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Physico-Mechanical Properties of Gamma Irradiated EPDM Rubber Nano composites Loaded with Fumed Silica and Zirconium Silicate

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ETHYLENE-PROPYLENE-DIENE rubber (EPDM/ nano-filler) composites were prepared and crosslinked by gamma rays. The composites either contain 40phr of fumed silica, 40phr of zirconium silicate, separately, or different ratios from both ranging from 30:10-10:30, respectively. The effect of the presence of these fillers in the composites on their electrical and thermal properties was studied. The mechanical properties namely, tensile strength (TS) and elongation at break at different irradiation doses has been measured. The best mechanical properties for the all irradiated composites were attended at 100kGy. The fumed silica increased the tensile strength and volume resistivity of the composites, but Zirconium silicate slightly increased the thermal stability and the former properties.

Keywords: Ethylene-propylene-diene rubber-Nano composites-transmission electron microscopy .

Due to the saturated chain backbone of EPDM, it can be prepared in different formulations, each one adequate to a specific application, in which characteristics such as resistances to ozone, heat and moisture, flexibility to low temperatures, wide range of tensile strength and excellent electrical insulation properties are required (Canaud *et al.*, 2001). These properties, being particularly important in the electrical sector, have stimulated an increasing utilization of EPDM-based compositions in medium and high voltage insulating cables and wires (Canaud *et al.*, 2001). On irradiation with ionizing radiation such as gamma rays or accelerated electrons, many properties of polymers are changed among which its electrical properties, e.g. conductivity and dielectric loss.

Inorganic fillers have a major role in improvement of the desired electrical and mechanical properties of polymers (Cherney, 2005 and Jeffry and Stephane, 1997). Due to the above attractive properties, thermal insulators based on EPDM are widely used in space vehicles to protect structures from high heat flux of propellant combustion (Zhao *et al.*, 2009). The major drawbacks of low physico-mechanical characteristics of EPDM are overcome by the combination of various particulate fillers as reinforced fillers. EPDM loaded with silica and fibrous fillers such as carbon fibers, aramid fiber or asbestos has shown best performance **(**Alemdar and Sain, 2007 and Jin, 2007). However, fiber filled insulation have the disadvantages of higher specific gravity and poor mechanical properties Although, silicas are widely used for the reinforcement of EPDM but in open literatures no much information are available on the effect of silica loadings on thermal and ablation characteristics of EPDM (Chaudhary and Jollands, 2008).

The influence of silica particles on the final properties of composites largely depends on their content, size, dispersing degree and interfacial adhesion with the polymer matrix. Zirconia-based ceramics are characterized by a unique combination of high strength, toughness and chemical resistance, which allows their use in harsh environments under severe loading conditions. Zirconia is very thermally stable to high temperatures (excellent thermal shock resistance), has low thermal conductivity, and is chemically inert. Therefore, in this work, the effect of fumed silica and zirconium silicate nanoparticles, on the thermal, electrical and mechanical properties of the γ-irradiated EPDM rubber composites was elaborated.

Experimental and techniques

Materials

EPDM (Buna G8850) was purchased from Lanxess, which contains (51%) ethylene content. Fumed silica was supplied by WACKER Chemie AG Company. It is synthetic, hydrophilic amorphous silica, produced via flame hydrolysis, refractive index 1.46, electric resistivity $>10^{13}$ Ω cm, BET-surface area 360-440m^2/ g and its commercial product name is HDK® T40. Zinc oxide and stearic acid were obtained from El gomhoria Company, Egypt. WingstayL antioxidant was obtained from ELIOKEM Company, France. The used Zirconium silicate was obtained from READE Company, USA.

Mixing and processing

Different Formulations of EPDM composites are listed in Table1. In these formulations, only the fumed silica and zirconium silicate content have been varied, while the content of the all other ingredients are kept constant. Mixing order of these ingredients to EPDM strongly influences the final properties of the vulcanizates; therefore the order of mixing is shown in Table1. Mixing of the EPDM with ingredients was carried out according to ASTM standard D3182-89 (Gajiwala and Himansu, 2004). The all ingredients of the selected formulation were accurately weighed and the mixing was carried out on a laboratory rubber mill type 150 with the following specification: the outside diameter is 470mm, Gear ratio= 1.14:1, working distance: 300mm and the speed of the slow roll is 24rpm. The temperature of the surface of the rolls was controlled during mixing and not exceeded 70^0C

TABLE 1. EPDM composites contain different concentrations of fumed silica and zirconium silicate.

Composite	EPDM (S40)	EPDM (S30/ Z10)	EPDM (S20/ Z20)	EPDM (S10/ Z30)	EPDM (Z40)
EPDM	100 phr	100 phr	100 phr	100 phr	100 phr
Wingstay L	1 phr	1 phr	1 phr	1 phr	1 phr
Zinc oxide	5 phr	5 phr	5 phr	5 phr	5 phr
Stearic acid	1 phr	1 phr	1 phr	1 phr	1 phr
Fumed silica	40 phr	30 phr	20 phr	10 phr	0 phr
Zirconium silicate	0 phr	10 phr	20 phr	30 phr	40 phr

Preparation of samples for irradiation

The prepared masticated sheets were cut to slabs. Sheets of ~1mm thickness were covered from both sides with polyester sheets in two clean polished stainless steel sheets before being pressed in Carver hydrolic hot press at 105^0C for at least 2min and a pressure of 160kg/ cm^2 on the mold surfaces.

Irradiation procedure

Irradiation of samples was carried out, in a gamma chamber-4000A manufactured by Bhabba Atomic Research Center, India, at the NCRRT, Nasr city, Egypt. The dose rate was about 2.18kGy/ h.

Transmission electron microscope (TEM) measurements

The particle size of the fumed silica and zirconium silicate was determined using TEM model JEM100CS, JEOL Electron Microscope, Japan, working at

acceleration voltage of 80kV and fitted with a digital camera (DXM1200, Nikon). Samples were prepared by the deposition of a dilute solution containing the nanoparticles specimen onto a 200mesh copper grid, which was coated with a carbon support film.

Scanning electron microscope (SEM) measurements

The morphology of samples was studied using JEOL JSM 5400 high resolution (Shimadzu Co., Japan), the orientation of photomicrographs was kept constant through the study. The surfaces of the samples were coated with thin film of gold (about 300-400µm thickness) using a vacuum evaporation technique.

Mechanical measurements

Dumbbell-shaped specimens were cut from the sheets using a steel die of standard width of 4mm and length of 15mm. The measurements of the mechanical properties, was carried out using a mechanical testing machine of hung-Ta Model HT-9112, Taiwan.

Tensile strength (TS)

The TS of a sample at break point can be calculated as follow:

$T.S = L / T.W$ kgf/ cm^2.

where, L: load in kgf necessary for causing break. T: thickness of sample in cm, W: width of sample in cm.

Elongation

The elongation at break point is expressed by the relation:

$E = L - L_0 / L_0$, %.

Where, L_0: The length of the sample between benchmarks, L: The length of the sample between benchmarks at the moment of rupture.

Thermogravimetric analysis (TGA) measurements

The thermal behavior of the samples was tested by applying the TGA technique using a TG-50 instrument from Shimadzu, Japan. Measurements were carried out under inert atmosphere of nitrogen gas. The wt of samples ranged from 3-5mg encapsulated in aluminium pans and heated from room temperature up to 600^0C and the rate of heating was 10^0C.

Electrical measurements

The volume resistivity of the samples was studied using Keithley electrometer, model 6517(Ohio, USA). The electrometer is connected externally with a Resistance Fixture, model 8009. The measurements of volume resistivity have been carried out at room temperature on a circular type probe of diameter 63.5mm and the average thickness (mm) of the tested sample has to be introduced at first to the data system of the electrometer.

Results and Discussion

Transmission electron microscopy (TEM)

The nanoparticle size was determined by measuring the number of contiguous pixels that meet the classification criteria for a particular micrograph. The number of pixels (or rather the area) is then converted into the equivalent circular diameter assuming a hard, uniformly spherical particle. Therefore, the particle size does not consider any pronounced faceting. The results after analysis show that the particle size of both fumed silica and zirconium silicate are 27.8nm, 40.5nm, respectively.

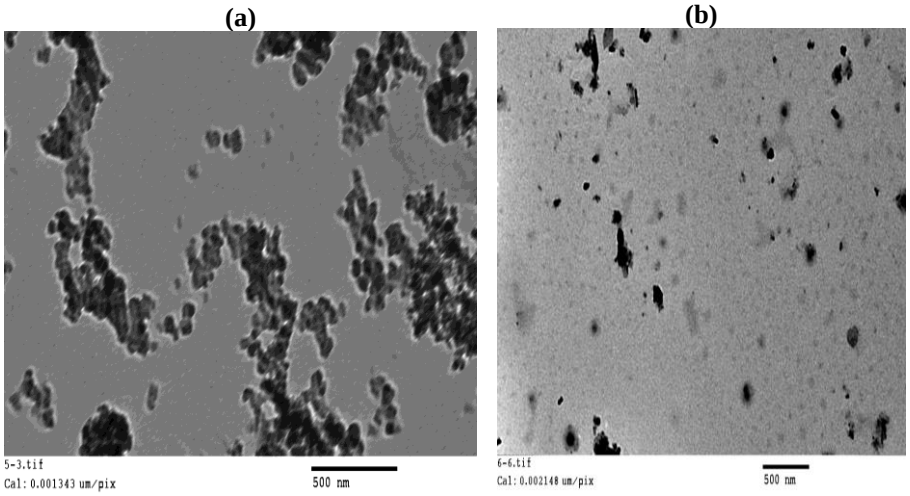

Fig. 1. TEM image of (a) fumed silica and (b) zirconium silicate.

Mechanical properties

One of the clearest effects of irradiation of elastomeric due to their cross-linking and degradation is the change in TS. Fig. 2. shows the variation of TS as a function of irradiation dose of EPDM rubber loaded with 40phr fumed silica, 40phr zirconium silicate and composites containing 10, 20 & 30phr fumed silica

with 30, 20 & 10 zirconium silicate, respectively. It can be seen that the values of TS for the all compositions increased with increasing irradiation dose reaching their maximum values at about 100kGy and after that, the values of TS decreased slowly on increasing the irradiation dose up to 200kGy. Irradiation causes two competing reactions in the rubber matrix namely; cross-link formation and scission of the macromolecular chains, depending on the total irradiation dose, dose rate and irradiation condition (in presence of air or inert gas). The reduction in tensile value may be due to the chain scission which is predominant at higher irradiation doses leading to polymer degradation.

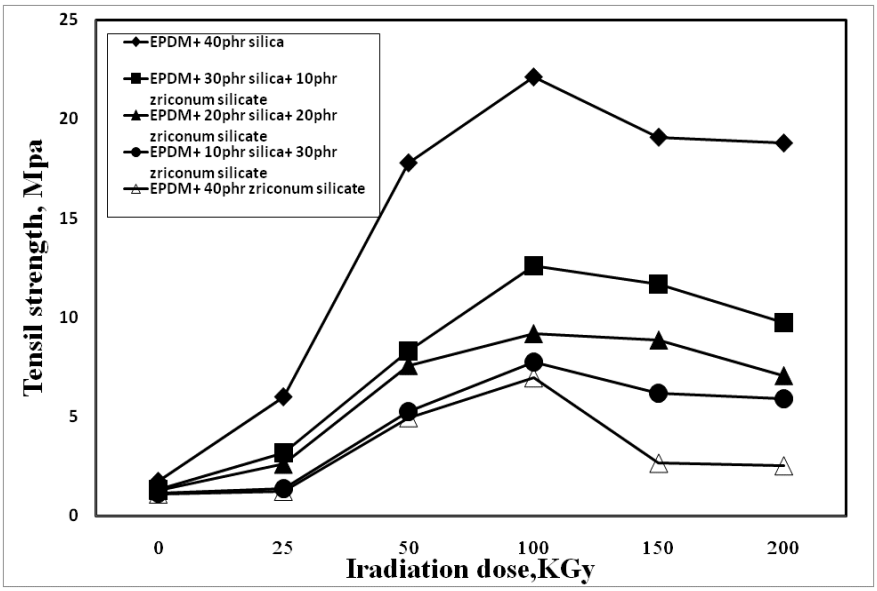

Fig. 2. Relation between irradiation dose and TS of EPDM rubber loaded with fumed silica and zirconium silicate with different concentrations.

Also from Fig. 2. it can be seen that the composite contain 40phr fumed silica has the highest TS. On other hand, the composite containing 40phr zirconium silicate has the lowest TS. However by partially replacement of zirconium silicate with fumed silica, the TS increased. According to the concepts of critical inter-particle distance and Percolation theory, the composites containing silica particles with a small size, a proper grafting degree and a primary-particle-level dispersion state are reached to improve the mechanical properties of composites and at the same time maintain the properties of matrix because of the low threshold values of silica content. Fig. 3. illustrates the

variation of the percentage of elongation at the break point (E%) as a function of irradiation dose of the former composites. It can be seen that, when increasing the irradiation dose, from 0-25kGy, the values of E% increased due to initial cross-linking occured after that, at higher irradiation doses, the crosslinking density icreased and hence, the values of E% for the all compositions decreased with different rates depending on the susceptibility of each formulation to the irradiation dose to form free radicals. Therefore, cross-linking density increased and hence, the hindrance of the mobility of the molecular chains increased which resulted in decrease in E%. Also it can be seen that the composite containing 40phr fumed silica has the lowest elongation, where the composite containing 40phr zirconium silicate has the highest elongation and by partially replacement of zirconium silicate with fumed silica elongation decreased.

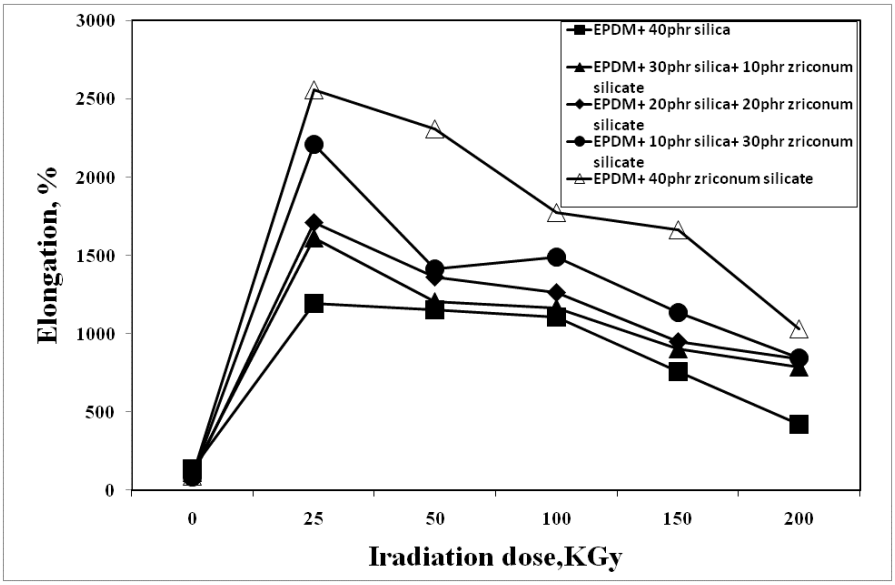

Fig. 3. Relation between irradiation dose and elongation of EPDM rubber loaded fumed silica and zirconium silicate with different concentrations.

Electrical properties

The volume resistivity at room temperature (~300K) of EPDM rubber loaded with fumed silica and zirconium silicate with different concentrations and irradiated to 100 kGy is presented in Table 2. It is known that the straight radiation of polymers, i.e. in absence of any kind of additives as well as oxygen, is accompanied by increasing their electrical conductivity. The radiation

induced conductivity arises from the creation of mobile charge carriers, namely, electrons, holes, and ions. It would be expected that the level of charge carriers to be a function of the polymer structure as well as the irradiation dose and the included fillers, i.e. type of filler, its particle size, content, dispersing degree and the interfacial adhesion between the filler and the polymer. Therefore, the high volume resistivity of the all composites is due to the high resistivity of the fillers and the rubber as well as the irradiation dose. The sample containing 40phr fumed silica has the highest volume resistivity and the partial replacement of zirconium silicate by fumed silica increased the volume resistivity of the composites containing zirconium silicate.

TABLE 2. The volume resistivity (Ω cm) of EPDM rubber loaded with different concentrations of fumed silica and zirconium silicate .

Sample	volume resistivity at 0kGy	volume resistivity irradiated to 100 kGy
EPDM+ 40phr zirconium silicate	4.8×10^{12}	4.10×10^{12}
EPDM+ 10phr silica+ 30phr zirconium silicate	5.4×10^{12}	4.8×10^{12}
EPDM+ 20phr silica+ 20phr zirconium silicate	6.5×10^{12}	5.9×10^{12}
EPDM+ 30phr silica+ 10phr zirconium silicate	6.8×10^{12}	6.2×10^{12}
EPDM+ 40phr silica	5.7×10^{13}	5.1×10^{13}

Scanning electron microscope

The morphology of EPDM rubber loaded with different concentrations of fumed silica and zirconium silicate and irradiated to 100 kGy was examined by scanning electron microscope. Fig. 4. (a-e) shows the morphology of these samples. As seen in Fig. 4. (a-e), fumed silica and zirconium silicate were observed on the entire failure surface, and it seems that these reinforcement fillers were homogeneously dispersed in the rubber matrix. Due to the smaller size of the fumed silica nanoparticles, these could be observed more homogeneous on the failure surface by SEM and this homogeneity depends on the content of the fumed silica to zirconium silicate in the composites. The effect of irradiation is clear; the surface is homogeneous, smooth and exhibits no indication of phase separation due to the occurrence of cross-linking.

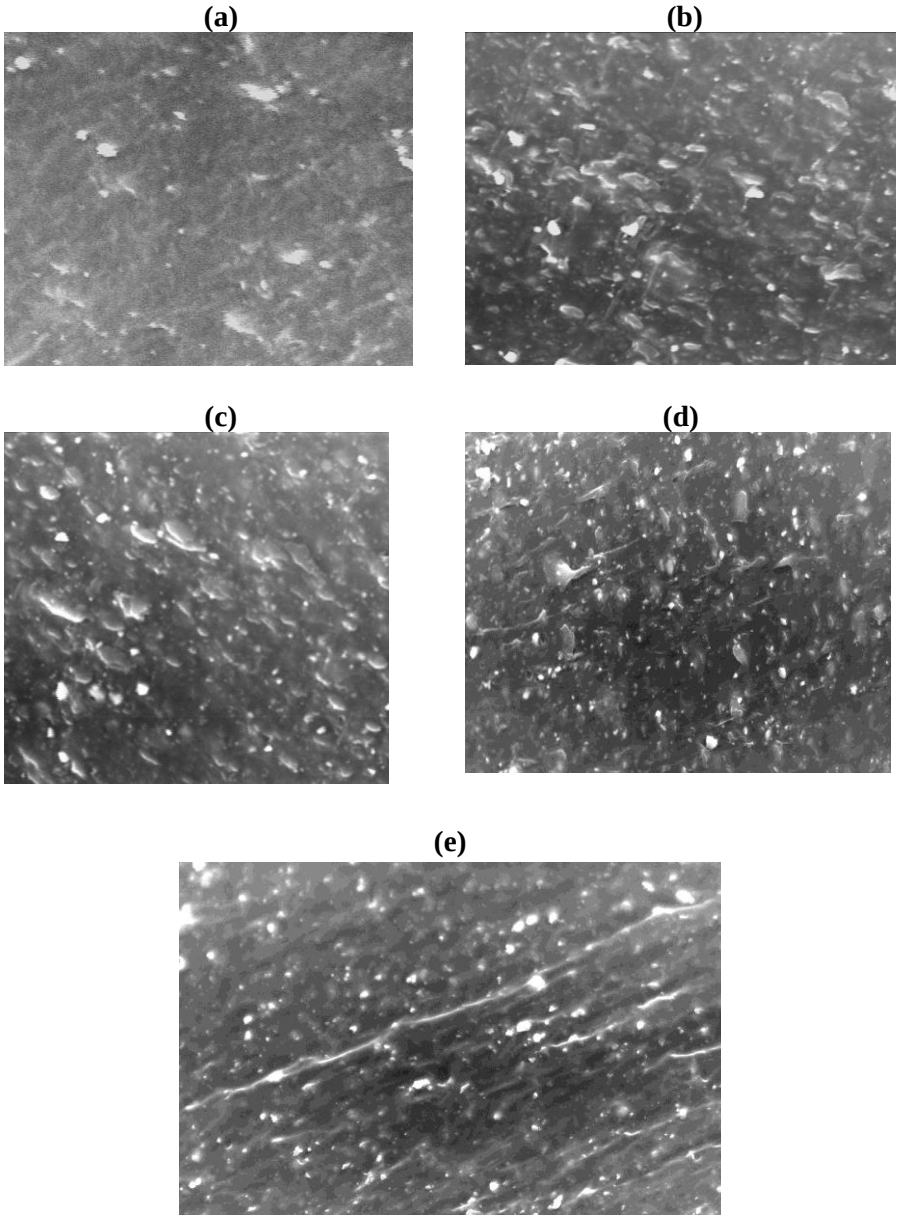

Fig. 4. SEM of loaded EPDM rubber and irradiated to 100kGy (a)40phr silica, (b)30phr silica+ 10phr zirconium silicate, (c)20phr silica+ 20phr zirconium silicate. (d)10phr silica+ 30phr zirconium silicate, (e)40phr zirconium silicate.

Thermal properties measurement

compared the thermal stability of sulphur, peroxide and radiation cured NBR/SBR vulcanizates, using TGA. They reported that the radiation cured formulation exhibited better thermal stability if compared to the other systems (Ahmed *et al.*, 2002).

Effect of silica loadings on thermal stability of EPDM composites

The effect of silica loading on the thermal stability of the EPDM irradiated to 100 kGy was characterized by TGA. The results shown in Fig. 5. & 6. are representing the TG and DTG curves of the samples, respectively. Besides, the data of TG-DTG curves of the all samples are presented in Table 3. The results showed that; the TG and DTG curves shifted to higher temperature with replacement fumed silica by zirconium silicate. T_{comp}(the temperature at which the weight loss was complete) and T_{max}(the temperature at which the rate of weight loss is maximum) of the composites increased indicating that the thermal stability of the samples increased with the increase of zirconium silicate content in the composites.

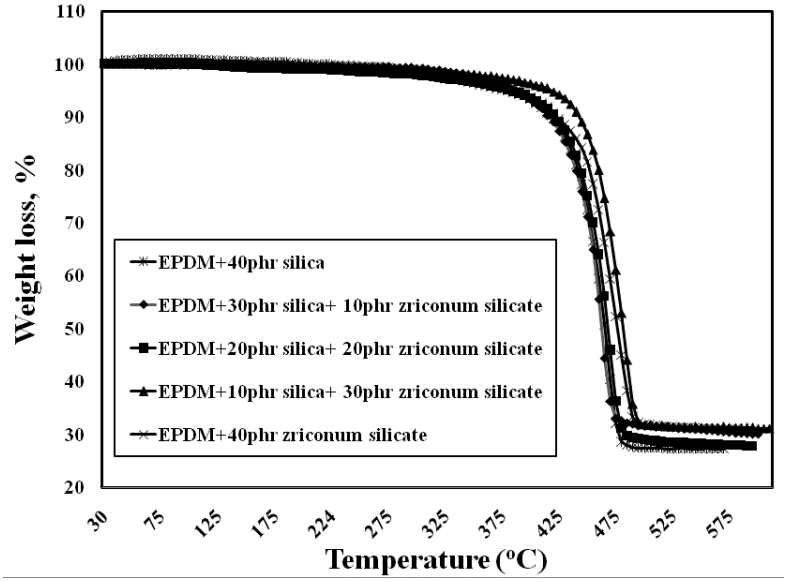

Fig. 5. TGA of EPDM rubber loaded with fumed silica and zirconium silicate with different concentration and irradiated to 100kGy.

All nanocomposites exhibit initial wt loss ($T_{10\%}$, temperature at 10 % wt loss) starting above 400^0C, indicating high thermal stability. $T_{10\%}$ of the composites increased with the increase of zirconium silicate content. However, the $T_{10\%}$, of the sample containing 40phr zirconium silicate is 15°C higher than that of the sample containing 40phr fumed silica. Silica is very hygroscopic materials and contained various amount of free and bond moistures (Jia *et al.*, 2008). Hence, the earlier commence of degradation and relatively higher wt loss at the early stage of heating of the samples containing fumed silica was due the vaporization of these moistures content. However, instead of the initial weight loss, T_{onset} (the temperature at which the sample starts decomposing.), T_{comp} and T_{max} of samples increased with the increase of zirconium silicate concentration in the EPDM composites, which is a confirmation of the thermal stability contributed by zirconium silicate.

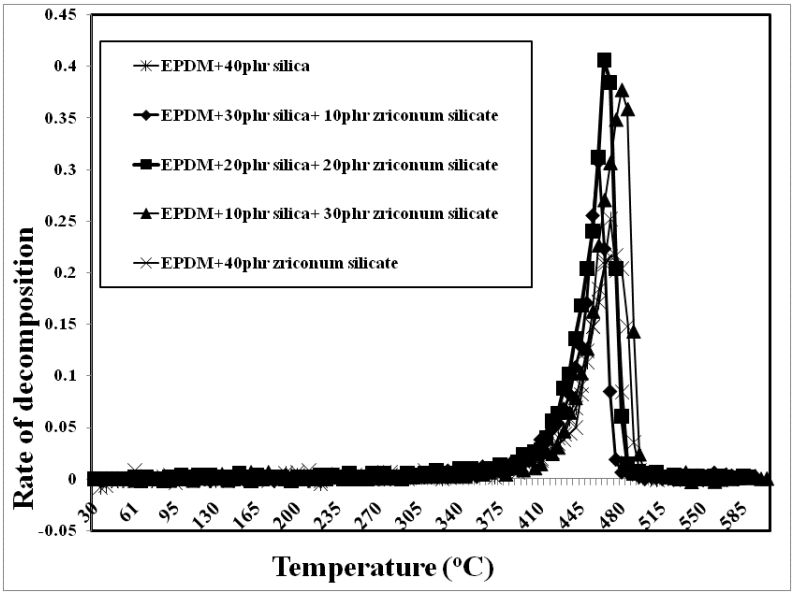

Fig. 6. Rate of decomposition of EPDM rubber loaded with fumed silica and zirconium silicate with different concentration and irradiated to 100kGy.

For example, the values of T_{onset}, T_{comp} and T_{max} of the sample containing 40phr of zirconium silicate are 22°C, 21°C & 16°C higher than those for the sample containing 40phr of fumed silica, respectively. The DTG curve of the sample containing fumed silica 40phr showed sharp peak at temperature, T_{max}, if

compared to that of sample containing zirconium silicate 40phr. The sharp peak indicates that the sample containing fumed silica 40phr decomposed at higher rate than that containing zirconium silicate 40phr. The considerable reduction in the decomposition rate of the sample containing zirconium silicate 40phr showed broad peak at T_{max} confirmed the thermal stability imparted by zirconium silicate, which may be the result of the decomposition of the samples at multiple overlapping steps. The results showed that the residue of the samples increased with the increase of the zirconium silicate content in the samples. EPDM is a hydrocarbon in nature and produce no char on decomposition (ASTM D3182-89, 1994). Zirconium silicate was very stable compound and did not decompose at the temperature of the experiment condition, resulted in increase in the char residue formation. The increase of char residues with increase of zirconium silicate content was also an indication of the formation of some stable compounds. The amount of char formation plays an important role in enhancing the thermal stability of the composites.

TABLE 3. Temperatures for different wt losses of EPDM rubber composites loaded with fumed silica and zirconium silicate with different concentrations and irradiated to 100kGy.

Sample ID	$T_{(10\%)}$ (°C)	T_{onset} (°C)	T_{comp} (°C)	T_{max} (°C)	Char Residue (%) at 600°C
EPDM+ 40phr silica	416	385	480	465	27.23
EPDM+ 30phr silica+ 10phr zirconium silicate	415	387	486	467	27.75
EPDM+ 20phr silica+ 20phr zirconium silicate	421	401	490	470	29.99
EPDM+ 10phr silica+ 30phr zirconium silicate	426	406	491	476	30.87
EPDM+ 40phr zirconium silicate	431	407	501	481	31.16

Effect of silica on the activation energy of thermal decomposition of the EPDM composites

The activation energy required for the decomposition of the materials is very important parameter for estimation the thermal stability and decomposition kinetic of the materials. Broido (1969) has developed a model in which the activation energy associated with each stage of decomposition is evaluated by this method.

The equation used for the calculation of activation energy (Ea) is:

$$\text{Lnln}\,(1/Y) = (-E_a/R)\,(1/T) + \text{constant}.$$

where, $Y = (W_t W_\infty)/(W_o W_\infty)$, Y is the fraction of the number of initial molecules not yet decomposed; W_t is the wt at any time t; $W\infty$ is the wt at infinite time (= zero), W_0 is the initial wt; E, R & T are the activation energy of thermal degradation, universal gas constant and absolute temperature, respectively.

A plot of lnln(1/Y) vs. 1/T gives an excellent approximation to a straight line. The slope is related to the activation energy. The procedure mentioned in (ASTM E 1641, 2007) was followed. Typical plots of lnln(1/Y) against 1/T are presented in Fig. 7. This figure showed the suitability of the Broido model for determination of the activation energy and decomposition kinetic as the experimental data best fit to equation1.

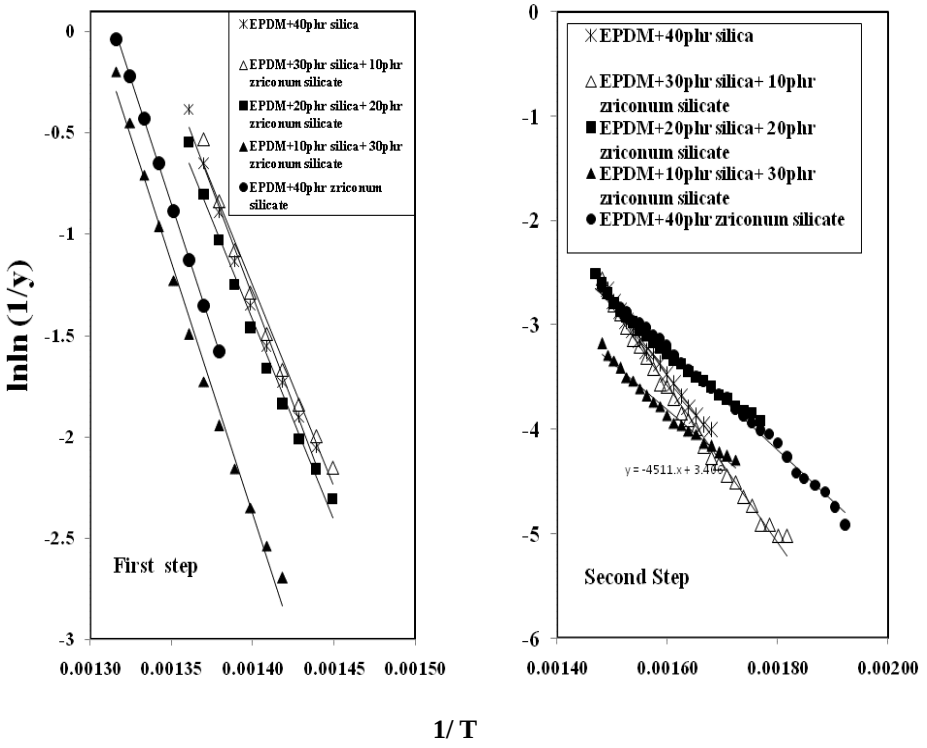

Fig. 7. Plot of lnln (1/y) versus 1/ T for conversion for EPDM rubber loaded with fumed silica and zirconium silicate with different concentration and irradiated to 100kGy.

The activation energy data calculated from the slopes of the straight lines in these figures are shown in Table 4. It is clear from Table 4. that the activation energy increased by increasing zirconium silicate loading. The increases in the activation energy indicate that some stable compounds formed during decomposition that required higher energy for thermal decomposition. The relative higher activation energy for decompositions related to higher thermal stability of the composites.

TABLE 4. Calculation of the activation energy for silica-EPDM composites.

EPDM composites	Slop of first step	E_a(kJ/mole)= slop*R
EPDM+ 40phr silica	7914	658
EPDM+ 30phr silica+ 10phr zirconium silicate	8062	670
EPDM+ 20phr silica+ 20phr zirconium silicate	8231	684
EPDM+ 10phr silica+ 30phr zirconium silicate	9362	778
EPDM+ 40phr zirconium silicate	9430	784

Conclusion

The effect of γ-irradiation on the values of TS for the all compositions increased with increasing irradiation dose reaching their maximum values at about 100kGy and after that, the values of TS decreased on increasing the irradiation dose up to 200kGy. Whereas the values of (E%) for the all compositions decreased with increasing of the irradiation dose. The composite containing 40phr fumed silica has the highest TS and by partially replacement of fumed silica with zirconium silicate, the TS decreased but the elongation relatively increased and the composite containing 40phr fumed silica has the lowest elongation over the all irradiation doses considered. The composites irradiated to 100kGy have the best mechanical properties; hence the thermal, electrical and morphology have been studied at that irradiation dose. It can be concluded that the volume resistivity increased with increasing the level of fumed silica. Hence, the composite containing 40phr of fumed silica has the highest volume resistivity. The TG and DTG curves shafted to higher temperature with replacement fumed silica by zirconium silicate, T_{comp} and T_{max} of the composites increased also. This indicates that the thermal stability of the samples is increasing with the increase of zirconium silicate content in the composites. All nanocomposites exhibit initial wt loss ($T_{10\%}$) above 400^0C, indicating high thermal stability. The temperature of the initial loss, $T_{10\%}$ of the composites

increased with the increase of zirconium silicate content; whereas the $T_{10\%}$, of the sample containing zirconium silicate 40phr was 10°C higher than that of the sample containing fumed silica 40phr. T_{onset},T_{comp} and T_{max} of the sample containing zirconium silicate 40phr are 22°C, 21 °C & 16°C higher than those of fumed silica 40phr, respectively. The char residue formation and the activation energy of the composites have been increased with the increase of zirconium silicate content in the samples. The increase in the activation energy indicated that some stable compounds formed during decomposition that required higher energy for thermal decomposition. The relative higher activation energy for decompositions related to higher thermal stability. It can be generally concluded that EPDM/ fumed silica and zirconium silicate composites irradiated to 100kGy are characterized by having good thermal and electrical insulation properties and hence, it has wide industrial applications as good thermal and electrical insulating materials.

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الخـواص الفيزيوميكانيكيــة لمتراكبــات مــن مطــاط الإيثيلــين بـروبلين داين المشـعع بأشـعة جامـا و المحمل بالنـانو سـليكا و سليكات الزركونيوم

محمد منصور عبد العزيز ، هانى احمد عامر* ، منى كمال عطية و عبد الجواد محمد ربيع**

قسم كيمياء الأشعاع ، المركز القومى لبحوث و تكنولوجيا الأشعاع ، ص. ب: ٢٩ مدينة نصر ، *هيئه الرقابه النوويه و الاشعاعية ، مدينة نصر ، **قسم الكيمياء ، كلية العلوم ، جامعة عين شمس ، مصر.

تم تحضير متراكبات من الايثيلين بروبلين داين محتوية على نسب مختلفة من مواد مالئة متناهية الصغرمن السليكا و سليكات الزركونيوم بنسب مختلفة ٣٠:١٠ الى ١٠:٣٠ من كليهما و عينات تحتوى على ٤٠ جزء لكل١٠٠ جرام مطاط من كل منهما على حده وتم معالجتهم بأشعة جاما و دراسة تأثير المواد المالئة على الصفات الكهربائية و الحرارية للمتراكبات. بدراسة الخواص الميكانيكية عند جرعات إشعاعية مختلفة وجد أن المتراكبات عند ١٠٠ كيلو جراى هى الأفضل لجميع المتركبات و بقياس الصفات الكهربائية و الحرارية وجد أن المتراكبات المحتوية على نسبة أعلى من السليكا أكثر مقاومة كهربائية و المتراكبات المحتوية على نسبة أعلى من الزركونيوم أكثر مقاومة للحرارة و تتدرج تلك الصفات باختلاف نسب كل منه.