Thermal Stability and Rheological Properties of Polyethylene (PE)/Polyvinylchloride (PVC)/Wood Composites

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Abstract: This paper investigated the thermorheological properties, thermal properties and flame retardant properties of wood-plastic composites (WPCs). With the addition of wood flour (WF), the rheological behavior became complexity. The critical frequency of shear-thinning phenomenon of the melt viscosity was shifted toward lower value. The temperature dependence of elastic modulus, loss modulus became more serious with the addition of WF. The Cole-Cole plot indicated the existence of complex multi-phase structure in the WPC melt. The CONE calorimetry results showed that ammonium polyphosphate (APP) had good flame retardancy through promoting the formation of the intumescent carbon layer. The present study will supply good insight into the optimization of WPC formulation.

Keywords: WPC, molecular characteristic, thermorheological complexity, intumescent flame retardant, char residue.

INTRODUCTION

Recent years have witnessed the phenomenon that the environmental protection has increasingly caused worldwide concern. Wood-plastic composites (WPCs), the composites made of wood flour/fibre (WF) and thermoplastics [1-3], are by far regarded as an environmentally-friendly approach of using waste wood and recycled plastic materials, including polyethylene (PE), polyvinylchloride (PVC), polypropylene (PP), polylactide (PLA), and so forth. As compared to the solid wood, WPCs need substantially low maintenance cost. Due to the combination of characteristics from both plastics and wood, WPCs have gained wide application in our daily life ever since 1990s [4].

WPCs also have their disadvantages, such as, easy absorption of moisture, flammability, thermal expansion, creep (time-dependent deformation), as well as the difficult application of paint, etc. Those shortcomings limit the application of WPC to an extent. For facilitating the optimal processing conditions as well as improving the performance of the end products, additives (e.g., coupling agents, lubricants, mineral fillers, heat or UV stabilizers, colorants, etc.) are usually utilized, for instance, coupling agent is an efficient way to enhance the compatibility between hydrophilic wood and hydrophobic plastic [5, 6].

However, addition of the chemical additives will inevitably lead to the complication of the WPC formulae. As a result, making one property stand out is always coupled with reducing another property, since the physical and mechanical properties of WPCs depend heavily on the interactions between wood and thermoplastic materials [7-10].

Ayrilmis *et al.* [11] found that the modulus of rupture of the flat-pressed WPC panels containing fire retardants decreased with increasing WF content from 40 to 50 wt%, even though the modulus of elasticity increased with the increase in WF content. Zhang *et al.* [12] investigated the mechanical properties, flame retardancy and thermal degradation of PP/WF composites. Their findings showed that silica had a flame retardant synergistic effect with ammonium polyphosphate (APP); while the addition of flame retardants considerably reduced the mechanical properties of the composites. Ndiaye *et al.* [13] studied the PP/WF composites blended with maleated polypropylene (MAPP) and clay, and the inorganic filler (i.e., clay) were found to act not only as a flame retardant, but also as an adhesion promoter in the WPCs. Adhikary *et al.* [14] argued that the performance of WPCs could be optimized by adjusting the processing parameters and material formulations. An appropriate combination of HDPE, WF, lubricant, and MAH-g-PE contents provided the benefits of lower shear viscosity (favorable to processing) while maintaining the mechanical properties and surface smoothness of the extruded WPC profiles.

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As is well known, the materials' processing behaviors substantially affect the end-use properties. However, the investigation on WPCs' rheological properties, which is practically significant to understand the flow characteristics as well as the molecular structure involved in the WPCs, is still inadequate so far. Considering the limited influence exerted on the melt structure, rheological data would supply us good insight into the optimization of the processing variables of WPCs. In this work, thermorheological behavior, flame retardancy and thermal stability of the PE/PVC/WF composites were investigated in an attempt to facilitate the formula design as well as the optimization of processing parameters for WPCs, which will be instructive to the further study on "processing-structure-property" relationship for polymeric materials.

EXPERIMENTAL

Materials

High-density polyethylene (HDPE), model: 5000S, used in this work was manufactured by Maoming Petrochem. Co. Ltd., P.R. China, with a melt flow rate (MFR) of 1.2 g/10min (190°C/2.16kg). Low-density polyethylene (LDPE), model: 2426H, was provided by Lanzhou Petrochem. Co. Ltd., P.R. China, with MFR=1.83 g/10 min (190°C/2.16kg). Polyvinyl chloride (PVC) was obtained from the Chlor-Alkali Chemical Co. Ltd., P.R. China. Chlorinated polyethylene (CPE) was produced by Shengtai Chemical Co. Ltd. Wood flour (WF) was kindly provided by Guofeng Wood-Plastic Composite Co. Ltd. Ammonium Polyphosphate (APP) was manufactured by Tongli Auxillary Co. Ltd. Laroflex MP-45 was supplied by the BASF in Greater China. Maleic anhydride grafted polyethylene (MAH-g-PE) was supplied by Anbang Chemical Co. Ltd., with a grafting rate of 0.5%. Aluminum hydroxide, melamine and stearic acid used in the experiments were all provided by Guangfu Fine Chemical Research Institute. Calcium-Zinc heat stabilizer (model: KF-10) and

antioxidant (model: AT-10) were both manufactured by the Selo Plastics Co. Ltd. The detailed formulae of the samples are listed in Table **1**.

Samples Preparation

All materials were mixed in a high-speed mixer (model: SHR-10A, Zhangjiang Light Industrial Machinery Co. Ltd., P.R. China), then melt blended using a twin-screw extruder (model: SJSH-30, Nanjing Rubber & Plastics Machinery Co. Ltd., P.R. China). Testing samples (discs) with a thickness of 1.0 mm and a diameter of 25.0 mm were prepared by compression molding *via* a platen vulcanizing press (model: QLB-352) manufactured by Shanghai Rubber Machinery Co. Ltd., P.R. China.

Dynamic Rheological Characterization

Dynamic rheological properties were performed by a dynamic shear rheometer (DSR) (model: Gemini-HR Nano, Malvern Inc., U.K.) using parallel-plate geometry mode at three temperatures of 190, 210 and 230 °C. The frequency sweep was run from 0.01 to 10 Hz at a constant strain of 1%.

Thermogravimetric Analysis (TGA)

The thermogravimetric analysis (TGA) was carried out on a Perkin-Elmer Pyris-1 thermal analyzer at a heating rate of 20 °C/min under pure nitrogen (N_2) atmosphere. The measuring temperature ranges from the ambient temperature to 800 °C. Each sample weights about 5~7 mg.

Limited Oxygen Index (LOI)

Limited oxygen index (LOI) was obtained using the oxygen index tester, manufactured by Shangyuan Analytical Instruments Co. Ltd., China, at the ambient temperature.

Table 1:   Formulae of the WPC Samples Used in this Study (unit: phr).

Samples	HDPE	LDPE	PVC	CPE	Wood Flour	APP	Laroflex MP-45	MAH-g-PE	ATH	Melamine	Heat Stabilizer
M20L0	250	0	50	25	220	100	0	20	50	0	10
M20L20	250	0	65	10	220	125	20	20	30	0	17
M25L0	250	0	150	50	250	250	0	25	0	0	25
M25L40	250	0	150	50	250	250	40	25	0	0	25
M30L0	200	50	90	20	220	125	0	30	50	25	13
M50L0	200	50	90	20	220	125	0	50	50	25	13

CONE Test

The CONE calorimeter test was performed at the *State Key Laboratory of Fire Sciences* (SKLFS) in the *University of Science and Technology of China* (USTC). Samples with the dimensions of 100 × 100 × 3 mm^3 were exposed to a Stanton Redcroft cone calorimeter with a heat flux of 50 kW/m^2.

RESULTS AND DISCUSSION

Thermorheological Complexity

Dynamic rheological measurements have been proved to disclose the molecular structural details without causing destroy to the inner structure of polymer melt. The zero-shear rate viscosity (η_0), a rheological parameter characteristic of the molecular attributes, can be calculated by non-linearly fitting the *Carreau-A* Model [15] as follows:

$$\eta^* = \eta_0 \left[1 + \left(\lambda \gamma \right)^2 \right]^{(n-1)/2} \tag{1}$$

where η_0 (unit: Pa·s) is the zero-shear viscosity, λ (unit: s) is the characteristic relaxation time, γ (unit: Hz) is the shear rate and n is the power-law exponent describing the slope of the viscosity function with respect to shear rate (frequency) within the shear-thinning or power-law region [16]. The values of flow activation energy (E_a) can be obtained using the *Arrhenius* Equation [17]:

$$k = A \exp \left(-\frac{E_a}{RT} \right) \tag{2}$$

where k is the rate constant, with A the pre-exponential factor, E_a (unit: J/mol) the flow activation energy, R the molar gas constant (8.314 J·mol^{-1}·K^{-1}), and T (unit: K) the temperature.

Table **2** lists the parameters calculated by non-linear minimization procedures fitting the dynamic experimental data at each measuring temperature in accordance with *Eq.* 1. At a given temperature, η_0 for WPC (#M20L20) is larger than that of neat HDPE, suggesting that the motion ability of polymer chains of WPC (#M20L20) was harder than neat HDPE at the corresponding temperature. According to *Eq.* 2, the flow activation energy (E_a) can be obtained by linear fitting the experimental data of *ln k* versus *1/T* [17-19].

Figure **1** shows the frequency dependency of the complex viscosity (η^*) for neat HDPE and WPC (#M20L20). It can be seen that viscosity of neat HDPE displayed an obvious platform within the low frequency region. The critical shear point (γ_c) of neat HDPE can be readily estimated as, 0.89, 0.56 and 0.18 Hz at 190, 210 and 230 °C, respectively. Actually, no "leveling phenomenon" within the scope of #M20L20 was observed. Therefore, the critical shear point of WPC can possibly be shifted to an even lower value as compared to neat HDPE. It is believed that it is the addition of WF that causes the change in melt viscoelastic properties [20, 21], which further leads to the thermorheologically complex behavior characterized by the failure of time-temperature superposition (TTS).

The dependence of modulus (G' and G") on the frequency at various temperatures for neat HDPE and WPC (#M20L20) is presented in Figures **2** and **3**. It was shown that both G' and G" decreases with increasing temperature and decreasing frequency. Temperature dependence of the elastic modulus (G') of WPC (#M20L20) is obviously higher than that of neat HDPE, almost larger by one order of magnitude, which suggests that temperature exerts greater influence on the melt internal intermolecular bonding strength of WPC than that of the matrix resin [22]. The elasticity of WPC is increased as compared with neat HDPE in the

Table 2: Coefficients Obtained by Non-Linearly Fitting the *Carreau*-A Model

Materials	Temperature (°C)	η_0 (Pa·s)	λ (s)	n (-)	E_a (kJ/mol)
Neat HDPE	190	2520.1	13.8	0.805	
	210	1904.3	11.7	0.815	24.2
	230	1537.5	10.2	0.814	
WPC (#M20L20)	190	5912.3	42.8	0.708	
	210	3231.4	47.2	0.715	39.1
	230	2682.2	71.4	0.701	
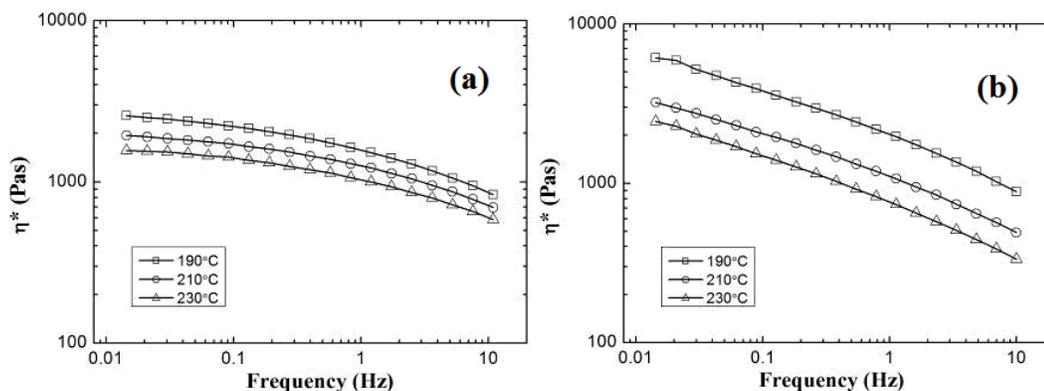
Figure 1: Frequency dependency of complex viscosity: (**a**) neat HDPE and (**b**) WPC (#M20L20).
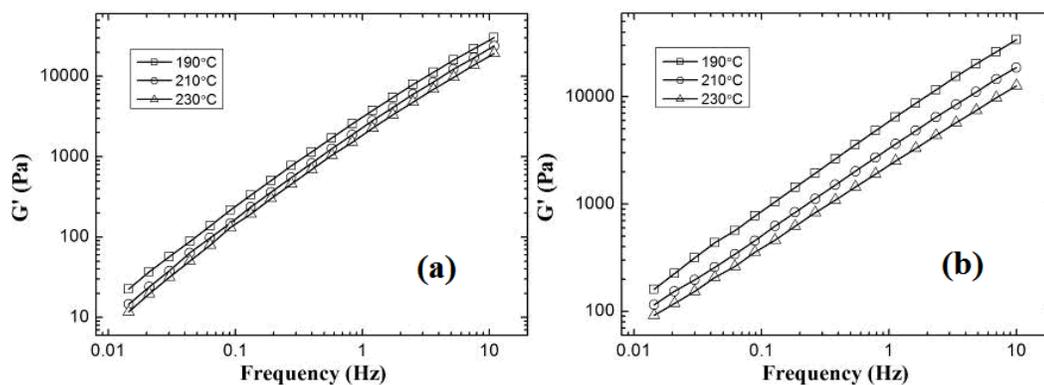
Figure 2: Variation of G' as a function of frequency: (**a**) neat HDPE and (**b**) WPC (#M20L20).
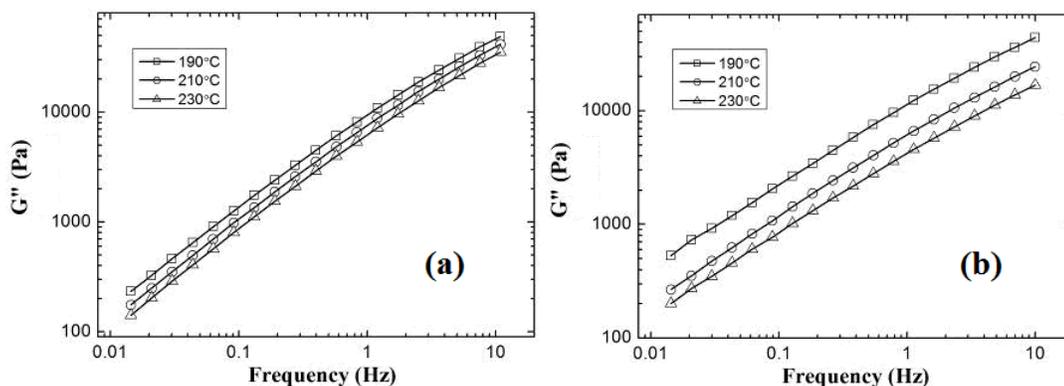
Figure 3: Variation of G" as a function of frequency: (**a**) neat HDPE and (**b**) WPC (#M20L20).

range of shear rates measured, it can be seen that the elasticity is remarkably enhanced with WF content, since modulus reflects the elasticity of the materials. In addition, from Figures **2** and **3**, it was also obvious that the curves of WPC appear to be more dispersive, which reflects that G' and G" values of WPC are more sensitive to temperature.

The Cole-Cole plot, consisting of the dynamic viscosity (η') and imaginary viscosity (η''), was a useful way to examine the miscibility of polymer blends and composites. If the curve takes the form of a semi-circular shape, it always suggests good miscibility of the blend. The Cole-Cole plot of neat HDPE displayed the semi-circular shape, while that of WPC did not, indicating the existence of complex multi-phase structure in the WPC melt. The phase separation possibly resulted from the incompatible polymers (such as, PE, PVC), which further leads to the thermorheological complexity and ultimately affected the melt flow characteristics during the processing operations.
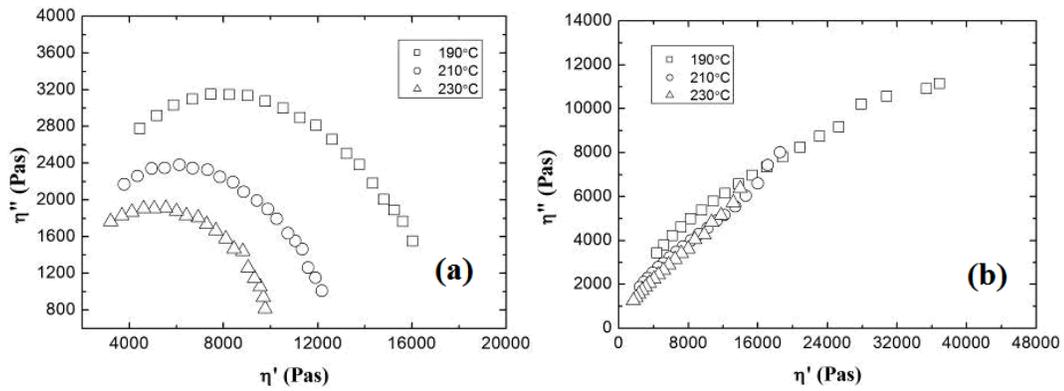
Figure 4: Cole-Cole plot of neat HDPE (**a**) and #M20L20 (**b**).

Thermal Stability

Figures **5** and **6** present thermogravimetric curves (TG and DTG) of the WPC samples, measured in nitrogen (N$_2$) atmosphere [23]. As can be seen in Figure (**5a**), #M20L20 (containing 20 phr Laroflex MP-45) stops losing weight after 500°C, while the weight of #M20L0 decreases continuously. Similarly, in Figure (**5b**), the corresponding weight loss of #M25L40 (containing 40 phr Laroflex MP-45) was smaller than that of M25L0 at the corresponding temperature. There was no obvious weight loss after 500°C for M25L40; whereas #M25L40 reaches its lowest weight after 590

°C. The reason could be due to the addition of Laroflex MP-45, which protects the carbon layer to stop degradation.

In Figure **6**, the first degradation peak can be attributed to hemi-cellulose (of WF) and inorganic filler, while the second one can be attributed to cellulose and lignin (of WF) and the matrix resin, according to previous work [24, 25]. We are in favor of one opinion that the free radicals, with the addition of Laroflex MP-45, protected the carbon chain, to inhibit the thermal degradation reaction, increase the char residue (CR) and stabilize the intumescent carbon layer. In Figure
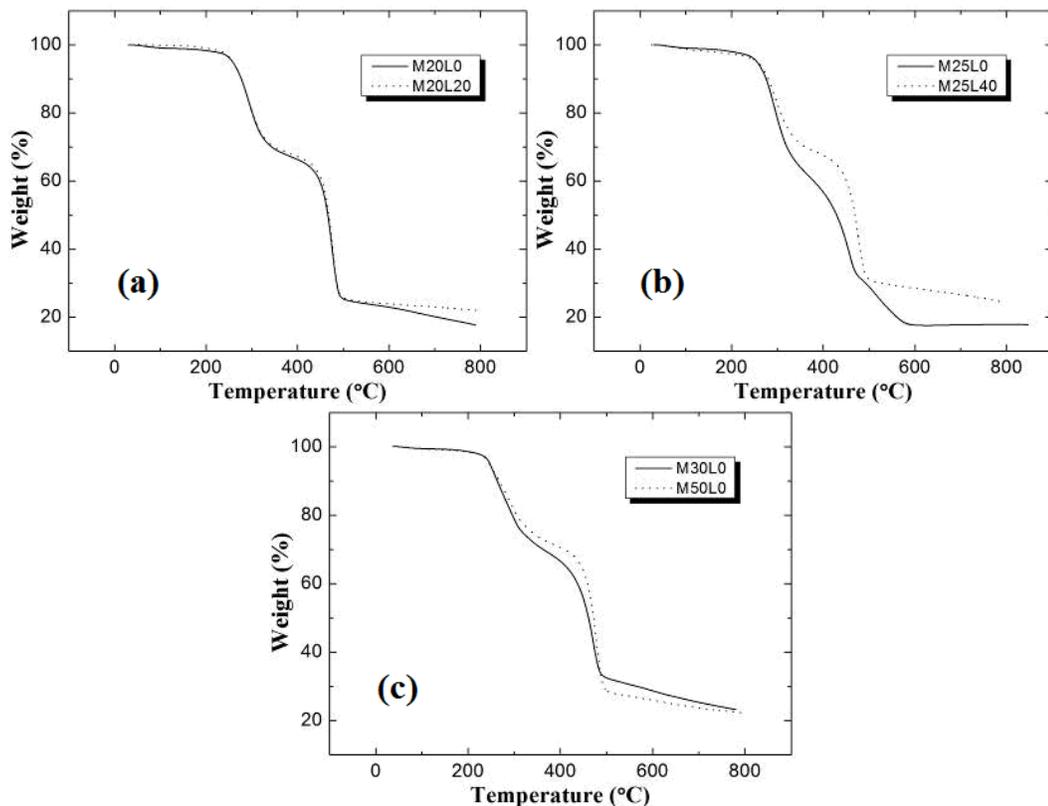
Figure 5: Comparisons of TG curves between M20L0 and M20L20 (**a**); M25L0 and M25L40 (**b**); and M30L0 and M50L0 (**c**).
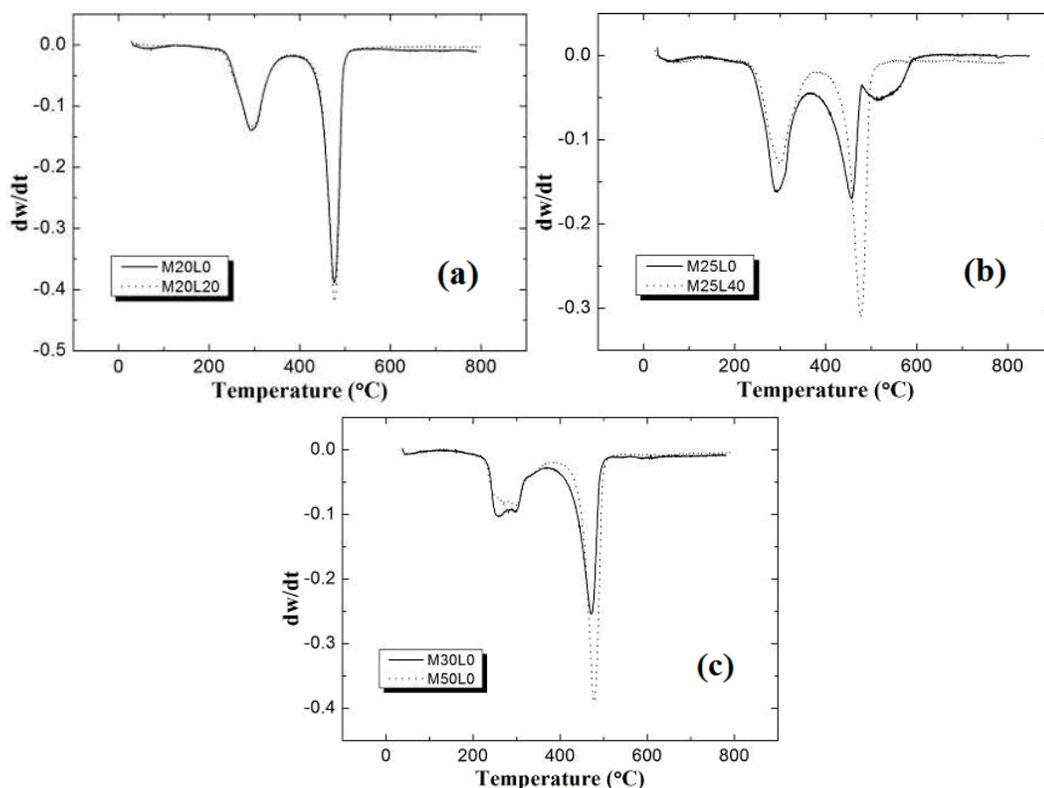
Figure 6: Comparison of DTG curves between M20L0 and M20L20 (**a**); M25L0 and M25L40 (**b**); and M30L0 and M50L0 (**c**).

(**6b**), as compared to #M25L0, the second degradation peak of #M25L40 increases by 20°C. That is to say, the addition of Laroflex MP-45 effectively improved the degradation temperature of the matrix.

In Table **3**, #M50L0 has lower initial degradation temperature (T_{in}) as compared to others, primarily owing to the high MAH-g-PE content. The comparison of CR values showed that M20L0, M25L40 and M30L0 have similar values in both nitrogen (N_2) and air atmospheres; while M20L20 and M50L0 have obviously lower CR values in the air atmosphere, which may be because the carbon layers continue to react with O_2 in the air at elevated temperatures.

Flame Retardant Properties

The evolution of total heat release (THR) and heat release rate (HRR) versus elapsed time for various WPC samples was illustrated in Figure **7**. Initially, HRR gradually increases with increasing temperature. Expansive carbon layer began to form, wood and plastic thermal degradation rate increases. During the course, moisture in the composite was evaporated together with the released gases (e.g., carbon monoxide, ethylene, etc) [26-28]. With the combustion of these gases, the HRR showed the first peak. With the gradual thickening of the expansive carbon layer, the HRR value decreased. Then, the material began to

Table 3:   Thermal Data Obtained by the TG Analysis

Samples	$T_{in.}$ (°C)	R_{max} (g/s)		T_{max} (°C)		Char Residue (%)	
		Peak 1	Peak 2	Peak 1	Peak 2	N_2	Air [*]
M20L0	237	0.140	0.390	297	475	14.2	13.5
M20L20	234	0.135	0.420	293	475	20.2	12.4
M25L0	237	0.162	0.170	295	455	17.6	21.1
M25L40	247	0.128	0.310	296	477	24.5	25.8
M30L0	233	0.104	0.255	258	472	20.5	19.4
M50L0	227	0.086	0.390	270	477	19.6	12.9

[*]Obtained by CONE measurement.
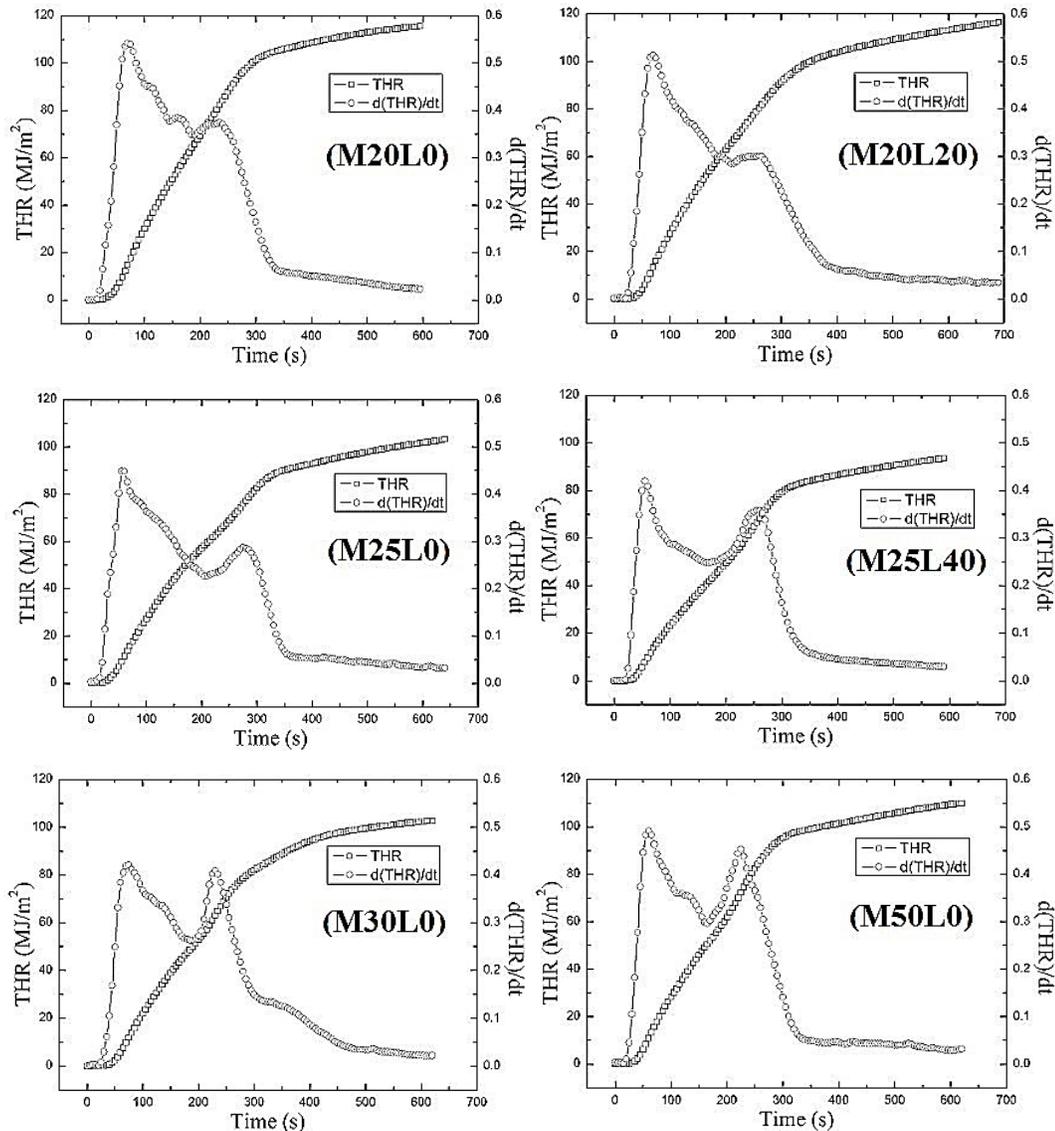
Figure 7: Evolution of THR and derivative THR with elapsed time obtained from the CONE measurements for various WPC samples.

swell and rupture, causing the gases generated inside the composite to be released from the gaps of carbon layer. This further accelerated the thermal degradation rate as well as the release of the combustible gas, and made the heat release rate achieve the second peak. As compared with M25L0 and M25L40, the second peaks of M20L0 and M20L20 are not apparent, because M25L0 and M25L40 containing more intumescent flame retardants formed thicker carbon layers during the combustion process.

Table **4** lists the CONE test results of various WPC samples. Among the samples, M25L0 and M25L40 have relatively lower THR, HRR, effective heat of combustion (EHC) but higher CR values (in air atmosphere), in spite of the similar limited oxygen index (LOI) values. This suggests that samples

#M25L0 and #M25L40 have better flame retardant properties. Table **4** also gives the measured LOI data of the samples. LOI of some samples reached 32 in our research. HDPE and wood flour are easily flammable materials. APP and other flame retardants obviously enhanced flame retardancy of HDPE/wood composites.

CONCLUSIONS

In this work, the thermorheological complexity, thermal stability and flame retardant properties of PE/PVC/WF composites were extensively explored. As compared to neat HDPE resin, the critical shear point of WPC was shifted to an even lower value in the viscosity-frequency plot. The temperature dependence of elastic modulus (G') and loss modulus (G") became more serious with the addition of WF. WPC behaved

Table 4: **CONE Calorimeter Testing Results**

Samples	M20L0	M20L20	M25L0	M25L40	M30L0	M50L0
LOI (%)	29	29	29	32	32	32
Time to ignition (s)	16	22	16	22	23	21
THR (MJ/m^2)	115.5	116.4	103.0	93.4	102.7	109.8
HRR (kW/m^2)	200.9	175.0	166.0	165.3	172.6	184.4
Peak HRR (kW/m^2)	545.7	516.3	471.9	430.5	426.1	495.0
EHC (MJ/kg)	27.1	26.1	23.9	23.3	24.3	26.3
Peak EHC (MJ/kg)	74.1	75.3	71.8	80.0	62.6	70.6
Carbon monoxide (CO) (kg/kg)	0.053	0.053	0.056	0.056	0.048	0.058
Carbon dioxide (CO$_2$) (kg/kg)	1.735	1.650	1.495	1.418	1.558	1.642

thermorheologically complex with multi-phase structure as indicated by the Cole-Cole plot. Laroflex MP-45 inhibited the thermal degradation and promoted the formation of char residue (CR). The CONE tests showed that APP had good flame retardancy through promoting the formation of the intumescent carbon layer. The current study will be practically significant to the optimization of WPC formulation.

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