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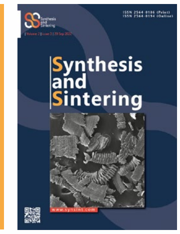

Research article

Investigation of flame retardancy properties of polypropylene-colemanite and intumescent flame retardant additive blends

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A B S T R A C T

Polypropylene (PP) represents a considerable proportion of polyolefins (PO) used in different industrial applications such as automobile components, textiles, packaging, insulation, medical devices, various housewares, and household appliances due to its efficient cost, desirable mechanical, thermal and electrical properties, easy processability and recyclability. Because of its carbonaceous structure, PP is a highly flammable material with an LOI value of 18 that presents a serious fire hazard. In this research, Intumescent flame retardant (IFR) and colemanite were added to polypropylene to compose 30% of the total mass of the polymeric compounds, and the synergistic effect of colemanite with intumescent flame retardant (IFR) additive in PP was investigated by limiting oxygen index (LOI), glow wire test (GWT), UL-94 test and mechanical properties measurements. The LOI, UL 94 and glow wire test results showed that colemanite had a significant effect on flame retardancy and LOI value which can reach 37.6% with a loading amount of 2 wt% colemanite at the total amount of flame retardant additives kept constant at 30 wt%. Additionally, the PP/IFR compounds passed UL 94 V0 rating and both 750 °C and 850 °C glow wire tests with 2–8 wt% colemanite loading. According to Thermal gravimetric analyses (TGA), the results indicated that colemanite improved the thermal stability of PP/IFR compounds and also promoted the formation of the char layer. When colemanite mineral is added to polypropylene without an IFR system, it has no effect on the flame retardancy properties of polypropylene. When all properties have been taken into consideration, colemanite can be used up to 4 wt% in IFR.

KEYWORDS

Flame
Flame retardancy
Polypropylene
Intumescent
Colemanite

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1. Introduction

Fire is a uniquely destructive force of nature and is known as a physical and chemical phenomenon. Three main components must be available for any combustion to take place these three components will form the fire triangle, oxygen, heat, and fuel. Fire starts with the interaction between the flame, fuel, and the surrounding that can be strongly nonlinear, and quantitative estimation of the process involved is often complex [1].

Fire is started by lightning or accidentally by people. It also creates large amounts of smoke pollution, releases gases, and also destroys our ecosystem dramatically. It is very important to provide protection from fires and to increase escape time when the fire happens. Plastics are generally flammable so it is necessary to add flame retardant additives to meet required standards. Therefore it is the main point to increasing flame retardancy properties of commercial plastic materials. Flame retardants are added to plastic material and other materials to inhibit, smoke suppress, or delay combustion. The main tasks of flame retardants added to plastics are as follows:

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Table 1. Properties of polypropylene.

Polypropylene material	Basic properties
Density (g/cm^3)	0.905
MFI (g/10 min)	25

- Increasing the ignition temperature
- Reduction of the rate of burning
- Reduction of the flame spread
- Reduction of the smoke generation

Flame retardant additives can be inorganic, organic, mineral-based, halogen, or phosphorus-based products that are used in consumer products. Lately, halogen-free flame retardants have been used instead of halogenated flame retardants. Halogenated flame retardants have intensive disadvantages such as the formation of smoke, toxic gasses, and corrosive decomposition product. In addition, the EU industry needs to find solutions to substitute the halogenated additives employed in the production of flame retardant plastic materials, used in an extensive range of commercial products, and markets such as household appliances, and the electronic sector. Unlike halogenated flame retardants, mineral-type flame retardants provide extremely low smoke values. They also show an environmentally friendly profile [2]. Minerals have been used as a flame retardant additives in plastic applications for many years. They have low toxicity, ready availability, and low cost. Halogen-free flame retardant alternatives are commercially available but due to the high filler content between 40–60% need to provide the same FR properties as a conventional halogenated flame retardant. The processability and final product properties are reduced whilst the cost of parts is considerably increased severely limiting the range of applications and hindering halogen-free flame retardant market introduction to replace dangerous halogenated flame retardants [3]. Intumescent flame retardant systems are used to increase the flame retardancy performance of polymers. They have recently found a place in polymer science as a method of providing flame retardancy performance of polymeric formulations [4, 5]. Colemanite ($2CaO.3B_2O_3.5H_2O$) is a naturally hydrated calcium borate able to release structural water according to endothermic degradation. The mechanism is ended with water and inorganic residue formation. It provides a protective layer on the polymer surface [6].

In this study, polypropylene homopolymer will be compounded with intumescent flame retardants (IFR) and colemanite to increase the flame retardancy properties of polypropylene material. According to a literature search, boron-based flame retardants can show effectiveness both in the condensed and gas phases. Especially, zinc borate uses as a flame retardant additive for polyvinyl chloride and engineering plastics. Boron-based flame retardant additives show endothermic reactions. In the first stage, these types of additives release water into the system and create a glassy coating on the polymeric surface for protection. Boric acid performs dehydrating and creating charring actions with oxygen-containing structures. Colemanite ($2CaO.3B_2O_3.5H_2O$) is a very important borate mineral that contains calcium and boron with 5 mol of crystal water. Therefore colemanite minerals have been chosen to create a synergistic effect with IFR in the polypropylene compound. It was expected that the colemanite performs endothermic. It will release water from its structure which creates a cooling effect during the fire. In terms of economic aspects, IFR is a higher price than colemanite so reduction of IFR in the polypropylene compound will bring cost reduction and effectiveness.

Table 2. Main compositions and basic properties of IFR PNP2D.

Appearance and compositions	Basic properties
Appearance	White, free-flowing powder
Phosphorus content (%)	21 ± 1
Nitrogen content (%)	18 ± 1
Average particle size (µm)	Approx. 15
Moisture content (%)	0.50
Density at 25 °C (g/cm^3)	Approx. 1.8
Thermal decomposition (°C)	≥ 250

2. Experimental

2.1. Materials

The polypropylene homopolymer of PP 578 L was used and supplied by Sabic (Netherlands). The IFR (Intumescent flame retardant) compound consisting of APP and synergist (JLS PNP2D) was supplied by JLS (China). The main compositions and properties of polypropylene, IFR PNP2D, and colemanite were given in Table 1, Table 2, and Table 3 respectively. Colemanite mineral was supplied by Eti Maden. All chemicals were used without further purification and the raw materials used in the experiment were not chemically treated before the testing.

2.2. Preparation of IFR and colemanite filled PP formulations

Before mixing and getting the colemanite dried the sample was heated to 100 °C for 4 h then the sample cooled slowly. Polypropylene (PP), Intumescent flame retardant (IFR), and colemanite were mixed in twin screw extrusion at different loading amounts to obtain seven other polymeric compounds. The total loading amount of IFR and colemanite was kept at 30 wt% mass of whole amounts of the compounds.

Table 3. Main composition and basic properties of colemanite.

Component	Content
B_2O_3	40.00 ± 0.50%
CaO	27.00 ± 1.00%
SiO_2	4.00 ± 6.50%
SO_4	0.60% max
As	35 ppm max
Fe_2O_3	0.08% max
Al_2O_3	0.40% max
MgO	3.00% max
SrO	1.50% max
Na_2O	0.50% max
L.O.I	25.00% max
Moisture	1.00% max
Bulk density	1.00 ton/m^3 max

Table 4. The specifications of the twin screw extruder.

Item	Units	PRISM TSE 24
Screw diameter	mm	24
Maximum screw speed	rpm	1000
L/D (shaft length/screw diameter)	-	28:1
Shaft center line distance	mm	
Channel depth	mm	5.15
Screw diameter/channel depth	-	5.15
Screw/barrel clearance	mm	0.2
Shaft center line distance	mm	18.75
Shaft center line/radius ratio	-	1.5625
Total volume	cm^3	511
Free volume	cm^3	228
Barrel peripheral surface area	cm^2	830
Surface area/total volume	m^2/l	0.14
Surface area/free volume	m^2/l	0.31
Max channel shear rate @ 1000 rpm	s^{-1}	244
Max power	kW	9

Colemanite was added to the system with mass fractions 2, 4, 6, 8, 15, and 30 wt%. The flame retardant PP compounds were produced using a co-rotating twin screw extruder at a temperature profile of 180, 180, 180, 185, 190, 190, and 195 °C. The twin screw extruder was an intermeshing co-rotating extruder. The brand name of the extruder is PRISM TSE24 IIC modular system, with 28:1 L/D. The extruder has two Brabender DSR28 volumetric feeders, degassing port, water bath, pelletizer, and also chopper. It is also equipped with seven modular barrel segments, 96 mm in length and an L/D ratio is 28:1. The specifications of the twin screw extruder are indicated in Table 4 [7–9]. The screw profile of the extruder was designed to achieve good melting and mixing of polymers by high shear and high residence time in the screw zones. Six zones are available in the extruder. The names of the zones are given below.

- Entrance of raw material and solid conveying zone
- Plasticizing zone
- Melt conveying zone
- Extra homogenization zone
- Degassing zone
- Pressure built-up zone

After the polymer comes out of the extruder, it goes to the water bath. The polymer spaghettis coming into the water bath is cut in the pelletizer unit. At the end of this process, polymer granules can be obtained by pelletizer unit.

Pellets were cut in a granulator unit and the injection molding machine (Arburg 370 S 800-150) was used to produce test samples according to ISO 20753:2018 [10]. Prepared compounds are indicated in Table 5.

3. Results and discussion

3.1. Measurement of density

Density values were measured with Mettler Toledo density test equipment according to the procedure explained in ISO 1183-1 Plastics-Methods for determining the density of non-cellular plastics-part 1: Immersion method [11]. Density test results of compounds indicated stable rise with rising colemanite amount. Colemanite and IFR have a higher density of 2.42 g/cm^3 and 1.8 g/cm^3 respectively. They have superior density than polypropylene because its density is 0.906 g/cm^3. The addition of colemanite and IFR to polypropylene concluded in compounds with superior density results. Moreover, even though total FR ratios were fixed due to the higher density of colemanite, increased colemanite content increased the density of compounds slightly. Density testing outcomes are indicated in Table 6.

3.2. Melt flow index (MFI) test results

Melt flow index (MFI) or melt flow rate (MFR) was measured by using the Zwick testing machine according to ISO 1133 test standard. The testing device is basically an extrusion plastometer operating at a fixed temperature [12]. Test temperature and weight are set to 230 °C and 2.16 kg, respectively. When IFR was added to polypropylene, the MFI value started to decrease from 22.35 to 14.88 g/10 min at 230 °C and 2.16 kg loading. When colemanite was added to polypropylene with IFR, MFI values began to increase from 14.88 to 21.8 g/10 min. After a 15% loading amount of colemanite, MFI values decreased from 21.40 g/10 min to 20.30 g/10 min. It can be said that the processability of colemanite is easy in PP compound due to its structure compared to other additives. According to the literature, it was demonstrated that

Table 5. Experimental design of colemanite and IFR filled polypropylene compounds.

Sample	PP (wt%)	Colemanite (wt%)	IFR (wt%)
Neat PP	100	0	0
PP70/IFR30/C0	70	0	30
PP70/IFR28/C2	70	2	28
PP70/IFR26/C4	70	4	26
PP70/IFR24/C6	70	6	24
PP70/IFR22/C8	70	8	22
PP70/IFR15/C15	70	15	15
PP70/IFR0/C30	70	30	0

Table 6. Density results of neat PPs, IFR, colemanite, and IFR/colemanite filled PP compounds.

Sample	Density (gr/cm^3)
Neat PP	0.9069
PP70/IFR30/C0	1.053
PP70/IFR28/C2	1.062
PP70/IFR26/C4	1.064
PP70/IFR24/C6	1.067
PP70/IFR22/C8	1.073
PP70/IFR15/C15	1.082
PP70/IFR0/C30	1.120

Table 7. MFI results of IFR, colemanite, and IFR/colemanite filled PP compounds.

Sample	MFI (gr/10 min)
Neat PP	22.35
PP70/IFR30/C0	14.88
PP70/IFR28/C2	21.80
PP70/IFR26/C4	22.00
PP70/IFR24/C6	21.00
PP70/IFR22/C8	21.40
PP70/IFR15/C15	20.30
PP70/IFR0/C30	22.00

MFI values of the final product were increased with increasing colemanite loading. Therefore, process temperature can be reduced to eliminate the risk of release of the IFR additives during compounding. In addition, it was observed minerals that have flat or lamellar structures can slide against each other during the application of shear forces, which causes to increase in flowability in the final compounds. The MFI testing outcomes were represented in Table 7.

3.3. Mechanical properties of tensile test

Tensile properties of polymeric compounds were investigated by using the Zwick–Z020 testing equipment according to ISO 527-1/2 1A Plastics-Determination of Tensile Properties-part 2: test conditions for molding and extrusion plastics [13]. At least 5 specimens were tested for each plastic compound. Testing outcomes are indicated in Table 8. Putting IFR in high quantity to PP material decreases strength and strain values due to the effect of mineral-like reinforcing effect. The important mechanical properties of neat PP, PP/IFR/colemanite filled compounds were also searched by discussing the effectiveness of colemanite content in the polymer matrix. Increasing the amount of colemanite content in polypropylene compounds increases the elastic modulus of neat polypropylene. The tensile strength value of polypropylene homopolymer decreased when IFR and colemanite were added to the polymeric system. No selective difference is observed in tensile strength values of only IFR and IFR/colemanite filled PP compounds. 30% colemanite-filled PP compound showed better tensile strength properties compared to IFR/colemanite filled PP compounds at different loading amounts. The strain value of neat polypropylene homopolymer decreased with increasing amounts of colemanite content in PP/IFR/colemanite compounds. No selective difference is observed on tensile strain values of 30% IFR and 30% colemanite in PP compounds. When the colemanite amount increased in the system, it resulted in an increase in the modulus of elasticity and decrease in tensile strength and strain at the yield point. According to the literature, Sahin observed that elastic modulus values increased with increasing of colemanite amount. It is generally believed that the polymer matrix around the inclusions can produce plastic deformation thanks to stress concentration. The elastic modules nonlinearly increased when the amount of IFR content in polypropylene compounds increased. IFR material has a stiffening effect on the final compound. This effect is related to the movement of the big chains of the matrix material blocked by IFR particles. The stiffness of filled PP compounds can be improved by adding IFR content to the PP compound. However, increasing IFR content leads to a decreased tensile strength of the PP compound. It is related to interfacial adhesion between inorganic materials and polymer matrices. If interfacial adhesion improves, tensile yield strength values can start to increase. Soykan and Veliyeva investigated the mechanical properties of colemanite compounded polypropylene composites. They have observed that the addition of the colemanite decreased tensile strength and tensile strain of composite due to ductile properties [14].

Table 8. Mechanical properties of colemanite and IFR filled PP compounds.

Sample	Elastic mod. (MPa)	Tensile strength at yield point (MPa)	Tensile strain at yield point (%)
Neat PP	1578 ± 10.69	35.7 ± 0.23	9.43 ± 0.06
PP70/IFR30/C0	2760 ± 102.7	26.4 ± 1.00	4.05 ± 0.12
PP70/IFR28/C2	2410 ± 50.4	24.2 ± 0.184	4 ± 0.068
PP70/IFR26/C4	2270 ± 115	24.0 ± 0.209	4 ± 0.082
PP70/IFR24/C6	2090 ± 138	23.7 ± 0.423	4.1 ± 0.063
PP70/IFR22/C8	2220 ± 72	23.9 ± 0.165	4.2 ± 0.060
PP70/IFR15/C15	2440 ± 101	23.4 ± 0.058	4.1 ± 0.068
PP70/IFR0/C30	2210 ± 62.2	24.1 ± 0.197	5.1 ± 0.14

3.4. Izod impact test results-notched

Izod notched impact strength of specimens was determined by Zwick–HIT–50 P test equipment according to ISO 180/A Plastics-Determination of Izod Impact Strength [15]. Izod impact testing outcomes of neat PPs, colemanite, and IFR filled PP compounds were indicated in Table 9. According to many investigations, the impact properties of mineral filled plastic compounds are bound up with particle size and amount of mineral in the system. Another item can be related to the improvement of the interface between plastic material and minerals. Oral [8] specified that the addition of calcium carbonate decreased the Izod impact properties of polypropylene but when maleic anhydrate polypropylene was added to the system, the interface

Table 9. Izod impact test results of IFR and colemanite filled PP compounds.

Sample	Izod impact strength (kJ/m^2)
Neat PP	3.21 ± 0.14
PP70/IFR30/C0	1.76 ± 0.14
PP70/IFR28/C2	1.83 ± 0.09
PP70/IFR26/C4	1.88 ± 0.12
PP70/IFR24/C6	1.76 ± 0.10
PP70/IFR22/C8	1.97 ± 0.10
PP70/IFR15/C15	1.77 ± 0.10
PP70/IFR0/C30	1.80 ± 0.10

Table 10. Heat deflection test results of colemanite and IFR filled PP compounds.

Sample	Heat deflection temperature (°C)
Neat PP	87.0
PP70/IFR30/C0	90.2
PP70/IFR28/C2	91.8
PP70/IFR26/C4	92.4
PP70/IFR24/C6	93.6
PP70/IFR22/C8	94.1
PP70/IFR15/C15	96.7
PP70/IFR0/C30	98.7

between calcium carbonate and polypropylene has been improved so it resulted in higher Izod impact values compared to neat polypropylene. It has been tested that the Izod impact value of PP homopolymer is around 3.22 kJ/m^2. Colemanite and IFR filled PP compounds showed a negative effect on Izod impact strength values at different loading amounts. No selective difference is observed on Izod impact values at 2%, 4%, 6%, 8%, and 15% colemanite loading amounts in PP compounds. The reduction of Izod impact strength with IFR and colemanite putting to PP can be examined by hindering chain versatility and decrease in fracturing energy according to neat polypropylene material. Izod impact properties of flame retardant additives can be improved with the addition of the impact modifiers but impact modifiers have a negative effect on flame retardancy properties so these types of additives weren't added to the compound.

Table 11. TGA results for colemanite and IFR filled PP compounds.

Sample	Residue at 450 °C (w%)	Residue yield at 800 °C (w%)
Pure Colemanite	77.76	75.46
PPH/IFR26/C4	13.34	12.81
PPH/IFR24/C6	14.50	13.43
PPH/IFR22/C8	14.57	14.06
PPH/IFR15/C15	19.08	17.24
PPH/IFR0/C30	26.04	23.03

3.5. Heat deflection temperature testing results

The heat deflection temperature (HDT) values of pure PP and IFR filled PP compounds were evaluated using a heat deflection tester according to ISO 75 standard [16]. This test is utilized to define the short period heat resistance of polymeric materials and to distinguish between materials that are able to sustain light loads at high temperatures and those that lose stiffness over a tight temperature range. The HDT values of neat PP and its flame retardant compounds with various amounts of colemanite are given in Table 10. Putting of IFR high amount into the plastic compound increased HDT from 87 °C to 90 °C. HDT values were increased essentially by the addition of an intumescent flame retardant additive. It was observed that different loading amounts of intumescent flame retardant and colemanite had an effect on the heat deflection temperature of PP compounds. This property will provide extra safety to PP compounds during the fire. PP70/IFR0/C30 sample has a higher heat deflection temperature than PP70/IFR30/C0, because the stiffness of the PP70/IFR0/C30 sample was higher than PP70/IFR30/C0 sample when the temperature

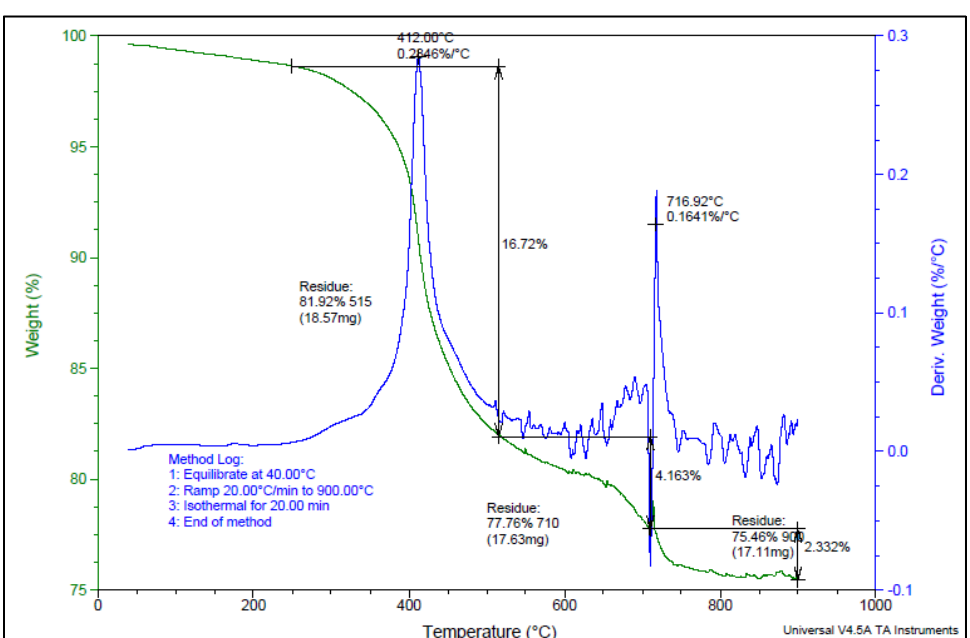

Fig. 1. TGA analysis of the colemanite sample.

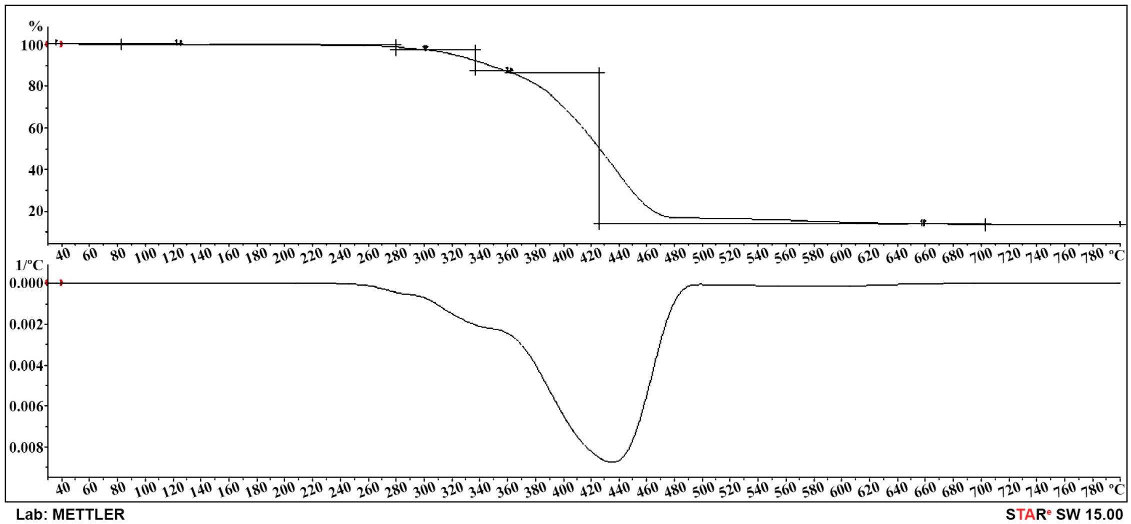

Fig. 2. TGA analysis of PPH/IFR26/C4 sample.

increased. The higher heat deflection temperature is related to the faster molding process in injection molding machines so energy efficiency can be achieved with colemanite filled IFR/PP compounds.

3.6. Thermal gravimetric analysis

Thermogravimetric analysis (TGA) was performed on a Q500 thermal gravimetric analyzer. Samples weighing about 20.0 mg were heated from room temperature to 800 °C at a heating rate of 20 °C/min in a. dynamic nitrogen atmosphere. The nitrogen flow rate was 50 ml/min The TGA graphs in below represent all colemanite filled flame retardant polypropylene compounds in Figs. 1–6. According to the TGA graph of colemanite, it started to decompose at above 225 °C. The amount of residue of colemanite was approx. 75 wt%. When graphs of the colemanite and IFR filled compounds were investigated, it was observed that these compounds shifted through higher temperatures compared to neat polypropylene and also char residue amount was higher than neat polypropylene. Char residue information was given in Table 11.

3.7. Limiting oxygen index

Neat PP can burn easily with a very fast combustion rate and it generates a large amount of black smoke during fire due to this reason flammability properties need to be improved. The flammability behavior of PP and IFR/colemanite filled PP compounds are indicated in Table 12 which shows the differences in LOI values towards colemanite content for the PP/IFR/colemanite compounds at an overall quantity of 30 wt% flame retardant ingredients fixed constant. It has been observed that the LOI grade of the IFR filled PP compound

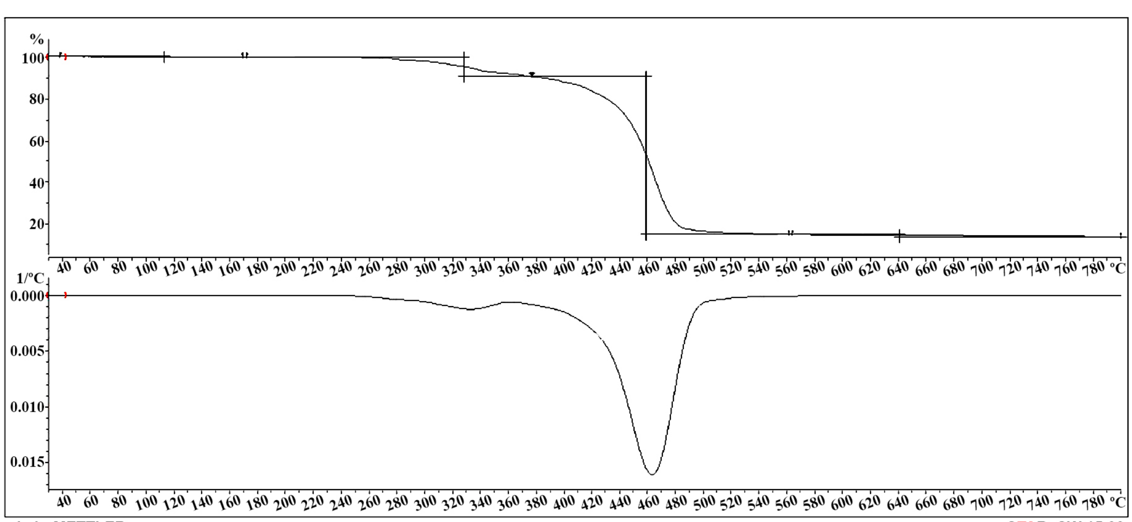

Fig. 3. TGA analysis of PPH/IFR24/C6 sample.

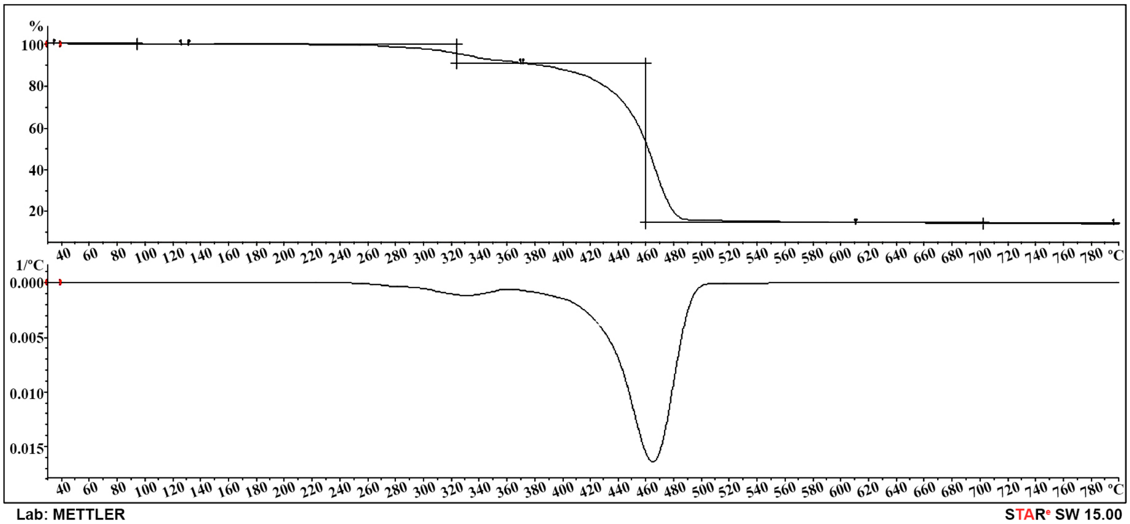

Fig. 4. TGA analysis of PPH/IFR22/C8 sample.

including 30 wt% IFR increased to 37.8 from 18% of neat PP. It showed that IFR can increase flame retardancy properties of PP when used alone in the PP compound. The addition of colemanite with IFR to PP compound showed better flame retardancy properties. The LOI values increased to 37.6% with a 2 wt% loading amount of colemanite in the compound. LOI values decreased rapidly after the addition of 8 wt% loading of colemanite. When the amount of colemanite caught up to 8 wt% the LOI value of the sample decreased to 33%. The results indicated that the replacement of IFR with colemanite at a reasonable amount can improve the flame retardancy properties of the PP matrix. A major cause for this event can be that, during the combustion, a superior amount of filler like colemanite makes the char too rigid and brittle so LOI values start to decrease when mica content increases in the PP compound. Huang et al. observed the same effect in their work with sepiolite and IFR [17]. A synergistic effect was observed between IFR and colemanite when colemanite was used at a reasonable amount in the PP compound. Isitman et al. also observed a synergistic effect between aluminum trihydrate (ATH) and colemanite in polyethylene compound. Partial replacement of ATH by colemanite brings higher LOI values at 10 wt% colemanite loading amount in the resin [18]. Atikler and coworkers observed that the LOI value reached up to 39.3% with a 2.1 wt% colemanite loading amount [19].

3.8. UL 94 flammability test (vertical)

The rate of burning and time of the burning of self-supporting plastics in a vertical position was measured by using a CEAST flammability tester. The CEAST flammability tester is equipped with a test chamber, text fixture, laboratory burner, gas supply, wire gauze, timing device,

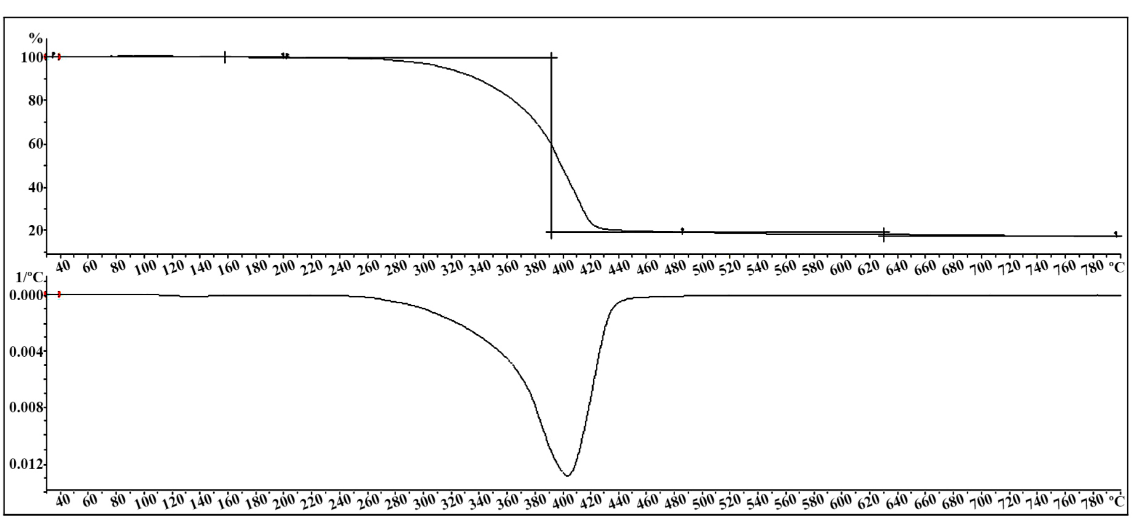

Fig. 5. TGA analysis of PPH/IFR15/C15 sample.

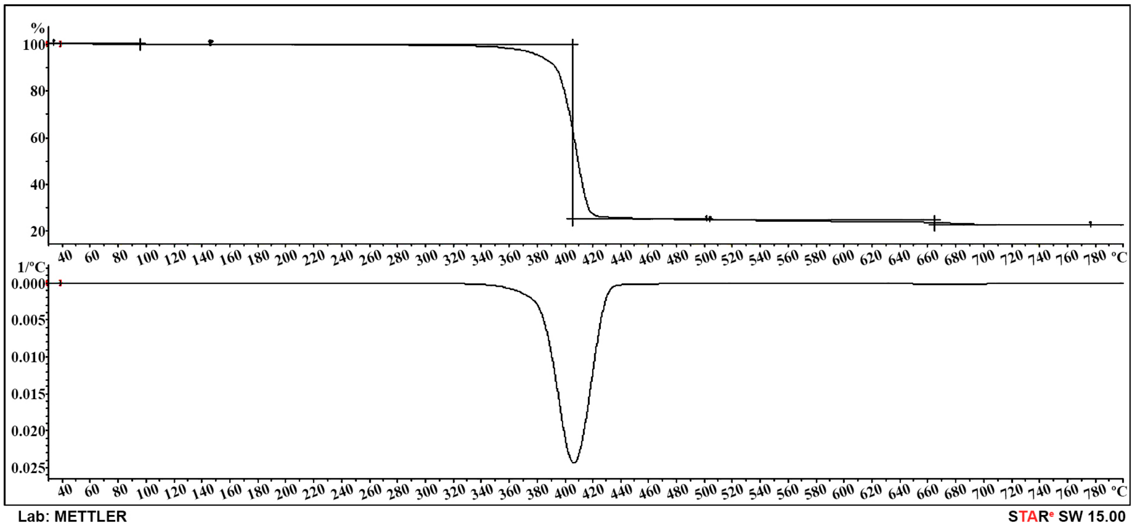

Fig. 6. TGA analysis of PPH/IFR0/C30 sample.

and flexible specimen support fixture. UL 94 flammability test results are categorized by burning rating V-0, V-1, or V-2 depending on the total burning time and dripping behaviors as described in the standard [20]. The best flame retardancy property of plastic materials is classified as the V-0 rating. UL 94 flammability testing is utilized to observe ignition performance and fire spread of polymeric parts. Flammability ratings are categorized as V0, V1, or V2 with UL 94 flammability testing. The best flammability property is obtained by a V0 rating.

V-0: Fire was applied to the test bar two times for overall 10 seconds. The burning of testing parts has to stop within 10 seconds. Flaming drips can't be allowed.

V-1: Fire was applied to the test bar two times for overall 10 seconds. The burning of testing parts has to stop within 60 seconds. Flaming drips can't be allowed.

V-2: Fire was applied to the test bar two times for overall 10 seconds. The burning of testing parts has to stop within 60 seconds. Flaming drips are allowed.

UL-94 flammability testing outcomes of the PP/IFR compounds toward colemanite amount are summarized in Table 13. When the amount of colemanite was adjusted in between 2 wt% and 8 wt% in IFR, samples of the PP/IFR compounds can achieve the V-0 classification. Nevertheless, a rising loading amount of 15% is classified as horizontal burning (HB). According to these results, a suitable amount of colemanite can create a synergistic effect in the PP/IFR/colemanite compounds more sufficient.

Table 12. LOI test results of neat PP, mica, and IFR filled PP compounds.

Sample	LOI (%)
Neat PP	18.0 ± 0.2
PP70/IFR30/C0	37.8 ± 0.2
PP70/IFR28/C2	37.6 ± 0.2
PP70/IFR26/C4	36.9 ± 0.2
PP70/IFR24/C6	34.9 ± 0.2
PP70/IFR22/C8	33.7 ± 0.2
PP70/IFR15/C15	24.9 ± 0.2
PP70/IFR0/C30	19.0 ± 0.2

3.9. Glow wire flammability tests

A glow wire test was carried out at 750 and 850 °C to observe the flaming and glowing properties of the end product. Testing plates can be used to observe mentioned features. GWT test results ensure a way of controlling and checking the capability of materials to remove fires and their capability not to manufacture flame particles capable of spreading flame. Glow wire test results are given in Table 14. IFR at 30 wt% loading amount decreased the burning time of

Table 13. UL 94 vertical test results of IFR, colemanite and IFR/colemanite filled PP compounds.

Sample	UL 94 Test-vertical				
	T1*	T2**	T3†	Dripping	Flame rating
PPH/IFR30/C0	0	2	0	No	V0
PPH/IFR28/C2	0	6	0	No	V0
PPH/IFR26/C4	0	7	0	No	V0
PPH/IFR24/C6	2	8	0	No	V0
PPH/IFR22/C8	2	6	0	No	V0
PPH/IFR15/C15	15	15	0	Yes	V2
PPH/IFR0/C30	Horizontal burning (HB)				

*T1: The burning time of samples with the flame after the first ignition.

**T2: The burning time of samples with the flame after the second ignition.

†T3: The burning time of samples without the flame after the second ignition.

Table 14. Glow wire test results of neat PPs, IFR/colemanite filled PP compounds.

Sample	Glow wire temperature (°C)	
	750	850
PPH/IFR30/C0	There is no glowing and no flashing.	There is no glowing and no flashing.
PPH/IFR28/C2	There is no glowing and no flashing.	There is no glowing and no flashing.
PPH/IFR26/C4	There is no glowing and no flashing.	Ignition time: 1 sec. Burning time: 20 sec. Cotton ignition didn't observe.
PPH/IFR24/C6	There is no glowing and no flashing.	Failed
PPH/IFR22/C8	There is no glowing and no flashing.	Failed
PPH/IFR15/C15	Failed	Failed
PPH/IFR0/C30	Failed	Failed

polypropylene compounds significantly. The glow wire test results indicated that the replacement of IFR with colemanite at a reasonable amount (2 wt%, 4 wt%, 6 wt%, and 8 wt%) can improve the flame retardancy properties of the PP matrix due to dehydration and better char forming. When the colemanite loading amount increased to 15 wt% in PP compounds, flame retardancy properties of the PP matrix decreased due to affecting the continuity of the IFR system as explained by Huang [17, 20]. According to test results, PP70/IFR0/C30 sample didn't pass both 750 °C and 850 °C. PP70/IFR0/C30 sample showed that colemanite can't show good flame retardancy properties when used alone in PP. The amount of IFR in the compound must be more than 20%. IFR is dominant flame retardant.

3.10. Morphology analysis of IFR and colemanite filled PP compounds

The char residue was obtained after the ash content test. The morphology analysis of ash residue was done by SEM. Fig. 7 shows

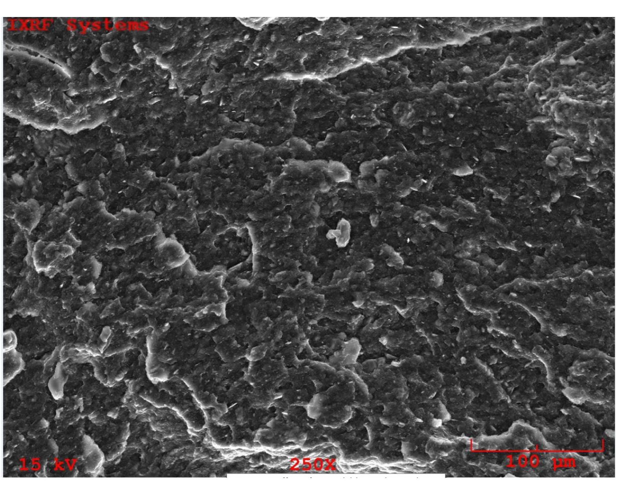

Fig. 7. SEM micrograph of PP70/IFR24/C6 sample.

the SEM picture of the char residue of 6 wt% Colemanite and 24 wt% IFR filled PP compound. It is observed that the structure of char residue of PP70/IFR24/C6 compound has a rigid structure. Having a rigid structure will prevent the flames from entering the polymer structure therefore flame retardancy of polymeric compounds can be enhanced.

4. Conclusions

Intumescent flame retardant and colemanite were mixed with PP material in co-rotating twin screw extrusion to create the flame retardant system and increase the flame retardancy of PP. The LOI, UL 94 and glow wire test results showed that colemanite had a significant effect on flame retardancy and LOI value which can reach 37.6% with a loading amount of 2 wt% colemanite at the total amount of flame retardant additives kept constant at 30 wt%. Additionally, the PP/IFR compounds passed UL 94 V0 rating and both 750 °C and 850 °C glow wire tests with 2–8 wt% colemanite loading. According to TGA analyses, the results indicated that colemanite improved the thermal stability of PP/IFR compounds and also promoted the formation of the char layer. When colemanite mineral is added to polypropylene without an IFR system, it has no effect on the flame retardancy properties of polypropylene. When colemanite was added to the system at a 30 wt% loading amount, the LOI value reached 19.0% but it wasn't enough to increase the flame retardancy properties of polypropylene because, for this reason, colemanite needs to be used with strong flame retardant additives to create a synergistic effect. In terms of mechanical properties, increasing the amount of colemanite content in polypropylene compounds increases the elastic modulus of neat polypropylene. The tensile strength value of polypropylene homopolymer decreased when IFR and colemanite were added to the polymeric system. The strain value of neat polypropylene homopolymer decreased with increasing amounts of colemanite content in PP/IFR/colemanite compounds. Izod impact values of flame retardant-filled PP compound increased with the addition of the colemanite. It has been observed that colemanite and IFR created a synergistic effect to increase the Izod impact strength of the compound. HDT values were increased essentially by the addition of an intumescent flame retardant additive. Consequently, IFR is an expensive flame retardant additive so it needs to be used with cheaper and more abundant minerals in the polymeric compounds. Since colemanite is a very common mineral in Turkey, a cost advantage can be achieved when they are used together with IFR. When all properties have been taken into consideration, colemanite can be used up to 4 wt% in IFR filled PP compound.

CRediT authorship contribution statement

Merve Zakut: Investigation, Methodology, Writing – original draft.
Nilgün Kızılcan: Resources, Supervision, Writing – review & editing.

Data availability

The data underlying this article will be shared on reasonable request to the corresponding author.

Declaration of competing interest

The authors declare no competing interests.

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