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Preparation and characterisation of Isophthalic-Bi_2O_3 polymer composite gamma radiation shields

M.R. Ambika[a], N. Nagaiah[a,*], V. Harish[b], N.K. Lokanath[c], M.A. Sridhar[c], N.M. Renukappa[d], S.K. Suman[e]

[a] Department of Physics, Bangalore University, JB Campus, Bengaluru 560056, Karnataka, India
[b] Department of Physics, Government First Grade College, Shimoga 577201, Karnataka, India
[c] Department of Studies in Physics, University of Mysore, Mysuru 570006, Karnataka, India
[d] Department of Electronics & Communication, SJCE, Mysuru 570006, Karnataka, India
[e] Radiation Safety Systems Division, BARC, Trombay, Mumbai 400085, Maharashtra, India

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ABSTRACT

Bi_2O_3 filled Isophthalic resin based polymer composites of different weight % (0, 5, 10, 20, 30, 40, 50 & 60) were fabricated by open mould cast technique. Gamma attenuation study was carried out using NaI (Tl) gamma ray spectrometer for Cs-137. The shielding parameters such as attenuation coefficient, HVL & λ were investigated. The distribution of the filler within the matrix was studied using Scanning Electron Microscopy. X ray diffractometer and Fourier Transform Infrared Spectroscopy were employed to study the structural changes if any. The thermal stability and mechanical strength of the composites were investigated using TGA & UTM respectively. Dielectric properties and AC conductivity were also studied using LCR meter. The composites are found to be thermally stable upto 200 °C. There were no such structural changes observed and all the composites show very low conductivity. The mechanical strength of the composites was found to increase upon adding the bismuth oxide with a slight decrease when the concentration of the filler exceeds 40 wt%. Attenuation results reveal that, the shielding efficiency increases with the increase of the filler wt% and are comparable to those of the conventional shielding materials. Hence, Bi_2O_3 filled composites can be used for gamma shielding applications.

1. Introduction

Unwanted exposure to high energy radiations is always harmful to both living and non-living systems. High energy radiations find their application in nuclear industries, medical, spacecraft, agriculture and so on; where in radiation protection is mandatory. Lead, because of its high density, low cost & radiation shielding ability, has traditionally been used in the form of sheets, plates, foils, laminates, bricks and blocks (Harish et al., 2012; Martin, 2006; Chilton et al., 1984). But, do lack in usage flexibility, chemical stability, mechanical strength, heaviness and moreover is toxic. In view of this, from the past few decades, research is going on to find an appropriate shielding material that can replace lead. Polymer composites have now become an attractive tool in the field of radiation protection. Several researchers have used different types of polymer as matrix and fillers as reinforcements depending upon their application. Gwaily (2002) has studied gamma attenuation property of Galena-rubber composites for 0.662 MeV gamma rays and has reported 29.5 m^{-1} as the linear attenuation coefficient and 0.023 m as HVL for the highest Galena filled composite (500 phr). In a study conducted by Hussain et al. (1997) polyethylene glycol with 40% lead oxide composite show greater shielding ability for Co-60 gamma radiations and have reported that, these composites can act as shielding materials against low dose rates. Harish et al. (2009) have fabricated lead oxide filled unsaturated polyester based polymer composites for shielding gamma rays of 0.662 MeV and have found linear attenuation coefficient of 0.206 cm^{-1} for the 50 wt% PbO composite. Their shielding ability has also been compared with the conventional materials and has proved to exhibit good shielding property for lower and medium gamma energies. Epoxy – Ferrochromium slag composites were investigated for X ray (85 keV), Gamma (1250 keV) and neutron (2–10 MeV) radiation shielding effects by Korkut et al. (2013). They have reported that, 50% FeCr slag in epoxy resin exhibits good shielding ability and hence find its application in radiotherapy rooms, nuclear industry and radioactive source containers. In a study, Dubey et al. (2014) have synthesised polydimethylsiloxane/Bi_2O_3 flexible polymer composites for shielding

* Corresponding author.
E-mail address: nagaiahn@rediffmail.com (N. Nagaiah).

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of soft gamma rays (Am-241) and have found 90% gamma attenuation for the 70% Bi_2O_3 filled composite. In addition, several other researchers have used styrene-butadiene rubber (Abdel Aziz et al., 1991), Polyaniline (Hosseini et al., 2014), high density polyethylene (Udagani and Seshadri, 2012), Epoxy (Eid et al., 2013), Silicone rubber (EI-Fiki et al., 2015), as matrix and metal/metal oxides as fillers and have mainly focused on attenuation studies. Only, a very few studies have been conducted to understand the structural, thermal and mechanical stability of the composites. All these are essential in order to evaluate any material as an efficient radiation shield.

In the present study, the authors have selected Isophthalic resin as matrix because of its superior mechanical and chemical properties with an added advantage that, they can be cured into either fibre or sheet form in variety of ways without altering the physical properties of the final composite (Hansmann, 2003; Scheirs and Long, 2003). Bismuth oxide is chosen as filler because of its high density, high melting point, low conductivity, available in fine powder form and moreover non-toxic. The authors have made an attempt to study the gamma attenuation, structural, ac conductivity, mechanical and thermal characteristics of the bismuth oxide filled polymer composites.

2. Experimental methods

2.1. Fabrication of composites

2.1.1. Materials

Isophthalic resin belongs to the class of thermoset polymers which consists of Isophthalic acid, maliec anhydride, propylene glycol and styrene in the molar ratio 0.5, 0.5, 1.0 & 1.2 respectively (Scheirs and Long, 2003). It is a pale coloured, viscous liquid with 45% styrene content and with an added advantage of superior mechanical and chemical properties. The resin was procured from M/s Ashland, India. The filler Bi_2O_3 is a high dense metal oxide with density 8.9 g/cc and melting point of 817 °C and was procured from Sigma Aldrich. Both the resin and the filler used for the synthesis were of analar grade.

2.1.2. Preparation of polymer composites

A simple open mould cast technique was used to fabricate bismuth oxide filled Isophthalic resin based polymer composites (Harish et al., 2012; Jose et al., 2012). Finely powdered Bi_2O_3 of different weight % (0, 5, 10, 20, 30, 40, 50 & 60) were dispersed into the matrix initially by an electric blender and then using an ultrasonicator. Later, 2% of catalyst & 1.5% of hardener were added for the completion of the reaction process. The sample was allowed to cure at room temperature for 12 h and then post cured in a vacuum oven at 80 °C for 6 h. The samples were then cut into the required dimension for further characterisation.

2.2. Scanning electron microscopy

The composites thus prepared were initially analysed using Scanning Electron Microscope (FEI Quanta 200 ESEM, Netherlands) at both low and high resolution to check the binding and dispersion of the filler within the matrix.

2.3. Gamma attenuation studies

A well calibrated (Evans, 1955) gamma ray spectrometer (REXON Components, USA) comprising of 3 in.×3 in. NaI(Tl) detector coupled to a photomultiplier tube, preamplifier, spectroscopy amplifier and PC based MCA with a good geometry set up was used to study the attenuation characteristics of the composites for 0.662 MeV gamma rays of Cs-137. The various shielding parameters such as linear attenuation coefficient (μ), mass attenuation coefficient (μ_m), half value layer thickness HVL ($x_{1/2}$) and relaxation length (λ) were evaluated using the following relation.

$$I=I_O e^{-\mu x} \quad (1)$$

Eq. (1) is the basic shielding relation, Where, I_O is the initial photon intensity, I is the transmitted intensity of photons coming out from an absorber of thickness × cm & μ is the total linear attenuation coefficient (cm^{-1}) which accounts for all the interaction processes such as photoelectric effect, Compton effect and pair production which are the three principal modes of gamma ray interaction (Martin, 2006; Evans, 1955).

2.4. X-Ray diffraction studies

X ray diffractometer (Miniflex II, Rigaku Corporation, Japan) was used to study the structural changes if any due to the incorporation of bismuth oxide into the matrix. The X ray diffraction patterns of the pure polymer and the polymer composites filled with different wt% of bismuth oxide were recorded using the Cu-Kα radiation at wavelength λ=1.5406 Å at room temperature. From the XRD pattern, the average crystallite size D and the micro strain were estimated using Scherrer's formula and W-H plots (Prabhu et al., 2014; Choudhury and Sarma, 2009).

2.5. FTIR analysis

The chemical structure and the bonding between the matrix and the filler were investigated using Fourier Transform Infrared Spectroscopy (PERKIN ELMER). The FTIR spectrum of each sample was recorded in the range of 300–4000 cm^{-1} using KBr pellets.

2.6. Thermogravimetric analysis

Thermogravimetric analyser (PERKIN ELMER) was used to investigate the thermal stability of the composites. Alumina crucible was used as a sample holder. The thermograms for all the samples were recorded at a heating rate of 10 °C/min in the temperature range from room temperature – 700 °C and were then analysed.

2.7. AC conductivity measurements

The dielectric properties and AC conductivity measurements were carried out using High Frequency LCR meter in the frequency range 100 Hz-6 MHz at room temperature. The dimension of the sample was 5 cm dia and 3 mm thick. The measured parameters such as capacitance (C_p), dissipation factor (D) & resistance (*R*) were used to calculate the dielectric constant (Ɛ'), dielectric loss (Ɛ") and AC Conductivity (σ_{ac}) of the polymer composites using the relations found elsewhere (Hussain et al., 2015; Pradhan et al., 2008).

2.8. Mechanical studies

The mechanical strength of the composites was determined using Universal Testing Machine (UTM) (LR 50K-Lloyd Instruments, UK). Tensile and compression measurements were carried out as per the ASTM standard test D 3039/D 3039 M and D6641/D6641M-09 respectively. The test specimens for the tensile and compression measurements were in the form of rectangular bars of dimension 250 mm×25 mm×2.5 mm and 150 mm×25 mm×2.5 mm respectively placed in between the grips of the UTM and was pulled (tensile) and compressed (compression) until failure due to the application of load.

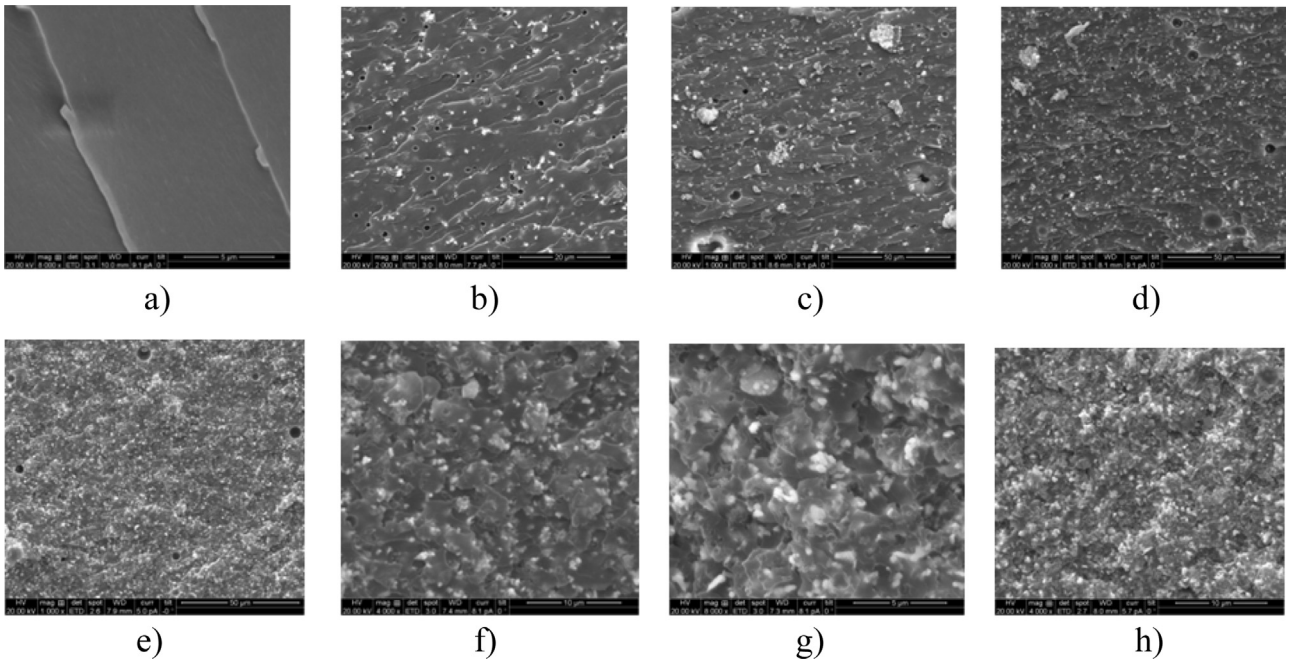

Fig. 1. (a–h) SEM images of ISO+Bi2O3 Polymer composites for 0, 5, 10, 20, 30, 40, 50 & 60 wt% respectively.

3. Results and discussion

3.1. SEM

The morphology and distribution of bismuth oxide–Isophthalic resin based polymer composites were investigated by SEM images (Fig. 1. a–h)..

The SEM image of pristine sample shown in Fig. 1a. clearly shows the presence of graded ridges. In addition, there are lighter streaks that depart from the ridges which may be attributed to the detachment of ribbon like strands from the ridges during fracture, which is a common fracture mode in neat thermosets (Harish et al., 2009). Fig. 1b–h represent the SEM images of ISO+Bi_2O_3 polymer composites. It is clear from these figures that, there is uniform distribution and good dispersion of the filler within the matrix. The radial fronts with the filler particles at one end are linked with one or the other particles along the path of the ridges which may be due to the crack propagation. In addition, it is also observed that, there are less pronounced ridges with increase in the filler loading due to decrease of tension in the matrix resulting in a smoother surface. On the other hand, Fig. 1g & h for the composites with ≥50% filler loading show the agglomeration of filler particle to form bigger particles and this has to be minimised for the better performance of the composites.

3.2. Gamma attenuation studies

Gamma attenuation study helps to evaluate the shielding performance of the polymer composites. It is clear from Fig. 2. that, the linear attenuation coefficient (μ) of the ISO+Bi_2O_3 polymer composites increases with increase in the filler concentration which is due to the increase in the density of the composite as μ is a density dependent factor (Harish et al., 2012). The shielding parameters such as μ_m, HVL & relaxation length have been evaluated as these are essential from practical point of view. The HVL values & the relaxation lengths are found to decrease with increase in the filler wt% and are as shown in Fig. 3 and they range from 7.11–2.77 cm and 10.26–4.0 cm respectively. The mass attenuation coefficient of the composites shown in Fig. 4. is density independent. μ_m also increases with increase in the concentration of the filler particles and this may be attributed to the increase in the bismuth content, a high Z metal that favours attenuation of gamma radiations.

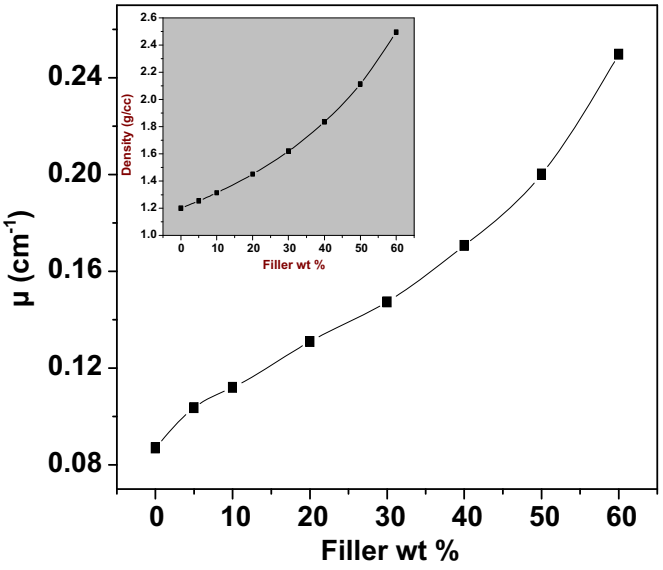

Fig. 2. Variation of linear attenuation coefficient of the composites w.r.t filler wt%.

All the composites do exhibit good shielding ability for the gamma rays of energy 662 keV. The shielding performance of the ISO+Bi_2O_3 polymer composites is compared with that of the conventional shielding materials and is as shown in Fig. 5. and is evident that, it is more than that of steel and concrete and almost corresponds to that of Baryte..

3.3. X Ray diffraction studies

The X ray diffraction spectra of the ISO resin and the polymer composites are shown in Fig. 6. A broad peak centred around 20.32° and diffused scattering from 32° to 60° as witnessed in Fig. 6a indicates the amorphous nature of the pristine sample (ISO+0%Bi_2O_3). This may be attributed to the intermolecular interaction within the long polymer chain structure with relatively large number of defects (Hemalatha et al., 2014). The XRD pattern of the polymer composites filled with

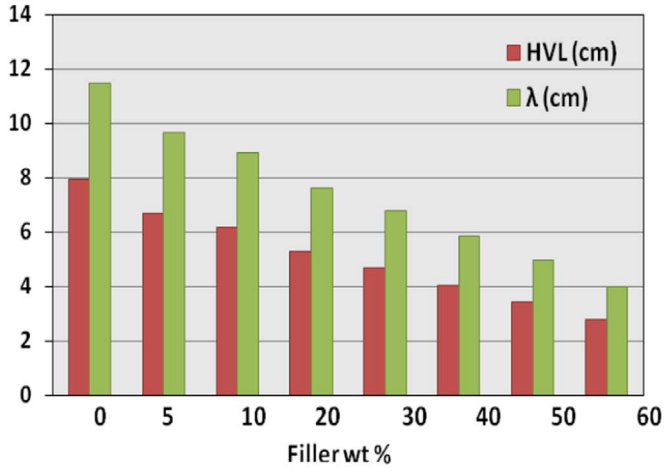

Fig. 3. HVL and relaxation length of the ISO+Bi_2O_3 polymer composites.

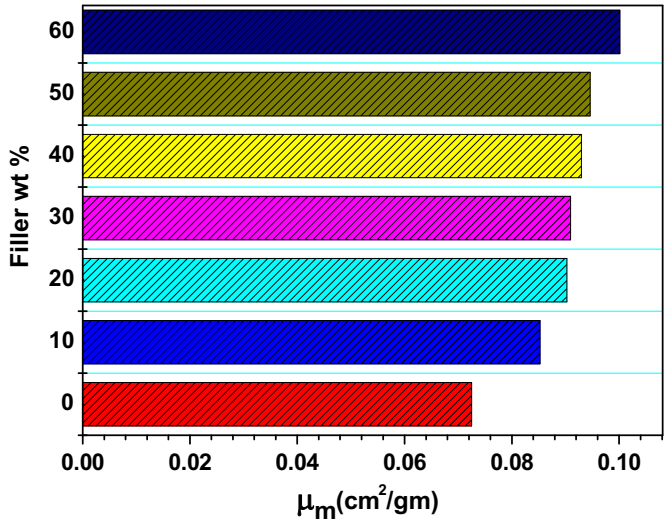

Fig. 4. Mass attenuation coefficients of ISO+Bi_2O_3 polymer composites.

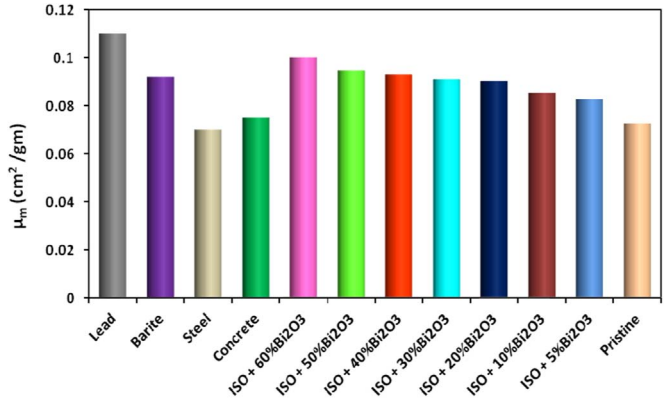

Fig. 5. Comparison study of the shielding ability of polymer composites with conventional shielding materials.

Bi_2O_3 reveals various prominent peaks at 23.86°, 27.42°, 33.24°, 37.66°, 42.34°, 46.4°, 47.43° & 52.34° as shown in Fig. 6b. In all the samples, these peaks are found to remain in the same position. The prominent peaks observed are in good agreement with the standard PDF Database (JCPD file No.41-1449). The other peaks observed in the samples may be due to the scattering from polymer layers..

The calculated values of the crystallite size and strain using Scherrer's formula and W-H plot are as shown in the following Table 1. The crystallite size of the filler within the polymer matrix using Scherrer's formula is found to be in the range of 23.47–42.29 nm and the average particle size (D) is 33.03 nm. As per the W-H plots, the crystallite size ranges from 27.63–44.36 nm with a mean of 37.06 nm. There is no such considerable difference in the crystallite size of the filler particles established from the above two methods. The slight difference in the size may arise due to the consideration of both strain and crystallite size effect in W-H method (Hemalatha et al., 2014; Choudhury and Sarma, 2009).

3.4. FTIR analysis

Fourier Transform Infrared spectroscopy (FTIR) spectra of both pristine and bismuth oxide filled composites which were recorded in the range of 400–4000 cm^{-1} are shown in Fig. 7. Figure shows the presence of hydroxyl group and is witnessed by a broad peak obsereved in the range of 3594–3558 cm^{-1}. A strong band appeared at 1724 & 2930 cm^{-1} confirms the presence of –C=O and symmetric –CH stretching. The peak due to the polystyrene is found in the range of 1602–1594 cm^{-1}. A strong peak at 1272 cm^{-1} indicates the presence of aromatic ketone groups. A medium absorption bands at 1458 & 750 cm^{-1} can be attributed to the mono substituted benzene rings, whereas the peak at 706 cm^{-1} is due to the iso or meta substituted benzene rings. The presence of unsaturated carbon bonds are confirmed by the presence of weak absorption bands at 1070, 1138 and 3072 cm^{-1} (Harish, 2013; Dholakiya, 2012). In almost all the samples, the position of all the peaks is found to be the same. There is neither the peak shift nor a new peak or the disappearance of the peaks. This indicates that, the polymer filler interaction may not be a chemical interaction instead a physical type of interaction..

3.5. Thermogravimetric analysis

The thermal stability of the composites was investigated by thermogravimetric analysis. The results are shown in Figs. 8 and 9. The thermograms of the composites with low filler concentration reveals that, the TG curves of the polymer composites shifts towards the lower side of the temperature and this may be due to the reason that, the phenolic functional groups generated may effectively retard the copolymerisation between the styrene and polyester double bonds which decreases the degree of crosslinking in turn resulting in lowering of thermal stability (Rubab et al., 2014). However, there is no such dramatic decrease in the stability for the higher wt% of the composites (≥ 50%). It is also evident from the thermograms that, there exists two stages of degradation and is as shown in Figs. 8 and 9. The first stage of degradation ranges from 200 –320 °C which may be due to the loss of the polymer content such as water (Pradhan et al., 2008), whereas, the second stage ranges from 340–700 °C which may be due to the presence of unreacted styrene which was added to the polyester (Binu et al., 2014). The residual weight % left over above 700 °C indicates the amount of bismuth oxide present in the sample.

The initial degradation temperature (IDT) values are found to decrease from 238–210 °C with increasing filler content which may be due to the filler particles of bigger size, thereby decreasing the cross link density of the polymer (Dholakiya, 2012). It is also evident from the derivative plots as shown in Fig. 9 that, the temperature at maximum rate of degradation T_{max} is observed to decrease from 308–270 °C and corresponding maximum rate of degradation R_{max} also decreases with increase in the filler concentration. Since, there is no such noticeable decrease in the thermal stability, the composites exhibits good thermal stability upto 200 °C.

3.6. AC conductivity

A good shielding material in addition to its shielding ability should also be a good insulator. But, the polymer composite which has been

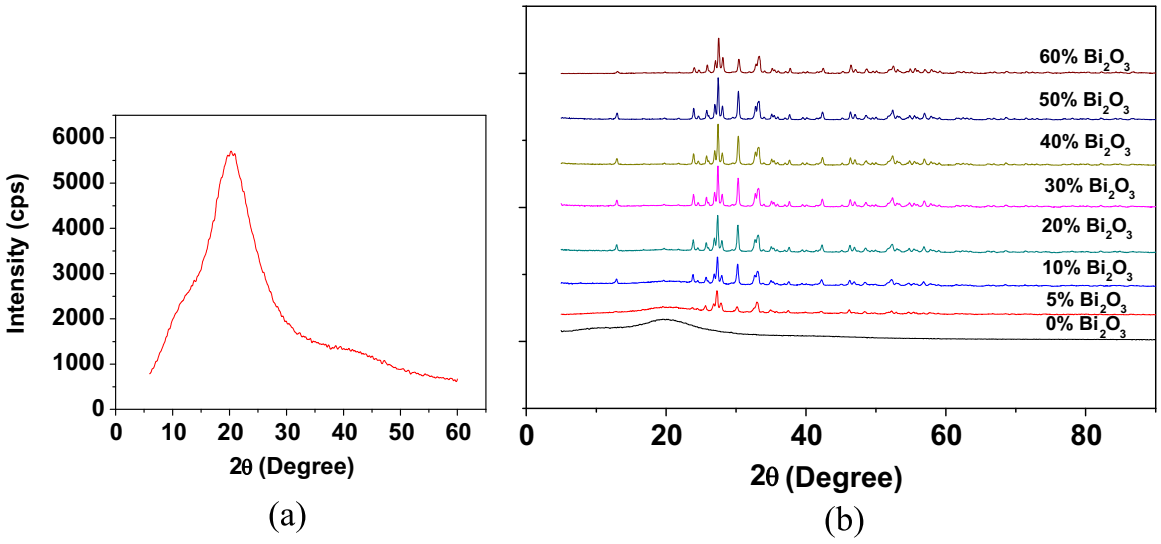

Fig. 6. XRD Spectra of (a) ISO resin (b) ISO+Bi_2O_3 polymer composites.

Table 1
Crystallite size and strain of the polymer composites.

Sample	Crystallite size D (nm)		Average strain×10^{-4}
	Scherrer's formula	W-H plot	
Pure Polymer	0.702	–	–
ISO+5% Bi_2O_3	23.47	27.63	8.39
ISO+10% Bi_2O_3	29.84	34.17	4.50
ISO+20% Bi_2O_3	31.99	36.01	8.15
ISO+30% Bi_2O_3	35.11	41.28	4.72
ISO+40% Bi_2O_3	32.13	34.70	2.79
ISO+50% Bi_2O_3	36.37	41.4	4.14
ISO+60% Bi_2O_3	42.29	44.36	4.21
Average	33.03	37.06	

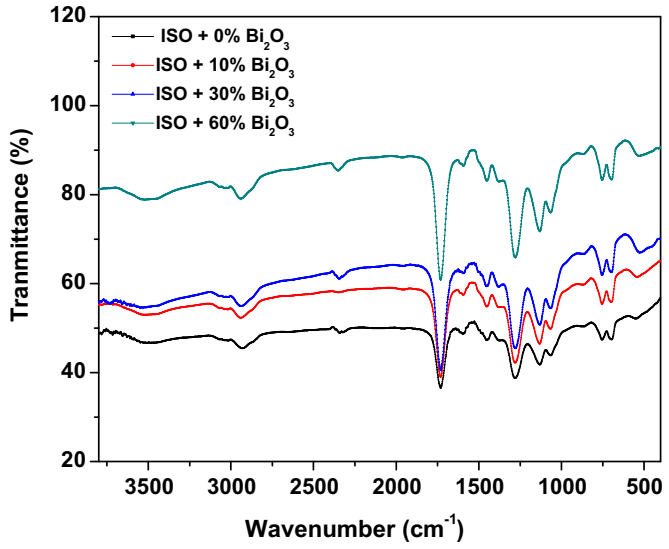

Fig. 7. FTIR spectra of pristine and Bi_2O_3 filled polymer composites.

prepared do consist of bismuth oxide which is a high Z metal oxide and one may expect some conductivity. Therefore, in order to quantify the amount of conductivity, ac conductivity and dielectric measurements were carried out in the frequency range 100 Hz–7 MHz. Figs. 10–12 show the frequency dependence of dielectric constant, dielectric loss and AC conductivity. Fig. 10 shows an increase in the dielectric constant with increase in the wt% of the filler concentration, this

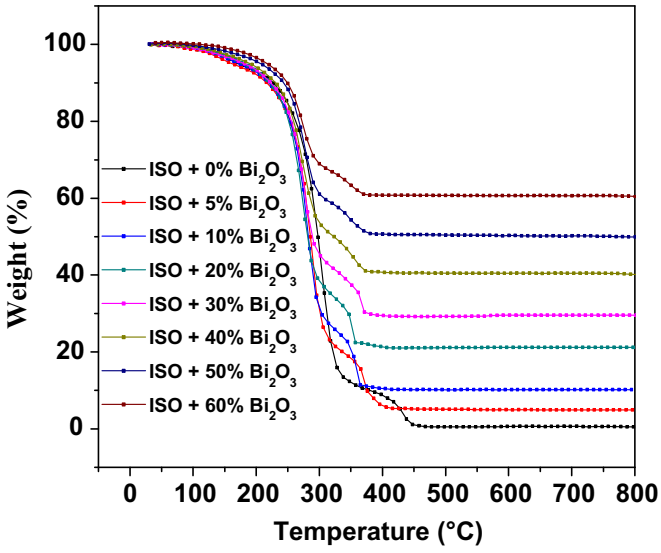

Fig. 8. TG curves of the ISO+Bi_2O_3 polymer composites.

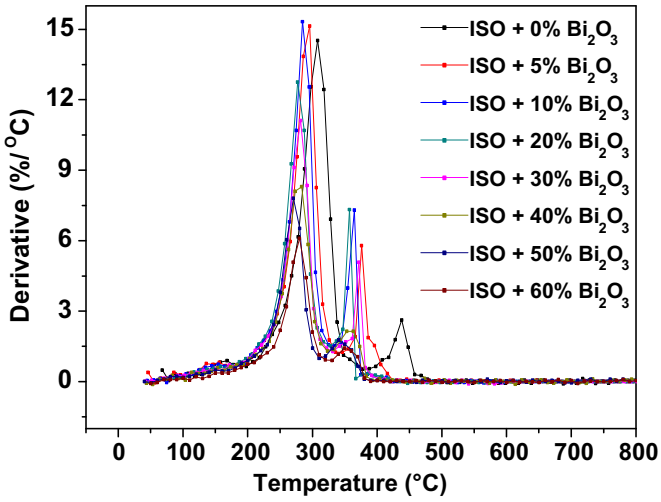

Fig. 9. Derivative TG curves of the ISO+Bi_2O_3 polymer composites.

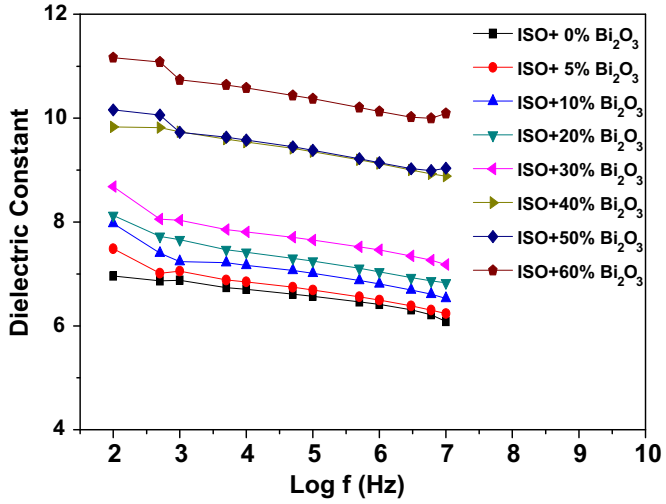

Fig. 10. Study of dielectric constant with respect to frequency.

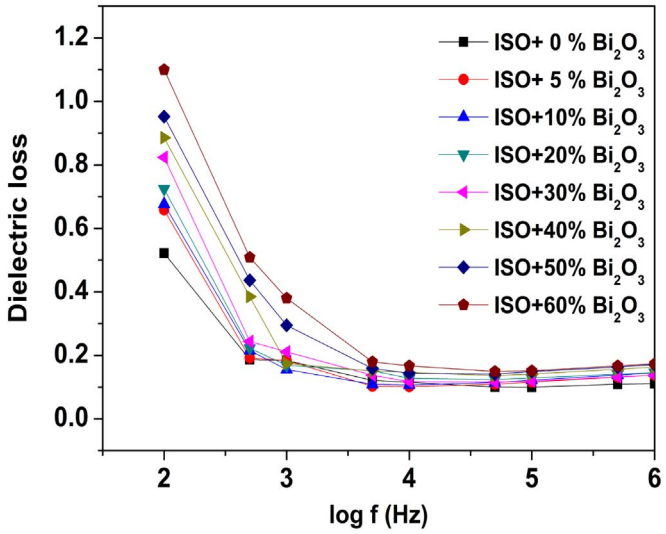

Fig. 11. Study of dielectric loss with respect to frequency.

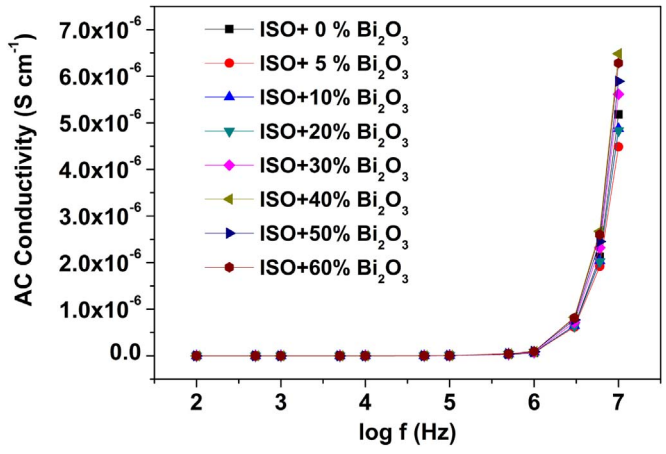

Fig. 12. AC conductivity of ISO+Bi_2O_3 polymer composites.

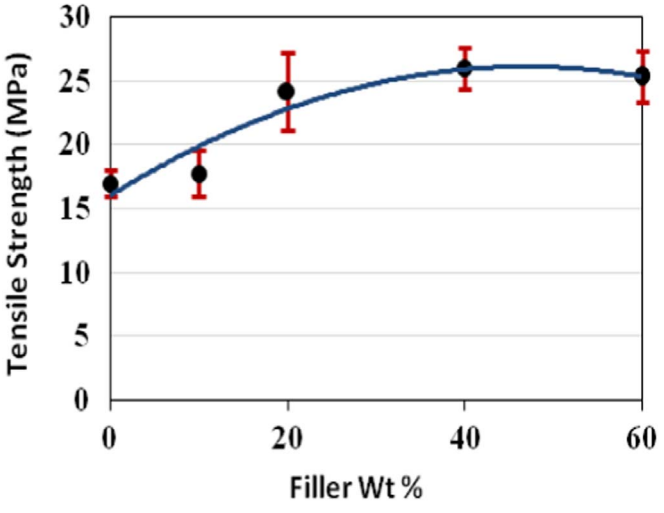

Fig. 13. Tensile strength of ISO+Bi_2O_3 polymer composites.

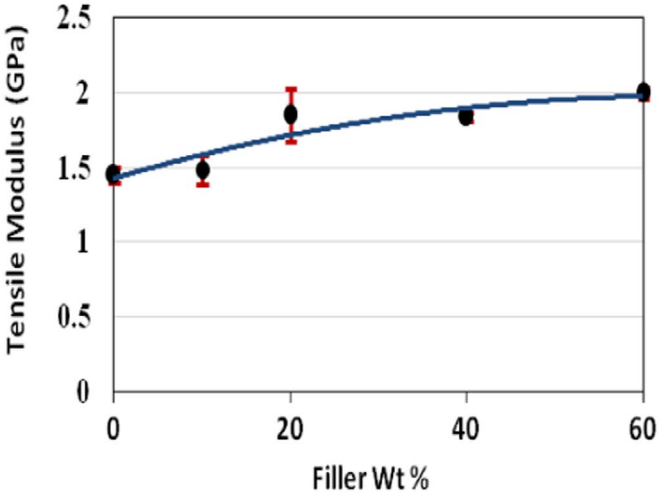

Fig. 14. Tensile modulus of ISO+Bi_2O_3 polymer composites.

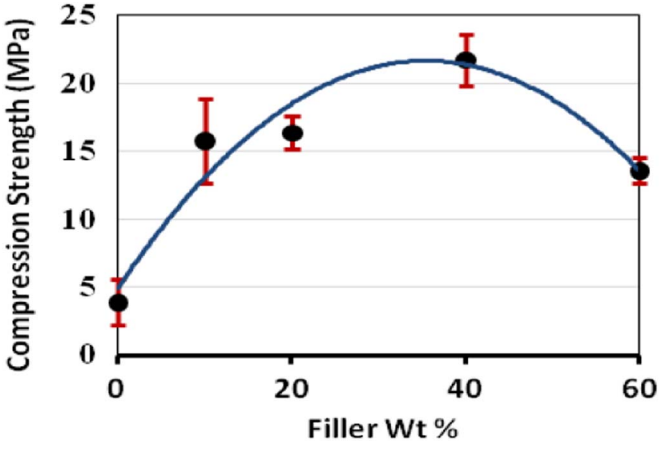

Fig. 15. Compressive strength of ISO+Bi_2O_3 polymer composites.

may be due to the high dielectric constant of bismuth oxide than the polymer isophthalic resin (Barber et al., 2009). In almost all the samples, initially, there is slight decrease in the dielectric constant in the low frequency region and then almost remains constant which may be attributed to the fact that dipolar polarisation of the matrix and the interfacial polarisation (due to the fillers and polymer matrix) become less capable to orient themselves in the direction of the alternating field as the frequency raised (Hussain et al., 2015; Kondawari et al., 2014; Mohapatra et al., 2008). It is also evident from Fig. 11 that, the dielectric loss also found to increase with the increase of filler wt% but, decreases with increase in frequency. The larger value of Ɛ" may be attributed to the mobile charges within the material (Nithya et al.,

2011). On the other hand, it can be seen from Fig. 12 that, the composite samples including the pristine do not exhibit any ac conductivity and this trend continues upto the frequency 1 MHz. Thereafter, it increases sharply with a very narrow range of frequency in almost all the samples in order. The latter may be due to the formation of conducting phase by the bismuth oxide filler particles. Thus, the polymer composites exhibit good dielectric property.

3.7. Mechanical studies

The primary function of a radiation shield is to attenuate the incoming radiation and hence importance is given to its radiation attenuation characteristics. However, understanding some of its mechanical properties, would provide insight into its response to any unexpected mechanical forces that it may be exposed during its operation and during the fabrication of the shield itself. The tensile and compressive strength and modulus was measured using a computer controlled UTM. Fig. 13 is a plot of variation in tensile strength as a function of percentage of filler, while Fig. 14 is a plot of variation in tensile modulus. The tensile strength increases by 56.2% from 16 MPa to 25 MPa, while the tensile modulus increases by 42.8% from 1.4 to 2 GPa with addition of Bi_2O_3 to polyester resin. The increase in these properties are linear, however as is evident from the plot there was variation observed in the samples. These variations could be attributed to the variations in the manufacturing process of these samples. Ultrasonic technique was used to disperse Bi_2O_3 into polyester resin, however during ultrasonication very fine bubbles are formed, which during the process of curing may not have been released completely in all the samples. During the ultrasonication process the dispersion of bismuth oxide powders will be enhanced, however the percentage of dissolved oxygen increases. This results in fine voids within the polymer matrix. These fine voids also have a tendency to affect the mechanical property of the polymer composites (Berhanu et al., 2014).

Unlike tensile strength and modulus, the compressive strength shows an increase upto 40% filler content and beyond this point it drops significantly as is evident from Fig. 15. The increase in compressive strength is quite linear from 4 MPa to 22 MPa at 40% loading of Bi_2O_3. The plot clearly suggests that there is a critical loading percentage of filler for polyester-Bi_2O_3 composite, in this case it appears to be at 40%. Beyond this percentage the compressive strength drops linearly. The presence of Bi_2O_3 beyond 40% could be acting as stress concentrators leading to rapid cracking during compression loading of these test specimens.

4. Conclusions

ISO-Bi_2O_3 polymer composites upto 60 wt% were fabricated successfully by open mould cast technique. Ultrasonicator was effective in dispersing the filler into the matrix upto 50 wt% and the same was confirmed through SEM images. The XRD & FTIR analysis revealed neither the peak shift nor the disappearance of peaks. The average crystallite size is found to be 33.03 nm & 37.06 nm as per Scherrer's formula & W-H plots respectively. This slight difference may be due to the consideration of both strain and crystallite size effect in W-H method. The TG curves of all the composites show the presence of two stage degradation. The initial degradation temperature is in the range of 210–238 °C which proves to exhibit good thermal stability upto 200 °C. Dielectric studies and ac conductivity measurements revealed that, though bismuth oxide was added to the matrix, the polymer composites show very low conductivity which is almost negligible. The filled composites have found to retain their mechanical strength upto 40 wt% of Bi_2O_3 and decreases slightly upon increasing further. The gamma attenuation results reveal that, the linear attenuation coefficient increases with increase in the filler wt%. The HVL thickness of the maximum filled composite is found to be 2.77 cm. The shielding ability of the polymer composites is comparable to that of the conventional shielding materials. As per structural, electrical, mechanical, thermal & gamma attenuation properties are concerned, ISO-Bi_2O_3 polymer composites prove to serve as an effective gamma radiation shields.

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