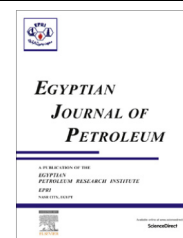




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## FULL LENGTH ARTICLE

# Flammability properties of polypropylene containing montmorillonite and some of silicon compounds

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Received 10 October 2015; revised 5 February 2016; accepted 10 February 2016

### KEYWORDS

Flame retardant;  
Polypropylene;  
Montmorillonite;  
Elongation;  
TGA

**Abstract** New polypropylene (PP) blends were prepared using phenyltrimethoxysilane (PTMS), glycidoxypolytrimethoxysilane (GPTMS) and potassium-montmorillonite (K-MMT). Polymers A (K-MMT/PTMS), B (MMT/GPTMS) and C (modified-MMT) were prepared using sol-gel technique then incorporated in the PP matrix by different concentrations. Fourier transform infrared spectroscopy (FTIR), thermogravimetric analysis (TGA), and differential scanning calorimeter (DSC) were used to investigate the structure and thermal stability. The mechanical properties (tensile strength/TS and elongation/%) were measured. The flame-retardant properties of the unblended and blended (PP/A, PP/B and PP/C) were estimated by limiting oxygen index (LOI), flame chamber (UL/94) and oxygen bomb calorimeter (OBC) tests, whereas the thermal stabilities were investigated using TGA were carried flammability out to detect the properties of the blended PP.

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## 1. Introduction

Polypropylene/polypropene (PP) is a thermoplastic polymer and has different applications as automobiles, textiles, medicine, furniture, electronic casings, automotive products, interior decoration, and relatively low cost [1–5]. The melting point of PP is ranged from 145 to 195 °C [6]. Due to the chemical constitution of the polymer it is easily flammable and so flame retardancy becomes an important requirement for PP

[7]. At temperature above 100 °C polypropylene dissolves in aromatic hydrocarbons, such as benzene and toluene. Montmorillonite (MMT) can be defined as an aluminosilicate layer mostly present in clays and most often used in the preparation of polymer nanocomposite. The surface of MMT is hydrophilic and therefore not suitable for the preparation of nanocomposites with most of the commercial polymers. To ensure good intercalation the surface of MMT platelets has to be modified. There are several ways of modifying MMT, but the most common method is to use a cation. New polymers can be generated by adding MMT at different amounts. These polymers can be characterized by decreasing at the peak heat release rate (pHRR), an increase in the percentage of ash residue after the combustion and the change in the char structure [8–19].

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Peer review under responsibility of Egyptian Petroleum Research Institute.

<http://dx.doi.org/10.1016/j.ejpe.2016.02.003>

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Also, modified polymer/clay materials increase physical, thermal and mechanical properties radically [20–27]. The sol-gel process involves the growth of inorganic networks through the formation of a colloidal suspension (sol) while gel can be formed during the sol gelation and form a network in a continuous liquid phase [28,29]. The sol-gel derived products have numerous applications such as: synthesis of the lightest materials and some of the toughest ceramics, optics, and electronics [30–32]. The technique of the sol-gel process depends on two steps: hydrolysis and condensation reactions [28]. The mechanisms of hydrolysis and condensation, and the factors (organic radical of the OR-group, solvent, catalyst, concentration and temperature) that have a bias toward linear or branched structures are the most critical issues of sol-gel science and technology [33,34]. In the present work, the PP/A, PP/B and PP/C blends have been prepared and their mechanical (tensile and elongation), thermal stability (TGA and DSC) and flammability (LOI and UL/-94) tests have been investigated. This study showed that, as the percentage of K-MMT, PTMS and GPTMS polymers increased compared to PP, the ignition properties increased, and vice versa.

## 2. Experimental

### 2.1. Materials

Polypropylene was purchased as pellets from the Oriental Petrochemicals Company (OPC), Egypt. Its specifications are shown in Table 1. Potassium montmorillonite (K-MMT, with a cation exchange capacity of 97 meq/100 g) were kindly provided by Ke Yan Company. Glacial acetic acid (99%) and ethanol were purchased from Fluka, while GPTMS (97%) and PTMS (94%) were purchased from Alfa Aesar and Aldrich, respectively.

### 2.2. Polymer preparation

The sol-gel technique was used to prepare the Polymers. Distilled water (2.5 ml), ethanol (2 ml), acetic acid (1 ml) and the clay (K-MMT, 7 gm) or silane coupling agents (PTMS, 7 gm or GPTMS, 7 gm) were mixed together in a round flask using a magnetic stirrer for 24 h to form A, B and C-polymers, respectively, then washed with ethanol several times and left to dry at 80 °C for 1 h.

### 2.3. Blended polymer preparation

Different percentage of A, B and C-polymers were blended with PP using Newplast twin screw extruder (made in India)

at zone temperatures 185 °C, 190 °C, and 190 °C for zone 1, zone 2, and zone 3, respectively, then blend them together with twin screw at speed 35 rpm, then preheat using a Morgan press injection instrument (made in USA) at 160 °C for nozzle zone and barrel zone and were injected with 120 MPa of injection pressure, 90 MPa of holding pressure, holding time of 20 s and cooling time was taken as 10 s to produce  $7.5 \times 7.5 \times 0.2 \text{ cm}^3$  and  $10 \times 1 \times 0.2 \text{ cm}^3$  molds. Samples of pure PP were processed in the same way and used for comparison.

### 2.4. Fourier transform infrared analysis (FTIR)

FTIR spectra for samples performed using Nicolet 380 Spectrometer-USA that is equipped with zinc selenide crystal in the spectral range  $4000\text{--}400 \text{ cm}^{-1}$  [39]. To ensure reproducible contact between the crystal face and the fabric, a pressure of about 18 kPa [40] was applied to the crystal holder. The FTIR absorbents frequencies for the treated samples are recorded with an average of 32 scans using a resolution of  $4 \text{ cm}^{-1}$ .

### 2.5. Thermo-gravimetric analysis (TGA)

TGA was used to determine the mass loss of a sample exposed to prominent temperatures using DTG-50 thermal analyzer (Shimadzu) after placing the sample in open platinum pan in nitrogen gas. The sample is heated by a furnace while the loss or gain of sample weight is monitored by a sensitive balance. Weight, temperature, and furnace calibrations were carried out within the range of the TGA (25–850 °C) at scan rates of  $10 \text{ °C/min}$  [41–43].

### 2.6. Differential scanning calorimeter (DSC)

DSC instrument was used to characterize the thermophysical properties of polymers such as; glass transition ( $T_g$ ), melting temperature ( $T_m$ ), and heat of melting ( $\Delta H_m$ ), using DSC-50 Shimadzu Instrument-Japan. The heating rate was  $10 \text{ °C/min}$ , a flow rate of 30 ml/min, nitrogen gas was used as a carrier gas. Scans were carried out at a temperature range of 30–650 °C [44].

### 2.7. Mechanical tests

Tensile strength and elongation break, % tests were carried out in a universal testing machine, model Z010, Zwick, Germany with load cell of 10 kN according to ASTM D638-02 [45].

### 2.8. Limiting oxygen index (LOI)

According to ISO 4589 LOI was determined for all samples. The lower concentration of oxygen required to sample ignition was measured using a Rheometric oxygen index instrument [46]. Both nitrogen and oxygen were connected to the apparatus through pressure regulators. Mixture of  $\text{N}_2/\text{O}_2$  atmospheres was continuously sent through the glass chamber. Samples were cut with dimension  $15 \times 5 \text{ cm}^2$ , and then clamped in the holder vertically in the center of the combustion

**Table 1** Specification of polypropylene [35–38].

Properties	Value	Unit
Density @ 23 °C	0.905	$\text{g/cm}^3$
Melt flow (210 °C)	12	$\text{g/10 min}$
Tensile strength @ Yield (50 mm/min)	33	MPa
Tensile elongation @ Yield (50 mm/min)	8	%
Flexural modulus (1% Secant), @ 1.3 mm/min	1350	MPa
Heat deflection temp. @455 kPa	104	°C

column. The top of each sample was ignited using a propane gas burner. Three or four replicates were tested for each sample to obtain the average LOI value [39].

### 2.9. Flame chamber (UL/94)

UL/94 is considered as a preliminary step toward obtaining plastic recognition. The samples were tested in a horizontal setting to determine the rate of burning according to ISO 3795 [47]. Sample with dimension  $10 \times 1 \times 0.2 \text{ cm}^3$  is held at one end in the horizontal position. The free end of the samples was exposed to flame (with length 3.8 cm) for 15 s then removed and the flammability properties of the samples were studied.

### 2.10. Oxygen bomb calorimeter (OBC)

OBC is used to calculate the heat of combustion of the polymers (as; powder, textile, liquid and polymer). The Model Parr 6200 instrument (made in USA) was used. It has two valves in the top head; one to fill the bomb by oxygen and close automatically after 1 min when the pressurized oxygen supply is shut off and the other to give a chance for the gases from the bomb to release and vent through this adjustment needle valve. The head of the bomb cell has two electrodes connected by the wire together from the inside which used to contact with cotton wire connected directly to sample in a crucible. The sample weights range from 0.6 to 1.5 gm with a maximum energy release of 8000 calories per charge. The sample is pressed into a tablet in a container by applying the force of a pressure piston. In the lower part, the container is covered with an easy-to-remove plate. The tablet is weighed using high accuracy weighing-scales to the precision of  $\pm 0.0001 \text{ g}$ . The bucket has three dimples in its bottom to rest on the supporting pins of the jacket and the jacket has three supporting pins at its bottom to hold the bucket. The bucket has screw stirrer that stirs the water (2L) to keep the surrounding temperature of bomb at room temperature ( $21 \text{ }^\circ\text{C}$ ) and is supported with a thermometer to control the water temperature. According to a standard method (EN ISO 1716) heat of complete combustion of testing material [48–50] was determined on gram samples tested when using high-pressure oxygen bomb calorimetry according to a standard method [51].

## 3. Results and discussion

### 3.1. Characteristics of A, B and C-polymers

#### 3.1.1. Infrared spectroscopy

The measured spectral frequencies for A, B and C-polymers are shown in Fig. 1. The broad absorption peak in the range  $3610\text{--}3640 \text{ cm}^{-1}$  arises from the  $\text{—OH}$  bonding of physically adsorbed water molecules and the peak between  $1620$  and  $1635 \text{ cm}^{-1}$  arises from the bending modes of  $\text{—OH}$  bond. The peak rising at  $1425 \text{ cm}^{-1}$  is corresponding to Si–phenyl group [52]. The peaks observed in the range of  $1050\text{--}1055 \text{ cm}^{-1}$  are related to the asymmetric, symmetric and the bending modes of  $\text{≡Si—O—Si≡}$  which refer to the polymerization of the silanol groups ( $\text{≡Si—OH}$ ) resulting in a three dimensional network [53]. At  $1633.41 \text{ cm}^{-1}$ , C-polymer has the lowest intensity percentage compared to the other. In case

of A-polymer, there is a peak at  $1431.89 \text{ cm}^{-1}$  due to the presence of deformation C—C at phenyl group, but not found in B and C-polymers. There is no epoxy  $\text{CH}_2$  stretch seen at  $3050 \text{ cm}^{-1}$  in B-polymer. This further confirms the full epoxy opening in B-polymer.

The intensity of OH group at A, B and C-polymers is varied (51.4632%, 60.9704% and 54.1013%, respectively), while C-polymer records the lowest intensity (81.2581% and 69.6339%) compared to A-polymer (87.9556% and 76.9443%) in case of asymmetric and stretch vibrations of  $\text{CH}_2$  and C=O (see Fig. 2). A deformation band of the C—C ring has appeared at wavelength  $1431.89 \text{ cm}^{-1}$  with intensity 78.9902%, while disappearing at other samples, this returns to the presence of the phenyl ring. A decrease in the intensity of the later peaks during the sol-gel reaction indicates the formation of Si—O—Si asymmetric bonds. Finally, the deformation band of the C—H ring has appeared only at wavenumber  $740.53 \text{ cm}^{-1}$  with intensity 76.3885% in the case of A-polymer and disappeared at the other.

#### 3.1.2. TGA characterization

TGA is one of the most commonly used techniques to evaluate the thermal stability of different materials and indicates the decomposition of polymers at various temperatures [54,55]. The decomposition behavior of A, B and C-polymers from 25 to  $850 \text{ }^\circ\text{C}$  in nitrogen atmosphere was examined by TGA as shown in Fig. 3. The initial decomposition temperature can be defined as the temperature at which the weight loss of a sample reaches 5 wt%. Up to 140, 130 and  $206 \text{ }^\circ\text{C}$ , the initial decomposition of A, B and C-polymers have been occurring, this return to dehydration at this stage. In case of A-polymer: the second stage is the main pyrolysis stage, since the siloxane and benzene ring start to cleave from  $140 \text{ }^\circ\text{C}$  to  $721 \text{ }^\circ\text{C}$ . Up to  $850 \text{ }^\circ\text{C}$  A-polymer has no change at its weight residue (84.5%). The B-polymer has three pyrolysis stages; up to  $130 \text{ }^\circ\text{C}$  the water is evaporated at the first stage. The second and third stages are decomposed very fast between  $130\text{--}721 \text{ }^\circ\text{C}$  due to the cracking of epoxy ring and dehydroxylation of the aluminosilicate. The final weight loss at temperature  $850 \text{ }^\circ\text{C}$  was 15.5%. Finally, the C-polymer is a hydrophobic water absorbent, in spite of having approximately 4–6 wt% water residing between montmorillonite crystallites is lost [56]. Up to  $216 \text{ }^\circ\text{C}$ , the dehydration of water is achieved at the first stage. At temperatures between 216 and  $682 \text{ }^\circ\text{C}$ , dehydroxylation of the aluminosilicate occurs and the organic constituent decomposes [57]. The weight residue is 90.1% compared to the others (A and B-polymers) at the third stage which ranged between 682 and  $850 \text{ }^\circ\text{C}$ . No change has been observed at the last stage of all samples.

#### 3.1.3. Differential scanning calorimeter (DSC) measurement

By DSC instrument the glass transition temperature ( $T_g$ ) can be measured, the data are tabulated in Table 2. The heat flow in milliwatts (mW) is plotted versus temperature in Fig. 4. According to DSC results the value of  $T_g$  is changed between A ( $77.3 \text{ }^\circ\text{C}$ ), B ( $33.9 \text{ }^\circ\text{C}$ ) and C ( $41.4 \text{ }^\circ\text{C}$ ) polymers, which prove the effectiveness of the sol-gel technique to synthesis primary materials. This may return to different percentages of volatile water or solvent. Up to  $100 \text{ }^\circ\text{C}$  the area under the peak are shallow except C-polymer that has deep area. Finally, there is a new peak with broader exotherm found in B-polymer at

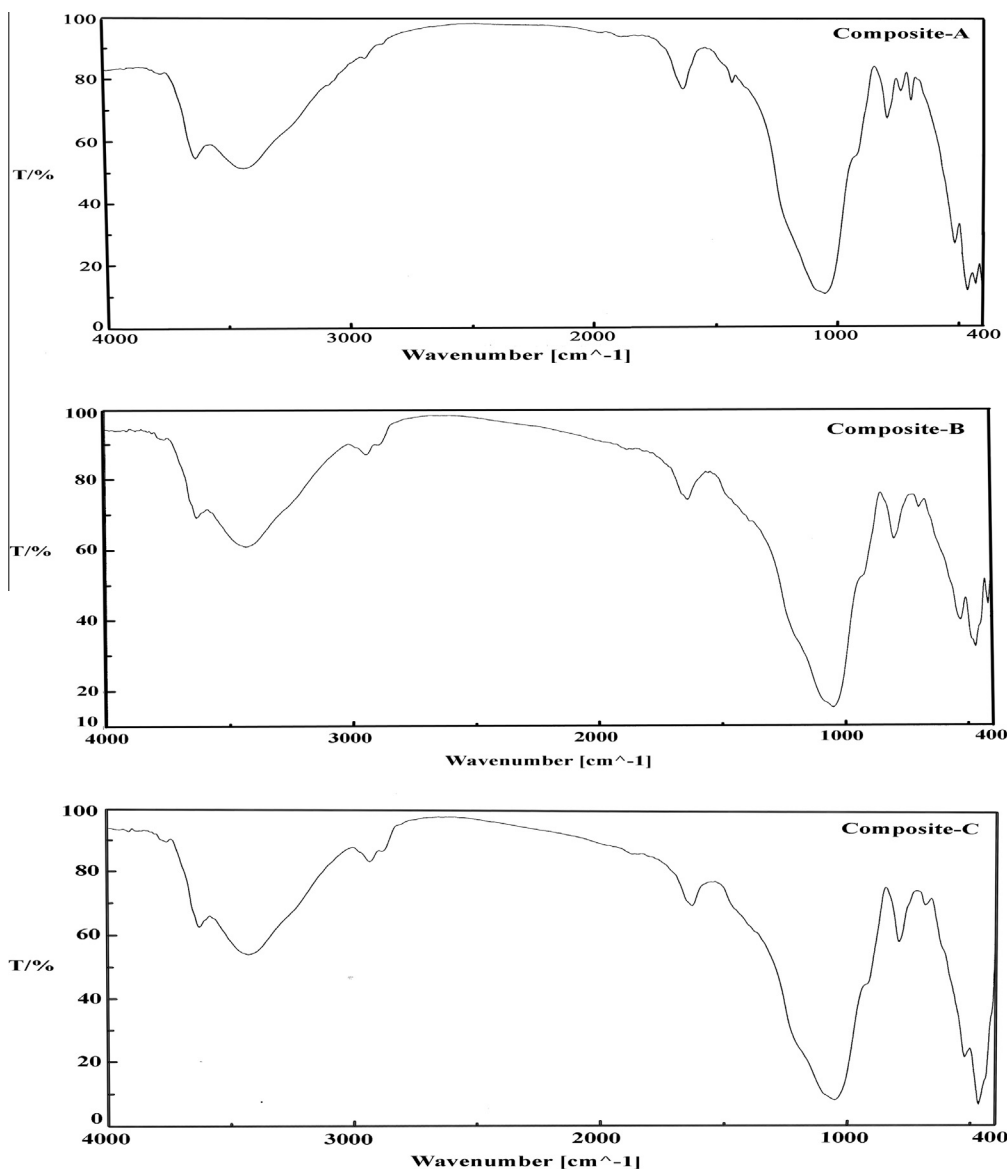


Figure 1 FTIR for A, B and C-polymers after drying at 80 °C for 1 h.

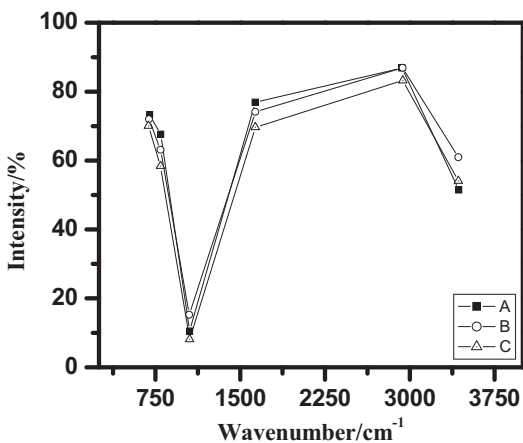


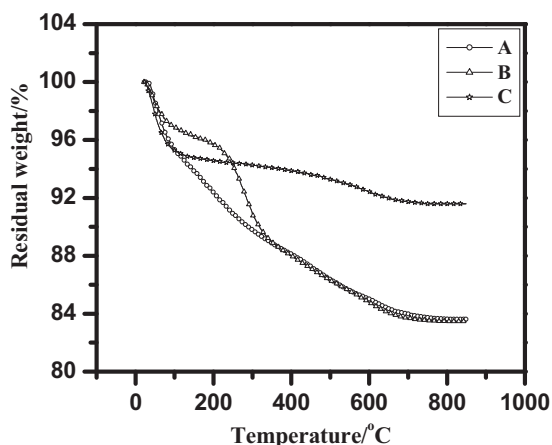
Figure 2 Relation between intensity and wavenumber for the A, B and C-polymers.

307.5 °C but does not appear in the case of A and C-polymer primary products.

### 3.2. Characteristics of PP/A, PP/B and PP/C blends

#### 3.2.1. Mechanical properties of PP/A, PP/B and PP/C blends

The ability to resist breaking under tensile stress (TS) is one of the most important and widely measured properties of materials used in structural applications. The effects of the different percentages of the polymers on TS (Kgf) and E (%) are shown in Table 3. Firstly, blending PP with 2.5% of A-polymer (P-1) records the maximum value in tensile strength (1144 Kgf) with the lowest elongation (2.73%) compared to the others. It may return to the presence of benzene ring which including short bond need high force to be cracked. In case of PP/B blend, the tensile strength increases as the percentage of B-polymer increases. While, in the case of PP/C blend, as the content increased, the tensile strength initially increased and then

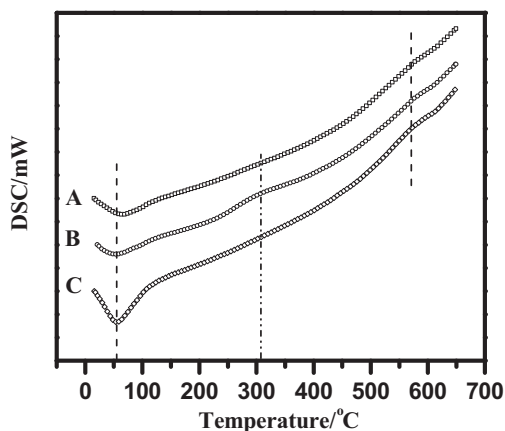


**Figure 3** Thermogravimetric analysis of A, B and C-polymers prepared by sol-gel technique in an  $N_2$  gas atmosphere and ambient temperature.

**Table 2** Values of glass transition temperature, peaks and heat of fusion for A, B and C-polymers.

Polymers data	$T_g/$ $^{\circ}C$	Peak1/ $^{\circ}C$	$\Delta H_1/$ $J g^{-1}$	Peak2/ $^{\circ}C$	$\Delta H_2/$ $J g^{-1}$
A	77.3	–	–	579.5	+12.67
B	33.9	307.5	+21.1	577.5	+7.70
C	41.4	–	–	571.2	+14.6

$T_g$  is the glass transition temperature,  $\Delta H_1$ , is the first heat of fusion, and  $\Delta H_2$  is the second heat of fusion.



**Figure 4** DSC thermogram of A, B and C-polymers.

decreased. The maximum value in tensile strength (579 Kgf) was achieved when the concentration of M-2 was 5 wt%. Finally, the tabulated data illustrate that, the mechanical characteristics of PP improved without affecting elongation by blending with different wt% of A, B and C-polymers, since polymer-A has the best tensile results compared to the others.

### 3.2.2. Thermogravimetric analysis (TGA)

The detailed variations of  $T_{10\%}$  and  $T_{50\%}$  for PP and blend samples are summarized in the Table 4. It illustrates that, the PP sample had no char residue as it degraded completely at higher temperatures (750 °C), while, the char residue increased

**Table 3** Effect of polymers on mechanical properties of PP.

Samples			Tensile (Kgf)	Elongation (%)
PP			360	3.51
PP/Poly. A	P-1	2.5%	1144	2.73
	P-2	5%	973	4.41
	P-3	7.5%	644	3.82
PP/Poly. B	G-1	2.5%	548	5.31
	G-2	5%	613	4.12
	G-3	7.5%	650	3.26
PP/Poly. C	M-1	2.5%	413	3.86
	M-2	5%	579	4.84
	M-3	7.5%	501	3.75

as the percentage of polymers increased. Since, the P-3 blend has the highest char residue compared to all the samples and increased by 10 wt% compared to unblended one, it may be returned to hydrogen bonding interactions between the end groups of the P-polymer and the MMT platelet surfaces.

### 3.2.3. LOI and flame chamber

The limited oxygen index and UL-94 tests are widely used to evaluate the flame retardant property of materials. All samples of unblended (PP) and blended with polypropylene with different weights were tested in the LOI and results are shown in Table 5. The LOI value of PP sample is 17.9% and increased when mixed with different weight percentages of A, B and C-polymers, which indicates a reduction in ignitability. An improvement is observed by increasing the weight percentage of polymers with PP. As shown in Table 4, the LOI values of P-3, G-3 and M-3 blends have the highest values of LOI (21.3%, 20.4% and 20.1%) compared to unblended and other blend samples (P1-2, G1-2 and M1-2) also.

UL/94 is a method to determine the horizontal burning rate of materials after exposing to a small flame for 15 s. Five samples were tested for each formulation. The final results are tabulated in Table 5. It is clear that the results compatible with LOI values, on another meaning, the rate of burning (RB) recording the lowest value when the PP is blended with the highest percentage of new polymers due to ignite the sample at long time, which lead to improve the physical characteristics (smoking and dripping). According to Eq. (1), when the burning time increases the burning rate decreases and vice versa:

$$\text{Rate of burning (RB)} = (60 \times D)/T \quad (1)$$

where RB is the rate of burning/mm  $\text{min}^{-1}$ ,  $D$  is distance of sample burning/mm,  $T$  is time of burning/min.

The previous data indicated that, the burning rate decreased by 16.8%, 13.3% and 27.1% in the case of M-3, G-3 and P-3, respectively.

### 3.2.4. Oxygen bomb calorimeter (OBC)

The data of adiabatic bomb calorimeter test of pure PP and blended with different weight percentages of A, B and C-polymers are tabulated in Table 6. PP has the lowest heat of combustion (3634.520 Cal/g) compared to the other samples due to the purity of polypropylene which has the melting point range from 160 to 165 °C (return to crystallinity and atactic material), while the corresponding values for treated samples; M-1, G-1, and P-1 with the lowest weight percentage have

**Table 4** TGA results of PP and A, B, C-polymers.

Samples	Begins of the weight loss/°C	$T_{10\%/^{\circ}\text{C}}$	$T_{50\%/^{\circ}\text{C}}$	Residue at 450 °C/%	Residue at 750 °C/%	
PP	180.9	336.8	404.4	14.9	0.0	
PP/Poly. A	P-1	206.9	301.5	388.5	4.3	2.0
	P-2	211.6	308.6	400.3	14.9	6.6
	P-3	277.4	360.3	424.0	28.5	9.7
PP/Poly. B	G-1	220.1	360.6	421.1	14.6	1.4
	G-2	221.2	391.3	440.0	34.7	2.9
	G-3	191.7	296.9	392.8	14.6	7.8
PP/Poly. C	M-1	202.1	349.7	416.2	21.6	1.8
	M-2	206.8	371.0	432.7	32.9	4.0
	M-3	197.3	309.6	392.6	16.7	5.8

Onset is the temperature at a 10% mass loss; mid is the temperature at a 50% mass loss.

**Table 5** LOI and UL/94 results of flame retardant of the unblending and blending with A, B and C-polymers.

Samples	Perc. (%)	LOI (% O <sub>2</sub> )	$\Delta$ LOI (% O <sub>2</sub> )	UL/94			Observed dripping	Smoking	
				dis/mm	Time/s	RB/mm min <sup>-1</sup>			
PP	100	17.9	–	70	165.5	25.4	Dripping	Heavy	
PP/Poly. A	P-1	97.5/2.5	19.3	1.3	70	172.6	24.3	No dripping	Slight
	P-1	95/5	20.2	2.2	70	210.6	19.9	No dripping	Slight
	P-3	92.5/7.5	21.3	3.3	70	224.6	18.5	No dripping	Slight
PP/Poly. B	G-1	97.5/2.5	19.0	1.0	70	178.2	23.6	No dripping	Slight
	G-2	95/5	19.6	1.6	70	187.2	22.4	No dripping	Slight
	G-3	92.5/7.5	20.4	2.1	70	190.8	22.0	No dripping	Slight
PP/Poly. C	M-1	97.5/2.5	19.5	1.5	70	184.2	22.8	No dripping	Slight
	M-2	95/5	19.9	2.0	70	187.0	22.5	No dripping	Slight
	M-3	92.5/7.5	20.1	3.0	70	199.4	21.1	No dripping	Slight

**Table 6** Gross heat combustion of unblending and blending samples with different weight percentage of A, B and C-polymers.

Samples	Initial weight/gm	Ash residue/gm	Average weight loss/%	Temp. rise/°C	Acid calculation	Gross heat/Cal g <sup>-1</sup>	
PP	0.9967	0.0025	99.9	1.569	5.756	3634.520	
PP/Poly. A	P-1	1.0047	0.0167	98.3	4.654	17.073	10723.307
	P-2	1.0038	0.0359	96.4	4.567	16.754	10531.699
	P-3	1.0097	0.0540	94.6	4.516	16.567	10353.180
PP/Poly. B	G-1	1.0054	0.0128	98.7	4.708	17.292	10840.961
	G-2	1.0093	0.0237	97.6	4.657	17.085	10681.879
	G-3	1.0037	0.0485	95.2	4.489	16.471	10354.016
PP/Poly. C	M-1	1.0077	0.0104	99.0	4.713	17.291	10827.777
	M-2	1.0163	1.0163	97.5	4.676	17.154	10651.062
	M-3	1.0030	1.0030	95.7	4.473	16.709	10322.838

the highest gross heat composition 66.4%, 66.5% and 66.1%, respectively. This may be, return to completely inter-chemical reaction between molecules of PP and polymers. On the other hand, increasing the weight percentage leads to decrease in the heat of combustion in the blend samples M-3, G-3, and P-3 (–4.9%, –4.7% and –1.6%).

#### 4. Conclusions

(1) In conclusion, I was able to prepare different PP-Polymers with various properties which can be applied in various applications according to the desired industrial products.

- (2) The tensile stress was very dependent on the composition of the blends.
- (3) Results from the LOI and UL-94 tests show that the LOI value of M-3, G-3 and P-3 is enhanced to 20.1%, 20.4% and 21.3%, respectively, from 17.9% (unblended PP) and the UL-94 rating is decreased to 18.5 mm/min compared to pure PP (25.4 mm min<sup>-1</sup>), due to the long time of burning.
- (4) The results gained from OBC test, illustrate the main effect of the A, B and C-polymers on the PP, since the gross heat recorded the highest values increased in case of M-1, G-1 and P-1 blends. These results go with the results of LOI, UL/94 and mechanical test.

## Acknowledgments

The author would like to thank Prof. Dr. Mohamed A. Nour (Fire and Explosion Protection Laboratory) and Prof. Dr. N. A. El-Zaher (Textile Metrology Laboratory) National Institute for Standards (NIS), Ministry of Higher Education and Scientific Research, Egypt for their cordial cooperation and help to do this work well.

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