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# Effect of flame retardants and 1% stabilizer on thermal decomposition and physics properties of thermoplastic polymers due to TGA and DTA tests

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# ABSTRACT

This work introduces seven various polymer samples blended in a twin-screw extruder with flame retardants (FR), and an additive, which is a 1% Stabiliser, to investigate thermoplastic polymer melting, thermal decomposition, and melting moderates. The reduction method is used to analyze its melting points, physics and chemical properties, and thermal decomposition. Thermogravimetric Analysis (TGA) and Differential Thermal Analysis (DTA) tests are known as thermal analysis tests. It was established that halogenated flame retardants have little effect on reducing the rate of gas escape from polymer melt which affects the viscosity, as it was found. Thus, the APP and FR245 in Polymer help in some polymer leftovers. All samples were treated with flame retardants and no clay, melted completely, at the end of the test some samples did change. All these results investigated by (the XRD) test either, Scanning Electron Microscopic (SEM) or Digital images have been used also applied Limiting oxygen index (LOI) test, in the previous work for the same samples of polymers to verify the results and record a more precise outcome to draw the relationship between melting and dripping behavior via the following studies due to the furnace test which is modulated and calibrated. As a result, the addition of clay together with conventional flame retardants enhances the overall thermal stability forming the polymer. The compatibles help in improving the dispersion of the clay in polymer and reduce the rate of gas escape from polymer melt affecting the viscosity.

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# 1. Introduction

The present study is part of a larger project exploring the production of fire-retardant synthetic nano/micro composite fibers. This work started with and concentrated on Polypropylene (PP) results of TGA and DTA tests analysis as shown in Fig.1. PP polymer is a useful commodity polymer mainly used in clothing, furniture, floor coverings, medical, geotextiles, and automotive applications, due to its low cost, lightweight, good mechanical properties and low reactivity towards other chemicals. Polypropylene (PP) had higher values for tensile strength at break, the Polypropylene polymer degradation is occurring at high temperatures. The main advantage is that PP being an addition polymer made from the monomer propylene, is unusually resistant to many chemical solvents, acids, and bases.

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Nomen	clature:			
Α	Compatabiliser	С	Stabiliser	
В	Nanoclay	D	Pelletising	
Pb	Polybond	TGA	Thermogravimetric Analysis	
FR	Flame retardants	DTA	Differential Thermal Analysis	
LOI	Limiting oxygen index	SEM	Scanning Electron Microscopic	
PP	Polypropylene	Tg	Glass Transition	
Nul	local Nusselt number along with the heat source	C		

Additive flame retardants if used, are required in large amounts (>20% w/w) to provide the required fire protection to products [1], this flame retardancy effect increases with increasing irradiation and vanishes with decreasing irradiation [2-3]. However, such high levels of additives cause polymer processing problems, in particular for their extrusion into thin films or fibers. The flame retardancy is sensitive to modification of the flame retardant, the use of synergists/adjuvants, and changes to the polymeric material.

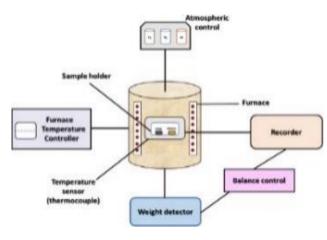


Figure 1.PP polymer DSC analysis

A detailed understanding facilitates the launch of tailored and targeted development [3]. TGA and DTA tests still require some amount of conventional flame retardants. It must be noted, however, that with this approach of using flame retardants as additives, the polymer content in the formulation is reduced compared to the unmodified polymer. In [4] earlier publications they have demonstrated that nano clays can be nano dispersed in polypropylene with proper choice of compatibility, and the compounded polymer can be extruded into fibers [2, 4]. Nano clays, although increasing the thermal stability of polypropylene and helping in char formation [5], do not reduce the flammability of PP fibers to a large extent [2]. Clay, nano clay, and a small amount of flame retardant (5%), when added together to PP containing certain compatibilizers, the extruded fibers could selfextinguish. In the previous work, only ammonium polyphosphate was used [4], whereas in this study different phosphorus. The main aim of this work is to understand the mechanism of combined action of different types of flame retardants on thermal stability and melting of PP. Several polypropylene samples containing compatibilizer, clay, stabilizer, and different flame retardants have been compounded in a twin-screw extruder and their thermal.

# 2. Experimental work



**2.1 Materials and methods** The following materials were obtained from commercial sources: the seven different thermoplastic Polypropylene polymers (PP) samples were tested after being blended with UV-Stabilizer (Nor) and Flame Retardants.

*The Polymer Provision:* 7 polypropylene samples composition (wt %) and additives blended with 1% Stabilizer (Nor 116), and 5% FR as shown in Table 1, the samples composition and named. These samples were required for many tests such as the LOI test in addition to the XRD test and DSC test for PP polymer only [6]. It was recorded that the DSC result of PP polymer showed the value of 105.93 °C for Glass Transition (Tg) and the Heat flow is 1.7261 mV, as shown in Fig. 2.

Table 1. Mass percentages of various components in the PP
formulations

Sample	pp %	Graft 1%	FR 5(%)			
PP-Nor116	100					
PP-APP 107	096	Polybond				
PP-NOR-NH	091	Polybond	APP			
PP FR 107	091	Polybond	1FR245			
PP Amgard 107	091	Polybond	Amgrad NH			
PP 3OB	096	Polybond				
PP APP 3OB	092		App			
<b>Note</b> : 1 FR = APP, NH, FR245, also PP-NOR-NH is PP 107, FR245.						

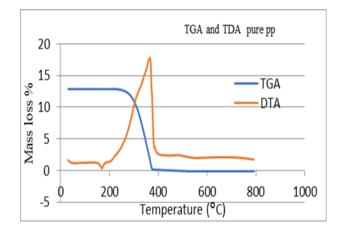


Figure 2. TGA and DTA curves results obtained from computer

Both of these two tests give a more precise outcome by using the Scanning electron microscopic (SEM) images [10], moreover, UL-49 burning and flammability test [12], also for applied TGA and DTA test in this work, and the furnace test [8, 9]. Fig.1, a block diagram of thermogravimetric and analysis principle.

As recorded in Table 1, coarse mono filaments (strands) blended to the PP wt %; stabilizer 1 wt %; and FR 5 wt %. However, the nano clay could be

used which is Cloisite 20A, Southern Clay Products, USA, is a montmorillonite clay modified with dimethyl, dehydrogenated tallow quaternary ammonium chloride. This modified clay was chosen because of its nonpolar alkyl substituents. A Thermoelectric Prism Europa 16 twin screw extruder with a temperature profile over six heating zones between 179–1900C was used for compounding. Polymers (diameter  $1.8 \pm 0.2$  mm) were collected before palletizing.

# 2.2 Measuring properties

# a) Characterization of the char residue

Selected samples are the presence of maleic-anhydride grafted polypropylene, APP, and clay in PP (sample 3 (PP-NOR-NH)) has encouraged char formation [10] whereas no char could be seen for the control sample 3, which when still a molten polymer, whereas no char could be observed for samples containing clays excluding samples 1, 2 and 6

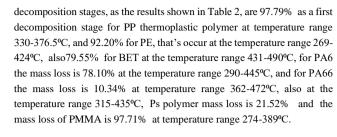
where char could be found. These have also been included to improve the dispersion of the clay [3], the dispersion and heat diffusion appear to be much better for samples 3 and 4. The differences are due to the effect of different flame retardants on the dispersion of the clay [5, 3] Obvious char formation is not apparent until both clay and flame retardant are present together, this behavior is not unexpected as clay presence changes melting and residual char behavior of polymer by reducing its thermoplastic.

#### b) Characterization and testing

Differential thermal techniques such as TGA-DTA (TA, SDT 2960) have been used in this work. TGA and DTA tests, (Table 2 and Table 3) were conducted using a standard procedure [3]. The melting and degradation behaviors of each sample, Table 1 was observed and noted. The general objective of this test is to determine and measure two reactions which are the chemical and physical reactions that occur on samples, also this measurement could measure temperature different between the sample and the reference material (empty pan) as function of temperature or time by DTA, and the DTA used to know and measure the release of heat such as endothermic reaction or absorption of heat such as exothermic reaction. To determine the mass changes of the samples as a function of temperature or time by measuring the mass loss by TGA, and to calculate the sample char residue produced at the end of this test at 600 °C. This test cured out by heating the samples from room temperature  $\approx 20^{\circ}$ C up to 700°C at 10°C/ min as a rate of heating in air (atmospheric Oxygen), the different polymers sample weight is 10mg. The next typical curves to shown TGA- DTA curves and PP polymer as mentioned in Fig. 3 analysis of pure pp to compared with 7 polymers samples with FR and additives recorded in Table 3. Also, all these TGA and DTA results shown in Fig.3 which shows more particulars and details. For these 7 pure polymers, TGA and DTA data are recorded in Table 2, for thermal decomposition analysis.

The seven pure different thermoplastic polymers samples, starting with PP thermoplastic polymer investigation as shown in Fig. 3, also as recorded in Table 2; PE, PET, PA6, PA66, Ps and PMMA start to melt at 330, 269, 431, 290, 362, 315°C and 274°C respectively.

The initial straight line up to these melt temperatures indicates no mass loss, and then these polymers samples start to loss the weight. The initiation of mass loss (expressed in weight percentage) was measured from the temperature were the mass loss starts, mass loss as a first and second



# 3. Results and discussion

The TGA and DTA curves as seen in Fig. 3 (a-g) to show the 2 stages of mass loss, also the endothermic and exothermic chemical reactions of the 7 polymers different types in air atmosphere (100 ml/min) at 10  $^{\rm OC}$ /min heating rate.

#### 3.1 TGA curves analysis

TGA analysis are polymers thermal analysis decomposition. The seven different thermoplastic polymers samples, starting with the PP investigation, also as recorded in Table 3, PE, PET, PA6, PA66, Ps and PMMA start to melt at 330, 269, 431, 290, 362, 315°C and 274°C respectively.

The initial straight line up to reaction indicates release of heat, for PP. Sample that's occur at 170°C (from 151°C -184°C) (lower than 330°C).

This mean for all polymers sample that endothermic reaction occurs from the glass to the rubbery or soften thermoplastic states, also for BE sample that's happened at 123°C (from 109-137°C) (lower than 269°C), for BET sample that's occur at 255°C (from 230°C -265°C) (lower than 431°C), for PA6 its 223°C (from 191°C -244.5°C) (lower than 290°C), for PA66 at 266°C (from 243°C -289°C) (lower than 290°C), for PS that's happened at 415.9°C (from 405°C -425°C) and for PMMA that's occur at 339.39°C (from 333°C -344°C). As seen in Fig. 3 a, b, c, d, e, f and g, for the seven pure polymers samples of PP., PE, PET, PA6, PA66, Ps and PMMA the DTA curve does not show any significant change up to151°C, 109°C, 255°C, 191°C, 243°C, 415.90°C and 339.39°C respectively; however for PP polymer one endothermic reaction peaks at 170°C, can be seen in Fig. 3a, this endothermic peak could be due to softening or rubbery of pp. polymer. Also, one exothermic peak at 366°C relate to first and second stages mass loss in the TGA analysis, and this peak at 366°C represents melting of the pp. polymer; however, for PE one endothermic reaction peak at 123°C, can be seen in Fig. 3b, this endothermic peak could be due to softening or rubbery of PE. polymer. Also, two exothermic peaks at 260°C and 419°C relate to the first and second stages mass loss in the TGA curve, and these two peaks represent melting of the PE. Polymer, also for PET polymer one endothermic reaction peaks at 255°C, can be seen in Fig. 3c, this endothermic peak could be due to the softening or rubbery of PET .



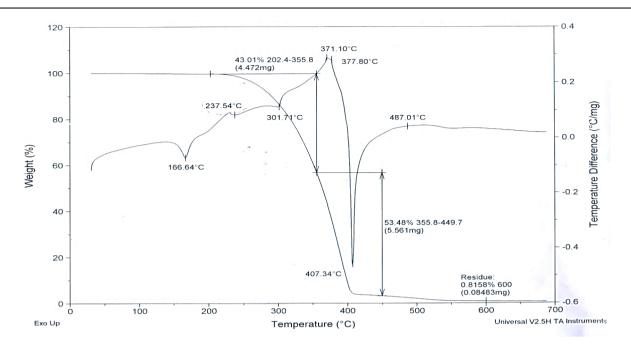


Figure 3A. The TGA and DTA curves of the 2 stages

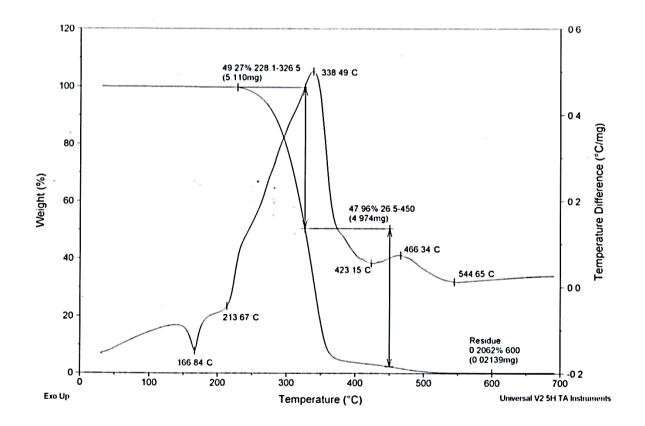


Figure 3B. The TGA and DTA curves of the 2 stages



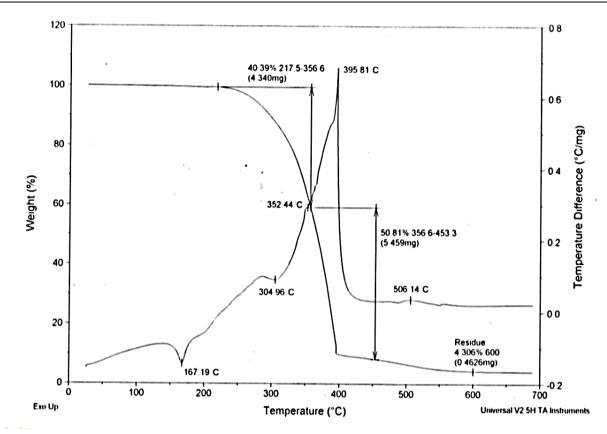


Figure 3C. The TGA and DTA curves of the 2 stages

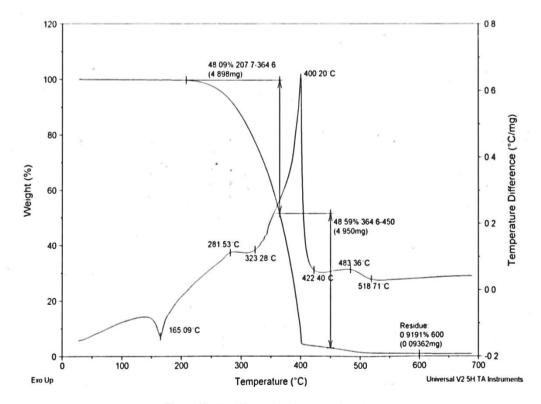


Figure 3D. The TGA and DTA curves of the 2 stages



**Table 2.** Data and TGA of simples

DTA			TGA						
Sample	Endo.	Exo.1	Ехо. 2	Exo . 3	Exo. 4	Onset degrad	ation		
	stat max. °C	°C	°C	°C	°C	Mass loss% Stage l Tem.range °C	Mass loss% Stage 2	Resedue at 600 C the end %	
1- PP.	170	366				97.79% 330-376.5	2.07% 380-540		0%
2- PE.	123	260	419			92.20% 269-424	5.72% 438.5-536		0.20 %
3- PET.	255	126	411	422	529	79.55% 431-490	9.05% 432-500		0.17 %
4-Nylon6	223 60.7 420	381	497	537		78.10% 290-445	6.33% 514-445	1.42 %	
5-Nylon66	266 381 412 468	392	447	493	525	10.34% 362-472	11.92 483-594	0%	
6- Ps.	415.9	441.08	498.28			90.24% 315-435	4.24% 439-515	0.85 %	
7- PMMA	339.39	380.24				97.71% 274-389		0%	



		DTA				TGA		
sample	Endo.	Exo.1	Exo. 2	Exo. 3	Mass loss%			
	stat max.	°C	°C	°C	Stage 1 Temperature. range °C	Stage 2	Residue at 600 C the end of line	
5	167.26		301.3		44.85%	45.87%	4.36%	
	313.22	132.45			235.2-364.6	364.6-450		
	338.09							
4	165.09	281.53	400.20	483.36	48.54%	48.22%	1.68%	
	320.61				207.7364.6	364.6-450		
1	166.84 423.15	338.49	466.34		49.27%	47.96%	0.21%	
					228.1-326.5	26.5-450		
3	167.19	352.44	395.81	506.14	41.17%	49.56%	3.66%	
	304.96			v. small	229.9-352.2	352-450		
7	167.36 303.27	314.07	356.69	450.02	41.87%	45.98%	27.44 %	
	338.28				235-330	353-450		
6	167.63	341.82	487.53	577.57	41.00%	54.42%	1.47%	
	209.79				218.4-331.8	331.8-450		
	285.23				210.4-351.0	001.0400		
	395.25							
	528.15							
2	166.64	371.10	377.60	487.01	41.47%	55.17%	0.67%	
-	237.54	571.10	577.00	407.01	43.01-355	355-450	0.07%	
	301.71				12.01-222	333-130		
	407.34							

Table 3. Record the TGA and DTA analysis results of 7 polymer samples

Also 4 exothermic peaks at 126°C, 411°C, 422°C and 529°C relate to first and second stages mass loss in the TGA curve, and these 4 peaks represents melting of the polyester (PET) polymer, for PA6 two endothermic reaction peaks at 60.7°C and 420°C, can be seen in Fig. 3d, at these two endothermic peaks could be due to softening or rubbery of Nylon 6 polymer.

Also 3 exothermic peaks at 381°C, 497°C and 537°C relate to first and second stages mass loss in the TGA curve, and these 3 exothermic peaks represents melting of the Nylon 6 polymer, also for PA66 polymer, 3 endothermic reaction peaks at 381°C, 412°C and 468°C, can be seen in Fig. 3e, at these 3 endothermic peaks could be due to softening or rubbery

of Nylon 66 polymer. Also 4 exothermic peaks at 392°C, 447°C, 493°C and 525°C relate to first and second stages mass loss in the TGA curve, and these peaks represents melting of the Nylon 66 polymer, also for PS. polymer, two exothermic peaks at 441.08°C and 498.28°C relate to first and second stages mass loss in the TGA curve, and these two peaks represents melting of the Ps. polymer, can be seen in Fig. 3f, and for PMMA one exothermic peak at 380.24°C relate to first and second stages mass loss in the TGA curve, and this peak represents melting of the PMMA polymer, can be seen in Fig. 3g. Since the thermal analysis studies will be uses to find out the processing temperature of these formulations, the melting point



gives the temperature at the polymer can be processed in the extruder, also it gives the temperature value, useful to know which polymers can be used in melting and dripping tested by using the UL-94 and the Furnace testes. The temperature at 50% mass loss gives the burn temperature. At this temperature, polymer samples will be burn in UL-94, to observe the melt/drip behavior studies of the polymers, also to compare all these processing temperature results with polymers samples with different FR. and additives results, therefore in previous work, the effect of the additives/fillers on various physical and melt dripping behaviors intensity of polymers was explored, in [10, 12, 13, 15].

#### 3.2 DTA curves analysis

The DTA curve shows the endothermic and exothermic reactions. After comparing TGA and DTA curves can be seen in Fig. 3a, b, c, d, e, f and g, and in Table 4 that the TGA curves, at the temperatures lower than melting temperatures, the sample not melting yet, but its soften or rubbery or flexible thermoplastic states, and in another hand, the DTA curve shows the endothermic reaction indicates release of heat, for sample no. 5 that's occur at 167.26°C (from 182.9°C -151°C) (lower than 235.2°C), this mean -for all polymers sample-, that endothermic reaction occurs from the glass to the rubbery or soften thermoplastic states, also for sample 4 that's happened at 388.77°C (from 150.1-178.5°C) (lower than 207.7°C), for sample 1 that's occur at 389.59°C (from 148.3°C -182.9°C) (lower than 228.1°C), for sample no. 3 its 223°C (from 151°C -184.7°C) (lower than 229.9°C), for sample no. 7 at 266°C (from 147.5°C -179.4°C) (lower than 235°C), for sample no. 6 that's happened at 415.9°C (from 194.2°C -182.9°C) (lower than 218.4°C). Sample no. 2 that's occur at 339.39°C (from 153.7°C -176.7°C) (lower than 43.01°CAs seen in Fig. 4, a, b, c, d, e, f and g, for the seven deferent polymer samples no. 5, 4, 1, 3, 7, 6 and 2, the DTA curve does not show any significant change up to 182.9°C, 150.1°C, 148.3°C, 151°C, 147.5°C, 194.2°C and 153.7°C respectively; however for sample no.5 shown 3 endothermic reaction peaks at 167.19°C, 331.03°C and 381.4°C can be seen in Fig. 3a, only these 2nd and 3rd endothermic peak could be due to softening or rubbery of number 5.

Also, three exothermic peak at 303.65°C, 364.65°C and 395.97°C relate to first and second stages mass loss in the TGA curve, and this peak at 303.65°C or 364.65°C represents melting of this sample.

However, for sample of number 4, these 3endothermic reaction peak at 166.55°C, 321.51 and 374.85°C can be seen in Fig. 3b, these 2nd and 3rd endothermic peak could be due to softening or rubbery of sample no. 4. Also, for sample no. 1; two exothermic peaks at 260°C and 370.30°C relate to first and second stages mass loss in the TGA curve, and these two peaks represents melting of this sample; either for sample 1; one endothermic reaction peaks at 389.59 °C, can be seen in Fig. 3c, this endothermic peak could be due to softening or rubbery of sample no.1. Also in sample 7, 2 exothermic peaks at 972.84 °C and 450°C relate to first and second stages mass loss in the TGA curve, and these 2 peaks represents melting of sample no. 7, for this sample two endothermic reaction peaks at 390.94°C and 339.73°C, can be seen in Fig. 3d, at these two endothermic peaks could be due to softening or rubbery of polymer sample no.7. Also sample no.6; one exothermic peak at 805.94 °C relate to first and second stages mass loss in the TGA curve, and this exothermic peak represents melting of this sample, also for this sample, 6 endothermic reaction peaks at 391.6°C, 389.87°C and 270.55°C, can be seen in Fig. 3e, at these 3 endothermic peaks could be due to softening or rubbery of this sample. Also sample no.3; one exothermic peak at 922.35 °C relate to first and second stages mass loss in the TGA curve, and this peak represents melting of this sample, either for this sample no.3; two endothermic peaks at 390.58°C and 309.67°C relate to first and second stages mass loss in the TGA curve, and these two peaks represents melting of this sample can be seen in Fig. 3f and for sample no.2 one exothermic peak at 868.12 °C relate to first and second stages mass loss in the TGA curve, and this peak represents melting of the this sample, can be seen in Fig. 4g also for this sample 3 endothermic reaction peaks at 166.3°C, 237.54°C and 296.88°C.

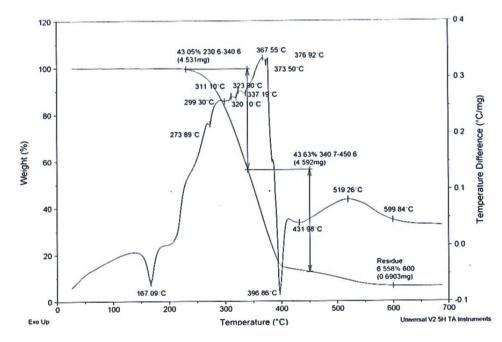
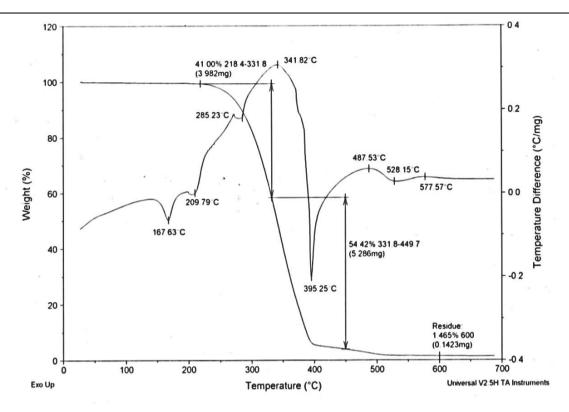
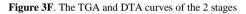


Figure 3E. The TGA and DTA curves of the 2 stages







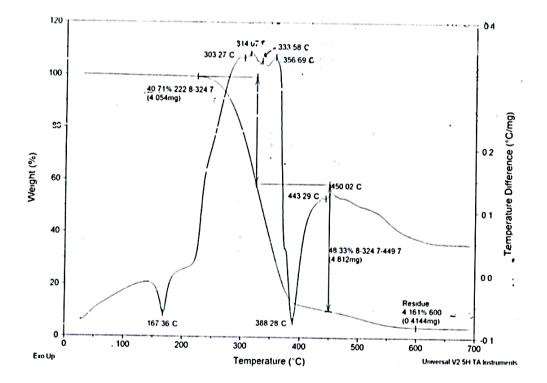


Figure 3G. The TGA and DTA curves of the 2 stages



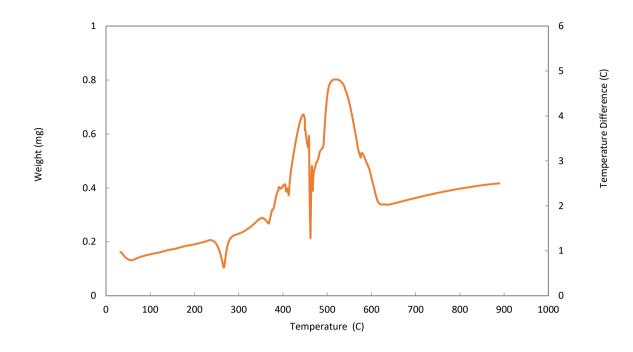


Figure 4. Shows DTA analysis with TGA and DTA of sample no. 5, Nallon 66\_02 size 12.9875 mg

## 4. Conclusions

The dispersion of nano clay can be improved by compounding polymerclay samples [3] and by adding compatibilizer. The effect of different flame-retardant types on thermal stability, flammability and char formation tendency of Optical micrographs of tape samples showing the effect [10] of Polybond (pb) on clay dispersion. Polypropylene (PP) is studied. PP, compatibilizer, Clay, stabilizer and different flame retardants have been compounded in a twin-screw extruder to produce polymers with improved thermal and flame-retardant properties. TGA and DTA thermal analysis has been used to study thermal properties. All flame retardants acting in the condensed phase (phosphorus- and nitrogen- containing) [5] lowered the rate of decomposition, whereas halogenated flame retardants had little effect. Addition of 1% compatibilizer with 3% clay in sample 3 has caused micro dispersion of the clay particles [10].

However, by increasing level of compatibilizer to 3%, the dispersion at the microlevel has improved as observed by absence of clay layer. The samples where compatibilizer and clay have been masterbatches prior to dilution and blending. Flame retardants increased the thermal stability of all samples and helped in char formation [3]. The decomposition significantly reduces the molecular weight, followed by easy flowing of polymer melts.

Up to this period, this research completed for the recent developments in the area of science polymer – nanocomposites have suggested that by the addition of just a small quantity (< 5%) of organically modified layered silicate nano clay (montmorillonite) [3] to a PP polymer matrix could enhance many of the properties of that polymer, including the fire performance [1].

polypropylene (PP) the lack of polar groups in the polymer chain makes direct intercalation or exfoliation of the nano clays almost impossible [1] without the use of a compatibilizer [2-3]. Maleic anhydride-grafted polypropylene (PP-g-MA) can be used as a compatibilizer, which enhances the interaction between the clay and polymer with strong hydrogen bonding between -OH or -COOH and the oxygen groups of clay [5, 3]. Watched the effect of flame retardancy, FR, and additives on the Polymer burning behaviors, Chemical and Physical properties, and then dripping to try to stop the fire and save the life. Therefore, it's very important to collect and record all the results related to these aims nano clav is Cloisite 20A. Southern Clay Products, USA, is a montmorillonite clay modified with dimethyl, dehydrogenated tallow quaternary ammonium chloride [3]. This modified clay was chosen because its nonpolar alkyl substituents) are taken to mean conditions fulfilling [3]. Future research can now use the newest factor discovered and reported in USA and Dubai conferences this year 2023, applying the newly discovered factor which effects drawing the relationship between melting and dripping behavior by using only moderated furnace test results recorded in 2011.

In

# **Declaration of competing interest**

The authors declare no conflicts of interest.

# Funding source

This study didn't receive any specific funds.

#### Data availability

The data that support the findings of this study are available from the corresponding author upon reasonable request.



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