

# Thermal Properties and Fire Retardancy of Polypropylene/Wood Flour Composites Containing Eco-friendly Flame Retardants

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**Abstract:** Thermal properties and the flame retardancy of polypropylene/wood flour (PP-WF) composite were improved by adding aluminum hydrogen phosphonate (AHP) and triphenyl phosphate (TPP) flame retardants. PP-WF composites containing 30 wt% of AHP and 30 wt% of TPP achieved UL-94 HB standard with the horizontal burning rate of 20.8 mm/min and of 11.5 mm/min, respectively. Thermal properties of PP-WF composite with and without flame retardant, as well as the interaction between PP-WF and flame retardant were investigated through TGA analysis. The flame retardant mechanism is mainly in condensed phase for AHP and in gas phase for TPP. The residual char after the decomposition of PP-WF/AHP composite was analyzed by FTIR to understand how the formation of char affected on the flame retardancy and thermal stability PP-WF composite.

**Keywords:** Fire retardants, Polypropylene/wood flour composite, Thermal property, Halogen-free flame retardant

## Introduction

Natural resources cannot give enough materials with the rapid development of the world. Therefore, wood plastic composite (WPC), a type of composite that is environmentally friendly and widely used in many fields, is one of the most promising candidates that can replace or reduce the use of materials absolutely based on natural resources. Generally, polypropylene, polyethylene, and polyvinyl chloride are used in WPC [1,2]. Among them, polypropylene (PP) is the most common polymer and has been widely used in thermoplastic industry [3-5].

PP-WF composite has the advantages of plastic such as being easy to shape, having a variety of colors, being fungus and insect resistant in comparison to wood products, and allowing the reuse of the excess wood flour from other industries (sawdust, wood chips). Therefore, it has become a very useful material in many fields, especially in the construction industry (window/door profiles, railing, and floors) as well as outdoor produces (worktop, fencing) [6].

The incorporation of WF into the PP disrupted the homogeneity of the matrix and led to poor interfacial adhesion between the WF and the matrix. The addition of maleated polypropylene to the formulation improved the WF dispersion in the PP matrix [7]. Nonetheless, the high content of WF presents the high flammability of PP-WF composite. This is the biggest restriction for further expansion of the composite applications. One of the effective ways to improve the fire retardancy is to add flame retardant (FR) additives to the composite. Originally, the well-known flame retardant additives were halogen FRs. In spite of their highly effective flame retardancy, they contain

halogen compound that generates large amounts of smoke and toxic gases. To protect and limit the effects on human health as well as to follow legal requirements, the main flame retardants currently used for PP-WF composite are mostly non-halogenated compounds (such as metal hydroxides, nitrogen, and/or phosphorus compounds as well as intumescent flame retardant (IFR) [8-11]. IFR, composed of three components: acid source (most commonly ammonium polyphosphate), a carbonization agent (pentaerythritol), and a blowing agent (melamine), is one of the most widely used systems because of its effective flame retardancy and less smoke during combustion. However, IFRs are sensitive with moisture and consequently reduce their applications [12]. Another widely used flame retardant is metallic hydroxides, which act via the mechanisms of dilution and cooling, produce less toxic gases and smoke than halogenated FRs. Nevertheless, metal hydroxides also have a disadvantage when using in large amounts to provide fire retardancy by negatively affecting the processing and mechanical properties of WPC [8]. Other additives are phosphorus-containing compounds, which are not only environment - friendly but also highly effective against fire, can be referred to as aluminum polyphosphate - APP, diammonium phosphate - DAP, or melamine polyphosphate - MPP.

However, except for the flame retardant studies of APP [13-15] and some studies of DAP and MPP used as flame retardant additives for WPC [15-17], no studies have yet been conducted on flame retardancy and thermal properties as well as the flame retardant mechanism of other single-component phosphorus compounds such as triphenyl phosphate (TPP) or aluminum hydrogen phosphonate (AHP) although their high flame retardancy was announced (when used for polyethylene terephthalate) [18]. Therefore, the main purpose of this study was to investigate the flame

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retardancy, thermal properties, and flame retardant mechanism of polypropylene/wood flour composite using TPP and AHP flame retardants.

## Experimental

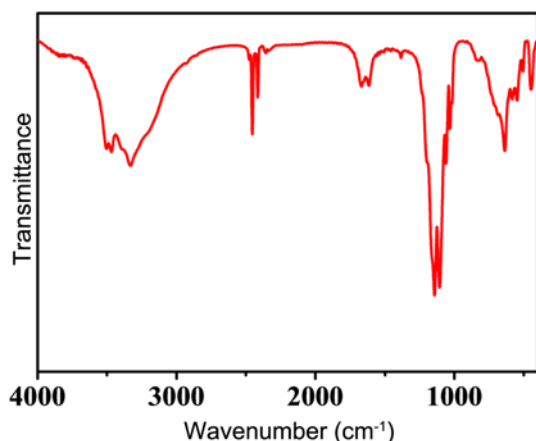
### Materials

Polypropylene (PP 1102K,  $M_w=250.000$ ,  $M_n=67.000$ , melt flow index of 4.0 g/10 min) was supplied by ThaiLan Polimaxx. Wood flour (E 35) with a mesh size ranges from 50 to 80 was purchased from La Sole Est Srl Company. Triphenyl phosphate (TPP) was purchased from Aldrich. Aluminum trinitrate nonahydrate was purchased from Xilong Chemical Co., Ltd. Phosphorus acid was supplied by Shang Hai Jinshan Tingxin Chemical Reagent Company.

Aluminum hydrogen phosphonate (AHP) was synthesized in the laboratory by the reaction between aluminum trinitrate nonahydrate and phosphorus acid at 90 °C for 24 hours. The precipitation was filtered off and washed with distilled water three times. A white solid product was obtained upon drying (70 % yield) and analyzed by FTIR (Figure 1). The spectrum had the main absorption peaks of AHP due to  $PO_3^{2-}$  and P-H at 1142-1105  $cm^{-1}$  and 2449  $cm^{-1}$ , respectively.

### Preparation of Composites

Wood flour was washed with warm water to remove impurities and then dried in an oven at 70 °C to constant weight. To limit moisture absorption, polypropylene, wood flour, TPP, and AHP were dried at 60 °C for 2 hours. PP-WF composite samples were prepared according to the composition in Table 1. The mixtures were mixed with high intensity mixer for 3 minutes. The Haake PolyDrive twin screw extruder was used to mix the composite mixtures, the mixing temperature was 170 °C, the screw speed was 50 rpm, and the mixing time was 5 minutes. After that, the PP-WF composite samples were molded and placed into a hot press machine at 170 °C for 7 minutes and the forming



**Figure 1.** FTIR spectrum of AHP.

**Table 1.** Main formulation for PP-WF and PP-WF/FR composites (by wt%)

Samples	PP-WF	AHP	TPP
PP-WF	100	0	0
PP-WF/AHP <sub>30</sub>	70	30	0
PP-WF/TPP <sub>30</sub>	70	0	30

PP-WF: 50-50 (wt-wt).

bars were obtained and used for further analyses.

### Characteristics of Composites

#### Thermogravimetric Analysis

The thermal properties of the composite PP-WF with and without FR additive were investigated using the TGA 2050 thermogravimetric analyzer, TA Instruments-USA. The samples were performed under air atmosphere in the temperature range of 30-700 °C at a heating rate of 20 °C/min.

#### Fourier Transform Infrared Spectroscopy

The samples were mixed with KBr powder, pressed into pellets, and investigated by a Thermo Scientific Nicolet 380 FT-IR Spectrometer - USA. All the spectra are the average of 32 scans taken in the wavenumber range of 4000-400  $cm^{-1}$ .

#### Scanning Electron Microscope

The scanning electron microscope (SEM) images of the outer surface morphology of residual char were taken by using a FE-SEM (FE SEM S4800 Hitachi, Japan) at an acceleration voltage of 15 kV. The samples were sputter-coated with a conductive layer of platinum prior to analysis.

#### Flame Retardancy Test

The flame retardant performance was determined by Limiting oxygen index (LOI) and UL-94. The LOI (Qualitest - USA) was determined according to ASTM D2863 for five samples with dimensions of 130×10×3 mm<sup>3</sup>. Five specimens for each composite sample with test bars of 127×12.7×3 mm<sup>3</sup> were tested for fire resistance by vertical burning (UL-94 V) and the horizontal burning (UL-94 HB).

UL-94 HB was performed according to D635-98 standard procedure: the sample is held horizontally at one side and at the other side of the sample is applied to burner flame for 30s at 45-degree angles. The ranking is determined by observing the speed of flame propagation and is classified with HB rating if:

- The samples may not have a horizontal burning rate exceeding 40 mm/min over a 75 mm span for samples having a thickness of 3.0 mm, or
- The samples stop to burn before the flame reaches the 100 mm mark.

UL-94 V according to ASTM D 3801 procedures: a burner flame is applied to the sample for ten seconds and then removed until flaming stops at which time the flame is reapplied for another ten seconds and then removed. Vertical ratings are classified:

- UL-94 V-0: Samples must not burn with flaming combustion for more than 10 seconds after either test flame application and the total flaming combustion time must not exceed 50 seconds for each set of 5 samples. No flaming drips are allowed.
- UL-94 V-1: Samples must not burn with flaming combustion for more than 30 seconds after either test flame application and the total flaming combustion time must not exceed 250 seconds for each set of 5 samples. No flaming drips are allowed.
- UL-94 V-2: Samples must not burn with flaming combustion for more than 30 seconds after either test flame application and the total flaming combustion time must not exceed 250 seconds for each set of 5 samples. Flaming drips are allowed.

#### Mechanical Testing

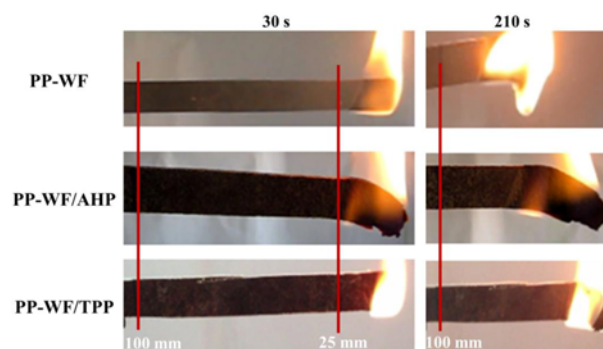
A Universal Testing Machine AG-X plus Shimadzu was used to test the flexural modulus and flexural strength (ASTM D790-00). An unnotched Charpy impact strength test (ISO 179-1/1eU) was carried out on a Zwick HIT25P Shimadzu using a Ceast Resil Impact tester with hammer energy of 5 J for each sample.

## Results and Discussion

### Flame Retardancy

The flame retardancy of PP-WF composites was investigated through the horizontal and vertical UL-94 tests (UL-94 HB and UL-94 V). The samples were initially tested for UL-94 HB. If they achieved UL-94 HB standard, they would be tested for UL-94 V. The test results are summarized in Table 2 and Figure 2.

A major drawback of PP-WF composite is a highly flammable material and then the flame retardant additive needs to be added in order to delay or even extinguish the burning process. The PP-WF composite without flame retardant achieved UL-94 HB with the horizontal burning rate of 28.7 mm/min and it completely burned to the sample holder clamp with low LOI value of 18 %. The presence of AHP and of TPP improved the flame retardancy of PP-WF composite. They satisfied UL-94 HB standard: their burning rates were 20.8 mm/min with LOI value of 19 % for 30 wt% AHP, and 11.5 mm/min with LOI value of 21 % for 30 wt% TPP loading. The UL-94 test results showed that the fire



**Figure 2.** Images of UL-94 HB of PP-WF, PP-WF/AHP, and PP-WF/TPP.

resistance of the PP-WF composite has improved with the presence of flame retardant additives. However, the improvement of fire resistance of two PP-WF composites with two flame retardant additives was different (shown by the difference in fire rates and LOI values), the cause may be due to the differences in the flame retardant mechanism of two flame retardant additives.

### Thermal Behaviors

The thermal properties of PP-WF composite with and without flame retardant additive were investigated by thermogravimetric analysis (TGA) in the air environment. The TGA curves are shown in Figure 3 and the results are summarized in Table 3 which includes the values  $T_{10}$ ,  $T_{50}$ , and  $T_{80}$  (corresponding to the temperature at which 10, 50, and 80 % mass loss occurred), and the charred residue at 500-600 °C.

Based on Figure 3, it was found that the decomposition of PP-WF composite without flame retardant occurred in two steps: the mainly first step (297-420 °C) with the mass loss 84 % corresponded to the thermal degradation of PP, hemicellulose, and cellulose components of wood flour; the second step (485-510 °C) was the decomposition of the lignin [17,19].

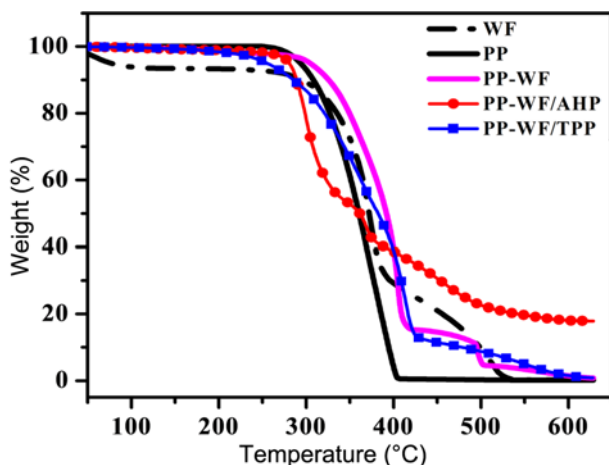
From the TGA curve of PP-WF composite with the presence of AHP flame retardant, the decomposition step slowly began at 277 °C with lower mass loss (64 %) in the temperature range of 277-420 °C.  $T_{10}$  and  $T_{50}$  values decreased (289.5 and 356.2 °C) compared to those of PP-WF

**Table 2.** UL-94 results and LOI values for PP-WF and for the mixtures of FR with PP-WF

Sample	PP-WF/FR (wt/wt)	LOI (%)	UL-94 Rating		
			UL-94 HB		UL-94 V
			Result	Burning rate (mm/min)	
PP-WF	100.0/0.0	18	HB	28.7	Not achieved
PP-WF/AHP <sub>30</sub>	70.0/30.0	19	HB	20.8	Not achieved
PP-WF/TPP <sub>30</sub>	70.0/30.0	21	HB	11.5	Not achieved

**Table 3.** Thermal stability parameters

Sample	PP-WF/FR (wt/wt)	T <sub>10</sub> (°C)	T <sub>50</sub> (°C)	T <sub>80</sub> (°C)	Residue at 500 °C	Residue at 600 °C
PP-WF	100.0/0.0	325.3	392.3	409.0	5.7	1.5
PP-WF/AHP <sub>30</sub>	70.0/30.0	289.5	356.2	552.7	23.3	18.1
PP-WF/TPP <sub>30</sub>	70.0/30.0	287.5	382.5	417.2	8.4	1.5

**Figure 3.** TGA thermograms of PP, WF, PP-WF, PP-WF/AHP, and PP-WF/TPP in air.

composite sample without flame retardant additives (325.3 and 392.3 °C), respectively. However, T<sub>80</sub> value of PP-WF composite with AHP (552.7 °C) was significantly higher than that of PP-WF composite without flame retardant (409.0 °C). Not only that, the presence of AHP flame retardant also extremely increased the residue of char at 500-600 °C (23.3-18.1 % for PP-WF/AHP<sub>30</sub> and 5.7-1.5 % for PP-WF).

For PP-WF composite with TPP flame retardant additive, one-step of decomposition at 239-430 °C and almost no residual char was observed at 650 °C. Similar to the case for the composite with AHP, T<sub>10</sub> (287.5 °C) and T<sub>50</sub> values (382.5 °C) of PP-WF/TPP<sub>30</sub> were reduced compared with those of PP-WF. Unlike the case of AHP, however, which significantly improved the residue char at 500-600 °C, the presence of TPP flame retardant additive only slightly improved the residue char at 500 °C (8.4 % compared with 5.7 % in PP-WF composite without flame retardant additive), while the residue char of PP-WF composite with TPP flame retardant was equivalent to that of PP-WF composite sample without flame retardant additive at 650 °C.

Similar with the UL-94 test results above, the TGA results showed that there was a difference in the flame retardant mechanism of AHP and TPP. In the case of PP-WF composite containing AHP, the first step of the thermal degradation has lower initial decomposition temperature compared to that of PP-WF. This may due to the simple

volatilization of AHP and/or more complicated decomposition of the products resulted from the interactive reaction between decomposed AHP and PP-WF. This step probably makes a contribution to the formation of significantly higher residual char that protects the composite from heat, oxygen, and flammable gases. From these results, it can be concluded that the flame retardancy of AHP mainly acted in the condensed phase mechanism. With the TPP flame retardant, it also improved the fire retardant property of PP-WF and UL-94 HB was achieved with 30 wt% loading of TPP. However, the presence of TPP did not improve the residual char after the decomposition of PP-WF composite (the residual char at 650 °C of PP-WF/TPP composite was equivalent to that of PP-WF composite without flame retardant additive). From these results it can be concluded that the fire retardant mechanism of TPP mainly is the gas phase (the decomposition of the TPP additive generates free radicals such as PO·, PO<sub>2</sub>·, HOPO·, etc., which capture free radicals maintaining combustion such as H· and OH·; thereby protecting the composite from the fire) [20,21].

To investigate whether the interaction between PP-WF composite and the flame retardant additives or not; the calculated and experimental TGA curves of PP-WF composite containing flame retardant were studied. The calculated TGA value is determined according to the following formula [22,23]:

$$C = \frac{F \times \text{wt}\%FRs + W \times (100 - \text{wt}\%FRs)}{100}$$

where C is calculated TGA value, F is TGA value of flame retardant additive, W is TGA value of PP-WF composite.

Figure 4 shows that with both flame retardants used, there were significant differences between the two calculated and experimental TGA curves of the PP-WF composite containing the additive. If there is no interaction, the calculated and experimental TGA should be the same. The onset temperature of PP-WF/TPP composite of the experimental TGA curve was higher than that of PP-WF/TPP composite of the calculated TGA. The main step decomposition (from 230 to 432 °C) also shifted to higher temperature than that of the calculated curve (from 206 to 416 °C).

AHP leaved significant residual char of approximately 73 % at 600 °C. With the addition of 30 wt% AHP, the main decomposition step of PP-WF/AHP experimental curve is in the temperature range of 270-600 °C, while that of PP-WF/AHP calculated curve is 147-600 °C. These results show that

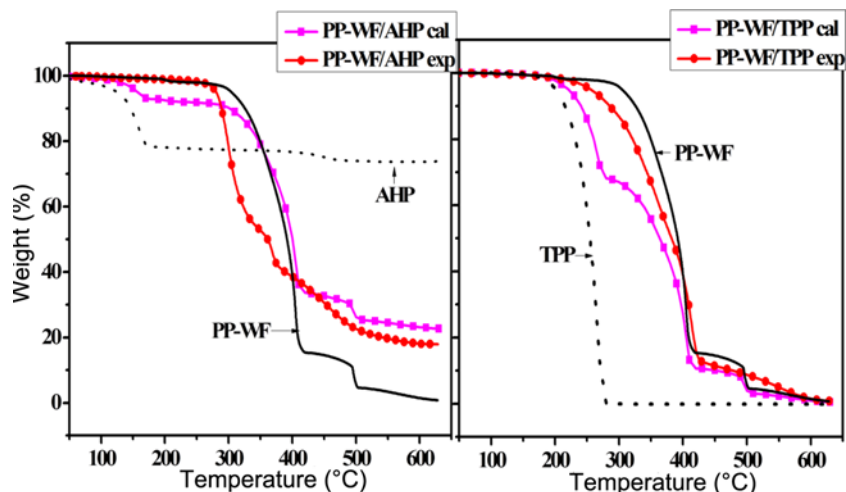


Figure 4. Experimental and calculated TGA thermograms for PP-WF/AHP and PP-WF/TPP.

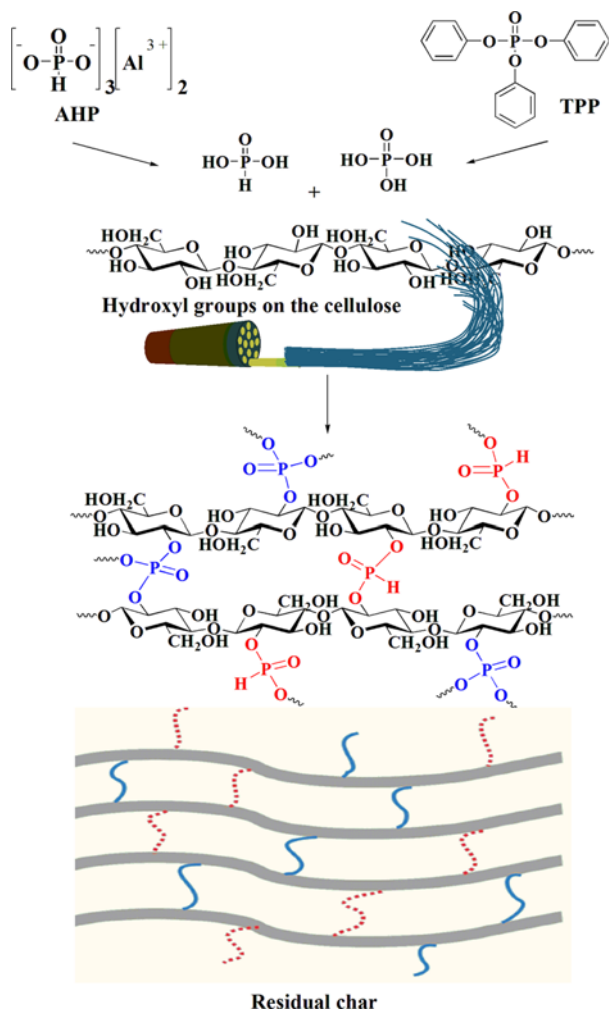


Figure 5. Possible scheme for the main decomposition of AHP, TPP, and PP-WF and interaction between them (the reaction between phosphoric acid and primary hydroxyl groups during thermal decomposition is cited from literature [16]).

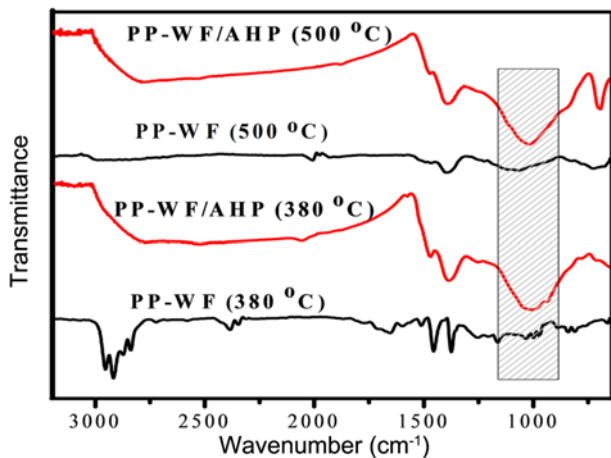
there is an interaction between the decomposition products of FR and PP-WF composite and/or the decomposition products of PP-WF that significantly contributes to the formation of more thermally stable char residue protecting the material from further degradation. In the vapor phase, TPP releases active radicals that act as scavengers for H<sup>•</sup> and OH<sup>•</sup> during combustion. Decomposition of AHP or TPP also produces acid phosphoric/phosphorus and can react with the OH groups of the wood flour to form phosphate esters [16,24]. Phosphate esters play a role as the barrier preventing PP-WF composite contacts with the heat source and flammable gases, thereby contributing to the flame resistance process for PP-WF composite (Figure 5).

**Characterization of the Residual Char**

The AHP flame retardant additive improved the flame retardancy of the PP-WF through the condensed phase and significantly increased amount of residual char (Table 3, Figure 3). To further investigate the composition of the residual char of PP-WF composite and PP-WF composite containing AHP after thermal decomposition, the solid char layers at 380 and 500 °C were conducted by FTIR analysis. The results are shown in Figure 6. In comparison with FTIR of the residual char of PP-WF composite, the FTIR analysis of remaining char layer of PP-WF/AHP composite has appeared the new peaks at wavenumbers 1180-1070 cm<sup>-1</sup> (vibrations of P=O, P-O-C, and/or P-O-P). These peaks are attributed to the formation of polyphosphates during the thermal decomposition of AHP and/or the phosphorus-containing products obtained from the interaction between of PP-WF and AHP. The above results implied that there was the formation of phosphorus-containing moieties and/or their derivatives acting as the residual char protecting polymer from fire.

Figure 7 shows that there was a formation of a heat-resistant residual char during the thermal degradation of PP-



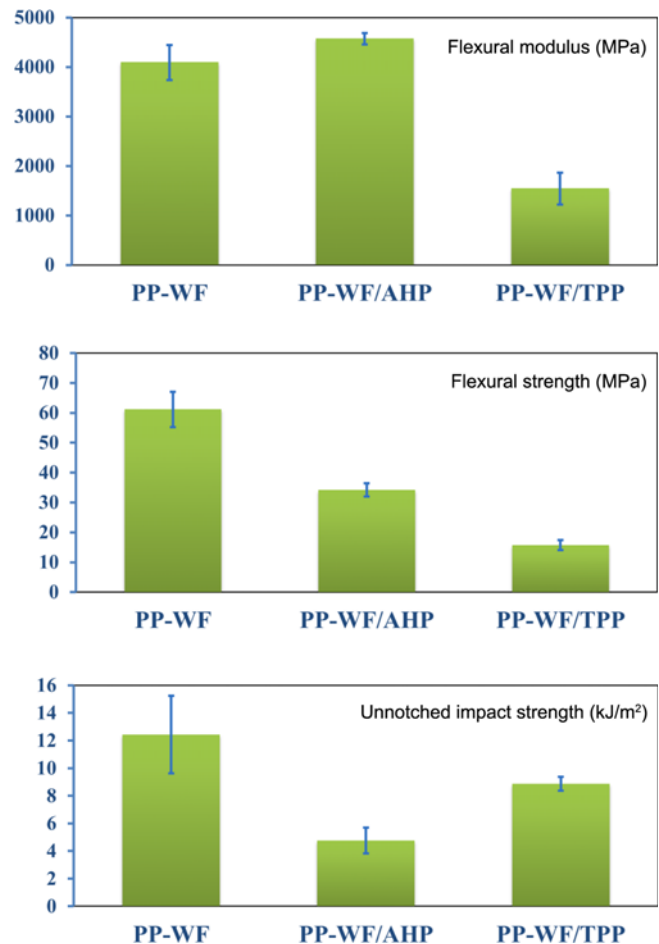


**Figure 6.** FTIR spectra of charred residues of PP-WF and of PP-WF/AHP obtained after combusting in the furnace at 380 and 500 °C.

WF/AHP. This result is in agreement with the TGA results discussed earlier. However, the brittle, less compact and condensed char of PP-WF/AHP is not good enough to act as an effective protective barrier that could prevent the transfer of mass and heat in the solid phase during combustion. Furthermore, PP-WF is a highly flammable material and therefore, it is difficult to find a sufficient halogen-free flame retardant for PP-WF composite.

### Mechanical Properties

High loading levels of flame retardant have effect on the mechanical properties of composite material, the interaction of the flame retardant and polymer matrix has less compatibility, and then the composite could lead to breakage. Figure 8 represents that loading of AHP or TPP caused the decreasing trend of flexural and unnotched impact strength. As we known, TPP not only acts as a flame retardant but also serves as a plasticizer. Thus, the melt mixing process of PP-WF/TPP is more successful and this leads to a higher impact strength than that of PP-WF/AHP.

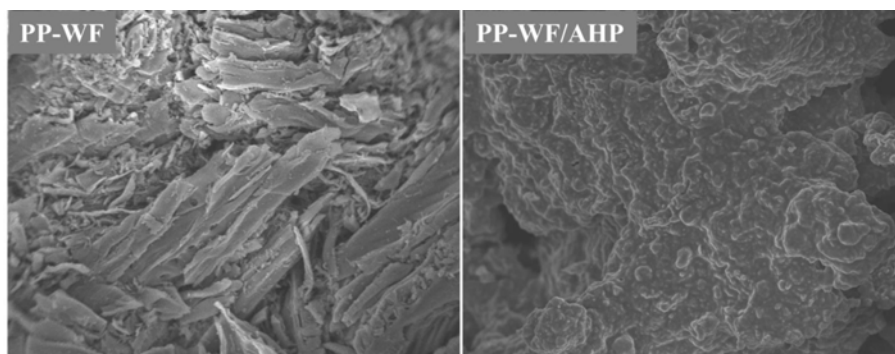


**Figure 8.** Mechanical properties of PP-WF, PP-WF/AHP, and PP-WF/TPP.

However, the high loading of TPP or AHP resulted in a decrease of the mechanical properties.

### Conclusion

The flame retardancy and thermal stability of two flame



**Figure 7.** SEM images of charred residues of PP-WF and of PP-WF/AHP obtained after combusting in the furnace at 500 °C.

retardant additives AHP and TPP applied for PP-WF composite have been demonstrated through the UL-94, LOI, SEM, and TGA test. PP-WF composites with a loading of 30 wt% AHP and of 30 wt% TPP achieved UL-94 HB standard. The main flame retardant actions of AHP and of TPP were studied. With TPP, the flame retardant mechanism worked mainly via gas phase with the formation of free radicals such as  $\text{PO}^\cdot$ ,  $\text{PO}_2^\cdot$ , and  $\text{HOPO}^\cdot$  that capture the  $\text{OH}^\cdot$  and  $\text{H}^\cdot$  sustaining the fire. Meanwhile, the main flame retardant mechanism of AHP acted in the condensed phase mechanism via the formation of a char layer. There was an interaction between flame retardant additive with PP-WF composite, which occurred through the reaction between phosphoric/phosphorous acid (produced during the decomposition of AHP and TPP) with the OH groups in the cellulose of wood flour. This interaction led to the formation of phosphorus-containing and/or their derivatives existing in the residual char. However, PP-WF is a highly flammable material, and the less compact and less condensed char of PP-WF/AHP is not good enough to act as an effective protective barrier that could prevent the transfer of mass and heat in the solid phase during combustion. The high loading (30 wt%) of TPP or AHP also resulted in a decrease of the flexural and unnotched impact strength.

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