Impact of Antimony Trioxide on the Physico-Mechanical and Flammability Properties of Irradiated Recycled Rubber Powder and Waste Polyethylene Composites

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ABSTRACT: The physicomechanical behavior and flammability properties of waste polyethylene and recycled waste rubber powder blend (WPE/RWRP/MA) filled with antimony trioxide (Sb_2O_3), at different contents 5, 10, 15, and 20 wt %, and irradiated with different gamma radiation doses, namely 50, 75, 100, and 150 kGy, were investigated by thermogravimetric analysis (TGA), differential scanning calorimetry (DSC) and limiting oxygen index (LOI) measurements. Tensile strength, elongation at break, elastic modulus, hardness, swelling properties, and electric conductivity behaviors were also investigated. Experimental results emphasized that Sb_2O_3 particles retard degradation and flammability of the composite. The LOI increases from 16 to 20.9 with 20 wt % Sb_2O_3 specimen irradiated with 150 kGy. Improvement in thermal stability at conditions of 15 wt % Sb_2O_3 and a dose of 75 kGy was demonstrated by TGA.DSC measurements showed that T_m significantly increased in the presence of the filler. Mechanical characterization tests showed a significant increase in tensile strength within the range 5–10 wt % Sb_2O_3. © 2011 Wiley Periodicals, Inc. J Appl Polym Sci 124: 4098–4106, 2012

Key words: waste polyethylene; waste rubber; gamma irradiation; fire retardant; limiting oxygen index; antimony trioxide

INTRODUCTION

The management of end-of-life tires is a great environmental challenge. Recent European regulations forbid the burning and landfilling of tires and set recycling objectives. Mechanical recycling, i.e., the grinding of tires and separation of metal and rubber components, is one of the most interesting waste management approaches. Various research articles and patents showed the possibility of recycling ground tire rubber (GTR) powders as functional fillers in host polymers. Nevertheless, while some applications already exist in the case of thermosets (athletic tracks, pavements, playgrounds,. . ., etc.),[1] no effective applications were found for thermoplastic/GTR blends due to the very poor mechanical properties of such composite materials,[2,3] which were attributed to the lack of interfacial adhesion between the rubber particles and the thermoplastic matrix.

Various research articles focus on solutions to compatibilize the two phases, the ultimate objective being to obtain a "thermoplastic elastomer" like behavior by combining rubber elasticity and thermoplastic matrix properties. Many processes have been carried out to improve the mechanical behavior, such as the control of GTR particle morphology, mean particle size or specific surface area,[4–6] the oxidation or chlorination of GTR particle surface,[7–9] the devulcanization of GTR particles using various physical, chemical, or biological processes[10,11] or the addition of a third polymer or reactive molecules as maleic anhydride or methyl methacrylate.[6,12,13] This last approach led to better results in terms of mechanical properties. The approach which gave the best results consisted of performing crosslinking radical reactions at the interface between rubber particles and host thermoplastic matrix. Such reactions are initiated by free radicals obtained either by the decomposition of a peroxide during melt blending, or by gamma irradiation of the blend.[14–15] Polymer processing by ionizing radiation is environmentally and energetically safe as it does not need solvents or initiators at high temperature and allows one to avoid degradation phenomena and other side reactions typical of polymer processing in the melt.

Antimony trioxide (Sb_2O_3) is one of the most important and widely used flame retardant additives, more often in combination with halogen compounds. However, the future use of these flame retardants faces some questions as the public reluctant perception of the environmental impact of recycling,

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toxicity, and combustion of certain halogenated flame retardants has become progressive.[16,17]

This work aimed at extending the knowledge of the synergistic impact of gamma irradiation in presence of antimony trioxide particles on the physico-mechanical and flammability properties of recycled rubber powder/waste polyethylene blend.

EXPERIMENTAL

Materials

Recycled waste rubber powder RWRP, was kindly provided by Narobine Company, Cairo, Egypt, of particle size 80 mesh (150 mm) of unclassified ground rubber from the tread and sidewalls of passenger and truck tires. Waste polyethylene (WPE) was obtained from the local market. The reactive compatibilizing agent maleic anhydride (MA) and antimony trioxide (Sb_2O_3) were obtained from Merck, Germany. Other chemicals ingredients were of commercial grade.

Sample preparation for irradiation

The preparation of WPE/RWRP composites was done on a Brabender-like apparatus. After melting WPE, RWRP was added into the apparatus, mixed for about 10 min within a temperature range of 170–175°C to disperse RWRP uniformly into WPE. Also, (MA) was added at 2%. The antimony trioxide (Sb_2O_3) was thereafter added at 5, 10, 15, and 20 wt % into the blend. After mixing, the samples were hot pressed at about 175°C under 10 MPa for 5 min into sheets of suitable thickness and size for analysis. The weight ratio of WPE/RWRP in all composites was 3 : 2.

Gamma irradiation

The samples were submitted to gamma irradiation in air, using gamma cell type 4000 A, India, at room temperature and ambient humidity. The absorbed doses were 50, 75, 100, and 150 kGy at irradiation dose rate $\sim$ 5 kGy/h. Irradiation was carried out at the National Center for Radiation Research and Technology, Atomic Energy Authority, Cairo, Egypt.

Mechanical testing

Tensile properties of the composites were determined by using Houns Fild computer aided testing machine, England. The ISO 37-1977 (E) and ISO 34-1975 (E) standards were followed in measuring tensile strength and elongation at break, respectively.

Hardness measurements

Samples of at least 1.25 mm in thickness with flat surface were cut for hardness test. The measurement was carried out according to ASTM D 2240 using 306L type D Durometer. The unit of hardness is expressed in (D Shore).

Thermal analysis

The thermal properties of all composites were investigated by means of the DSC Shimadzu Type DSC-50 system in a nitrogen atmosphere at 20 mL/min, within the temperature range from ambient to 200°C at a heating rate of 10°C/min. Thermogravimetric analysis (TGA) was performed with a Shimadzu TGA-50 system, Kyoto, Japan, heated within the temperature range 20–600°C at 20°C/min, under a controlled dry nitrogen flow of 20 mL/min.

Limiting oxygen index

The minimum oxygen concentration required to sustain burning was measured. Samples were held vertically in an oxygen index system instrument (Type HC-2, Jiangning Analysis Instrument Factory, Nanjing, China). All tests were carried out according to the standard oxygen index test ISO 4589-1984.

Swelling measurements

For swelling determination on plastic/rubber composites, a molded or pressed specimen, three pieces of sample of approximately uniform size and weight ($\sim$ 0.5 g) were accurately weighed (W_1) and immersed in 50 mL of benzene at room temperature for 72 h. Aftermath, the sample was taken out and put between two pieces of filter paper, then put between two sheets of glass (each weighted 98.4 g), kept for 5 s, then transferred to a weighing bottle and reweighed (W_2). The swelling percent (Q) was calculated as below, eq. (1):

$$Q\% = [W_2 - W_1/W_1] \times 100 \quad (1)$$

Electric conductivity

Volume resistivity was examined by the Electrometer 6517, Keithly Instruments Ohio, and USA. The electrometer is connected externally with a resistance text fixture Model 9008 for automatic display of the volume resistivity reading. The measurement of volume resistivity was carried out at room temperature on circular type probes of diameter (mm).Conductivity of each specimen was calculated by eq. (2):

$$\text{Conductivity} = L/R.A \quad (2)$$

where, L is the thickness of the sample (cm), R is the reading resistance (ohm^{-1} cm^{-1}), and A is the area of the sample (cm^2).

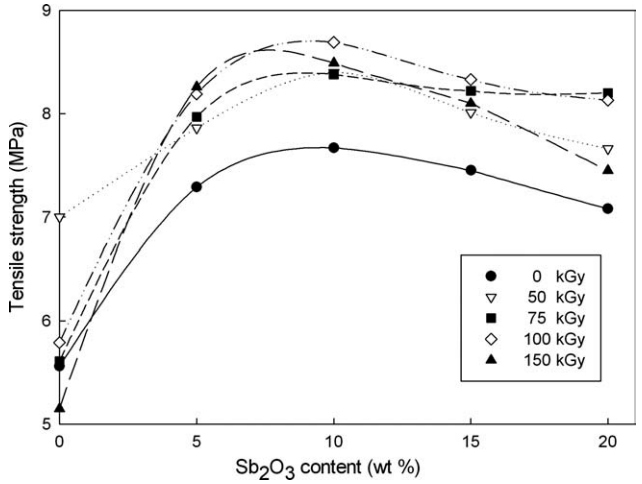

Figure 1 Effect of Sb_2O_3 content on the tensile strength of WPE/RWRP/MA 60/40/2 wt % at different gamma radiation doses.

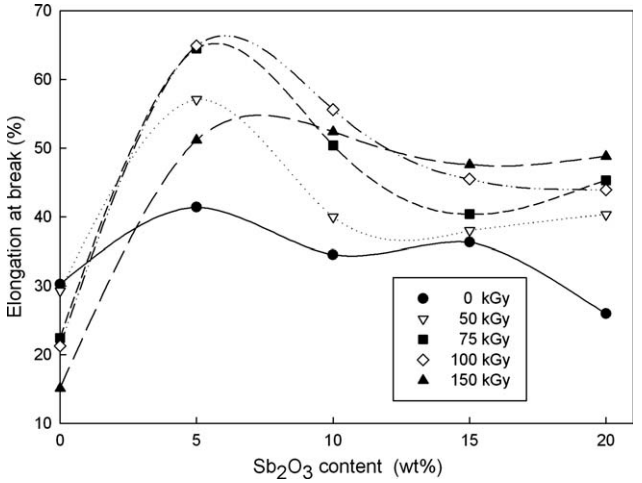

Figure 2 Effect of Sb_2O_3 content on the elongation at break of WPE/RWRP/MA 60/40/2 wt % at different gamma radiation doses.

Morphological characterization

An ISM-5400 scanning electron microscope (JEOL, Tokyo, Japan) was used for morphological observation of fracture samples in liquid nitrogen and coated with gold before microscope testing.

Infrared spectroscopic analysis

The infrared spectrawere obtained by using an FTIR spectrophotometer, Mattson 100 Unicam (England), over the range of 500–4000 cm^{-1}. A dry constant weight from each composite was ground with 3 mg of KBr and then pressed to form disks. The samples for IR analysis were first dried in a vacuum oven at 80°C for 2 h.

RESULTS AND DISCUSSION

Mechanical testing

The mechanical properties of composites depend on many factors: the aspect ratio of the filler, the degree of dispersion of the filler in the matrix, and the adhesion at the filler–matrix interface. To assess the effect of the flame-retardant Sb_2O_3 on the mechanical properties of the blend, tensile strength, and elongation at break were measured; the results are numerated in Figures 1 and 2, respectively. Evidently, filler load up to 10 wt % improved the mechanical parameters, as tensile strength raised from 5.2 to 7.5 MPa and elongation at break from 30 to 40%; however, by further increase in filler load the yields of both parameters prone to reduce as a result of bringing about weakness due to difficulties in dispersing filler particles. The attempt with 20 phr Sb_2O_3 lowered elongation at break to rather loser value than that of gum blend.

Figures 1 and 2 show also tensile strength and elongation at break as functions of varying load and radiation dose, respectively. Both parameters, by $\sim$ 5 wt % content, improved due to radiation crosslinking in polymer matrix up to 100 kGy, giving values of $\sim$ 8.5 MPa and $\sim$ 65%, respectively. The phenomenon my be accounted for a competition that takes place between the growing disturbance to the crystalline portions, and hence softening of blend components as filler content augments up to $\sim$ 5 wt %, and the appreciable increase in radiation crosslink density up to $\sim$ 100 kGy. The assumption may be supported by ΔH_m recorded data, Table II. In fact with any further increase in filler loading the molecular mobility decreased due to the formation of physical bonding between the filler particles and rubber chain, stiffening the matrix.[18]

Blend elastic modulus improved by nearly 20 wt % by adding Sb_2O_3 up to 10 wt %, Figure 3. This

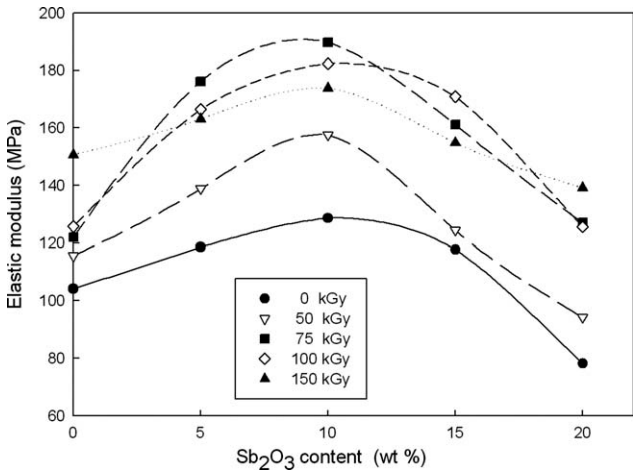

Figure 3 Effect of Sb_2O_3 content on the elastic modulus of WPE/RWRP/MA 60/40/2 wt % at different gamma radiation doses.

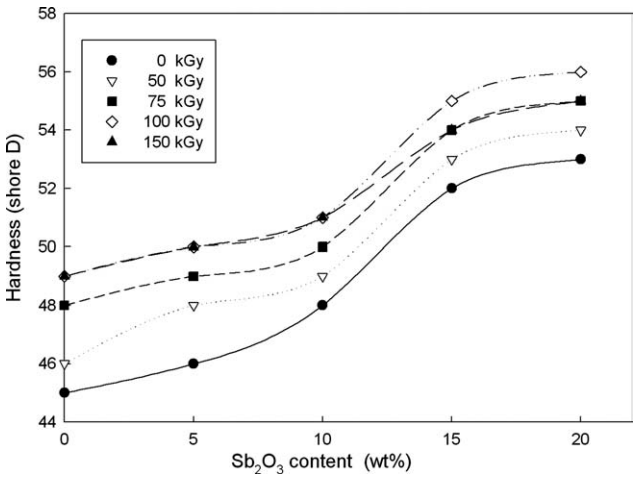

Figure 4 Effect of Sb_2O_3 content on the hardness of WPE/RWRP/MA 60/40/2 wt % at different gamma radiation doses.

could be a result of the intercalation of filler particles in polymer matrix. The modulus thereafter decreased gradually as Sb_2O_3 content increased suggesting perturbation in the semicrystalline portions. Evidently, from Figure 3, elastic modulus of 10 wt % filler load specimen increases with radiation dose up to 75 KGy by nearly 50% indicating that crosslinking is the predominant process beyond which all specimens revealed degradation and hence reported decreases. Apparently, a dose of 100 kGy may lower the maximum attained modulus by nearly 5%, whereas 50% increase in the dose may only lead to 10% loss.

Hardness measurements

Variation in hardness of loaded and unloaded blends as a function of radiation dose is illustrated in Figure 4. The data of unirradiated loaded composites are also introduced in the same figure for comparison. Hardness values of the latter samples effectively increased with increasing the degree of load. Consistent increase in hardness was observed over the load rang as dose increased up to 100 kGy, above which it tends to decrease. On the other hand, a marked increase was observed in hardness at similar filler content on increasing radiation dose from 50 to 150 kGy. The data clearly indicate that the main contribution to hardness is correlated with filler content. The observed improvement by loading and irradiation within the cited ranges accords with the attained enhancement in the examined mechanical parameters.

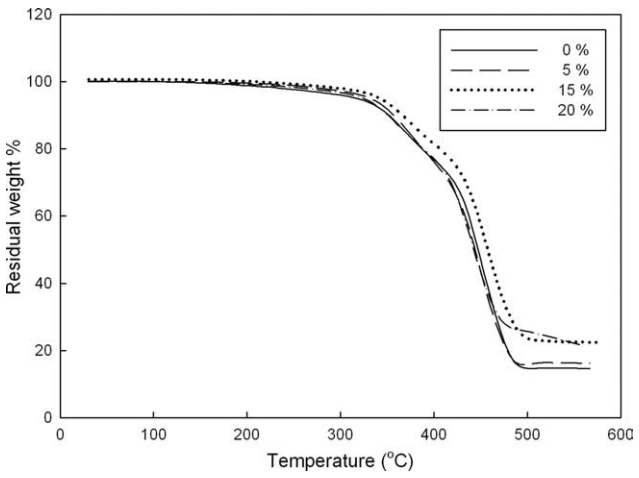

Figure 5 TGA thermograms of nonirradiated WPE/RWRP/MA 60/40/2 wt % at different Sb_2O_3 content.

Thermogravimetric analysis

The TGA thermograms of the unirradiated and irradiated samples are shown in Figure 5. The WPE/RWRP/MA/Sb_2O_3 composites generally show higher thermal stability than WPE/RWRP/MA blend and degrade in one step. The Sb_2O_3 load increases the thermal stability as indicated by elevation in the degradation onset temperature. The temperature of 10% mass loss, $T_{10\%}$, has been taken as a measure of the onset of degradation; the typical values are listed in Table I. $T_{10\%}$ increased from 348°C for neat blend to 355°C for the loaded blend with 5 wt % Sb_2O_3. Interestingly, further increase in load content up to 15 wt % elevated the onset temperature. This behavior may be attributed to progressive interfacial linking, which is mainly accompanied with enhanced thermal stability. By 20 wt % load the onset temperature tended to decrease.

Table I show the data obtained by the variation of Sb_2O_3 content against temperature at different radiation doses, 75–150 kGy. Remarkably, the irradiated samples exhibit higher thermal stability as represented by $T_{10\%}$ than that reported for the

TABLE I
TGA Results of the Thermal Degradation of Nonirradiated and Irradiated WPE/RWRP/MA (60/40/2) wt % Composites at Different Sb_2O_3 Contents

Sb_2O_3 content %	Dose (kGy)	T_{onset} (°C)	$T_{0.5}$ (°C)	Charred residue at 600°C (wt %)
0	0	348	445	14.6
5	0	355	447	16.3
	75	360	449	18.4
	150	358	457	18.3
15	0	366	458	22.4
	75	369	459	23.9
	150	357	445	27.4
20	0	351	442	21.8
	75	356	444	22.3
	150	353	442	21.5

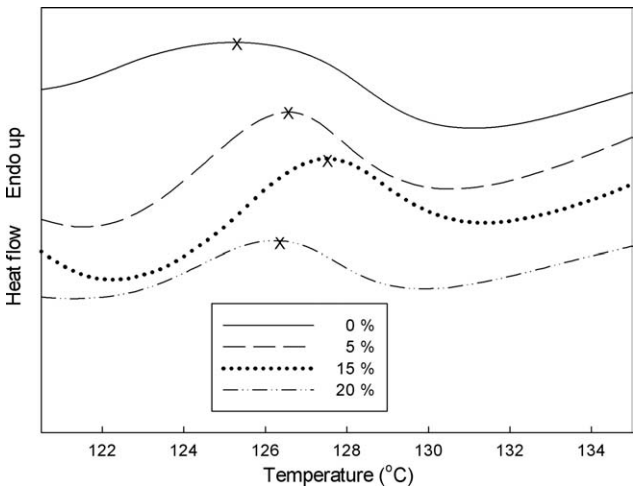

Figure 6 DSC thermograms of nonirradiated WPE/RWRP/MA 60/40/2 wt % at different Sb_2O_3 content.

unirradiated ones. Also, Table I summarizes the thermal decomposition temperatures of composites irradiated with various gamma radiation doses. As expected, radiation crosslinking improved the thermal stability of unloaded and loaded composites. Thus, the results fairly support the observed mechanical behavior.

Differential scanning calorimetery

Figure 6 demonstrates the DSC thermograms of composites with varying Sb_2O_3 contents. The endotherms show well-defined peaks whose melting temperatures and enthalpy are enumerated in Table II. The blend melting temperature 125°C has been explicitly shifted to higher values by compounding with the filler. For specimens containing 5 wt % Sb_2O_3 the recorded T_m was 129°C whereas for 15 and 20 wt % slightly reduced to 127.3 and 126.2°C, respectively. The general increase in T_m may be attributed to the crystallization effect of Sb_2O_3 which can act as a nucleating agent increasing the rate of crystallite formation while reducing crystallite size.

TABLE II
DSC Data of Unirradiated and Radiated WPE/RWRP/MA (60/40/2) wt % Composites at Different Sb_2O_3 Contents

Sb_2O_3 content %	Dose (kGy)	T_m (°C)	ΔH_m (J/g)	Onset temperature
0	0	125	1.37	120.9
5	0	129	2.4	125.2
	75	127.2	1.8	122.9
	150	126.5	1.6	122.0
15	0	127.3	2.2	124.0
	75	126.2	2.2	122.3
	150	125.7	2.5	121.7
20	0	126.2	1.8	122.9
	75	129.5	0.52	126.6
	150	127.2	1.66	124.2

On the other hand, the slight decrease in T_m recorded by increasing Sb_2O_3 content may be accounted for two morphological effects (a decrease in lamellar thickness) and the thermodynamic factors (polymer–polymer interactions). The macromolecular chain motion was apparently restricted due to physical bonding interactions of filler molecules with substrate backbone.[19]

As shown in Table II, ΔH_m value, onset temperature and T_m are significantly increased with the load 5 wt % by favoring crystallization. While, further increase in Sb_2O_3 led to reductions. It becomes difficult to disperse filler molecules in the WPE matrix and hence hindering crystallization. Variation of mechanical properties as a function of Sb_2O_3 filler load well confirmed this standpoint. The mechanical properties increased with the addition of 5 wt % Sb_2O_3 and then decreased with further addition of filler load.

Figures 7 and 8 display the melting endotherms of composites irradiated with 75 and 150 kGy. The endotherms show well-defined peaks whose melting peak temperatures and enthalpy values are summarized in Table II. The melting curves displaced towards lower temperatures as radiation dose increases. This difference in crystalline population can be attributed to the decrease in molecular weight of the polymer matrix due to radiation chain scission,[20] which lowers the onset temperatures. Sb_2O_3 beyond 5 wt % resulted in decreases in melting and onset temperatures. This may be ascribed to restriction in the spherulitic growth and hence reduction in the degree of crystallinity.

Limit oxygen index

Table III presents the limiting oxygen index (LOI) values of the compatibilized gum blend and its

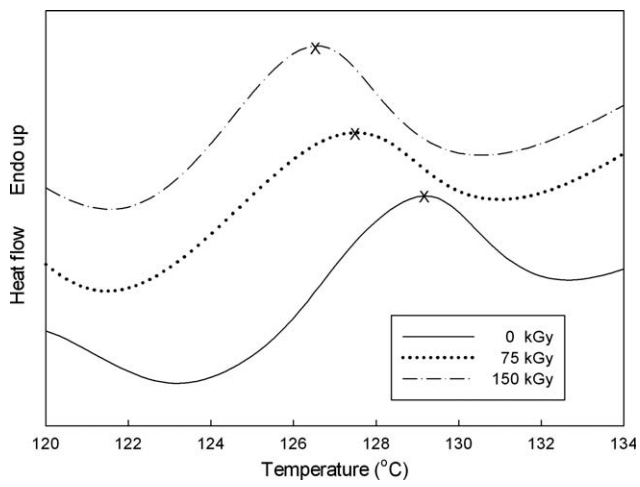

Figure 7 TGA thermograms of nonirradiated WPE/RWRP/MA 60/40/2 wt % at various radiation doses.

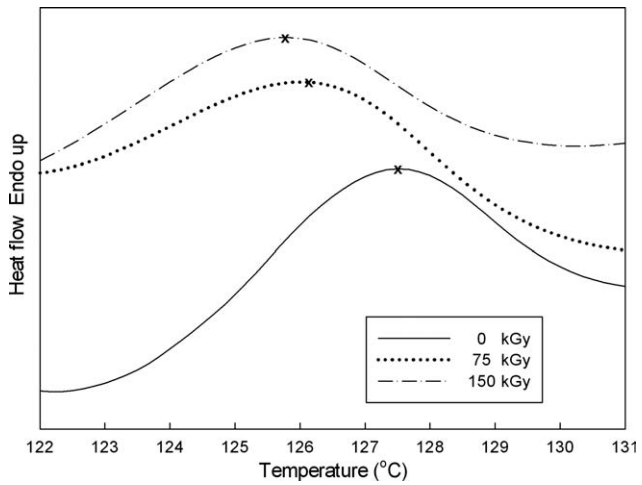

Figure 8 DSC thermograms of nonirradiated WPE/RWRP/MA 60/40/2 wt % at various radiation doses.

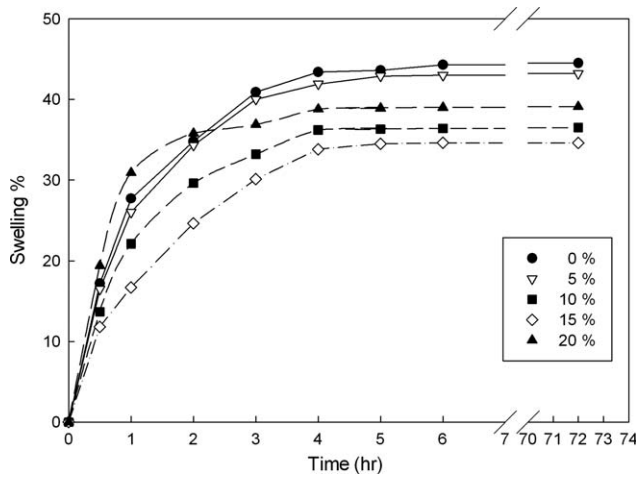

Figure 9 Effect of Sb_2O_3 content on the swelling percent of WPE/RWRP/MA 60/40/2 wt % in benzene over time.

unirradiated and irradiated composites. A remarkable increase in LOI value from 16 to 20.9 was observed for the 15 wt % loaded blend after irradiation. Whereby Sb_2O_3 by itself has no perceptible flame-retardant effect on its own; however, it has been found to provide fire retardancy to polymer in some special cases; it was reported to be effective even in the absence of halogen.[21] The flame retardancy was accounted for simple physical dilution of the flammable substrate and formation of impermeable glassy layer or coating on the burning surface at ignition temperature.[22]

The maximum improvement in LOI value is indicated for the 15 wt % loaded specimen irradiated with a dose of 150 kGy. Therefore, it may be conceivable to assume that by further increase in radiation dose higher LOI levels could be probably attained. At such degree of filler load, assisted with network rupture via the relatively high dose degradation, excess Sb_2O_3 molecules can be released to play the integral role in flame retardance. Comparatively, the LOI levels of unirradiated gum blend demonstrated dependence on filler load suggesting tendency to decrease above 15 wt % as chances for agglomeration increases and hence restricting the particulates mobility.

TABLE III
LOI Data of Nonradiated and Irradiated WPE/RWRP/MA (60/40/2) wt % Composites at Different Sb_2O_3 Contents

Sb_2O_3 content %	Dose (kGy)	LOI
0	0	16
5	0	19.4
	50	19.6
	100	19.8
	150	19.9
15	0	20.4
	50	20.8
	100	20.8
	150	20.9
20	0	20.2

Swelling behavior

Figures 9 and 10 show the variation in blend swelling percent (Q%) in benzene as a function of filler content and radiation dose and its composites loaded up to 20 wt %, respectively. Generally, swelling percent decreases with increasing Sb_2O_3 content and the exposure dose. The increase in radiation dose leads to a crosslink copolymer, and thus minimizes the free space available for swelling and reduces the swelling rate of the blend, which is ascribed to the relatively long time needed for relaxation of the tight network. Moreover, the pristine blend exhibited higher swelling values through the

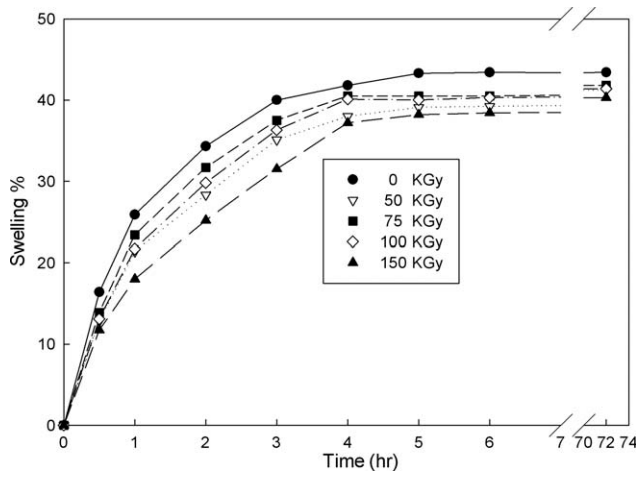

Figure 10 Effect of radiation dose on the swelling percent of WPE/RWRP/MA 60/40/2 wt % in benzene over time.

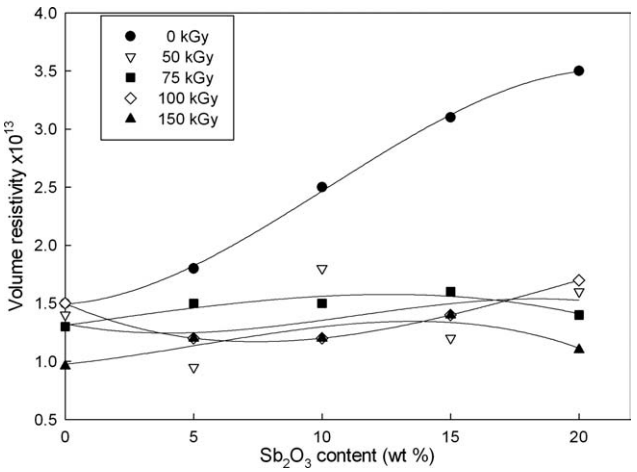

Figure 11 Variation of volume resistivity of WPE/RWRP/MA 60/40/2 wt % at different contents of Sb_2O_3 by various radiation dose.

entire radiation range with respect to the loaded ones whose swelling values decreased at similar radiation dose with increasing load. Apparently, the presence of filler particles in its aggregate "structure" produces physical bonding with the formed interphase.

Electric conductivity

Variation in volume resistivity as a function of the Sb_2O_3 content and radiation dose is presented in Figure 11. Notably, the resistivity of the unirradiated specimen increases with the increase in load Sb_2O_3. This may indicate that filler particles inherently impede conductivity, and impediment is associated with filler content. By irradiation, the rubber moiety experiences radiation inductive chain scission.[23] Accordingly, it can be assumed that neat matrix may exhibit an extent of carbon black release that enhances conductivity. Conductive networks may be formed not only due to the physical contact of particles but also due to particles being separated by a small distance (<10 nm) when an electron can hop the interparticle gap. The observation has been confirmed as conductivity markedly grew up by the considerable increase in dose up to 150 kGy. On the other hand, almost invariable data were maintained as irradiated gum blend was loaded. The significant increase in conductivity by irradiation at similar filler content ratio and the relative consistence in the parameter values by increasing the filler content may suggest that the impeding role of the additive is more pronounced that the carbon black within the cited ranges of loading and dose. The observed

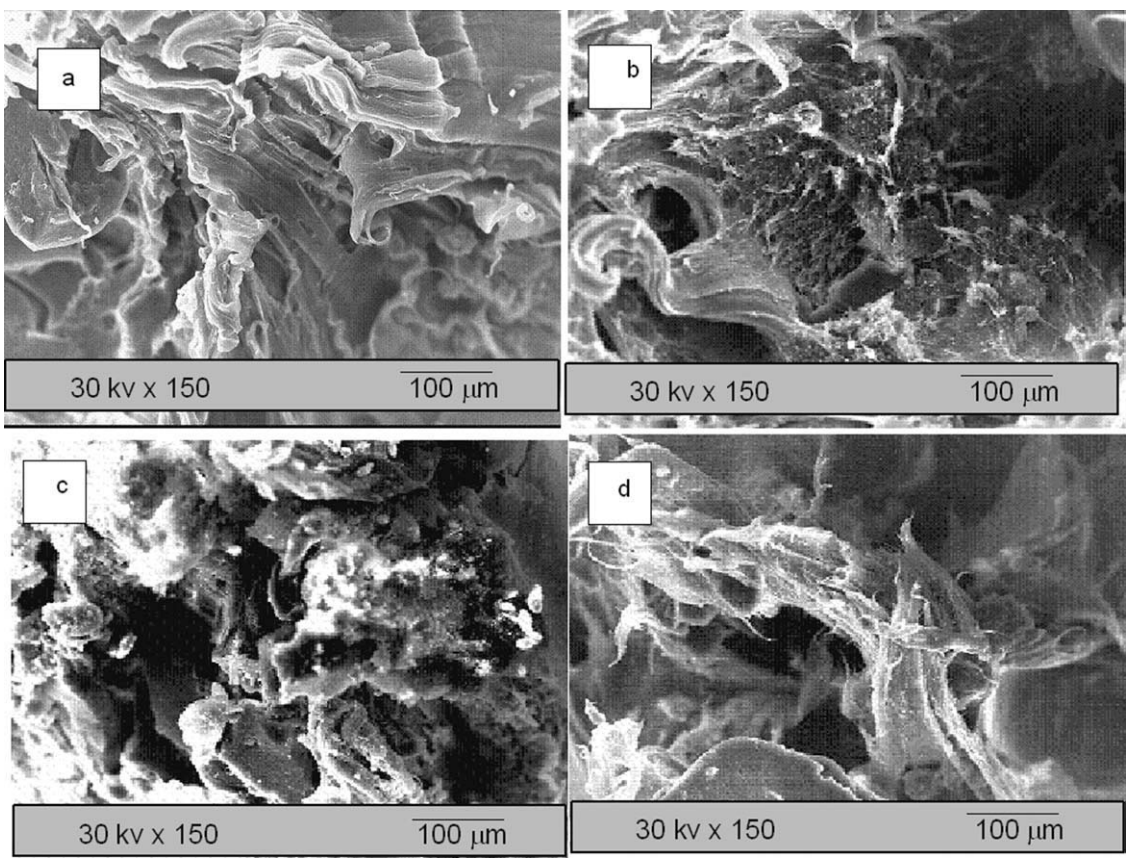

Figure 12 SEM micrographs of tensile fracture surface of WPE/RWRP/MA 60/40/2 wt % composite at different contents of Sb_2O_3: (a) blank, (b) 5%, (c) 15%, and (d) 20%.

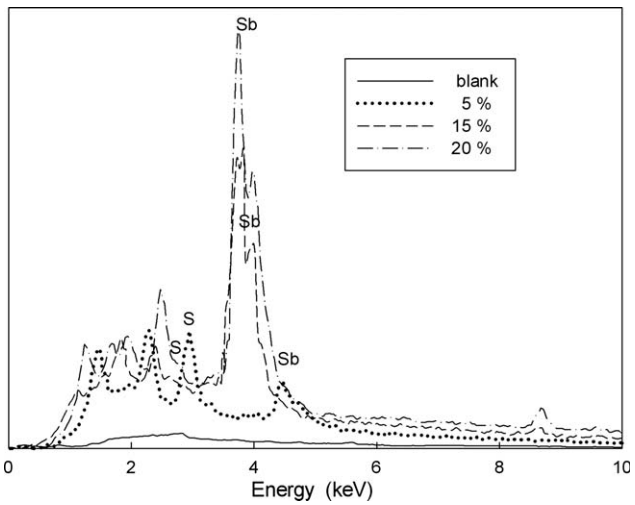

Figure 13 EDX patterns of fracture surface of WPE/RWRP/MA 60/40/2 wt % composite at different contents of Sb_2O_3.

decrease in resistivity within the radiation dose range, 0–50 kGy, is due to the proportions of formation and breakdown of conductive networks present in the system. Radiation-induced crosslinks may also strengthen the conductive networks imply restrictions on polymer chain mobility.[23] The results obtained derive further evidence on the variation experienced by the matrix microstructure under conditions of higher degree of degradation in presence of variable filler content.

Effect of Sb_2O_3 on phase morphology

Figure 12 shows the scanning electron micrographs of the unirradiated blend containing varying content of Sb_2O_3. Figure 12(a) of the blend shows an irregular domain size and a coarser and poorer distribution of RWRP in the WPE matrix. Figure 12(b) of 5 wt % Sb_2O_3 shows better strength with many tear lines and homogeneity than for unloaded blends with a higher dispersed domain size and pore size. However, Figure 12(c,d) of 15–20 wt % Sb_2O_3 shows a coarse and unstable particle structure and an uneven distribution of the dispersed filler phase. This indicates low adhesion between phases, giving rise to poor stress transfer across the interface. The presence of many tear lines on the tensile fracture surface of polymer composite, Figure 12(b,c), indicates increased interaction between phases, thus improving compatibility and consequently enhancing the mechanical properties. Figure 13 shows the EDX spectra of the mineral in the composite matrix at 5, 15, and 20 wt %. The presence of Sb indicates that Sb_2O_3 was distributed in all cases. Figure 14 represents the EDX spectra of the composite containing 20 wt % Sb_2O_3 irradiated with 75 and 150 kGy doses. Obviously, the presence of Sb corroborates that Sb distribution in the composite is still confirmed.

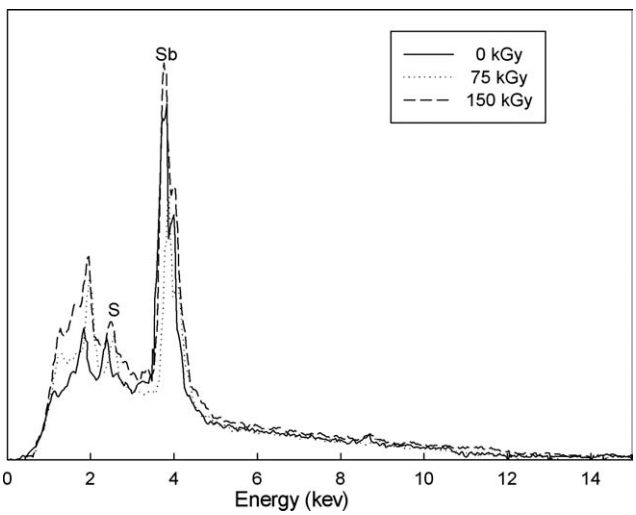

Figure 14 EDX patterns of fracture surface of WPE/RWRP/MA 60/40/2 wt % composite at different radiation doses.

FTIR analysis

FTIR spectroscopy is a good technique for estimation of the chemical and physical changes that occur in the polymeric materials due to irradiation.[24] The degree and extent of oxidation reaction can be easily determined spectroscopically throughout the following up for IR band intensity C=O and O–H. Also, the degree of radiation-induced crosslinking can be estimated directly by evaluating the decrease in the IR intensity of the C=C band.[25]

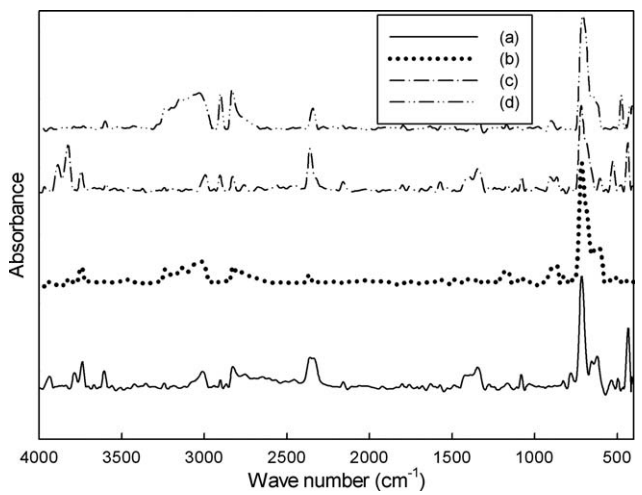

Figure 15 FTIR spectra of WPE/RWRP/MA/Sb_2O_3 60/40/2 wt % at various radiation doses. (a) WPE/RWRP/MA. (b) WPE/RWRP/MA/Sb_2O_3. (c) WPE/RWRP/MA/Sb_2O_3 exposure to radiation dose at 75 kGy. (d) WPE/RWRP/MA/Sb_2O_3 exposure to radiation dose at 150 kGy.

The composite 60/40/2/15 wt % was gamma irradiated with various radiation doses 75 and 150 kGy analyzed by FTIR spectroscopy. Post irradiation IR absorbance peaks at 968 and 1168 cm^{-1}emerged in Figure 15. The appearance and the increase of absorbance intensity with dose of other peaks at 1791 and 3124 cm^{-1} are attributed to formation C=O and O—H groups. It can be seen that the intensity of the peak at 1616 cm^{-1} constantly decreased over the cited range of irradiation at a result of involvement of the C=C group in radiation crosslinking.

This decrease in peak intensity is accounted for generating growing amount of free radical with dose; thus provides further opportunity of crosslinking after formation of higher number of free radicals.

CONCLUSIONS

It has been emphasized that gamma irradiation of WPE/RWRP/MA/Sb_2O_3 composites effectively contributed to the mechanical properties: tensile strength, elongation at break, and elastic modulus within 5–10 wt % Sb_2O_3 load. Remarkable increases in hardness and resistance to swelling in benzene and volume resistivity were also reported by elevating Sb_2O_3 content up to 20 wt % within the total integral radiation dose range, up to 150 kGy. Sb_2O_3 level has also been proved to influence the fire safety properties of the blend system as Sb_2O_3 particles relatively retarded the degradation and flammability of the composite. LOI data of irradiated specimens with a dose of 150 kGy augmented from 16 to 20.9 by a load of 15 wt %.The SEM micrographs inferred that irradiation facilitated the compatibility among blend components via crosslinking which in its turn is supported by the presence of the reinforcer. Enhancement in thermal stability was contributed by 15 wt % filler load irradiated with a dose of 75 kGy. Similarly, DSC measurements showed significant elevation in T_m in the presence of Sb_2O_3.

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