"The Effect of Fly-ash on Morphological Characterization of Fly-Ash Based Polymer Composites"

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Abstract:

In this study the effect of fly-ash on structure and surface morphology of different type of fly ash reinforced polystyrene composites have been investigated .XRD analysis is reported the crystalline index increases with increasing the concentration of the fly ash. FTIR spectra of pure polystyrene pure fly ash and fly ash based composites have been observed and analyzed. The shifting of parent peaks were observed in composite films. The shifting may come from the results of physical bonding between polystyrene and fly ash.

Keywords: FTIR, XRD, PS, FA.

1. Introduction

The composite concept allows to produce stiffer ,stronger and tough materials with improved elevated temperature capability. Polymers have low strength and stiffness and are ductile .In resent time researchers are exploring the possible use of waste materials like fly ash in composites to produce good materials for wide range of application (Chauhan et al. 2010; Singla Manoj et al 2010). The fly-ash is produced as industrial by product from the combustion of coal in thermal power plant and properties of fly-ash depend primarily on the type of coal burned, the type of combustion of equipment used and the type of flyash collection mechanism (Celik Ozlem et al. 2008). The major component of the fly-ash is silica and alumina and oxides of iron, calcium, magnesium along with elements like Ti, carbon, Mg. etc. (Singla Manoj et al. 2010).b Fly ash can increase the strength and making these light weight composites very desirable for the automotive , aircraft and aerospace industries (Abdullah et al. 2010).In developing composites using most environmentally friendly wastes as reinforcing fillers and thermosetting polymers as matrixes. Recent investigations of polymer- based composite materials have opened new routes for polymer. Formulations and have allowed the manufacture of new product with optimal properties for special applications (Imoisiliet al.2012)

2. Experimental

2.1 Materials

For the preparation of the polymeric composite films ,the commercially available polymer Polystyrene obtained from-and fly ash in powder form has produced from thermal plant, Singroli (M.P.) used without further purification.

2.2 Preparation of specimen

Fly ash in different concentration was added to the polystyrene to yield the composite of required concentration and labled as Pure polystyrene(PS),Pure fly ash (FA), 1% FA & 99% PS,3%FA & 97% PS and 8%FA & 92% PS. For 1, 3 and 8 weight percentage concentration of fly ash respectively. The sol-gel method (Bajpai R et al. 2002; Ramrakhiani M et al. 2005; Patel Arunendra Kumar et al. 2011) has been utilized to prepare the composite films of pure polystyrene(PS) and fly ash based composite films. 1,2 Dichloroethane has been taken as solvent. Fly ash and polystyrene were mixed in solvent 1,2 dichloroethane with the help of electric motor operating at 200 rpm in air atmosphere at 60^0C for 6 h. A known quantity of homogeneous solution poured in glass mould of size 5x5 cm^2 and kept in an electric oven at 70^0C temperature for 24h. Samples of 4x2 cm^2 size and 1mm thickness were cut from the pallets and kept in the air tight polyethylene bags.

2.3 FTIR Spectroscopy

The structural characterization of pure polystyrene, fly ash powder and their composites of various ratio were recorded with NICOLET 6700 (Thermo scientific) with resolution of 4 cm^{-1}

2.4 X-ray diffraction

The X-ray diffraction studies of prepared pure polystyrene ,pure fly ash and fly ash reinforced polystyrene composites were carried out witht the RIGAKU 18KV diffract meter, X-ray powder diffract meter with CuKα radiation (wave length 0.15405 nm) performed at 40kV and 100 mA. The diffraction pattern is recorded in the 2θ range from 10^0 to 70^0 with scanning speed of 2^0 per minute.

The crystallinite has been measured on the premise that increasing amorphousness tends to broaden the line width whereas increasing crystallinity increasing the intensity. The height (C_rH) of the main peak above its adjacent minimum represents the crystallinity of the sample and the width (AmW) of the peak at this adjacent minimum is considered

to represent the amorphousness of the sample. The crystallinity CrI, is calculated from the Eq.2:

$$CrI = [1 - t x$$
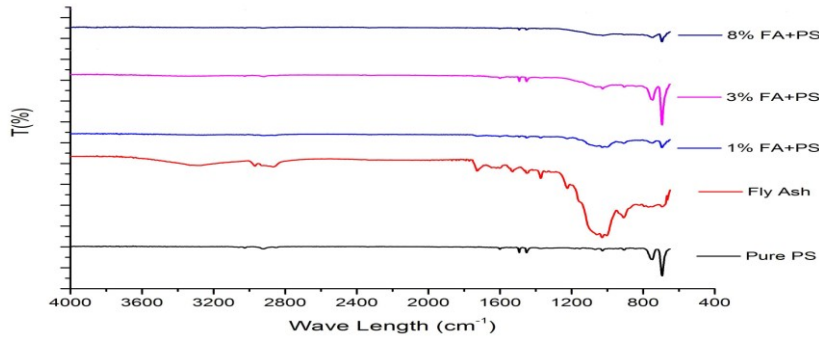
$$(A_m W)/(C_r H \quad ……………………………………...(2)$$

Where t is the scale factor relating the height of $C_r H$ to full scale (total blackness)(statton 1967).From the diffraction patterns of the various samples, the inter planer distance d was computed for different peaks. Bragg's equation 3 was used to determine the distance between two successive planes from which the X-rays were diffracted:

$$2d\sin\theta = n\lambda \quad …………………………………………………...(3)$$

Where λ is the X-ray wavelength, θ is the Bragg's angle and n is the order of diffraction .d was calculated from the first order (n = 1) for various peaks obtained in the diffraction patterns (Eq. 4):

$$d = \lambda/(2\sin\theta) \quad …………………………………………………………….(4)$$

The relationship between the crystallite size and the X-ray line broadening was determined from the Scherrer 's equation:

$$D = k\lambda / \beta\sin\theta \quad ………………………………………………………… (5)$$

Where, D is crystallite size , λ is the wavelength, β is the angular line width of half maximum intensity , and θ is the Bragg's diffraction angle ,k is the constant with a value of 0.89 (Dubey et at. 2008)

Fig.1 FTIR analysis of pure polystyrene(PS),Fly ash(FA) powder,1% FA and PS,3%FA and PS,and 8%FA and PS

3 Results and Discussion:

3.1 FT- IR Spectroscopy

The FT-IR spectrum of pure polystyrene in (fig .1) indicates the details of functional groups present in the synthesized polystyrene. The absorption band indicating CH bands appear at approximately 1000 cm^{-1} for the in plane bends and about 675 cm^{-1} for the out of plane bend.(Sarkarmoinuddin et al.2013). Vinyl stretch at 910 and 1000 cm^{-1} and aeromatic ring stretch at 1496 cm^{-1} are present.The band at 1427 cm^{-1} attributed to Phenyl (Yongfeng Li et al.2011). The FT-IR spectra of fly ash shows the three characteristic bands centered around 1100, 1000 and 660 cm^{-1}. The strong and broad band at 1000 cm^{-1} is due to (Si-O-Si) asymmetric stretching vibrations and the other two bands at 1100 and 660 cm^{-1} are attributed to (Si-O) and (Al-O) vibrations respectively (CelikOzlem et al. 2008) .A band towards 1453 cm^{-1} has been assigned to the presence of sodium bicarbonates. Stretching and deformation modes of water were detected at 3500and 600 cm^{1}(Fernandez Jeinenez et al. 2005).FT-IR spectra of various concentrations of polystyrene–fly ash composites have been taken. For 1 weight percent concentration of fly ash the peaks appeared at 780 and 1024 cm^{-1} representing Al-O vibration and SiO_2 asymmetric stretching respectively. The shifting in the pecks of characteristic frequency of polystyrene with respect to the pure polystyrene is observed at frequencies: 1350 and 1380 cm^{-1} representing the presence of Phenyl band and aromatic ring stretch respectively .These shift also observedfor higher concentration.

3.2 X-ray diffraction:

The structure and morphology is basically dependent upon regular arrangement of the atomic system or crystallinity. Polymers are in general amorphous orsemi crystallinematerial. Crystallization of polymer depends on the degree of missibility and mobility of crystallizable and non crystallizable component in the specimen.

The X-ray patternof pure polystyrene,fly ash powder and fly ash reinforced polystyrene composites are showen. In all the samples based on the fly ash reinforced polystyrene composites a broad peak observed between 15^0 to 24^0 .The fly ash powder gives the broad peak at 26.6^0 to 26.9^0 . In pure polystyrene a broad peak observed between 11^0 to 27.5^0 , yielding crystallinity index (C_rI) = 44.86%.Since the fly ash contains SiO_2 , Al_2O_3, $Ca(Co)_3$, Fe_2O_3 and other constituents which contributes to

crystalline phase, so reinforcement of fly ash within polystyrene increases the crystallinity of the prepared composites. The C_rI increases from 44.86 to 52.53% for the one weight percent concentration of fly ash in polystyrene .The interplaner distance "d" also increase from 4.43 A^0 to 4.50 A^0 and crystallite size (D) increase from 4.48nm to 8.87 nm. These parameters also increase for 3 wt. %.The higher concentration of fly ash (8 wt. %) within polystyrene gives the crystallinity index 31.33%,interplaner distance 4.59 A^0 and crystallite size 11.56 nm.
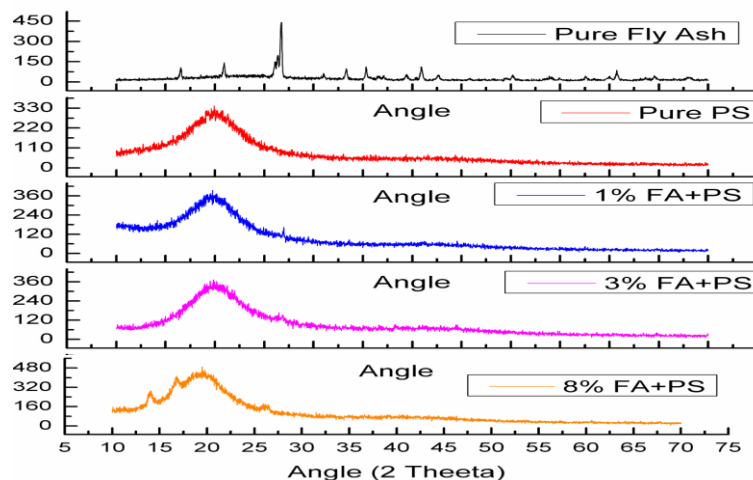
Fig.2 X-Ray diffraction pattern of pure fly ash,pure polystyrene,1%fly ash and PS,3%fly ash and PS,8%fly ash and PS

Conclusion

The outcome of the studies undertaken for the structural and morphological behaviour of fly ash reinforced polystyrene composite can be formed.

(1) The FT-IR studies of pure polystyrene, fly ash and fly ash reinforced polystyrene composite confirms the formation of new peaks and shifting of existing peaks which occurs in the prepared composites thus there is a better linkage of flyash molecules with polystyrene molecules confirming the development of stable composites.

(2) The XRD measurement indicates that there is a formation of crystalline region.Crystallinity Index and crystalline size also increases with increasing the concentration of fly ash .

All these studies confirms that the addition of fly ash within the polystyrene causes the enhancement in crystallinity and morphology yielding better microchemical properties.

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