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Research Article

The Effect of Ultrafine Magnesium Hydroxide on the Tensile Properties and Flame Retardancy of Wood Plastic Composites

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The effect of ultrafine magnesium hydroxide (UMH) and ordinary magnesium hydroxide (OMH) on the tensile properties and flame retardancy of wood plastic composites (WPC) were investigated by tensile test, oxygen index tester, cone calorimeter test, and thermogravimetric analysis. The results showed that ultrafine magnesium hydroxide possesses strengthening and toughening effect of WPC. Scanning electron micrograph (SEM) of fracture section of samples provided the positive evidence that the tensile properties of UMH/WPC are superior to that of WPC and OMH/WPC. The limited oxygen index (LOI) and cone calorimeter test illustrated that ultrafine magnesium hydroxide has stronger flame retardancy and smoke suppression effect of WPC compared to that of ordinary magnesium hydroxide. The results of thermogravimetric analysis implied that ultrafine magnesium hydroxide can improve the char structure which plays an important role in reducing the degradation speed of the inner matrix during combustion process and increases the char residue at high temperature.

1. Introduction

Wood plastic composites (WPC) are widely used in many fields such as construction material, furniture, and automotive parts, owing to their good performance and because they are environment friendly [1–3]. What is more, it is an effective way to alleviate the shortage of wood and the white garbage-waste plastics. However wood plastic composites (WPC) are mainly composed of wood and plastics, which are easily combustible materials. It is essential to improve the flame retardance of WPC [4–6]. The traditional method is to introduce halogen-contained flame retardant to matrix. However, WPC treated with halogen-contained flame retardant would produce harmful hydrogen halide and dense smoke. Intumescent flame retardant (IFR) and inorganic flame retardant are effective halogen-free flame retardants [7–10]. Complex flame retardant composed of montmorillonite, magnesium hydroxide, and ammonium polyphosphate was incorporated into polypropylene to improve its flame retardancy [11]. Magnesium hydroxide is an effective inorganic flame retardant and possesses the characteristics of flame retarding, smoke suppression, promoting charring, and so forth [12–14]. However the addition amount of magnesium hydroxide is over 50% (based on matrix) when the flame retardancy of materials satisfies the government's request, which will result in incompatibility and deteriorate the processing and mechanical properties of WPC. Reducing its particle size and modifying its surface with couple agents are the effective ways to improve compatibility [15]. Layered double hydroxide nanoparticles were used to improve the thermal stability and flame retardancy [16]; nanofibril and nanosphere of polyaniline were used to prepare flame retarding and electrical conductive epoxy resin [17]; magnetic iron-core carbon shell nanoparticles were prepared to reinforce the conductivity and reduce the flammability of epoxy resin [18]; the thermal stability and flame retardancy of HDPE/EVA/EG composites can be improved with decreasing particle size of expandable graphite [19]. The limited oxygen index (LOI) of polymer treated with magnesium hydroxide can increase substantially when the size of magnesium hydroxide is less than 1 micrometer; what is more, the effect of strengthening and toughening will appear when the superfine magnesium hydroxide was introduced to the polymer [20, 21]. Other studies implied that the effect of superfine magnesium hydroxide on improving the flame retardancy of matrix is better than that of ordinary magnesium hydroxide [22, 23].

This work is devoted mainly to studying the effect of ultrafine magnesium hydroxide on the tensile properties and flame retardancy of WPC system based on tensile test, LOI, and cone calorimeter. SEM and thermogravimetric analysis are also employed to further understand the mechanism of improving tensile properties and flame retardance of WPC when ultrafine magnesium hydroxide incorporated into matrix.

2. Experimental

2.1. Materials. Poplar powder (60–80 mesh, smashing of poplar wood and sieving), high density polyethylene (HDPE PE-LA-50D012), was supplied by Nanzhou Petrochemical Co. of China petroleum; polyethylene grafted by maleic anhydride (PE-1040) was supplied by Guangzhou aiqing sea additive Co; ordinary magnesium hydroxide (analytical reagent) was supplied by Tianjin Institute of Fine Chemicals (particle size 0.5–2.0 μm); ultrafine magnesium hydroxide was prepared in our laboratories (particle size 100–200 nm).

2.2. Sample Preparation. Poplar wood powder (60–80 mesh) was dried in oven for 6 hours and preserved in desiccator. High density polyethylene (HDPE) and polyethylene grafted by maleic anhydride (2% based on the amount of wood plastic composites) were completely plasticized with two-roll mixer (XK 160, Qingdao, China) under 150–160°C and then mixed with wood powder and magnesium hydroxide under 150–160°C for 10 min. The mixed samples were pressed into 3 mm sheet in a vulcanizing press machine (DLB 500 × 500,Wuxi, China) at 150–160°C, and samples of various sizes were obtained according to the testing standard.

2.3. Measurements. Tensile strength and elongation at break were investigated with an electronic tensile testing machine (WDW-50E, Jinan, China) according to GB/T 1040-92; the sample is in dumbbell shape according to GB/T 1040-92, and tensile rate is 50 mm/min.

LOI was performed by an oxygen index tester (JF-3, Nanjing, China) according to ISO 4589. The sample was 130.0 × 6.5 × 3.0 mm^3.

Cone calorimeter (Fire Testing Technology Limited, UK) was used to evaluate the flammability and smoke production following the procedure defined in ASTM E 1354. The samples were put in horizontal orientation at an incident flux of 35 kW/m^2. Samples size is 100.0 × 100.0 × 3.0 mm^3, and the samples exposed to the incident irradiance are 88.4 cm^2. Data were collected once every 5 seconds automatically. All data were treated with Microsoft Excel and the combustible parameters were obtained.

The fracture sections of broken samples resulting from tensile test were characterized by a scanning electron microscopy (FEI QUANTA 450 scanning electron microanalyzer, USA); the fracture section of samples was coated with a conductive gold layer.

The thermal stability of samples was studied by a synchronous thermal analyzer (NETZSCH STA449 F3, Germany). All measurements were conducted under static air and the weight of sample was 9.0±0.5 mg; the test temperature range was from 40°C to 800°C at heating rate 10°C/min, and the pan used was ceramic pan.

3. Results and Discussion

3.1. Tensile Properties. Tensile strength and elongation at break of all the samples are given in Table 1.

The data presented in Table 1 indicate that tensile strength and elongation at break increase when the content of ultrafine magnesium hydroxide (UMH) is no more than 30% (based on the amount of wood plastic composites); the tensile strength and elongation at break of wood plastic composites containing 30% UMH are 23.08 MPa and 7.6%, respectively; however the tensile strength and elongation at break decrease substantially when the content of UMH is over 30%; particularly the testing samples cannot be made owing to its fragility when the content of UMH is 60%, which may be explained when the content of UMH is below the critical value. The dispersion of UMH particles is good; the tensile performance improved owing to the strengthening and toughening effect of UMH. However, the particles of UMH will agglomerate and the dispersion will deteriorate when the content of UMH is above the critical value. Compared to ultrafine magnesium hydroxide, the tensile properties of wood plastic composites containing ordinary magnesium hydroxide (OMH) decrease substantially when the content is over 20%; the tensile strength and elongation at break of wood plastic composites containing 30% OMH are 15.38 MPa and 4.35%, respectively. The tensile strength and elongation at break of wood plastic composites containing UMH are larger than those of wood plastic composites containing OMH. This can be explained by the fact that the compatibility of UMH and WPC is better than that of OMH and WPC owing to their smaller size and good dispersion. Specific area increases quickly with the size of particles decreasing; higher specific area enlarges the contacting interface between magnesium hydroxide and wood powder or plastics, which favor the bonding strength between magnesium hydroxide and matrix. On the other hand, many microdeformation zones can be produced under stress field, which can absorb some energy and transfer exterior stress. So ultrafine magnesium hydroxide can improve the tensile properties of WPC when the content is below critical value.

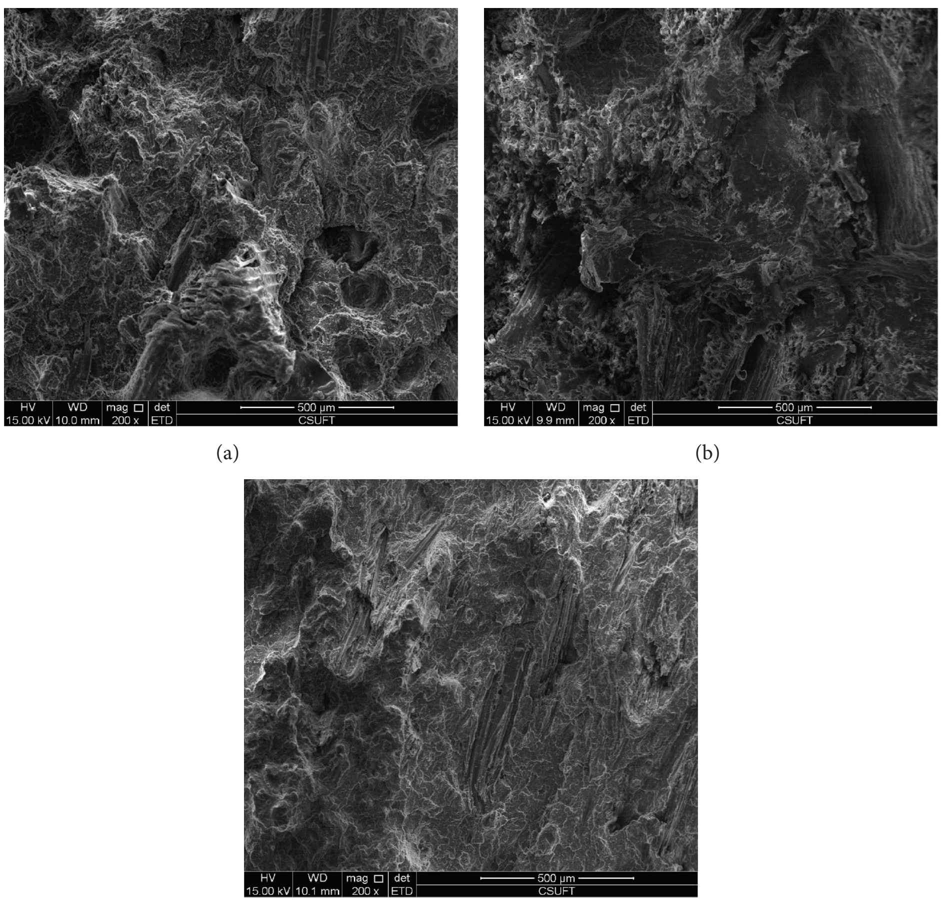
3.2. SEM Analysis. Different morphologies that are observed in the SEM image of fracture section are illustrated in Figure 1.

Many holes can be observed from the SEM of fracture section of pure WPC, which are the trace of fiber being pulled during the tensile test. The SEM microstructure of pure WPC indicated that wood powder is not dispersed evenly in the HDPE matrix; the compatibility of wood fiber and HDPE is not good owing to the polarity difference. Larger holes can be observed from the SEM of fracture section of WPC containing 30% OMH; the trace of pulled fiber is more obvious and serious, which indicated that the compatibility

Table 1: Tensile test results of WPC containing different content of magnesium hydroxide.

Sample	Wood powder (g)	HDPE (g)	OMH (g)	UMH (g)	Tensile strength (MPa)	Elongation at break (%)
WPC1	40	60			17.81	6.75
WPC2	32	48	20		17.80	4.40
WPC3	28	42	30		15.38	4.35
WPC4	24	36	40		14.58	3.10
WPC5	20	30	50		13.83	2.70
WPC6☆	16	24	60		—	—
WPC7	32	48		20	22.58	7.35
WPC8	28	42		30	23.08	7.60
WPC9	24	36		40	18.72	6.30
WPC10	20	30		50	18.53	5.90
WPC11☆	16	24		60	—	—

☆WPC6 and WPC11 cannot be made for the testing samples owing to their fragility.



(a) (b)

(c)

Figure 1: SEM microstructure of fracture section of samples: (a) pure WPC, (b) WPC containing 30% OMH, and (c) WPC containing 30% UMH.

is worse when 30% OMH incorporated into WPC, which lead to the tensile strength and elongation at break decrease substantially. The SEM of fracture section of WPC containing 30% UMH is quite different compared with that of pure WPC and WPC containing 30% OMH. The number and the size of holes decreased obviously, the dispersity of wood fiber and UMH is improved substantially, no agglomerate phenomenon of wood fiber can be observed, and most of wood fiber remained in the HDPE matrix and shared the stress. What is more, small particles of magnesium hydroxide possess good dispersity and compatibility in the matrix, which provided strengthening and toughening effect, so the tensile strength and elongation at break increase substantially. The SEM analysis is positive evidence that UMH can improve

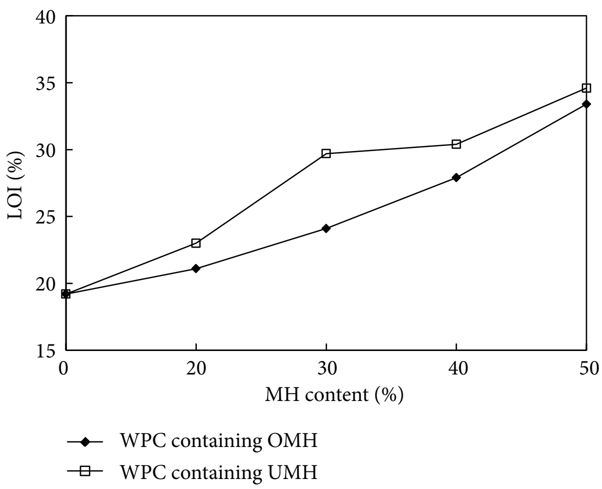


Figure 2: The effect of MH content on the LOI of WPC.

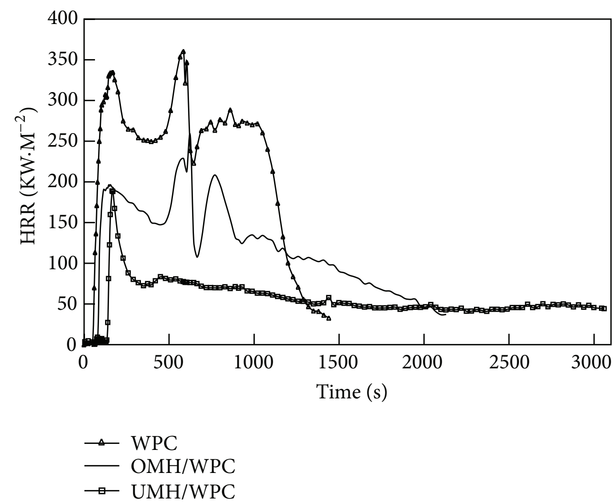


Figure 3: HRR curves of WPC, OMH/WPC, and UMH/WPC.

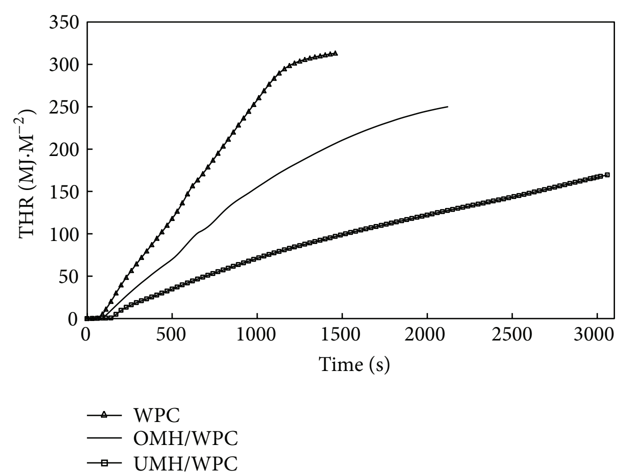


Figure 4: THR curves of WPC, OMH/WPC, and UMH/WPC.

the tensile strength and elongation at break when its content is below critical value.

3.3. Limited Oxygen Index. Limited oxygen index (LOI) of WPC containing different content (10%, 20%, 30%, 40%, 50%, and 60%, based on the amount of wood plastic composites) of magnesium hydroxide (MH) is illustrated in Figure 2.

The LOI curves implied that higher content of MH favors improving the flame retardancy of WPC; particularly the LOI increases more quickly when the content of UMH or OMH is larger than 20%. It is interesting to compare the LOI of WPC containing UMH and OMH, when the content of magnesium hydroxide is no more than 30%. The LOI difference between them is large, and the maximum difference (ΔLOI = 5.6) occurred when the content is 30%. With the content increasing further, the difference of LOI diminished, which can be explained by the fact that the dispersity of UMH is much better than that of OMH when the content is below critical value. Good dispersity contributes good flame retardance. However, the particles of UMH will agglomerate and the dispersity will deteriorate when the content of UMH is above the critical value.

3.4. Cone Calorimeter Test. WPC, UMH/WPC, and OMH/WPC are represented by the samples of pure WPC and WPC containing 30% ultrafine magnesium hydroxide and WPC containing 30% ordinary magnesium hydroxide, respectively. They are chosen to undertake further studies because they represented the typical composition of pure WPC and WPC containing two different sizes of magnesium hydroxide. Cone calorimeter test is a small-scale test, but it has good correlation with real fire disaster, compared with LOI test; the data obtained from cone calorimeter can provide plentiful information on fire. Some important combustion performance parameters are given in Table 2. The time to ignition doubled when 30% UMH incorporated into WPC; it implied that the ignition of UMH/WPC is more difficult than that of WPC and OMH/WPC. The heat release rate (HRR) and total heat release (THR) are presented in Figures 3 and 4. HRR is recognized as the most important parameter to measure the developing and spreading of fire; it provides an indication of the likely size of fire. The maximum of HRR is called pk-HRR, which indicates the danger of materials combustion in fire disaster. THR is the total heat released during the whole combustible process; most of THR will be absorbed by the matrix and accelerate its degradation to produce more combustible materials and speed up the fire spread in disaster. So HRR, pk-HRR, and THR are the critical parameters to evaluate the risk of fire disaster.

The results of Figure 3 show that pk-HRR of UMH/WPC is the smallest (188.59 KW/M^2), which decreased 48.4% compared with that of the WPC (365.27 KW/M^2) and decreased 26.8% compared with that of the OMH/WPC (257.60 KW/M^2). It is interesting to observe that the shape of HRR curves is distinct. The HRR curve of UMH/WPC is different from that of WPC and OMH/WPC. The HRR curve of WPC has two sharp peaks and one shoulder peak. The HRR curve of OMH/WPC has three peaks. The HRR

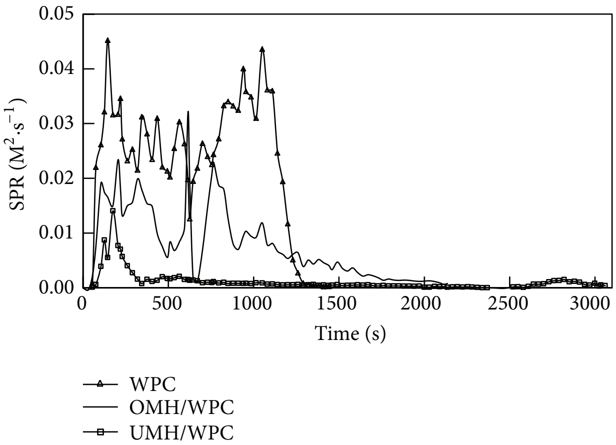


Figure 5: SPR curves of WPC, OMH/WPC, and UMH/WPC.

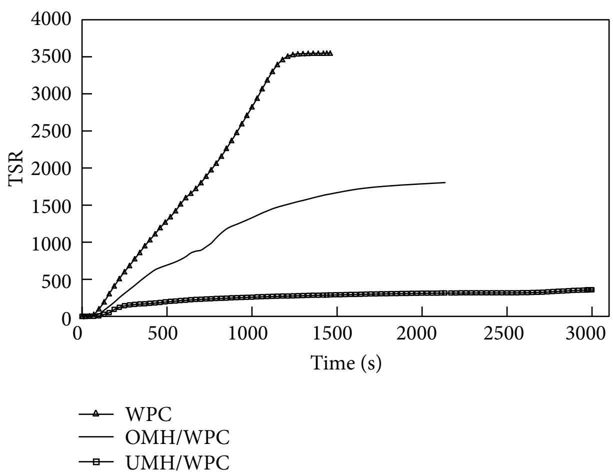


Figure 6: TSR curves of WPC, OMH/WPC, and UMH/WPC.

curve of UMH/WPC has only one peak. The shapes of first peak of OMH/WPC and UMH/WPC are different; the first peak of OMH/WPC reaches its summit and drops gradually; however the first peak of UMH/WPC reaches its summit and drops sharply, which indicated that the heat release of UMH/WPC is much lower than that of OMH/WPC during the initial combustion stage. The HRR curve of UMH/WPC nearly approaches being flat (50 KW/M^2) after the first peak; no other peak appeared. Figure 4 indicates that THR curve of UMH/WPC is much lower than that of WPC and OMH/WPC. The order of THR is as follows: WPC (313.04 MJ/M^2) > OMH/WPC (238.01 MJ/M^2) > UMH/WPC (118.41 MJ/M^2).

Smoke suppression is essential to consider for selecting an ideal flame retardant [24, 25]. Smoke production rate (SPR) and total smoke release (TSR) are two important parameters to evaluate the smoke release amount during combustion. SPR and TSR of WPC, OMH/WPC, and UMH/WPC are illustrated in Figures 5 and 6.

It is interesting that the shapes of SPR curves of WPC, OMH/WPC, and UMH/WPC are distinct; there are more than two peaks in SPR curves of WPC and OMH/WPC; however there is only one peak in SPR curve of UMH/WPC. The SPR peak value and TSR of UMH/WPC are 0.014 m^2/s and 356.8, respectively, which are much less than those of WPC (0.045 m^2/s, 3548.4) and those of OMH/WPC (0.032 m^2/s, 1804.0). The results imply that ultrafine magnesium hydroxide possesses obvious smoke suppression on WPC by decreasing SPR peak value and TSR. The smoke suppression of ultrafine magnesium hydroxide can be explained by the fact that ultrafine magnesium hydroxide can absorb smoke particles owing to its little particle size and large specific area.

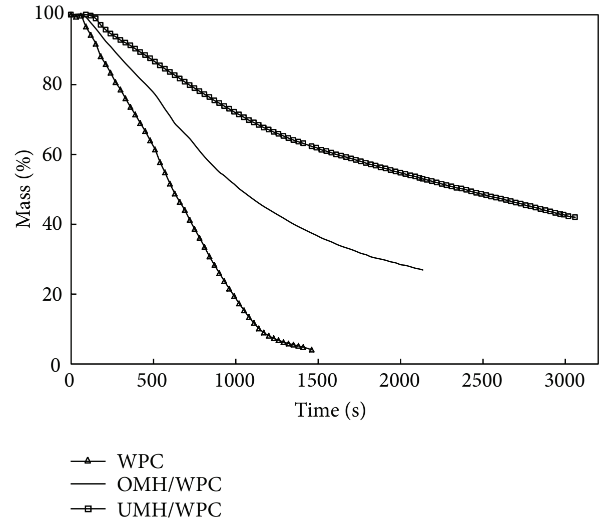


Figure 7: Mass curves of WPC, OMH/WPC, and UMH/WPC.

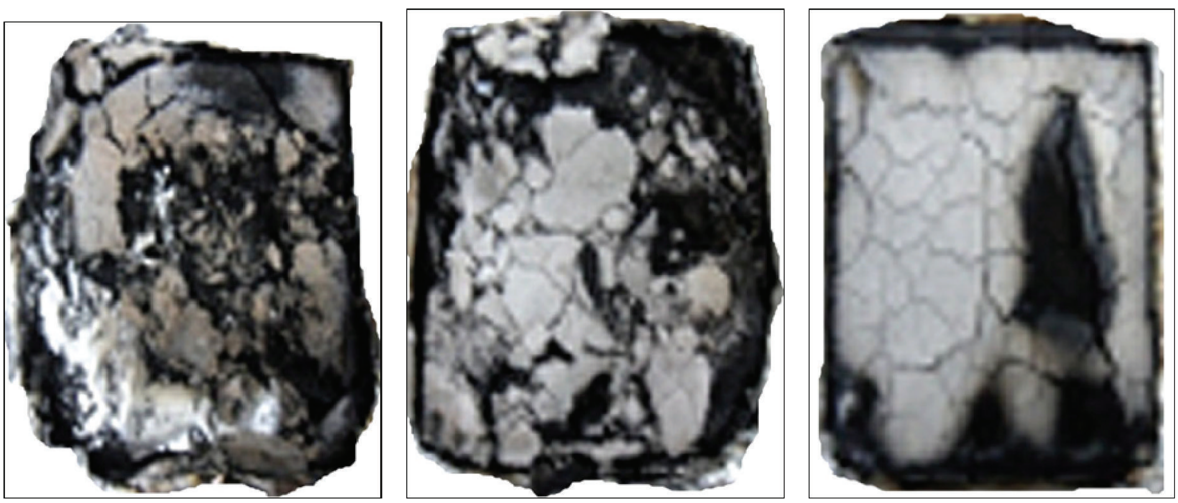
Mass loss rate and residual mass fraction reflect the thermal degradation rate and degradation behavior of materials under certain irradiance intensity; it is also an important parameter to evaluate the flame retardance of materials. Mean mass loss rate and residual mass curves are illustrated in Table 2 and Figure 7.

The mean mass loss rate of UMH/WPC (0.025 g/s) is the smallest compared to that of WPC (0.064 g/s) and that of OMH/WPC (0.041 g/s), which correspond to the mass curves in Figure 7. The mass loss of UMH/WPC (57.23%) is the smallest compared to that of WPC (95.85%) and that of OMH/WPC (73.10%) at the combustion termination. The time needed for the residual mass to become half of the original mass of WPC, OMH/WPC, and UMH/WPC is 615 s, 1025 s, and 2380 s, which implied that UMH can more effectively improve the thermal stability of WPC compared to that of OMH.

Effective combustion heat (EHC) indicates the contribution of degradation products to the combustion; lower EHC indicates that most of degradation products are incombustible gas. The mean EHC of WPC, OMH/WPC, and UMH/WPC is 30.40 MJ/kg, 28.01 MJ/kg, and 22.85 MJ/kg, respectively, which implied that ultrafine magnesium hydroxide can more effectively reduce the combustible gas during degradation process under heat compared to that of ordinary magnesium hydroxide.

Table 2: Combustion performance parameters of different compositions of WPC samples.

Combustion parameters	WPC	OMH/WPC	UMH/WPC
Time to ignition/s	51	78	133
Peak-heat release rate/KW·m^{-2}	365.27	257.60	188.59
Mean heat release rate/kW·m^{-2}	222.78	132.36	65.90
Mean effective combustible heat/MJ·kg^{-1}	30.40	28.01	22.85
Total heat release/MJ·m^{-2}	313.04	238.01	118.41
Peak-smoke produce rate/m^2·s^{-1}	0.045	0.032	0.014
Total smoke release	3548.4	1804.0	356.8
Mean mass loss rate/g·s^{-1}	0.064	0.041	0.025
Residual mass fraction/%	4.15	26.90	42.77



(a) (b) (c)

Figure 8: Digital photo of residue of (a) WPC, (b) OMH/WPC, and (c) UMH/WPC.

3.5. Combustion Phenomenon and the Morphology of Residue. The combustion phenomena of WPC, OMH/WPC, and UMH/WPC are distinct. Big flame accompanied with dense black smoke can be observed during the WPC combustion. The carbon layer formed and flame become small during the initial combustion stage of OMH/WPC; however formed carbon layer will collapse under the heat, the flame becomes larger, and combustion is exacerbated. Small flame accompanied with white smoke can be observed during the whole combustion process of UMH/WPC, which implied good flame retardance of UMH/WPC.

The digital photos of residue after combustion are illustrated in Figure 8. The distinct appearance of residue samples of WPC, OMH/WPC, and UMH/WPC can be observed easily. The residue of WPC is scarce. The residue of OMH/WPC increases obviously except that the exterior carbon layer was seriously damaged, which cannot insulate the heat and oxygen effectively. The residue of UMH/WPC maintained its original shape; continuous compact carbon layer plays an important role in insulating heat and oxygen, which decreases the HRR and MLR. The appearance of residue provides positive evidence that ultrafine magnesium hydroxide has better flame retardance compared to that of OMH when they are incorporated into WPC.

3.6. Thermogravimetric Analysis (TGA). Thermal analytical techniques are effective methods to investigate the thermal degradation behavior of flame retarding materials. The curves of TG are listed in Figure 9.

It can be seen from Figure 9 that the char residue of UMH/WPC (29.10%) is higher than that of OMH/WPC (27.02%) at 800°C and the char residue of pure WPC is the lowest (7.24%), which implies that ultrafine magnesium hydroxide is superior to ordinary magnesium hydroxide in protecting char and decreases its oxidization speed at high temperature.

Interactions between the compounds of a mixture can be revealed by comparing the experimental TG curve with a "theoretical" TG curve calculated as a linear combination of the TG curves of the mixture ingredients weighed by their contents [26]. The experimental and "theoretical" TG curves of UMH/WPC are presented in Figure 9. UMH/LDPE (theoretical)= $(1-\beta)\times$ TG(WPC) + $\beta \times$ TG(UMH), where TG (WPC) and TG (UMH) represent the experimental value of pure WPC and UMH in TG test, respectively; β represents the mass percent of UMH in the UMH/WPC ($\beta = 30\%$). UMH/WPC (experimental) represents the experimental value of UMH/WPC in TG test. The residual char of UMH/WPC (experimental) and UMH/WPC (theoretical)

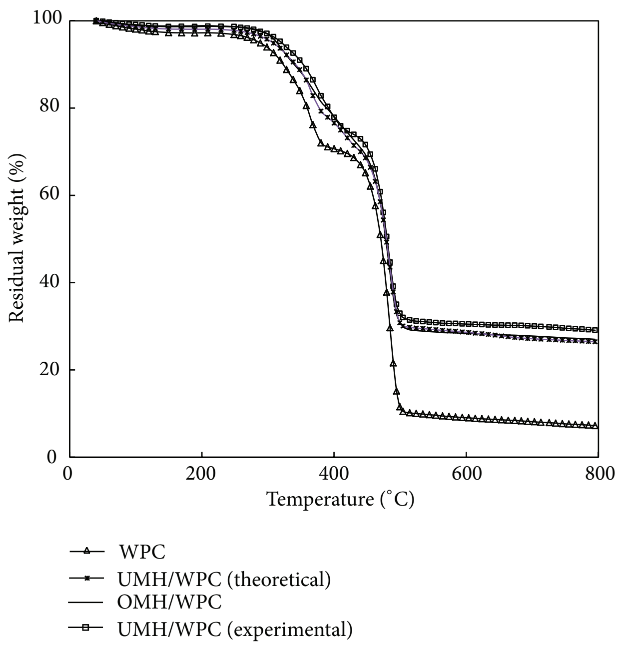


Figure 9: TG curves of WPC, OMH/WPC, and UMH/WPC (theoretical and experimental).

is 29.10% and 26.25% at 800°C, respectively. The comparison of theoretical and experimental TG curves provided positive evidence that ultrafine magnesium hydroxide can promote charring of WPC and increase the char residue at high temperature.

4. Conclusions

The tensile properties (tensile strength and elongation at break) of WPC improved when 30% ultrafine magnesium hydroxide is introduced to pure WPC system, while the opposite effect will occur when 30% ordinary magnesium hydroxide is introduced to pure WPC system.

The microstructure SEM image of fracture section provides the positive evidence that the tensile properties of WPC treated with ultrafine magnesium hydroxide are superior to pure WPC and WPC treated with ordinary magnesium hydroxide. Better compatibility plays an important role in the tensile strength and elongation at break of WPC.

The results of LOI and cone calorimeter showed that flame retardance and smoke suppression improved when ultrafine magnesium hydroxide was introduced into WPC system. Thermogravimetric analysis implied that ultrafine magnesium hydroxide can improve the char structure which plays an important role in reducing the degradation speed of the inner matrix during combustion process and increases the char residue at high temperature.

Conflict of Interests

The authors declare that they have no conflict of interests regarding the publication of this paper.

Acknowledgments

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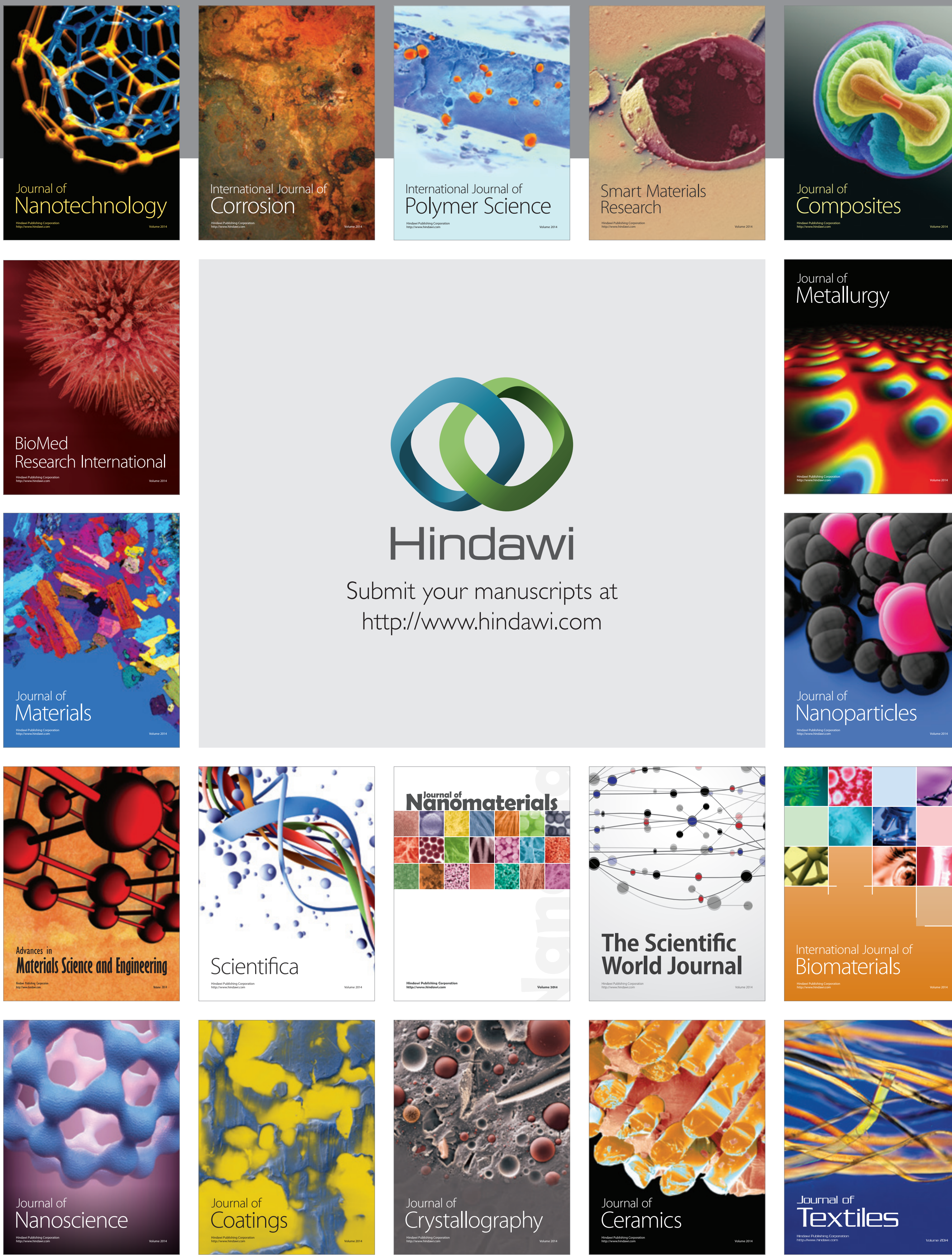
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