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Original Research Article

THE FLAMMABILITY OF POLYPROPYLENE NANOSIZED AMMONIUM POLYPHOSPHATE SYSTEM

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ABSTRACT:

The prepared nano sized ammonium polyphosphates (APP) from ammonium dihydrogen phosphate and urea in the presence of ammonia solution was evaluated as flame retardant for polypropylene (PP). The results of study show that negligible change has occurred on the mechanical properties with APP addition. Both the thermogravimetric analysis (TG and DTA) and the cone calorimeter method were used for evaluation of PP/APP system. The results of TG and DTA show that the addition of APP with different percentage (1,3,5,10%) leads to decrease in the T_{onset} and increase in the T_{outset} ; the calculated time of the complete degradation increases. This behavior means that the addition of different percentages of APP improve the flammability of PP. The total heat release (THR), the heat release rate(HRR), the mass loss rate (MLR), the time of ignition, the effective heat of combustion(EHC), and the smoke production and toxic gas formation were estimated from the profile of the cone calorimeter curves. From these parameters, it is observed that all the parameters decrease by adding APP to PP. From the analysis of data, it may be conclude that the addition of APP to PP improves the flammability without remarkable effect on the mechanical properties.

Keywords: Flame retardant, polypropylene, Ammonium polyphosphate, Thermal analysis, Cone Calorimeter

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I-Introduction

There are several methods (1-5) to enhance flammability retardation of the polymer materials. Among these P-N compounds gained a special attention because they are rich in nitrogen and phosphorous. Ammonium polyphosphate (APP) is useful compound for flame retardation of polymeric materials when it is mixed with the polymer. It is also environmentally friendly and of low cost.

There is an increase of demand to the polypropylene in industry for its excellent properties. The drawback for its uses, in some industries, is its flammability. In this field, Adivareku et al (6) synthesized a halogen free encofriendly phosphorous based flame retardant (FR) for polypropylene (PP). They found that the synthesized materials enhance the limited oxygen index (LOI) vlues of propylene and also arrests the antidripping properties. They also found that the thermal stability of PP increases with increasing the FR contents. Matko et al (7) prepared a flame retardant thermoplastic polymer containing recycled rubber, low density polyethylene, ethylene vinyl acetate copolymer and an intumescent additive system consisting of waste polyurethane foam and ammonium polyphosphate. They concluded that the process ability of compounds containing recycled materials is excellent and applicable as flame retardant thermoplastic rubbery material for engineering purposes such as floor covering or interior penel in construction of building.

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Ammonium polyphosphate $Al(OH)_3$ system, and APP/ ethylene diamine phosphate are suitable flame retardants(8,9). Reija et al (10) studied the effect of APP/nanoclay system on the flammability of the epoxy materials. They found that the addition of APP improves the flame retarding of the epoxy. Hupuarachchi et al (11) found that using $Al(OH)_3$ APP system as fire retardants for unsaturated polyethylene resin improved the thermal stability of the materials. Watanabe et al (12) prepared APP in different phases and used as flame retardants for polymer. Recently in 2013, Feng et al (13) concluded that the addition of intumescent flame retardant composite including APP decreased the rate of degradation of PP. Different modifications were carried out to increase the surface area of APP including chemical reaction or mixing to increase its efficiency when used as flame retardants materials (14-24).

The presence (25) authors prepared nonosized ammonium polyphosphate (APP) from ammonium dihydrogen phosphate, urea and ammonia solution. They characterized the product using x-ray diffraction, IR absorption, thermogravimetere analysis and electron microscope. The results of characterization show that the produced samples are in nono scale particles ranging from 58 to 81 nm, which exhibits large surface area.

The present work aimed to use the nanosized ammonium polyphosphate as flame retardant material for polypropylene.

II EXPERIMENTAL:-

II-1 Materials:

Polypropylene:

Polypropylene (PP) has a chemical formulaCH_2=CH-CH_3. According to IUPAC nomenclature, its molecular formula (C3H8)n ,its density 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. Polypropylene is one of the most commonly used thermoplastic materials owing to its easy processing and good mechanical strength. A major advantage of polypropylene is its ability to be used in a wide range of fibrous and non woven (26). Its advantage is:-

1) Pure polypropylene is highly flammable at room temperature and leaves little char.
2) The low melting point (160-170 ºC) is an advantage in many non woven processing steps
3) Its flammability, a very low limiting oxygen index (LOI) (17.4) relative to the other polymers as for PVC= (45), for PC= (26).
4) Polypropylene is burned with free flame without leaving a char residue its burning is accompanied by dripping and flowing of the flaming polymer which is a considerable hazard in itself. Therefore, a flame retardant showed not only cause extinction of the burning polymer, but also prevent the flaming dripping .Thus flame retardant a Polypropylene material is a requirement of many fields for their applications. On other hand. as far as the safety aspects and environmental problems are considered, the use of some of the flame retardant is limited for the evaluations of toxic gases and corrosive smoke during combustion

Ammonium polyphosphate:

It was prepared in the previous work (25) from Ammonium dihydrogen phosphate, urea and ammonia solution having nano-particle size.

II-2 Preparation of the samples

Before melt spinning the PP pallets were compounded with APP in twin screw co rotator extruder where RPM of screw was 60-80.The compounding of APP with PP was done by adding 1,3,5, 10 percentages concentration of APP. The temperatures of five zones (including untreated PP) in extruder were 175,185,195,210 and 220 ºC for zone 1,2,3,4 and 5 respectively. The melt spinning of

the PP pallets blended with APP was carried out on laboratory melt spinning machine supplied by Fair Deal Association, New Delhi, India. The temperatures of this spinning extruder zones maintained were 190,200 and 220º C for zone-I, zone-II and zone-III respectively (27). The temperatures were distinctly above the melting point of the PP pallets. The extruder zone was provided with a nitrogen gas to prevent oxidation of PP. The pallets melt forms a molten mass, which moves further to metering pump and then to Die head zone. The filaments extruded from the spinnerets were cooled down by blowing cool air in 1.5 meter long quench duct.

II-3 Characterization of APP/PP

IR spectra, Thermal measurements and morphology of (APP/PP) were performed by using the previous technique described elsewhere (25)

II-4: Flammability measurements

Combustion experiments were performed in a cone calorimeter (Fire Testing Technology, UK) at an incident heat flux of 35 kW/m^2 according to ISO 5600. The bottom and edges of each specimen with a dimension of 100 ×100 ×4 mm are wrapped with aluminum foil. All samples were run-in duplicate and the average value was reported. Various parameters can be measured including time of ignition (TI), heat release rate (HRR) as a function of time, peak of heat release rate (PHRR), mass loss rate, and so on. The experimental error of data from the cone calorimeter was about 5%

II-5 : Mechanical measurements

The impact strengths were measured with an XJJ-5 Charpy impact tester (Chengde Tester Co., Ltd.,China) at 25°C according to GB/T1043. Tensile tests were carried out on an Instron Universal Tester Machine (model 5566) with a cross head speed of 50 mm/min according to ASTM D 638.The tested parallel samples were 10, the presented results are average data

III- RESULTS AND DISCUSSION:-

III-1 IR spectrum:-

The IR spectra of untreated and treated samples are shown in Fig 1. The IR spectra show that different peaks appeared as follows:-

1) Absorption bands appeared at 3078, 2985, 2922, 1457, 1378, and 890 cm^{-1}; these bands may corresponding to the C-H bond of the saturated and unsaturated part of the PP chain (14).
2) Specific characteristic to the P-O and P=O attached with the APP were appeared at 1117, 1081 and 1079 cm^{-1}.
3) Absorption band appeared at ~ 1248 due to the interaction between the APP and PP (P-O , P-O-P and P=O)
4) Appearance of the bands at 2926 and 2853 corresponding to the formation of OH or N_H^{4+} derived from the ammonium part of APP
5) Important series of bands at 1158, 1000, 927,492 corresponding to the P-O and P-O-P indicate that there are an interaction between the PP and APP.
6) A series of bands appeared at 760,680 and 600 specified the APP molecules

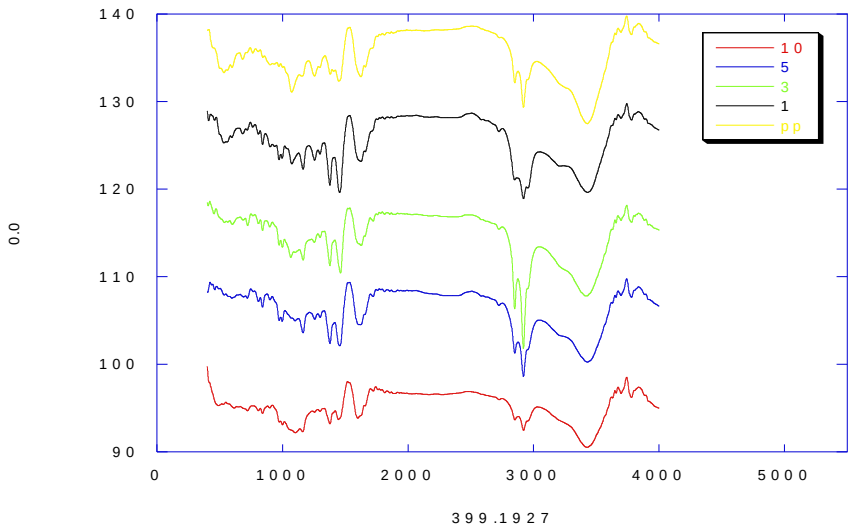


Fig 1 IR spectra of trated and untreated PP

III-2 The mechanical properties:-

Table 1 shows the tensile strength and the elongation values of the untreated and the treated PP. For PP, the mean value of tensile strength equals to 5417.97 N/mm^2 and the mean value of elongation is 14.66 %. The tensile strength for the treated PP with 1, 3, 5, and 10 % of APP changes slightly to become equal to 5161, 4635.516, 4854.973 and 5618 respectively. While the elongation mean values change to become equal to 15.3, 13.3, 16, and 20 % respectively as the APP added with percentages equal to 1, 3, 5, and 10 %. From these results, it is obvious that both the tensile strength and the elongation values changed slightly by adding APP up to 10 % which indicates unremarkable effect on the mechanical properties of PP.

Table 1. The mechanical properties of treated and untreated PP

Sample No.	(APP %)	Mean value of Tensile strength (N/mm^2)	Mean value of elon test %
1	0	5417.975	14.667
2	1	5161.443	15.333
3	3	4635.516	13.333
4	5	4854.973	16.000
5	10	5618.290	20.000

IV- The flammability evaluation of APP/polypropylene system:-

Different measurements were carried out to detect the suitability of nanosized APP as fire retardant for polypropylene to increase the ability of its applications. Thermogravemetric analysis and cone calorimeter are two preferred measurements for testing the validity of APP as flame retardant for polymers

TGA is a useful indicator of polymer decomposition and flammability behavior and it is a preferred technique for rapidly comparing and reading the thermal stability of various polymers(1,5,28). On the other hand, the cone calorimeter test is very useful to follow the flammability of polymer, where different parameters could be determined such as, heat release rate (HRR), total heat release (THR), effective heat of combustion (EHC), smoke production rate (SPR), average specific extinction area (ASEA) and carbon monoxide (CO) yield(29).

In the present work the two previous methods are carried to evaluate the addition different percentage of nanosized APP to polypropylene; the obtained results will be discussed.

IV-I: The thermogravemetric analysis:-

Fig 2 shows the thermal degradation curves for pp (0% APP sample 1, 1% of APP sample 2, 3% of APP sample 3, 5% APP sample 4 and 10% APP sample 5). The TG curves of the samples behave in a similar trend with difference in the T_{onset} (onset temperature) and the T_{outset} (outset temperature). They shows that the PP and PP/APP system degraded thermally in three sequence stages(30,31). The first stage starts from the room temperature and ends at the T_{outset} and represents the evolving of the volatile materials and the water content. The second stage starts at T_{onset} and ending at T_{outset} and represents the main degradation of the tested samples. The third stage starts at the T_{outset} and continues with slight decrease in the weight. This last stage represents the carbonization of the polymer chains and forming the char. The TG curves show that the addition of APP to polypropylene resulted in a decrease in the T_{onset} when adding 1, 3, 5 and 10 % of APP. On the other hand, the addition of APP to PP causes an increase in the T_{outset}. This behaviour means that the addition of APP is effective as flame retardant to PP. Taking in consideration that the T_{onset} is the starting of ignition and T_{ouset} is the ending of the ignition, the net time of ignition was calculated and given in Table 2. From the time of ignition, it is found that as the percentage of APP increases the time of ignition increases from 1.7 to 4 min. This finding also indicates that APP could increase the time of ignition, hence the spread of flame is decreased. The DTA curves of the five samples are represented in Fig 3. The profiles of the DTA indicate that the appearance of an endothermic peak at 460 ºC for pure PP, this peaks confirmed the TG behaviour. For the APP/PP system the DTA shows that the endothermic peaks shifted from 460 to 426 ºC for the sample containing 10 %. For comparison between the tested samples, Table 3. Summarizes the results of investigation.

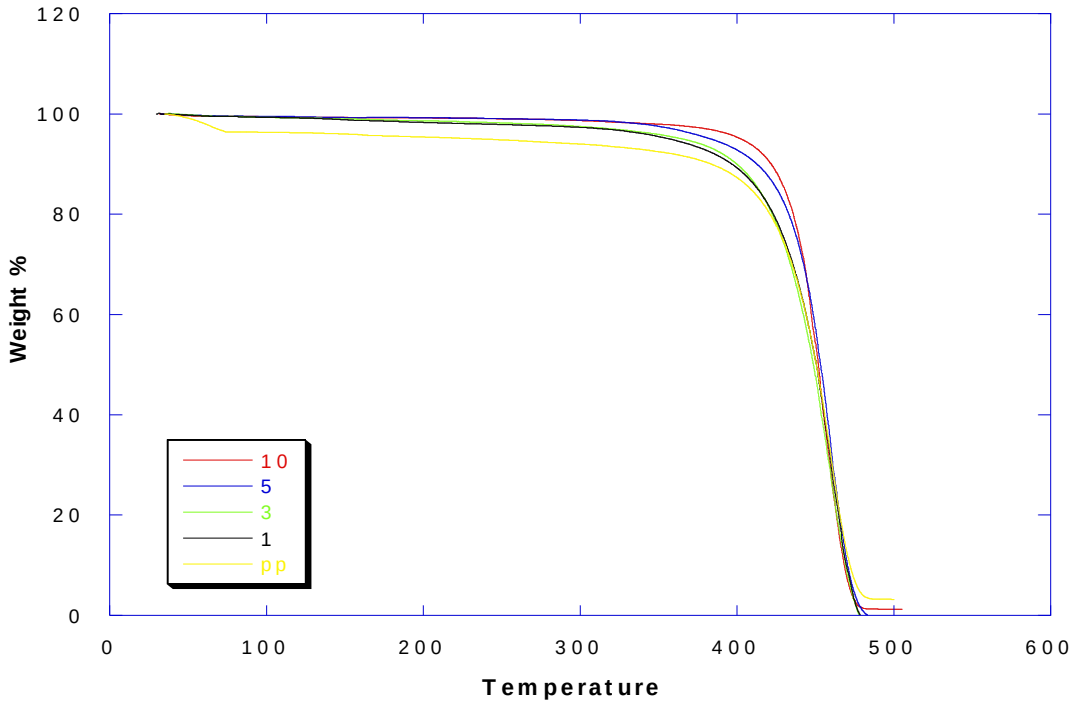


FIG 2 The TG curves of the treated and untreated PP

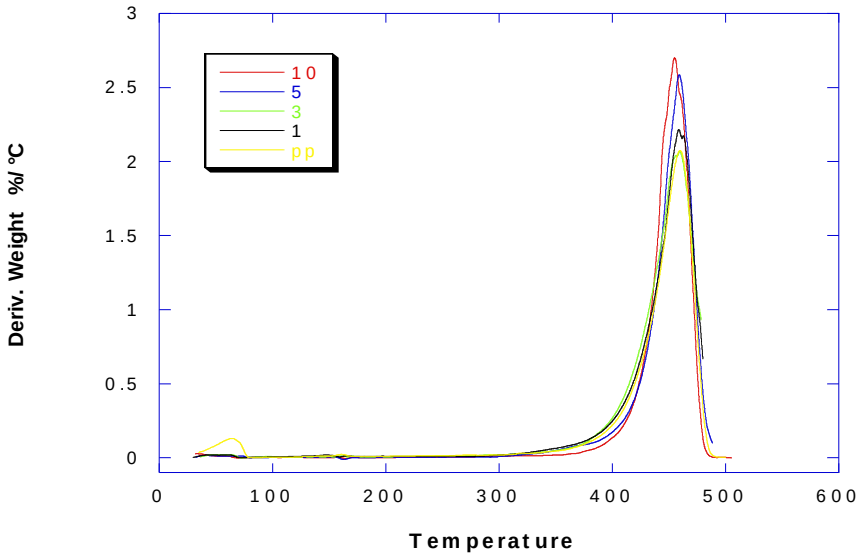


FIG 3 The DTA curves of the treated and untreated PP

Table 2: The T_{onset}, T_{outset} and the time of degradation of treated and untreated

Sample No.	APP %	T_{onset} °C	T_{outset} °C	Time of degradation(mi
1	0	410	427	1.7
2	1	400	440	4
3	3	404	438	3.4
4	5	400	423	2.3
5	10	385	420	3.5

Table 3: The values of the endothermic peaks of treated and untreated PP as calculated from the DTA

Sample No.	APP %	Endothermic peak °C
1	0	460
2	1	452
3	3	446
4	5	439
5	10	426

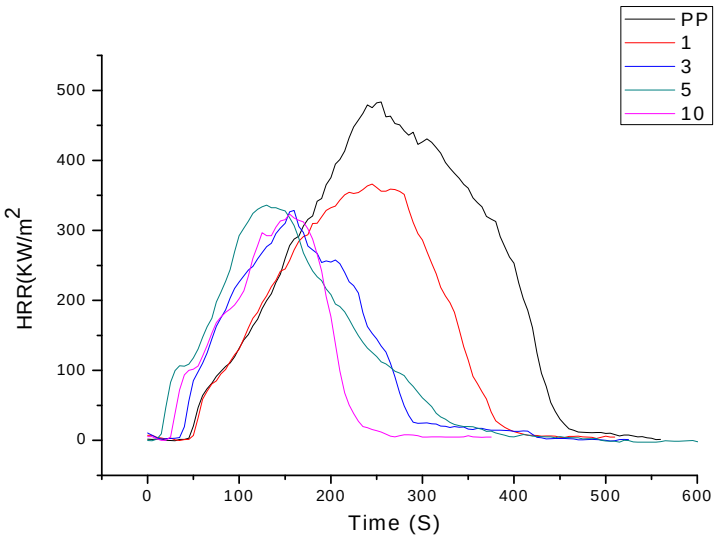
IV-2 The cone calorimetric measurements:-

In the present study, the flame performance of PP and PP/APP system was evaluated using a cone calorimeter. The flammability parameters, including heat release rate (HRR), total heat release, (THR) , mass loss (ML), mass loss rate (MLR), total smoke release (TSR), effect heat of combustion (EHC), mass loss rate (g/sim^2) (MLR), carbon monoxide yield (kg/kg), and carbon dioxide yield (kg/kg) were recorded to evaluate the burring behavior of PP and PP/APP system. The obtained results are given in Table 4, as extracted from Fig 4. This test was conducted according to the slandered of the International organization for standardization ISO 5660-1

Table 4. the data obtained from the cone – calorimeter for treated and untreated PP

the measurements	Sample 1	Sample 2 APP=1	Sample 3 APP=3	Sample 4 APP=5	Sample 5 APP=10
Total heat release (THR)	99.4	74.9	52.6	58.2	41.1
Heat release rate(HRR)	408.17	362.75	328.21	336.94	323.3
Time of ignition	38	49	36	15	26
Effective heat of combustion (EHC)	71.78	77.72	60.09	79.9	76.77
Co yield	0.0263	0.3803	0.2056	0.4791	0.3492
Co2 yield	5.1	36.07	16.63	25.72	13.81
Specific extinction area	3160.88	4402.4	18.03	1244.74	108.45
Mass loss rate	14.04	17.86	22.31	16.46	20.98
Total smoke release	7.9	7.6	3.7	4.8	5.3
Tot. smoke production	0	0	0	0	0

The total heat release (THR) for PP and PP/APP system in range of the added APP from 1 to 10 % is presented in Fig 4. THR represents flame spread through the bulk of material and it is recognized as an important parameter representing the total available energy within the medium. The curves of PP and the treated samples behave in similar trends with changes in the values of the THR. From the curves it is observed that the values of THR increase slightly through the first 100 sec then increase rapidly forming a maximum values at about 400 sec, then reach to steady state. The value of THR for PP sample equals to 115.4 MJ/m^2 and decreased to 74.9 as 1 % of APP is added to PP. This means that the addition of 1 % APP reduces the value of THR by 36 % and these percentages continue to increasing recording ~ 64 % at 10% of APP. In comparison, the values of THR for the treated samples are always less than that of PP without treatments. This behavior leads to suggest that the heat release of PP is suppressed at high concentrations of APP.



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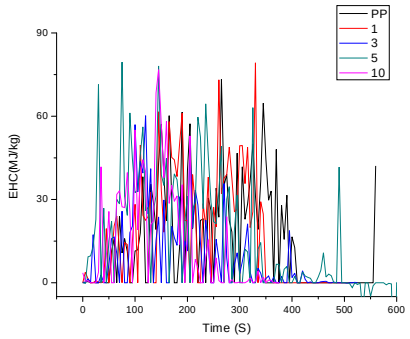

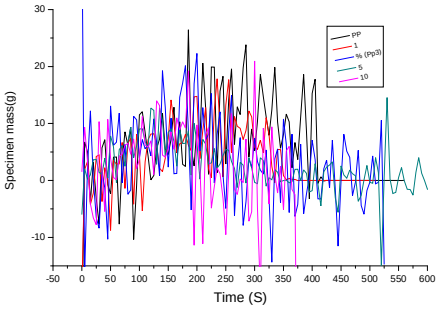

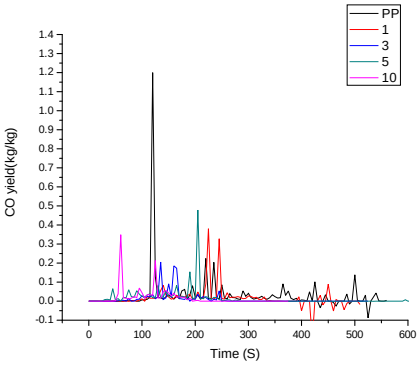

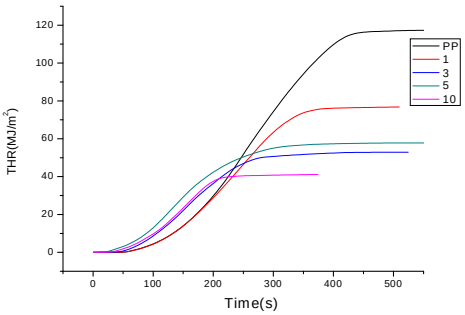

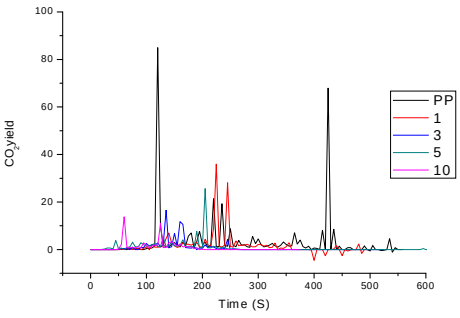

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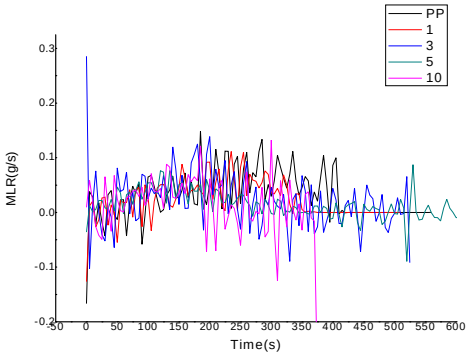

Figure 4. The cone calorimeter measurements of treated and untreated PP

The heat release rate (HRR) is the main parameter measured by the cone calorimeter. HRR curves for treated and untreated PP are shown in Fig 4 .The curve of the PP sample shows that the value of HRR increase gradually by time of ignition forming a maximum at 408.17 then decreased by time. This value decreases to become equal to 362.75 by adding 1 % APP. For the samples containing 3 %, 5% and 10% APP it is equal to 328.2, 336.94, and 323.31 respectively. This indicates that as the percentage of APP increases the mean peak of HRR decreases. The shape of the curve exhibits the glowing combustion of the char residue when the volatile parts had been burnt out. It is known that APP after degrades, gives phosphoric acid which catalysis the dehydration and carbonization of the polymers. This leads to the formation of a char layer at lower temperature. This layer forms a protection layer to reduce spreading of the flame (32).

The mass loss rate (g/sec.m^2) of the PP and PP/APP system are summarized in Table 4 as deduced from the curves. The curve of (MLR) for PP shows a dramatic decrease with time, reaching to the steady state after 400 sec. As 1 % of APP was added the values of (MLR) decreases to about 4.31 and forming a peak at 17.86 showing the half value of the pure PP. As the percentage of APP increases these value decreases reaching to the values of 2.84. This means that the addition of APP as flame retardant for PP reduces the value of the MLR by 50% as shown from the Figure 4.

Time of ignition is the time from the beginning of combustion to the start of sustained burning (33). It indicates the fire resistance performance of the material being tested. The longer it takes for a material to ignite, the more resistance the material is to burn. For PP samples treated with APP, the time to ignition was shorter than for the untreated control sample (Table 4). This may due to the presence of APP within the treated polymer. Also, it is proposed that APP promotes the degradation process of polymer and lowers its degradation temperature. It is noteworthy from the data of the time of ignition that the addition of 1% APP causes no significant change in the time of ignition while further addition causes reducing in the time of ignition, from 38 s for the control sample to 26 s for the sample containing 10 % APP. Also, it is observed that the flame out decreased from 456.6 to 255 sec by increasing the content of APP.

The effective heat of combustion (EHC) measured in the cone calorimeter corresponds mostly to the flame burning condition and thus to the combustion of volatiles from within the material (34). It is calculated by dividing the THR by the mass loss with a specific time. The mean EHC and peak EHC values of PP and PP/APP system are presented in Fig 4 and Table 4.The peak EHC for untreated PP equals to 71.78 and it increased slightly by adding 1 % APP and shows the same increase at 5 and 10 % of APP.

Smoke production and toxic gas formation play a critical role in the spread of the fire while the production of smoke reduces the visibility and thus reduces the chance for one to escape from the fire and hence, toxic gas becomes effective. So, it useful in this field to decrease the smoke

production and minimize the production of the toxic gases. For PP/APP system as the PP starts to flame the value of the total smoke production in presence of 1 % APP equals to 7.6 as recorded in Table 4, it decreases by adding 3 % , 5 % and 10 % of APP reaching to 5.3 recording a reduction in the total smoke production by 32 %. This reduction gives a chance for the persons to escape and facilitates the work of the fire men.

One of the most toxic gases released from burning polymer is carbon monoxide, CO. For untreated PP the value of CO yield equals 0.0263 (Kg/kg). This value decreases by treating PP with APP to become, 0.3803,0.2056, 0.4791, and 0.3492 for adding 1,3,5 and 10 % of APP. This decrease may due to the degradation of APP part by heating and released some inflammmmable gases containing phosphorous and nitrogen gases. These gases lead to the incomplete combustion of polymer (35).

From the parameters measured and estimated through the cone calorimetric test, it may be conclude that all the parameters decrease by adding APP to PP. These findings lead to suggest that the addition of APP with percentage up to 10 % improves the flame retardancy of PP without considerable changes in the mechanical properties. It is noteworthy that the same results were obtained by M.Wang et al(29) , when they used different percentage of APP to wood

V- CONCLUSION:-

From the evaluation of the prepared nanosized APP as flame retardant for PP, it may be conclude that:-

1) The addition of different percentages of APP to polypropylene improves the flammability without a considerable change in the mechanical properties.
2) The addition of different percentage of APP to polypropylene decreases the T_{onset} and increases the T_{outset} of the polymer which causes an increase in the time of degradation. This finding gives the chance for persons to escape the person and gives a long time to the fire men.
3) All the parameters of the cone calorimeter decrease. This behavior emphasizes the using of APP as flame retardant for PP.

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