystalline peak, indicated a better heat transfer from mold wall due to less sticking of the mixed material to grit paper. The addition of SiO_2 particles may induce a significant decrease in the crystallinity of composite material. Hence in other words intensity decreases with increases the filler concentration which shows the lower crystallinity due to d, spacing between the planes in the atomic lattice according to Bragg law ($n\lambda = 2d \sin \theta$, where n is an integer; λ is the wavelength of X-ray; d is the spacing between the planes in the atomic lattice; and θ is the angle between the incident ray and the scattering planes). It was observed from XRD results that the electrostatic interaction between the materials were very strong due to shifting of diffraction peak towards the small angle, shows the resulted material was intercalated composites.

FTIR SPECTROSCOPY ANALYSIS

The spectroscopy methods consist of dispersing a radiation from a source and passing it over a slit system that isolates a narrow frequency range falling on the detector. By using a scanning mechanism, the amount of energy transmission

through a sample as a function of frequency, known as the spectrum, is obtained and compared with the spectrum characteristic for each functional group of thermosetting resins. The FTIR pattern of fumed silica, pure unsaturated polyester sheet and unsaturated polyester-fumed silica composites with varying proportional of fumed silica filler content 2%, 4%, 6%, 8% by weight of resin are analyzed as shown in figure 2 (a) and figure 2 (b) .

FTIR spectra of pure unsaturated polyester resin were shown in figure 2 (a). Different peaks were observed representing the presence of various groups, i.e. C-H, C-N, C=O, N-O in the composites. The peaks obtained by FTIR spectra at 1934.96 cm^{-1} , 1797.66 cm^{-1} , 1685.66 cm^{-1} , 1551.98 cm^{-1} represents the presence of C-N group, C=O group, C-H group & N-O group respectively. It was observed that the peaks at 1685.66 cm^{-1} indicated stretching vibration modes where as peak 1551.98 cm^{-1} indicated deformation vibration mode.

FTIR spectroscopy pattern of unsaturated polyester contained 2% fumed silica (total weight of resin) was shown in figure 2 (b). The FTIR showed the different peaks at 3304.34 cm^{-1} , 2746.37 cm^{-1} , 2354.76 cm^{-1} , 1927.73 cm^{-1} , 1750.69 cm^{-1} and 1631.46 cm^{-1} indicated the presence of different groups, i.e. OH group, C-H group, C-N group & C=O group in composites. The peaks obtained by the FTIR spectra at 2746.37 cm^{-1} , 1750.69 cm^{-1} represents the presence of C-H group, C=O group respectively. The peaks obtained at 3304.34 cm^{-1} and 1631.46 cm^{-1} showed the presence of OH (alcohols) groups. It was observed that the peaks at 1750.69 cm^{-1} 3304.34 cm^{-1} indicated stretching vibration modes where as peak at 2746.37 cm^{-1} indicated symmetric stretching vibration mode.

FTIR spectroscopy pattern of unsaturated polyester containing 4% fumed silica (total weight of resin) was shown in figure 3 (a). Different peaks were observed representing the presence of various groups, i.e. N-H, OH, C-H, C=O in the composites. The peaks obtained at 2340.57 cm^{-1} and 1960.25 cm^{-1} showed the presence of C-N group of nitrile in the polyester. The peaks obtained by FTIR spectra at 3521.73 cm^{-1} , 3217.39 cm^{-1} , 2819.26 cm^{-1} , 1747.08 cm^{-1} , 1414.68 cm^{-1} represents the presence of N-H group, OH group, C-H group, C=O group and alkyl groups respectively. It was observed that the peaks at 3521.73 cm^{-1} and peaks at 1747.08 cm^{-1} indicated stretching in vibration modes.

FTIR spectroscopy pattern of unsaturated polyester contained 6% fumed silica (total weight of resin) was shown in figure 3 (b). The FTIR showed the different peaks at 3903.18 cm^{-1} , 3628.17 cm^{-1} , 2703.64 cm^{-1} , 2429.59 cm^{-1} , 2329.62 cm^{-1} , 2044.44 cm^{-1} , 1870.62 cm^{-1} , 1752.54 cm^{-1} ,

1626.27 cm^{-1} and 1456.64 cm^{-1} indicated the presence of different groups, i.e. N-H group, C=O group, C-H group, C=H group & $-\text{CH}_3-\text{CH}_2-$ group in composites. The peaks obtained at 3903.18 cm^{-1} , 3628.17 cm^{-1} , 2429.59 cm^{-1} due to the presence of N-H group in the polyester. The peaks obtained by the FTIR spectra at 1870.62 cm^{-1} , 1752.34 cm^{-1} in the presence of C=O groups due to maleic anhydride. It was observed that the peaks at 2429.59 cm^{-1} , 1870.62 cm^{-1} and 1752.34 cm^{-1} indicated stretching vibration modes where as peak at 1456.64 cm^{-1} indicated deformation vibration mode.

FTIR spectroscopy pattern of unsaturated polyester containing 8% fumed silica (total weight of resin) was shown in figure 3 (c). Different peaks were observed representing the presence of various groups, i.e. C=O group, C-N group in the composites. The peaks obtained at 2336.63 cm^{-1} , 2121.76 cm^{-1} and 1973.14 cm^{-1} due to the presence of C-N group of nitrile in the polyester. The peaks obtained by FTIR spectra at 3646.85 cm^{-1} represents the presence of C=O group. It was observed that the peaks at 3646.85 cm^{-1} indicated stretching in vibration modes where as peak 2121.76 cm^{-1} indicated isocyanides vibration mode.

“6. CONCLUSION”:

The XRD pattern showed that fumed silica powder exhibits highly amorphous behavior and showed highest amorphous peak at an angle of 22.793° . The pure unsaturated polyester exhibits crystalline behavior and showed crystalline peak at an angle of 4.962° . It is analyzed that XRD pattern indicated that the crystallinity decreases with increase in the filler content as the lower peaks are observed at various angles. In other words intensity decreases with increases of filler concentration.

The FTIR showed that the OH and Ester functional groups were present in the polymer structure (unsaturated polyester resin). It was observed from the absorption peaks that as the filler content increases functional group tends to decrease in the composite. The size of the peaks in the spectrum was a direct indication of the amount of the material present. It was visualized from the absorption peaks that the functional groups are present in the composite containing 2% and 4% filler (total weight of the resin). After 4% filler content, the functional groups tend to decrease in the composites as the gelation occurs vigorously in the composite.

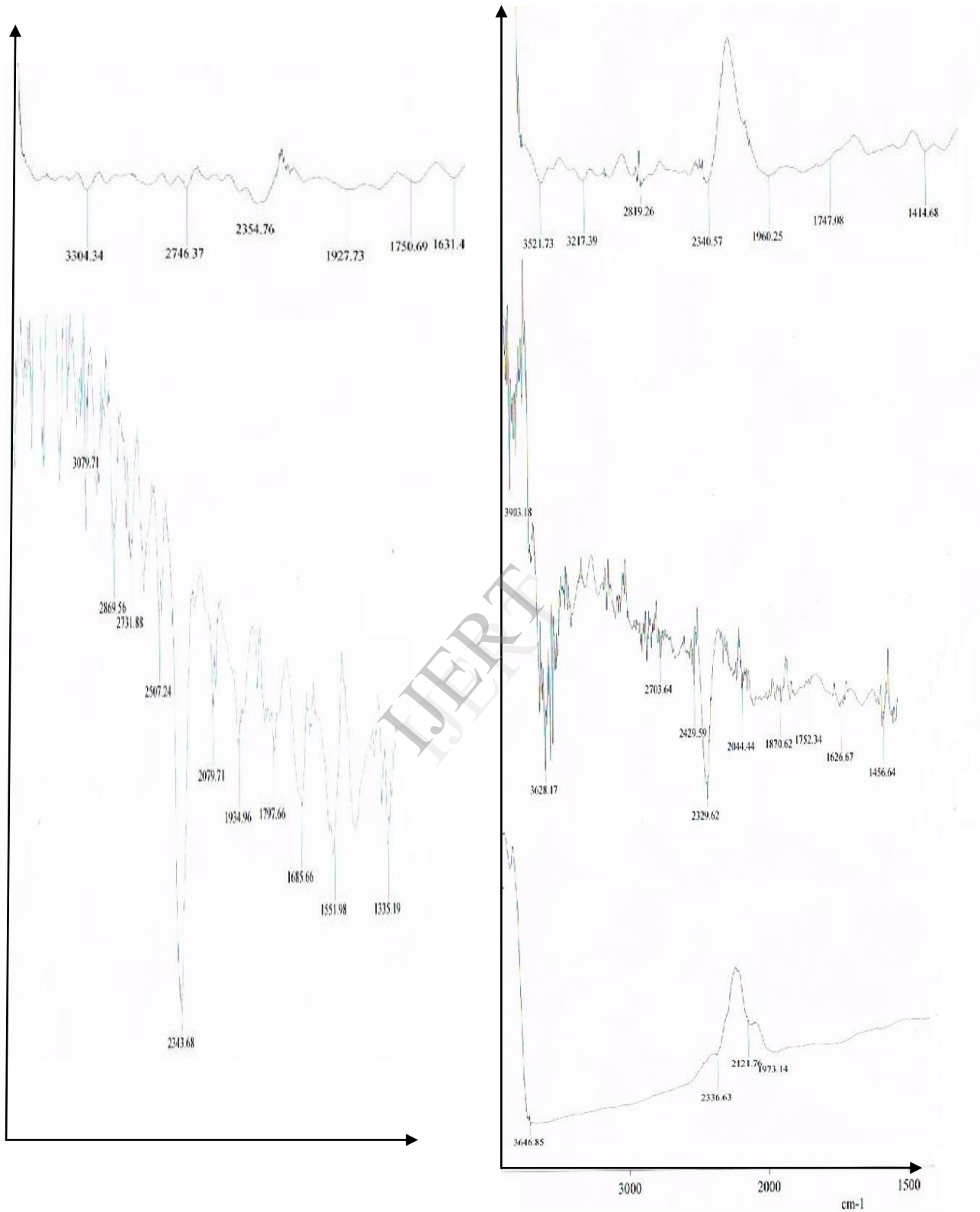


Figure 3: Spectroscopy pattern of composite sheets containing filler contents 4% (a), 6 % (b) and 8% (c)

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