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Fire Retardancy, Thermal Stability and Irradiation Studies of Modified Poly (Vinyl Chloride) composites for Industrial Applications

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Poly(vinyl chloride) (PVC) composites were prepared by compounding different concentrations of antimony trioxide Sb_2O_3 (ATO) and Organo-modified montmorillonite clay (OMMT) as fillers under the effect of gamma irradiation in order to improve its fire retardancy properties. The fire retardancy of the prepared PVC composites were evaluated by measuring ofthe limiting oxygen index (LOI). The mechanical properties, thermal stability using thermogravimetric analysis (TGA) and the electrical conductivity were also investigated. The results indicated that the addition of ATO alone up to 3 phr or the mixture of ATO 1.5 phr/ OMMT 1.5 phr enhances the fire retardancy properties, accompanied with a decrease in the mechanical properties, without effective change in the thermal and electrical conductivity. Moreover, it was found that the mechanical properties and the gel content of the prepared PVC composites were enhanced accompanied with a decrease in the swelling ratio, as a function of the irradiation with gamma rays up to 50 kGy.

Keywords: *Poly (vinyl chloride)/ Thermal stability/ Fire retardancy/ Irradiation*

Introduction

Poly(vinyl chloride) (PVC) composites were used for the cable insulation and the artificial leathers fabrication, in which they are characterizes by their good electrical insulating nature, acceptable thermal stability, easy and economic cost processing and their inherently flame retardant nature **[1]**. It was reported in previous studies that the PVC compounds have self-fire retardancy properties, due to the presence of about 50% of chlorine, which is the source of the hydrogen chloride gas production, which slows down the combustion process, shields the PVC composite surface from oxygen in the air, leading to stopping the burning progress.

PVC composites were modified by different methods in order to enhance their thermal stability and fire retardancy properties, which are required for different industrial applications. In general, the fire retardancy properties of polymeric materials can be enhanced by the use of two procedures: the introduction of additives during the hot mixing process and the introduction of the chemical compounds, which containing chosen metals, which can enhances the fire retardancy of the polymeric composites. These materials are boron, aluminum, phosphorus, antimony, chlorine and bromine [2-4].

Antimony trioxide (Sb_2O_3), aluminum hydrate ($Al(OH)_3$) and magnesium hydroxide ($Mg(OH)_2$) as inorganic compounds can be used to improve the fire retardancy of the PVC composites. In this regard, antimony trioxide (ATO) is preferred, because it contains about 83.53% antimony and 16.47% oxygen, which act as a co-synergist with halogenated flame retardant to enhance their effectiveness [5]. On the other hand, the organo-

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modified montmorillonite clay (OMMT) can be also used to improve the fire retardancy of the PVC composites [6],as it promotes the dehydrochlorination reaction of PVC and reduce the heat release rate for PVC composites [7,8], leading to hindering the ignition process[9, 10]. Moreover, the addition of OMMT enhances the thermal stability of PVC composite through delaying the allylic species loss (6). Moreover, the mechanical properties [11-13], gas permeability and other barrier properties were also enhanced [14].The interaction between gamma rays and polymers may lead to cross linking or chain scission processes, through formation of free radicals [15-17].The irradiation of PVC composites, as an example of solid polymer, predominant reactions can take place include cross linking, chain scission and dehydrochlorination processes depending on the irradiation doses [18, 19].

In the present work, different concentrations of ATO and OMMT as fillers were added to PVC composites in order to enhance their flame retardancy. The effect of the additives on the thermal stability, mechanical properties, gel and swelling ratios and the electrical properties was investigated. In addition, the effect of gamma irradiation on the physical and the mechanical properties of modified PVC composites were also studied.

Materials and Methods

Materials

A commercial formulation of PVC used as cable coating was supplied by the Electro Cable Co., Cairo, Egypt. A laboratory grade antimony trioxide (Sb_2O_3) as a flame-retardant filler with was supplied from Fluka Chemical Co., Germany. A commercial grade sodium- montmorillonite clay (MMT) was provided by Zhejiang Huate Group Co., China. Cetyltrimethylammonium bromide (CTAB)(laboratory grade) used for clay modification, was provided from Fluka, Germany. Toluene with a laboratory grade was purchased from El-Gomhoria Company, and it is used as a solvent for gel yield and swelling characterization.

Preparation of organo-montmorillonite clay (MMT)

For the preparation of the tertiary amine modified MMT clay, 5 g of both of MMT clay and cetyltrimethylammonium bromide (CTAB) were dissolved separately in water at 80^{o}C for half an hour. Then the dissolved (CTAB) was mixed together with the clay dispersion under continuous steering to obtain the modified clay (OMMT). The produced modified clay was then washed, filtered and dried in a vacuum oven at room temperature.

Preparation of PVC composites

The commercial PVC formulation batch was mixed with 1phr, 3phr of antimony oxide and 1, 3 phr mixture of ATO and OMMT clay with 50:50 ratio of both using a Laboratory Plastic order mixer at a mixing speed of 60 rpm for 5 min at 160^{o}C. The produced hot samples were immediately transferred from the mixer to an open two roll mill to make sheets. The sheets were then pressed at 170^{o}C for 5 min (after 2 min preheating) and at a pressure of 300 kg/cm^2 to obtain rectangular sheets of 1 mm thickness. The molded sheets were then immediately transferred to water-cooled press at the same pressure.

Gamma irradiation

The samples were irradiated with Cobalt-60 as a source of gamma rays (made in Russia) installed at National Center for Radiation Research and Technology (NCRRT), Cairo, Egypt. The irradiation process was carried out at a dose rate of 2.18kGy/h. The sheets were exposed to irradiation doses of 10, 20, 50 and 75 kGy.

Mechanical properties

The measurement of the mechanical properties were carried out using a microprocessor controlled tensile testing machine of Hung-Ta Model HT-9112 (Taiwan),with load cell 100 kgf and fitted with an extensometer model HT 8160. The crosshead speed was 500 mm/min.

X-Ray diffraction measurements

X-ray diffraction (XRD) studies were performed using Philips (PW 1390), using Nickel- filtered Cu-Kα radiation. The diffract grams were scanned from 2 to 40^o at room temperature.

Measurement of limiting oxygen index (LOI)

Limiting oxygen index (LOI) tests were conducted according to GB/T 2406—1993 by the use of an oxygen index instrument (JF-3), made by Nanjing Jiangning Analytical Instrument Factory, China. The tested samples were cut into dimensions of 50 mm by 150 mm and then clamped vertically to the holder of the LOI tester. Both nitrogen and oxygen were connected to the apparatus through pressure regulators. Before the ignition, the nitrogen/oxygen ratio should be set and pre-conditioned for 30 s. The minimum concentration of oxygen in a mixture of oxygen and nitrogen which is required to supports the combustion process under equilibrium conditions was measured for the tested sample. The average of three samples was taken for every sample to overcome the poor repeatability of data.

Thermo Gravimetric analysis (TGA)

The thermal behavior of samples was tested by applying the TGA technique using a TG-50 instrument from Shimadzu, Japan. The measurements were carried out under an inert atmosphere of nitrogen gas. The samples were encapsulated in aluminum pans and heated from room temperature up to 600^0C, with rate of heating of was 10^oC/ min.

Electrical conductivity measurements

The DC electrical conductivity was measured using of the two-electrode method, in which, the samples were painted from both sides with silver paste, and then they are placed between two brass electrodes to ensure a perfect composite-electrode contact. The values of DC electrical conductivity were calculated using equation (1):

EC = (I\V) (t\A) (1)

Where **I** represents the electrical current measured experimentally using a digital electrometer model 6517 type Keithley, **V** represents the applied voltage (20V), while **t** and **A** represent the thickness and the area of the tested sample, respectively. Where: Area = πr^2 for a circular disk, r typically 0.3 cm. All the measurements were taken at room temperature.

Gel content measurement

The samples were extracted in Toluene under boiling condition for 24 h. The insoluble part was dried under vacuum at 70 ^{0}C to a constant weight. The gel content was calculated using equation (2):

Gel fraction (%) = [(W_2/W_1)] x100 (2)

Where W_1 andW_2 represented the initial sample weight and the weight of the insoluble part.

Swelling measurements

The gel yield was allowed to swell in Toluene for 48 h at room temperature. The swollen samples were gently dried using tissue paper and weighed. The swelling ratio was calculated by using of equation (3):

Swelling ratio = (W_2-W_1)/ W_1 (3)

Where W_1 and $W_2$are represented the sample weights before and after swelling.

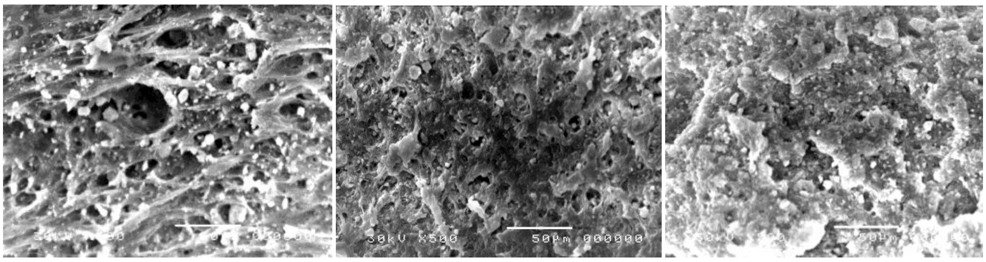
Scanning Electron Microscope (SEM)

The samples morphology was studied using JEOLJSM 5400 high resolution, Japan. The orientation of photomicrographs was kept constant through the study.

Results and Discussions

Scanning electron microscopy (SEM)

The morphology of PVC composite (a), PVC loaded with ATO3 phr (b) and OMMT1.5 phr with ATO 1.5 phr(c) was examined by SEM, as shown in Figure (1a-c).With addition of ATO as fine white inorganic filler, the surface of the samples showed a somewhat tough rupture, a brittle and rough fracture surface accompanied with appearing whiteness. While, the addition of OMMT clay, sharing with ATO (1:1) to the PVC composites, it was observed to be more rough and porous than the surface of PVC, accompanied with the formation of cavities, in which the OMMT clay acts as an internal lubricant, and decreasing the frictional energy between PVC particles, which leads to the formation of cavities on the surface of PVC composites.



(a) (b) (c)

Figure (1):SEM micrographs of unmodified PVC composite (a), after addition of ATO 3phr(b) and after addition of OMMT clay 1.5 phr/ ATO 1.5 phr(c)

X- Ray diffraction

The effect of the addition of ATO (3 phr) or ATO 1.5 phr/ OMMT 1.5 phr on the XRD of PVC composites is shown in Fig.(2). For unmodified PVC composites, a sharp characteristic diffraction peak can be seen at$2\theta = 29.807^o$, which represents their crystalline nature. With addition of ATO (3 phr) to the PVC composites, a new characteristic diffraction peak can be seen and shifted to lower value of $2\theta = 29.347^o$, accompanied with a decrease in the peak intensity by ~ 40%, as a result of the decreasing in the total crystallinity proportion of PVC composites and the change in the particles orientation. Moreover, with addition of ATO 1.5phr/ OMMT clay 1.5 phr to the PVC composites, a new characteristic diffraction peak, shifted to a lower angle value at $2\theta = 22.592^o$ can be seen, with a decrease in the peak intensity by ~ 50%, accompanied with an increase in the interlayer distance (d) from ~ 2.995 to ~ 3.933 nm, which indicated the intercalation of the OMMT clay by PVC macro chains[21].

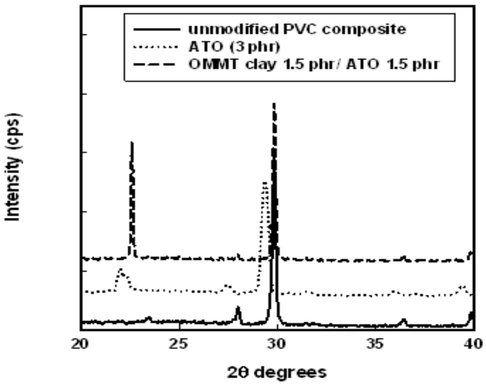


Fig.(2):X-ray patterns of PVC composite before and after the addition of different fillers

Effect of the composition on the mechanical properties of PVC composites

The effect of addition of different concentration of ATO or its mixtures with the OMMT clay on the mechanical properties of PVC composites were investigated as shown in Table (1).It can be seen that, the tensile strength (TS) and elongation at break (E%) of the PVC composites were decreased with increasing of ATO concentrations from 1-3 phr., due to the effect of ATO, which influences the oxidative degradation processes and catalyze the dehydrochlorination, leading to decreasing the overall mechanical properties [21].

On the other hand, with addition of the OMMT clay as a filler mixed with ATO (1:1),it can be seen that, the (TS)of the PVC composites were decreased by 21% and 28%, while, the(E%) were decreased by 24% and 25% by increasing of the OMMT clay concentration from 0.5 to 1.5 phr, respectively compared with the unmodified PVC composites. This could be attributed to the dispersing difficulty of the OMMT clay through the PVC composites, accompanied with the agglomerates of the clay [22].In addition, the poor bonding, between OMMT clay and PVC composites, leads to reducing of the overall mechanical properties, including the (TS) and the (E%), which is lower than that in the case of addition of ATO alone to the PVC composites.

Table (1): Effect of the addition of ATO and OMMT clay on the mechanical properties of PVC composites

Ingredients of PVC composites			TS (Mpa)	E (%)
Unmodified PVC comp.	-----	-----	16± 0.4	160± 3
Unmodified PVC comp.	ATO 1 phr	-----	15± 0.7	159± 1
	ATO 3 phr	-----	13± 1	144.3± 3
	ATO 0.5 phr	OMMT 0.5 phr	12.6± 0.8	122± 4
	ATO 1.5 phr	OMMT 1.5 phr	11.5± 0.2	120± 3

Effect of the Irradiation on the mechanical properties of PVC composites

The irradiation process can cause two competing reactions in the polymer matrix; crosslinking or chain scission processes, depending on the total irradiation conditions [23]. Therefore, due to the important effect of the irradiation on the mechanical properties of PVC composites, the (TS) and (E%) of the PVC composites containing different concentrations of ATO and OMMT clay, as the function of irradiation dose was investigated and shown in Fig.(3). It can be seen that the values of (TS) for all composites were increased with increasing of the irradiation dose, reaching their maximum values at about 50 k Gy, as a result of the irradiation crosslinking effect, and then it tends to decrease as it noticed with irradiation to 75kGy, in which the degradation process begins as a result of the radiation chain scission process.

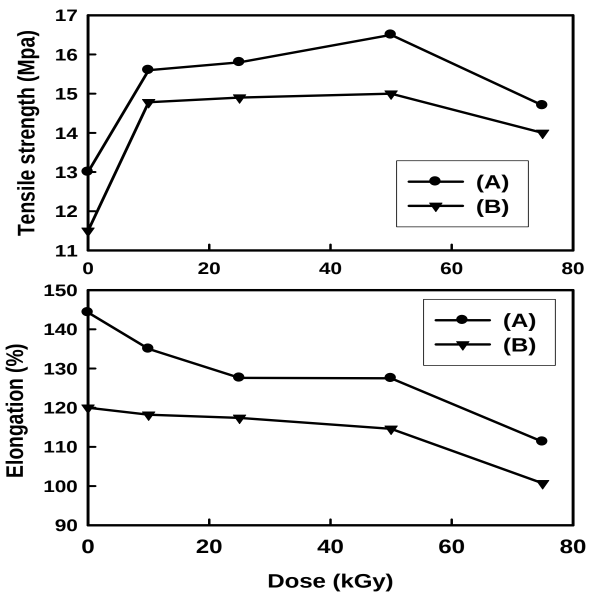


Fig.(3): Effect of irradiation dose on thetensile strength (Mpa) and elongation at break (%) of modified PVC composite by addition of ATO 3 phr (A) and OMMT clay 1.5 phr/ ATO 1.5 phr (B)

Figure(3)shows the effect of irradiation dose on the tensile strength (Mpa) and elongation at break of modified PVC composite by addition of ATO 3 phr (A) and OMMT clay 1.5 phr/ ATO 1.5 phr (B). It can be seen that the values of (E%) for the all PVC composites were decreased with increasing of the irradiation dose. This could be attributed to the increasing of the crosslinking density, leading to hindrance of the mobility of the molecular chains, resulted in the decreasing of overall (E%).

Swelling characteristics and gel content of modified PVC composites

The swelling ratio and gel content of the modified PVC composites with the ATO and OMMT after irradiation with different doses of gamma rays were investigated. It was mentioned that the crosslinking of the polymeric matrix prevents their extension and swelling. Therefore, the swelling ratios can represent the network chain density of the polymer matrix [24]. Figure (4) shows the relation between irradiation dose and the swelling ratios of PVC composites loaded with ATO 3 phr and ATO 1.5 phr with OMMT clay 1.5 phr. It can be seen that the attained swelling ratios for all PVC compositions have decreased with increasing of the irradiation dose, which indicates the crosslinking occurrence induced by irradiation process and formation of three-dimensional network structure of PVC composites. Moreover, the composite which contains ATO3 phr has attained lower swelling ratio than that which contains ATO 1.5 phr and OMMT clay 1.5 phr. This could be attributed to the poor bonding, between OMMT clay and PVC, which has a hindrance effect on the total crosslinking as a result of the irradiation effect.

Moreover, as a result of the irradiation process, PVC chains crosslink and form interpenetrated networks or polymeric gels which are difficult to dissolve. Therefore, increasing the gel contents indicates an increase in the crosslinking level in PVC composites [25]. Figure (4) shows the variation of gel content in toluene as a function of irradiation dose for the above samples. It can be seen that the values of gel content of PVC increases with increasing of the irradiation dose. On the other hand, the gel content values of the PVC composites which loaded with ATO 1.5 phr and OMMT clay 1.5 phr gave higher values than the composites which loaded with ATO 3 phr only, as result of the poor bonding, between OMMT clay and PVC, which decreases the total obtained crosslinking level.

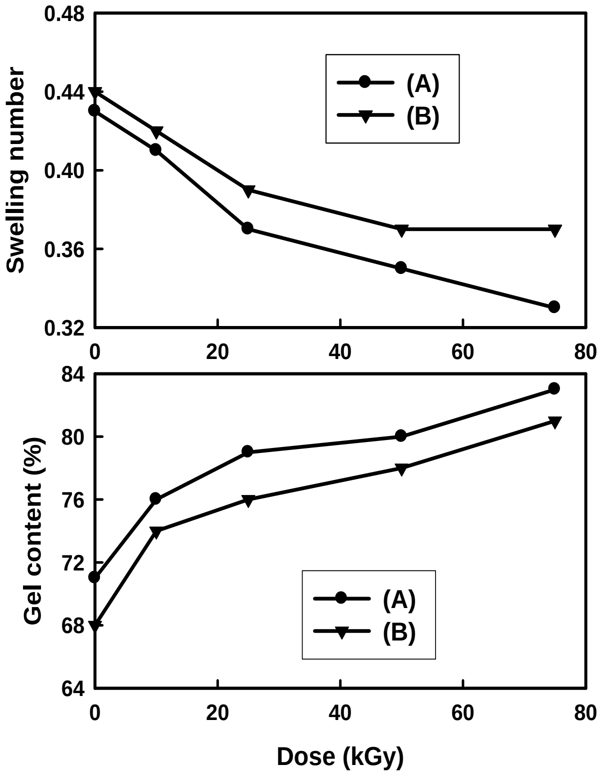


Figure.(4):The effect of the irradiation dose on the swelling numberand gel content of modified PVC composite by addition of ATO 3phr (A) and OMMT 1.5 phr/ ATO 1.5 phr (B)

Thermal properties of PVC composites

Figure (5)shows the TGA thermo grams and the corresponding rate of thermal decomposition reaction curves of unmodified PVC composite, after addition of ATO 3phr (A) and after addition of OMMT clay 1.5 phr/ATO 1.5 phr (B).The temperature parameters are illustrated in Table(2),in which the T_{comp} represents the temperature at which the weight loss was completely decomposed and T_{max} represents the temperature at which the maximum rate of reaction occurred. It can be seen that, the thermal decomposition of PVC composites goes through two main stages; the first stage (from 200–400^{o}C), represents the dehydrochlorination process, accompanied with the releasing of HCl gas, while, the second stage (from 400–560^{o}C) represents the condensation and fragmentation of polyene sequences to form aromatic compound [26, 27].With addition of the ATO (3 phr), two processes can be expected: in the first the ATO as an inorganic material with high thermal stability and great barrier properties can prevent heat from transmitting quickly and thus limit the continuous decomposition of the PVC composites. The second is the possibility of the reaction of the ATO with the evolved HCl gas as a result of the dehydrochlorination process, leading to the promoting early dehydrochlorination and early formation of char [28]. Thus, as result of the two expected possibilities, no noticeable change in the overall thermal stability of PVC composites can be observed by addition of ATO, in which, there was no noticeable difference in the onset, midpoint, end set and the T_{max} temperatures, that for both of two stages of thermal decomposition, except for the increase in the rate of thermal decomposition in the primary heating stage, accompanied with a slight increase in the final char residue by 3% at 600^{o}C as illustrated in Table(2).

In the case of the addition of the mixture of OMMT clay / ATO (1.5 phr: 1.5 phr) to the PVC composites, it can be noticed that there is a slight decrease in the thermal stability, especially in the first stage of thermal decomposition of the modified PVC composites than that for the unmodified one. This is illustrated in Figure.(5) and Table (2),in which, the onset, midpoint and end-set temperatures were 284, 293, 307 and 283, 295, 313 for the first stage of the thermal degradation, before and after addition of (ATO/ OMMT; 1.5 phr: 1.5 phr) to the PVC composite, respectively, while the final char residue was increased by 4% at 600^{o}C.This could be attributed to the effect of the alkyl ammonium modifier of OMMT clay, which can speed up the thermal degradation process of the PVC/OMMT clay composite **[29]**, due to the formation of alkene, tertiary amine and HCl, according to Hofmann’s elimination mechanism. The presence of the acidic sites can catalyze the polymer matrix degradation and accelerates the dehydrochlorination process of the modified PVC composites, resulted in a shift of initial temperature of degradation towards lower temperature region [30, 31].

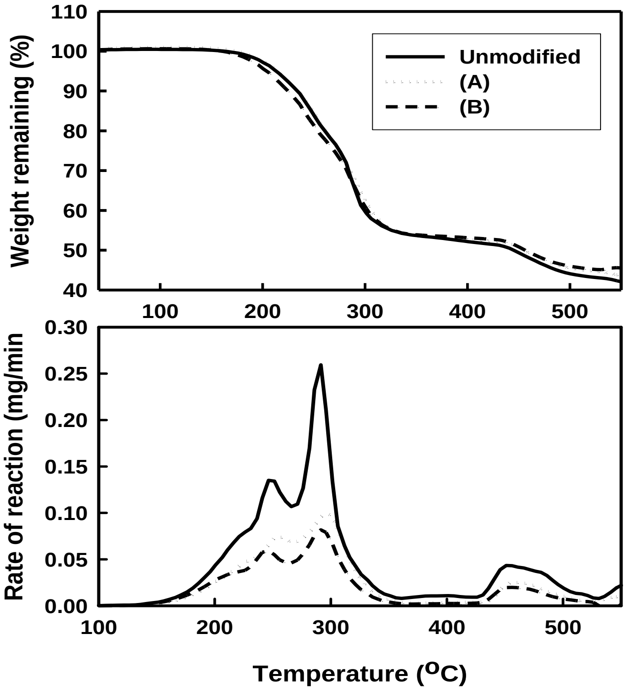


Figure.(5):The TGA thermograms and the corresponding rate of thermal decomposition reaction curves of unmodified PVC composite, after addition of ATO 3phr (A) and after addition of OMMT clay 1.5 phr / ATO 1.5 phr (B)

Effect of the modification on the LOI of PVC composites

Different polymeric materials can be classified based on their LOI, according to the required oxygen percentage to supports the combustion process under equilibrium into different categories, as illustrated in Table (3).

In the present work, the effect of addition of different contents of ATO, and OMMT clay to the PVC composites on the LOI was investigated. Table (4)presents the limiting oxygen index (LOI) of various samples of PVC; before and after addition of 1 and 3phr of ATO, 0.5phr of ATO /0.5 phr of OMMT and 1.5phr of ATO/ 1.5phr of OMMT clay, respectively. From the obtained LOI results, it can be seen that the addition of ATO to PVC composite enhanced the fire retardancy, in which the LOI result was increased from 20.8 for the PVC composite to 23 after the addition of 1phr of ATO and increases to 23.6 by addition of 3phr of ATO. This could be attributed to the reaction of ATO with the evolved HCl gas through the PVC burning process, and formation of antimony oxchlorides, which finally decompose to form antimony tri chloride ($SbCl_3$), that is the responsible of the flame reaction inhibition thought the scavenging of the free radicals and introducing of chlorine during combustion.

Table (2):Temperature parameters of the thermal decomposition of PVC composites

PVC Composites	Onset	Midpoint	Endset	Wt. loss (%)	$T_{10\%}$(°C)	T_{Comp}(°C)	T_{max} (°C)	Char Residue (%) at 600°C
Unmodified	284	293	307	24	239	492	293	39%
	441	466	492	8.1				
PVC/ ATO 3 phr	285	296	318	25	234	482	296	42%
	445	461	482	5.75				
PVC/ATO1.5phr/	283	295	313	23%	250	489	295	43%
OMMT1.5 phr	443	465	489	7%				

Table (3): Classification of materials based on the LOI values

LOI values	Flammability state
<20.95	Flammable
20.95	Almost stable
21- 28	Slow burning
28- 100	Self extinguishing
>100	Non flammable

Therefore, it can be concluded that ATO can be used as a co-synergist with halogenated flame retardants to enhance their effectiveness [32, 33].

On the other hand, by addition of OMMT clay, sharing with ATO to the PVC composites, the overall LOI results were decreased, but it still more than the virgin PVC composites, in which the rate of heat release was decreased compared with PVC composite, as a result of the producing of an external layer during the combustion process that acts as a heat and oxygen transport barrier, resulted in the delaying of the ignition process of PVC [7, 8].

Table (4):Effect of ATO and OMMT clay contents on the LOI of PVC composites

Ingredients of PVC composites			LOI
Unmodified PVC comp.	-----	-----	20.8
Unmodified PVC comp.	ATO 1 phr	-----	23
	ATO 3 phr	-----	23.6
	ATO 0.5 phr	OMMT 0.5 phr	21.4
	ATO 1.5 phr	OMMT 1.5 phr	22.6

Electrical properties of the modified PVC composites

The effect of the modification of PVC composites by addition of different concentrations from ATO and OMMT clay on their electrical conductivity (EC) was investigated. As illustrated in Table (5), it can be seen that the EC values of the PVC composites were slightly increased with increasing the ATO concentration from 1 to 3 phr. This could be attributed to the increase of charge carrier quantity and ionic charge due to penetration of antimony (Sb) into chain polymer of PVC composite[34].On the other hand, with addition of the OMMT clay sharing with ATO to the PVC composites, a slightlyultra-increasing in the electrical conductivity with replacement of ATO by the modified claycan be noticed. From Table(5), it can be seen also that the values of the electrical conductivity for all modified PVC composites show acceptable values for cable standards, according to ASTM requirements (ASTM D 257; EC $\geq 10^{-12}$) and they are suitable for cable applications and other industries which require insulting PVC composites[28].

Table(5): Electrical conductivity of unmodified and modified PVC composites modified by different concentrations of ATO and OMMT clay

Ingredients of PVC composites			Electrical conductivity ($\Omega^{-1}cm^{-1}$)
Unmodified PVC comp.	-----	-----	$1.3*10^{-13}$
Unmodified PVC comp.	ATO 1 phr	-----	$4.8*10^{-12}$
	ATO 3 phr	-----	$5.9*10^{-12}$
	ATO 0.5 phr	OMMT 0.5 phr	$6.5*10^{-12}$
	ATO 1.5 phr	OMMT 1.5 phr	$8.8*10^{-12}$

Conclusions

Due to the importance of the PVC composites in different industrial applications, especially in cable insulators and the artificial leather, so many studies focused on improving its properties such as the mechanical, thermal and fire retardancy properties. This article focused on improving the fire retardancy properties of the PVC composites through the addition Antimony trioxide and organo-modified montmorillonite clay and investigating the effect of the additives on the mechanical properties, gel content, swelling ratio, thermal stability and the electrical conductivity of the modified PVC composites. Moreover, the effect of the gamma irradiation on the mechanical, gel content and the swelling ratio were studied also. From the results, it can be concluded that the investigated fire retardancy using (LOI) measurement of PVC composites was enhanced, with keeping the thermal stability and electrical conductivity, accompanied with an acceptable decreasing in the mechanical properties. On the other hand, the irradiation processes up to 50 kGy enhanced the mechanical properties and these properties tended to decrease with higher irradiation doses. Also, the gel content was increased accompanied with a decrease in the swelling ratio due to the crosslinking formation of PVC composite, as a resulting effect of irradiation process.

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