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Studies on the flammability of polypropylene/ammonium polyphosphate and montmorillonite by using the cone calorimeter test

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Abstract: Fire performance of polypropylene (PP) containing different percentages of ammonium polyphosphate (APP) with montmorillonite (Mt) or treated montmorillonite (MtT) was carried out by using the cone calorimeter test (CCT). Different samples from ammonium polyphosphate and montmorillonite were mixed with 90% polypropylene. The characterization of the prepared samples indicates that there is incorporation between the components of the samples. Heat release rate (HRR), peak heat release rate (PHRR), average heat release rate (Av-HRR), and time to ignition (TTI) of the samples were obtained from the cone calorimeter test. Also, the fire performance index (FPI) and the fire growth rate (FIGRA) were calculated. The interpretation of the curves and the parameters results from the cone calorimeter test which indicates that the addition of montmorillonite to APP increases its action as a flame retardant for PP. Moreover, the samples contain acid treatment montmorillonite showed an increase in the efficiency of ammonium polyphosphate when used. This result may be due to an increase in the SiO_2 content by acid treatment.

Keywords: Flame retardant, montmorillonite, cone calorimeter test, thermal analysis, flammability, acid treatment

1 Introduction

Polypropylene (PP) is used in many industrial applications, such as automotives, furniture, electronic casings, indoor building decorates, and architectural materials as it is an environmentally friendly polymer [1,2].

Because PP consists chemically of carbon and hydrogen, it is easily ignited producing carbon dioxide and water vapor. The main drawback of using PP in special applications is its flammability; therefore, flame retardancy is an important requirement for PP [3]. The current literature survey indicates that there are general classes of flame retardants available today. There are the normal additions which are not chemically bonded to the polymer, and the reactive additives that are bonded to the polymer backbone through polymerization [4]. Different materials were used as flame retardants for the different polymers and the natural cellulosic materials such as wood and cotton. These materials includedorganic, inorganic and organic-inorganic substances. Also, they included some ores such as clays [5], zeolite [6]. In recent years [7-11] polymer/layered silicates nano-composites have attracted great interest, both in industries and academia. Montmorillonite (Mt) is commonly used as a nano-filler in the preparation of polymer nano-composites. Montmorillonite, and other layered silicate clays are naturally hydrophilic. This makes them poorly suited to mixing and interacting with most polymer matrices which are mostly hydrophobic. Moreover, the stacks of clay platelets are held tightly together by electrostatic forces. For these reasons, the clay must be treated before being used as a nano-composite. Between 2013-2017, Bettina and his co-workers focused their research on the formation of inttumescent flame retardants including ammonium polyphosphate (APP) and the different shapes of nano-particles of graphene [12-16]. They concluded that the presence of both APP and graphene improves the flammability of PP.

The efficiency of the flame retardant behavior of materials depends on the nature of the used materials

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as well as the conditions of the incorporation of the flame retardant materials into the polymer. To benefit the uses of PP in different applications, several paths were followed. First, modify the used materials, second related with how the flame retardant materials incorporated into the polymer and lastly related with the methods of the evaluation. In our previous work, ammonium polyphosphate (APP) was prepared in nano-sized particles to increase the surface area of particles [17]. Also, it was used as flame retardant for PP [18]. The evaluation results indicated that the APP could be used as a flame retardant for PP when mixed up to 10% of the APP without any remarkable changes in the mechanical properties. Hanna et al [19] showed that the efficiency of APP as a flame retardant can be increased by adding some clays materials such as kaolin and diatomite. They found that the addition of kaolin or diatomite to APP with 6% benefits the uses of APP where the results indicated that the time of ignition increased from 14 to 28 sec. Furthermore,they modified the clay materials before mixing them with APP, and they observed that the acid treatment of clays increased their action [20].

There are different methods for evaluation of the flame retardant of materials such as thermogravimetric analysis (TGA), limiting oxygen index (LOI) and cone calorimeter test (CCT). Among these methods, the cone calorimeter test is a largely accepted method for evaluation because this test gives different parameters such as heat release rate (HRR), average heat release rate (Av.HRR) total heat release (THR) and time to ignition (TTI) [21]. The interpretation of these parameters gives complete information about the behavior on the burning of the materials and could evaluate the system successfully [22].

The thermal decomposition (TGA and DTA) of the composite was studied previously [23] under the same composition. The results indicated that the addition of APP+ montmorillonite improves the thermal stability and increases the time of degradation.

To benefit natural montmorollonite (Mt) which is more abundant in Egypt, the present work aimed to study the effects of the addition of different amounts of montmorillonite (Mt) to APP (10%) relating to the flammability of PP and by applying the cone calorimeter test. Furthermore, an acid treatment on Mt was carried out to study the effects of this treatment on the efficiency of APP/Mt when used as a flame retardant for PP.

2 Experimental

2.1 Materials

2.1.1 Ammonium polyphosphate (APP)

Ammonium polyphosphate was prepared from ammonium dihydrogen phosphate and ammonia solution as described elsewhere [17]. The characterization of the produced APP indicated that the product has a particle size between 5 to 85 nm and is typical to form I of APP.

2.1.2 Polypropylene (PP)

Polypropylene (PP) has a chemical formula (CH_2=CH-CH_3), according to IUPAC nomenclature, its molecular formula is $(C_3H_6)_n$, its density is in the range of 0.855 to 0.946 $gm.cm^{-3}$ for the amorphous or the crystalline form. It melts at about 130-171ºC, (LG Chem, Ltd Seetc, N1600 B001046M, Korea).

2.1.3 Montmorillonite (Mt)

In this study, montmorillonite was supplied by the geology department of the National Research Center, which was derived from Wadi El Hamadiya region, Egypt. The sample was used without further treatment. The untreated montmorillonite, contains SiO_2=42.02, Al_2O_3=47.07, Mgo=0.028, CaO=0.035, K_2O=0.013, ZnO=0.006, TiO_2=0.76 and Fe_2O_3=0.005 wt%. Furthermore, the montmorillonite was exposed to H_2SO_4 treatment to increase the surface area. This treatment did not affect the mechanism of the flammability, but changed the structure and the orientation of the montmorillonite. The treated montmorillonite, contains SiO_2=55.32, Al_2O_3=28.88, MgO=0.021, CaO=0.013, K_2O=0.011, ZnO=0.006, TiO_2=0.25 and Fe_2O_3=0.004 wt% (10). It is noteworthy that the Mt analysis was carried out by XRF weight % as mentioned in the previous work.

2.2 Preparation of the samples

Different percentages of APP and unmodified montmorillonite (Mt) or modified montmorillonite (MtT) were prepared by a melt blending method using a newplast twin screw extruder (India) at a temperature of 185-190^{0}C. The twin screw speed was 30 rpm. The samples obtained from the extruder were preheated using a Morgan press injection unit at a temperature of 190-195^{0}C to produce 7.5

Table 1: The samples codes and preparation conditions of the samples.

Treated montmorillonite (MtT) %	Montmorillonite (Mt)%	APP %	PP %	Sample code
0	0	0	100	PP
0	0	10	90	APP10%
0	2	8	90	Mt2
0	4	6	90	Mt4
0	6	4	90	Mt6
0	8	2	90	Mt8
0	10	0	90	Mt10
2	0	8	90	MtT2
4	0	6	90	MtT4
6	0	4	90	MtT6
8	0	2	90	MtT8
10	0	0	90	MtT10

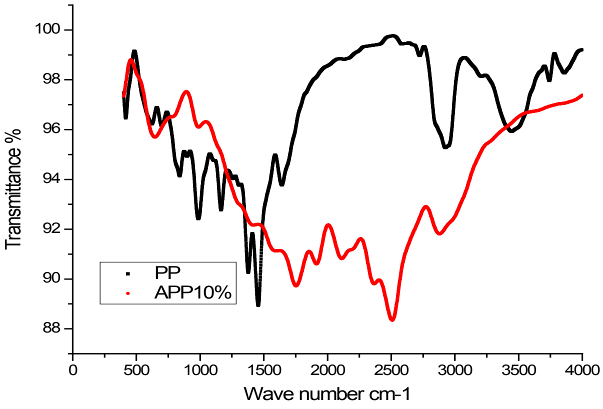


Figure 1: FTIR for PP and APP10%.

x7.5 x 0.4 cm molds. The samples codes and preparation conditions are listed in Table 1.

2.3 Measurements

FTIR spectra of the samples were obtained using a KBr disk technique and FTIR 6500 spectrometer (JASCO, Japan) in the range of 400-4000cm^{-1}. The surface morphologies of the samples were carried out using a JEOL JSM t20 scanning electron microscope (SEM) (JEOL, Japan) at an accelerating voltage of 5 kV. A cone calorimeter test was performed using fire testing technology Ltd equipment according to ASTM 1354-95. Samples, with dimensions of 7.5 x 7.5 x 0.4 mm^3, were tested horizontally under an incident flux of 35 kW/m^2. The cone calorimeter that was attached to a software program modified automatically to the standard dimensions 10*10*0.5 (22)

Ethical approval: The conducted research is not related to either human or animals use.

3 Results and Discussion

3.1 FTIR measurements

The IR measurements of PP and APP10% samples are shown in Figure 1. The IR spectra show that different peaks appeared as follows:

1. Absorption bands appeared at 3087, 2922, 1457, 1378 and 890 cm^{-1}. These bands may correspond to the C-H bond of the saturated and unsaturated part of the PP chain [23,24].
2. Specific characteristics of the P-O and P=O attached into APP appeared at 1117, 1081 and 1079 cm^{-1}.
3. Important series of bands at 1158, 1000, 927 and 492 cm^{-1} corresponding to the P-O and P-O-P indicate that there was an interaction between the PP and APP.

The FTIR absorption spectra of (Mt2, Mt 4, Mt 6, Mt 8 and Mt 10) and (MtT 2, MtT 4, MtT 6, MtT 8 and MtT 10) show new bands at 1165, 990, 931, cm^{-1} which are assignable to (Si-O-P), (Si-O-C) and (SiOH) respectively. This result confirms that there was an interaction between the montmorillonite (treated or untreated) with PP and APP as shown in Figures 2 and 3. This interaction may be due to two factors. The first factor relates to an electrostatic force between the montmorillonite and the surface of PP; the second factor penetrates the montmorillonite particles between the layers of PP.

3.2 Scanning electron microscope (SEM)

The morphology of PP, PP/APP, PP/APP+ montmorillonite or treated montmorillonite was studied, and the photos were shown in Figures 4, 5 and 6. The photo shows that the particles of PP were distributed uniformly, whereby adding 10% APP, the texture converted to layers. For the samples which contained montmorillonite or modified montmorillonite, the layer structure increased as eviudencedin Figures 5 and 6. It is observed that as the percentage of montmorillonite or modified montmorillonite increased, the clearance and the layers formation were enhanced.

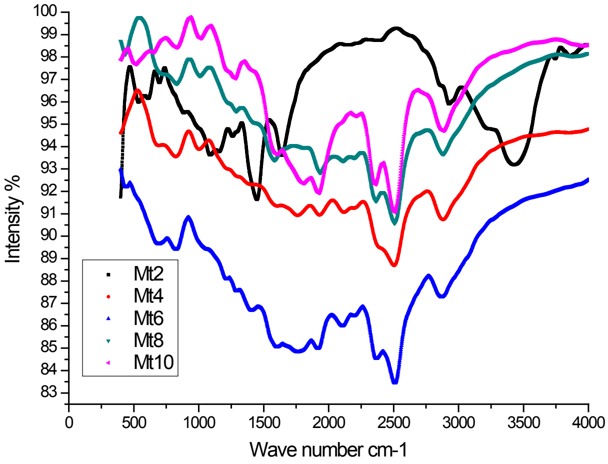


Figure 2: FTIR for Mt2, Mt4, Mt6, Mt8 and Mt10.

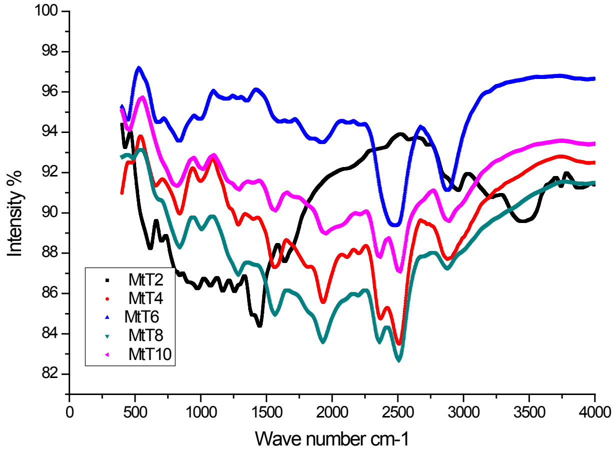
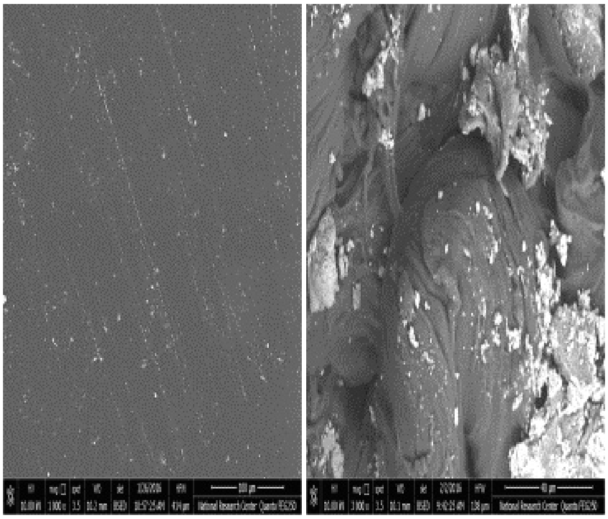


Figure 3: FTIR for MtT2, MtT4, MtT6, MtT8 and MtT10.



(a) (b)

Figure 4: SEM of a) PP and b) PP/10%APP. (The magnification equal 3000).

3.3 Cone calorimeter test

The cone calorimeter test was used to evaluate the flammability of the samples under investigation. This test represents a small scale testing configuration which provides important correlating parameters with a real fire scenario and gives different parameters. The heat release rate (HRR), the peak of the heat release rate (PHRRP) and the average heat release rate (Av-HRR) are quantitative measures of thermal energy released by a materialper unit area when exposed to a fire radiating at a constant heat flux (or temperature) usually at 35 kW/m^2. The time to ignition (TTI) corresponds to the period that combustible materials can withstand when exposed to a constant radiant heat flux before ignition and when undergoing sustained flaming combustion. The cone calorimeter data reported in Table 2 represent an average of the three replicate experiments.

The HRR value is considered to be the single most important fire reaction property as it represents the most valuable indicator of the fire hazard for combustible materials.

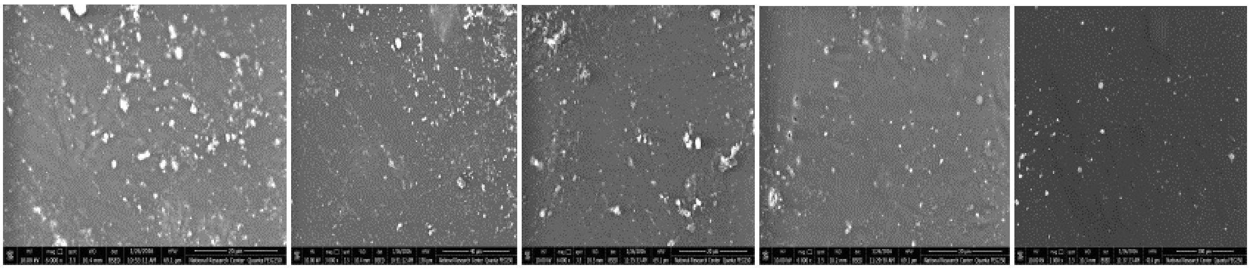
The variation of the heat release rate (HRR) followed the same trends, while its value increased with time and formed peaks and decreased to obtain the steady state at about 180 seconds as shown in Figure 7. The peak of the heat release rate equals 1104 KW/m^2 for the PP sample without any addition. By adding 10% APP to PP, the value of PHRR reduced to 925 KW/m^2 (16.2% reduction) as shown in Table 2. For the samples containing APP with Mt (Mt2, Mt4, Mt6, Mt8), the values of PHRR equaled 769, 765, 764 and 751 KW/m^2 respectively. This means that the values of reduction continue for the Mt2 reaching a nearly steady state for the other samples. For sample Mt10 which contained 10% Mt and free APP, the value of PHRR was equal to 1005 KW/m^2. This finding indicates that the mixture of APP with Mt is more effective than using APP that is free of Mt or, Mt free of APP. This may be explained by the mutual effects between APP and Mt where the presence of low Mt accelerated the degradation of APP, while the increase in the percentage of Mt decreased the degradation of APP due to the coating effect.

For the cone calorimeter test, the data shown in Figures 7, 8 and Table 2. For the samples containing MtT, it was observed that the reduction in the values of PHRR was greater than that obtained for the samples containing Mt.

Contrary, the average heat release rate (Av-HRR) Table 2 exhibits the same trend as that observed for PHRR, where the values of Av-HRR decreased from 447 to 221 KW/m^2. The reducing value for the sample Mt8 was higher than the other samples. This finding confirmed the behavior of

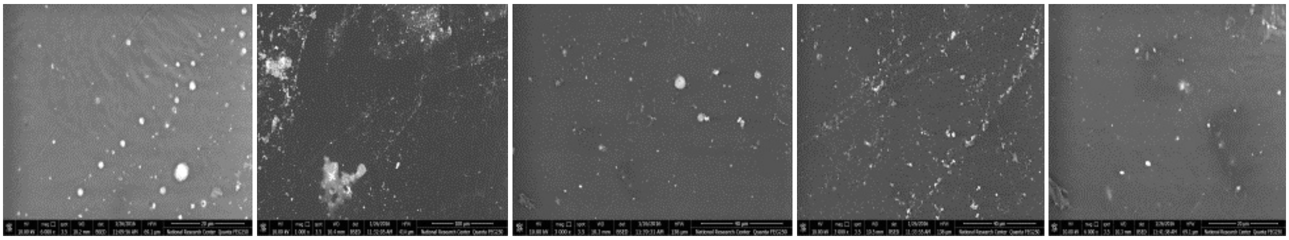
Table 2: Main cone calorimeter parameters evaluated of the samples.

FPI (sm2/kW)	**THR (MJ/m2)**	**FIGRA (kW/m2s)**	**AV. HRR (KW/m2)**	**PHRR (KW/m2) %**	**TTI (S)**	**Samples**
0.01268	106	13.8	447	1104	14	PP
0.02595	92	8.8	334	925 (16.2%)	24	APP10%
0.03251	66	8.09	232	769 (30.34)	25	Mt2
0.03529	65	7.25	228	765 (30.79)	27	Mt4
0.03665	64	7.20	226	764 (30.70)	28	Mt6
0.03862	62	7.17	221	751 (31.97)	29	Mt8
0.01791	99	13.08	359	1005 (8.97)	18	Mt10
0.04056	64	5.91	214	715 (35.23)	29	MtT2
0.04846	63	5.80	212	619 (43.93)	30	MtT4
0.05003	57	6.07	211	599 (45.74)	30	MtT6
0.05391	56	5.38	148	575 47.91	31	MtT8
0.02054	98	12.09	352	925 (16.21)	19	MtT10



(a) (b) (c) (d) (e)

Figure 5: SEM of a)Mt2, b) Mt4, c)Mt6, d)Mt8 and e)Mt10. (The magnification equal 3000).



(a) (b) (c) (d) (e)

Figure 6: SEM of a)MtT2, b) MtT4, c)MtT6, d)MtT8 and e)MtT10. (The magnification equal 3000).

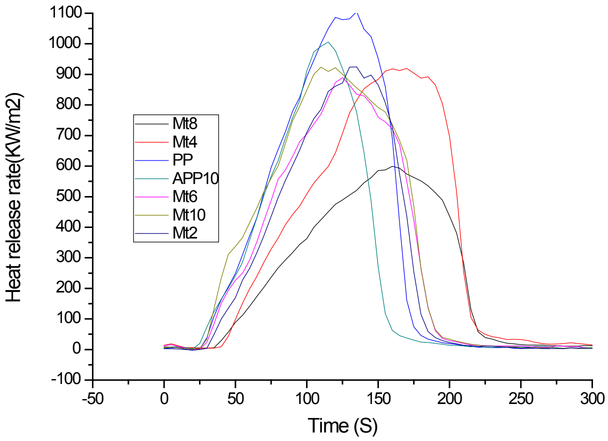


Figure 7: HRR of PP and APP10% and Mt2, Mt4, Mt6, Mt8, Mt10.

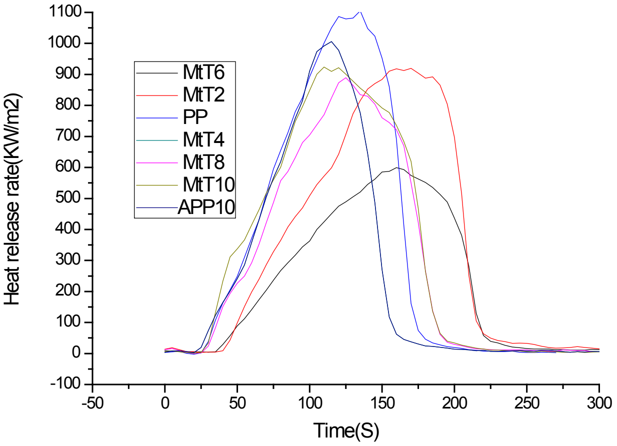


Figure 8: HRR of PP and APP10% and MtT2, MtT4, MtT6, MtT8, MtT10.

PHRR. For samples containing MtT, the curves and the parameters of the cone calorimeter test showed that:

1. There was a reduction in the values of PHRR for all samples
2. This reduction was larger than that obtained for the samples containing untreated Mt, where it recorded a reduction 35.23, 43.93, 45.70 and 47.91 KW/m^2 for the samples MtT2, MtT4, MtT6 and MtT8 respectively.
3. The average HRR for the samples from MtT2 to MtT8 decreased gradually as the percentage of treated Mt (MtT) increased with a recording of 148 KW/m^2 for sample MtT8.

As reported previously, PHRR is a measure of the progress of burning and the observation of the average HRR. It may be concluded that the addition of Mt to APP inhibits the spread of the fire due to the thermal decomposition of APP to produce nitrogen and phosphorus acid and the effect of the samples free of APP is minimal.

Ignition data are reported as time to substance ignition, which is defined as ignition with the assistance of a spark igniter. The time to ignition for all samples (APP10%, Mt2, Mt4, Mt6, Mt8, Mt10, MtT2, MtT4, MtT6, MtT8 and MtT10) is higher than that for the pure PP sample. These results are in agreement with the data obtained from PHRR values.

The fire growth rate (FIGRA), computed as PHRR/time to PHRR, provides the basis for estimation of both the predicted fire spread rate and the size of fire [21] and is a good indicator of the contribution to fire growth of materials. The FIGRA for all samples is shown in Table 2. The values of FIGRA were decreased from 13.8 to 8.8 for PP without any addition for the sample containing 10% of APP, while it decreased to 8.09 for the Mt2 sample. These decreases continue as the amount of Mt increases with a recording value equal to 7.17 for the Mt8 sample. As a comparison, the recorded value for the sample Mt10 was 13.08. This indicates that the mixture of APP and montmorillonite is more effective as a flame retardant. In all cases the samples which contained treated montmorillonite (MtT2, MtT4, MtT6, MtT8, MtT10), showed lower FIGRA valuesthan those for untreated montmorillonite. Also, the results indicate that the treatment of montmorillonite improved the action of montmorillonite when used with APP as a flame retardant material for PP.

Another important parameter is the the fire performance index (FPI) that was calculated as the ratio between the time to ignition (TTI) and the peak of heat release (PHRR):

$$\mathbf{FPI = TTI/PHRR}$$

This FPI value gives useful information about the degree of fire hazard [25-29]. The calculated values of FPI for all samples as shown in Table 2 are increased by adding APP or APP+ montmorillonite or treated montmorillonite. This indicates that the presence of montmorillonite increased this APP efficiency when used as a flame retardant for PP; however,this efficiency increased by the treatment of montmorillonite.

The values of THR for the samples are represented graphically in Figures 9 and Figure 10 for all samples (APP10%, Mt2, Mt4, Mt6, Mt8, Mt10, MtT2, MtT4, MtT6, MtT8 and MtT10) shows a lower value (92, 66,65, 64, 62, 99, 64, 63, 57, 56 and 98, respectively), in comparison to pure PP(106 KJ/m^2). It is very interesting that the value of the total heat release was decreased for all samples compared to pure PP.

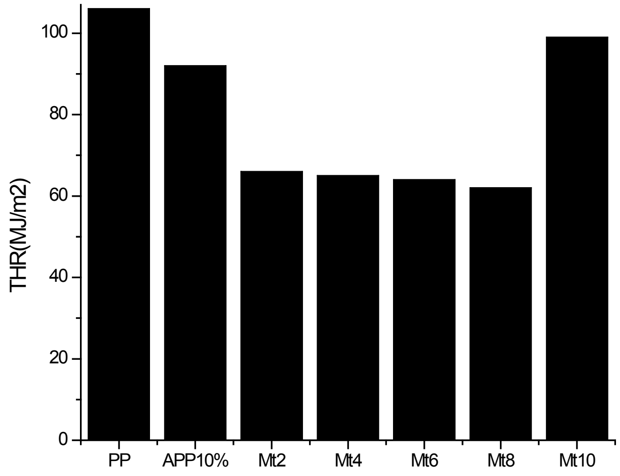


Figure 9: Total heat release of (PP, APP10% Mt2, Mt4, Mt6, Mt8, Mt10) samples.

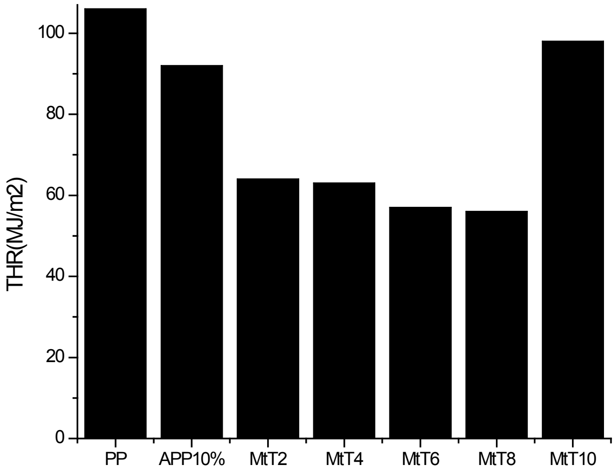


Figure 10: Total heat release of (PP, APP10% MtT2, MtT 4, MtT 6, MtT 8, MtT 10) samples.

4 Conclusion

Results conclude that the addition of montmorillonite to APP increases the effects of APP when used as a flame retardant of PP. On the other hand, the treatment of montmorillonite with H_2SO_4 raised the action of montmorillonite when added to APP. This may be due to the increase in the SiO_2 content as well as the high surface area resulting in acid treatment of montmorillonite.

Conflict of interest: Authors state no conflict of interest.

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