

Research Article

Thermal Stability and Flame Resistance of the Coextruded Wood-Plastic Composites Containing Talc-Filled Plastic Shells

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Talc is a popular filler for the fabrication of plastic composites. The presence of talc helps improve mechanical, thermal, and flame resistance properties of the composite. In this work, we report the influence of a talc-filled plastic shell layer on thermal stability and fire flammability of the core-shell structured wood high-density polyethylene (HDPE) composites manufactured through coextrusion. The result showed that morphological analysis of the char layer after combustion confirmed the formation of a continuous surface char layer with talc addition in the composites, helping block fire penetration and enhance overall fire resistance of the composites. The shell thickness averaged at 1.0 ± 0.2 mm, which represents a fair thick shell over a 10 mm thick WPC core layer. The surface of regular wood-filled HDPE showed large cracks, allowing more rapid fire penetration and reducing its fire resistance. At 800°C, average residual weight for all composite was $21.5 \pm 13.8\%$, most of which was attributed to the inorganic nonvolatile talc components. With the increase of talc level, THR values of coextruded WPC decreased from 302.47 MJ/m^2 (5 wt% of talc) to 262.96 kW/m^2 (50 wt% of talc). When talc content in the shell layer was less than 25 wt%, the flame resistance properties were slightly enhanced compared with the composites containing unmodified HDPE shells. When talc content in the shell exceeded 25 wt%, the composite's total heat release and its rate substantially decreased.

1. Introduction

Coextruded core-shell structured wood-plastic composites (WPCs) as an emerging type of green structural materials have been developed to enhance performance of wood/natural fiber-reinforced polymer composites [1–3]. The combined use of wood fiber and plastic through core-shell structure design can help reduce overall composite costs and improve stiffness and biological resistance. They are also advantageous in terms of melt processing and weather resistance. The presence of a plastic shell (with large thermal expansion and low modulus) over a WPC core in the coextruded WPCs can, however, negatively affect overall fire flammability and thermal stability of the composite [4]. A shell layer composed of properly filled plastics can help reduce flammability and thermal degradation properties of

the composite compared with coextruded WPC with unfilled shell and/or regular WPC without a core-shell structure.

Stabilization of shell layers using carbon nanotubes [5] as well as by combining wood and mineral fillers in the shell layer [6, 7] was attempted. Studies have also been conducted on the development of a stable shell layer over the coextruded WPCs by blending high-density polyethylene (HDPE) with additives (such as compatibilizers, photostabilizers, and nanosized TiO_2) using combination of various minerals with wood [8] and precipitated calcium carbonate (PCC) [9] in the shell layer. Thermal expansion of the coextruded WPCs as function of thermal expansion and modulus of HDPE shells containing glass fibers was also studied by a finite element model. However, more cost-effective materials and design are still needed as shell layers for the coextruded WPC.

WPCs are popular materials for interior panels for automobiles as well as construction deck boards. However, WPCs demonstrate poor fire resistance due to the extreme sensitivity of both plastic and wood to high temperatures. This limitation hinders the application of WPCs. Therefore, it is critical to improve WPCs' fire performance for overall safety and also to satisfy fire requirements and regulations, which will help to widen their industrial applications [10]. One possible method to improve WPC fire resistance is incorporation of fire retardants during, for example, the compression, extrusion, or injection molding stages of the WPC processing. Some of the most popular flame retardants are phosphorous based [11–15]. When ammonium polyphosphate (APP) was used as a WPC flame retardant, it formed a protective char layer, which significantly decreased flammability of the whole composite [16–19]. However, additional research and development of filler materials as flame-retardant shell layers for core-shell structured WPCs are still necessary.

Talc, a common mineral filler with a chemical formula $Mg_3Si_4O_{10}(OH)_2$ and density of $\sim 2.75 \text{ g/cm}^3$, is often used in fabrication of reinforced plastic composites [20–22]. Talc-filled shells in coextruded WPCs can enhance their mechanical and thermal expansion performance [23]. The growth of inorganic filler systems for plastic has been particularly rapid owing to the demand for high-performance materials and filler's environmentally friendliness compared with chemical fire retardants.

Some work had been done to study the effects of talc fiber on mechanical properties of final composites. However, talc has not been widely used as a reinforced filler for polymer resin systems as a shell layer of coextruded WPC especially on thermal behavior, and only limited amount of data, especially on antflammable properties, has been published on talc-filled HDPE composites with coextruded WPCs. Moreover, there is a lack of detailed investigation on the effect of talc at this point. Thus, the main objective this study described in this paper was to investigate the effect of incorporation of talc-filled shells on the thermal stability, morphology, and flammability of coextruded WPCs.

2. Materials and Methods

2.1. Raw Materials. Talc (with density of $\sim 2.8 \text{ g/cm}^3$, 325-mesh average particle size, and 30 wt% absorption rate) was purchased from the Fibre Glast Developments Co. (USA). Pine wood flour (WF 20-mesh size) was acquired from American Wood Fibers Inc. (USA). High-density polyethylene (HDPE) of AD60 grade with 0.7 g/10 min MFI at 190°C and 2.16 kg and density of $\sim 0.96 \text{ g/cm}^3$ was obtained from ExxonMobil Chemical Co. (USA). Maleic anhydride-grafted polyethylene (MAPE, Epolene™ G2608 grade) with MFI = $\sim 6\text{--}10 \text{ g/10 min}$ (190°C and 2.16 kg applied weight), molecular weight = 65000 g/mol, and an acid number = 8 mg of KOH per g of MAPE was bought from Eastman Chemical Co. (USA). Lubricant (TPW 306 grade) was from Struktol Co. (USA).

2.2. Composite Formulation. Several sample groups of coextruded WPCs were prepared for this work. The composite

TABLE 1: Formulation of core-shell (talc-filled shell) structured WPC.

	Core layer (wt%) ^a					Shell layer (wt%)		
	Wood	HDPE	MAPE	Lub	Talc	HDPE	Talc	Colorant
Core	55	33	2	5	5	0	0	0
A	55	33	2	5	5	98	0	2
B	55	33	2	5	5	93	5	2
C	55	33	2	5	5	88	10	2
D	55	33	2	5	5	83	15	2
E	55	33	2	5	5	73	25	2
F	55	33	2	5	5	63	35	2
G	55	33	2	5	5	48	50	2

^aFiller content was based on the total composite weight.

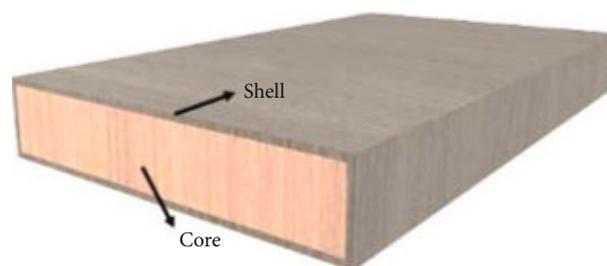


FIGURE 1: Coextruded core-shell WPCs with a talc-filled shell layer.

core consisted of HDPE, WF, lubricant, MAPE, and talc at 33:55:5:2:5 weight ratio. The shell was made with HDPE and talc with talc fill rates of 0, 5, 10, 15, 25, 35, and 50% by weight. A total amount of 2 wt% of a colorant (relative to the shell material weight) was also added to the shell. The formulation of coextruded WPC is shown in Table 1.

2.3. Composite Manufacturing. Coextrusion experiment was performed using a coextrusion system [6] with two feeders for plastic pellets and wood fiber/lubricant/talc mixtures, respectively. The extrusion system consisted of a 32 mm conical Brabender (USA) and the Leistritz Micro-27 twin-screw extruders for the shell and for the core, respectively. Specially made die was used to obtain a profile with 13 × 50 mm target cross-section size maintained by a vacuum sizer. The coextruded profiles were pulled by a downstream puller, going through a 2-meter-long water bath under constant water spray. Temperatures during coextrusion were 155 (at the feeder side), 160, 165, 170, 170, 170, 160, 150, 140, 135, and 155°C (at the die side) for the core. For different formulations, extrusion temperatures in the 150–165°C range were used for the shell.

2.4. Characterization

2.4.1. Composite Morphology Characterization. Sample morphologies were analyzed by scanning electron microscopy (SEM) (Model Hitachi S4800, Tokyo, Japan) operated at 15 kV accelerating voltage. To prepare the SEM samples, the WPC samples were treated in liquid nitrogen for 10 min and then impact-broken to two separate pieces. The

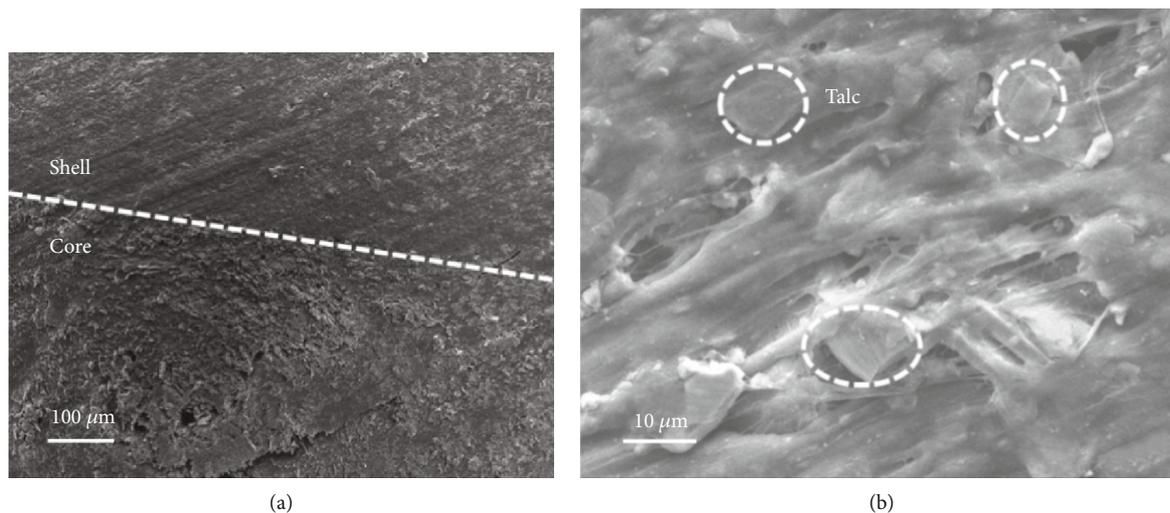


FIGURE 2: SEM micrographs of coextruded WPC with a talc-filled shell. (a) Core-shell interface. (b) Shell layer.

TABLE 2: Data obtained from TGA.

Name	T_o (°C)	WL_o (%)	T_p (°C)	WL_p (%)	T_s (°C)	WL_s (%)	Residue (%)	$T_s - T_o$ (°C)	$WL_s - WL_o$ (%)
AD60	459.0	30.13	470.5	65.74	480.4	95.42	0.83	21.4	65.29
Colorant	446.3	15.83	458.2	26.70	674.9	44.75	52.23	228.6	28.92
Talc	529.2	5.49	560.7	13.05	625.2	18.78	80.91	96	13.29
B	462.2	35.31	469.6	58.63	480.9	89.08	5.93	18.7	53.77
C	456.5	20.85	468.9	51.60	483.2	84.44	11.97	26.7	63.59
D	461.0	26.01	470.0	50.43	480.6	78.36	13.58	19.6	52.35
E	460.2	22.24	471.5	48.06	663.8	76.36	23.48	203.6	54.12
F	460.5	19.21	472.0	42.73	675.6	68.95	30.93	215.1	49.74
G	459.8	14.84	470.7	32.60	684.7	56.70	43.17	136.7	41.86
Core	341.3	0.18	465.4	71.72	478.0	82.56	12.76	136.7	82.38

fractured cross-sections were sampled and coated with Au to improve their conductivity during SEM runs.

2.4.2. Thermogravimetric Analysis (TGA). Thermal stability properties of various shells in comparison with the core composition were studied using a TA Instrument model Q50 analyzer (TA Inc., New Castle, DE, USA) with nitrogen. For each test, approximate 5 mg of samples was tested at a heating rate of 5°C/min and temperature range of 30–800°C. From each test run, the TG and differential TG (DTG) curves are plotted, from which characteristic temperatures were determined.

2.4.3. Flammability Test. Flammability tests were done using a Stanton Redcroft cone calorimeter manufactured by Fire Testing Technology Ltd. (London, UK). ISO 5660-1 standard procedure was implemented. Each sample was placed in an Al_2O_3 crucible (100 mm long and 100 wide) lined with Al foil. The assembly was mounted horizontally on the calorimeter loader and heated at 50 kW/m². The upper surface of the test sample corresponded to a temperature of ~780°C. Three measurements were performed for each sample group. Test result reproducibility was within ±7%.

The limiting oxygen index (LOI) was determined by igniting 10 × 0.65 cm sample pieces in a glass tube containing O₂/N₂ mixture.

$$LOI = \frac{\text{Volume of } O_2}{\text{Volume of } (O_2 + N_2)} \times 100\%. \quad (1)$$

2.4.4. Surface Char Morphology Imaging. Digital images were taken from the sample surface before and after flame testing to contrast the surface char layers among different shell formulations.

3. Results and Discussion

3.1. Sketch Map of Coextruded WPCs. Figure 1 reveals a sketch map of coextruded WPC with core-shell structure (talc-loaded shell layer). The shell thickness averaged at 1.0 ± 0.2 mm, which represents a fair thick shell over a 10 mm thick WPC core layer.

3.2. Morphology. SEM micrographs of coextruded WPCs containing talc-filled shells are shown in Figure 2. Figure 2(a) shows a significant difference in morphological

characteristics between composite cores and shell layers. The core material showed a typical impact-fractured rough surface with wood fibers, while the shell layer with pure HDPE exhibited a smoother surface (Figure 2(a)). The shell layer bonded to the core layer well without showing apparent separation between the shell and the core. Talc platelets (shown in Figure 2(b)) are clearly visible on the fractured sample surfaces of the shell layer, and the platelets had both in-plane and out-of-plane orientations as shown. Talc pull-out did not occur during the composite fracture judging by the absence of voids (Figure 2(b)).

3.3. Thermal Stability. Thermal stability data of pure and modified (with different talc contents) HDPE shells are presented in Table 2 and Figure 3. The TG and DTG curves of the shell layers containing different talc amounts are shown in Figure 3, in comparison with the WPC core. A strong DTG peak, appeared because of composite decomposition, was accompanied by a shoulder (shown with an arrow), which is typically attributed to talc decomposition in inert atmosphere. Similar peaks were observed for all samples, which indicates that even at different talc contents, composites had similar characteristics typical for polymer composites.

The TG curve indicated three obvious stages of pyrolysis. Accordingly, the DTG curve had two characteristic peaks (Figure 3). During the entire pyrolysis process, the initial temperature of pyrolysis with talc loading was close to 450°C (Table 2) and the initial temperature of pyrolysis for the core composite was 350°C. The elements of the WPCs started to react in this stage. The first pyrolysis peaks were observed between 450°C and 560°C, which represents a major stage of reaction wherein cellulose and hemicellulose began to decompose and lignin gradually softened, resulting in a number of products. The second pyrolysis peaks were observed between 470°C and 680°C, which represents a stage of carbonization of materials. In this stage, the weight loss of all composites reached a maximum. The temperatures between the beginning of reaction and the second peak were in the range of 10~230°C, and the weight loss was in the range of 10~90%. As talc content in the composite increased, residual weight also increased. Residual weight of the shell composite containing 10 wt% talc was 11.97%.

Data related to the composite decomposition is presented in Table 2. Decomposition onset was in the 459.0–462.2°C temperature range with the exception of the colorant and the core. Weight loss in this range, WL_o , was ~58% for all talc-containing composites. HDPE containing 10 wt% of talc showed higher values, which indicates the fastest initial decomposition rate. T_p indicates that the maximum decomposition rate of talc-filled composites occurred at $\sim 470.5 \pm 1.2^\circ\text{C}$. Decomposition of stand-alone talc occurred at higher temperature (equal to 560.7°C). At these temperatures, composite weight losses were above 32%. In this temperature range, because of the small talc content, weight loss associated with talc was ~13%. From peak to shift temperature, the composites exhibited rapid degradation in a narrow temperature range. Most shells showed a weight loss of almost 40% or higher at around 670°C as WL and T_s while the loading level was higher than 32 wt%. At 800°C, average residual

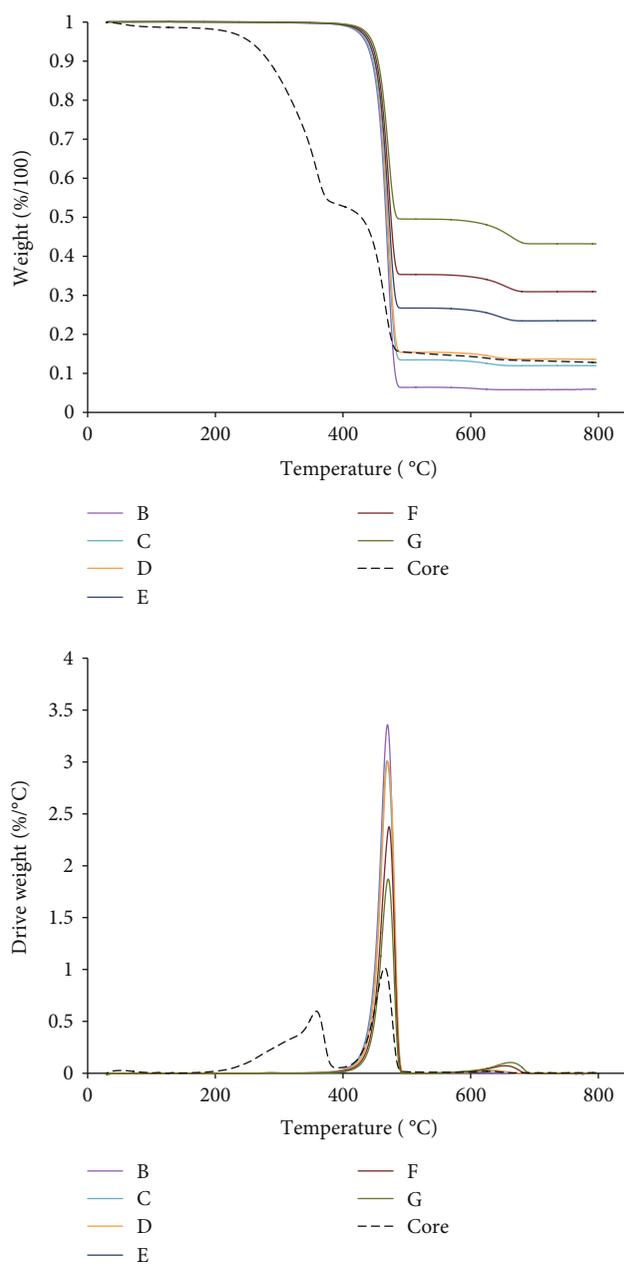


FIGURE 3: Decomposition curves obtained at a 5°C/min heating rate of core-shell composites with different talc contents in the shell in comparison with a material containing just the core (dashed curves).

weight for all composite was $21.5 \pm 13.8\%$, most of which was attributed to the inorganic nonvolatile talc components. Temperature and weight loss differences between the offset and shift points, $(T_s - T_o)$ and $(WL_s - WL_o)$, respectively, demonstrated most weight loss (~50%) occurred at $\sim 200^\circ\text{C}$ for most talc-filled composites.

3.4. Flame Resistance. Test parameters including peak heat release rate (PHRR), total heat release (THR), average heat release rate (AHRR), average mass loss rate (AMLR), average effective heat of combustion (AEHC), and average specific extinction area (ASEA) were obtained from cone calorimetry

TABLE 3: Summary of flammability performance.

Talc content (wt%) ^a	PHRR ^b (kW/m ²)	AHRR (kW/m ²)	THR (MJ/m ²)	TSR (m ²)
Core	313.5 (30.3) ^c	221.0 (22.1)	193.2 (18.7)	4167.0 (398.1)
0	805.1 (78.7)	286.5 (28.7)	295.0 (27.8)	6466.9 (637.7)
5	807.1 (79.7)	280.2 (27.5)	302.5 (30.1)	6476.0 (653.2)
15	805.1 (78.9)	286.5 (26.9)	295.0 (29.7)	6466.9 (612.8)
25	571.6 (53.8)	239.4 (21.1)	276.5 (22.5)	5938.6 (578.9)
35	476.3 (42.1)	215.7 (19.8)	260.9 (25.8)	5036.8 (511.2)
50	323.3 (31.5)	192.7 (19.6)	262.1 (26.3)	4708.1 (381.9)

^aFiller content was based on the total composite weight. ^bPHRR = peak heat release rate; AHRR = average heat release rate; THR = total heat release; AMLR = average mass loss rate; ASEA = average specific extinction area; AEHC = average effective heat of combustion. ^cNumbers in the parenthesis are standard deviation.

testing, and the data are shown in Table 3 and Figure 4 for the composites tested in this work.

The PHRR is a parameter that indicates fire size and its spread. For the composite containing over 15 wt% of talc in its shell spread, the PHRR values were significantly lower. The PHRR of the core alone was equal 313.48 kW/m². After the addition of the pure HDPE layer, however, the PHRR value of the composite increased to 805.11 kW/m², which indicates a negative effect of HDPE on WPC's flammability. Talc addition to the shell decreased the PHRR value of the composite. At talc shell content equal to 50 wt%, the PHRR value of the composite was 59.8% lower than the PHRR of the unmodified plastic shell. In addition, THR and AMLR also decreased as more talc was added. Thus, the addition of talc in the shell layer slowed down the combustion process of the core-shell structured WPCs.

Total smoke rate data for our composites is shown in Figure 4(a). TSR increased with time. Coextruded WPCs containing shells with different talc amounts demonstrated improved (by 13-15%) TSR values relative to unmodified WPCs. The composite core had a TSR of 4167 kW/m². The neat HDPE in the shell layer had a TSR of 6467 kW/m², and TSR values had little change for the talc-HDPE composites at 0, 5, and 15% talc level. After talc filled the shells at 5 wt% in the shell layer, the TSR for talc-filled composites declined from 6476 kW/m² (5 wt% of talc) to 4708 kW/m² (50 wt% of talc) as filler content increased. Figure 4(b) shows correlation between heat release and time and reflects flammability performance of the talc-filled composites. The talc filler in coextruded WPCs had an important influence on fire performance. The HRR increased, and the surface of the composite was easier in pyrolysis, producing more combustible materials. On the contrary, the composite having lower HRR or peak of HRR had a good flammability performance.

In general, PHRR increased as filler content in the coextruded WPCs was increased (see Figure 4(b) and Table 3). For the core composite, the PHRR was 331.48 kW/m². The higher PHRR value at pure HDPE in the shell layer (compared with the core-only data) was 805.11 kW/m², which indicates a negative effect of pure plastic coating on flammability of the composite. Coextruded WPCs with shell filler content at 5 wt% of talc had a PHRR of 807.07 kW/m², rising up 0.24% compared with pure plastic. The PHRR decreased as talc content in the shell increased above 5 wt%. AHRR

values of pure and modified (with different talc contents) HDPE are shown in Table 3. The AHRR of a fixed WPC core was 221.04 kW/m². The AHRR for coextruded WPCs containing unmodified HDPE slightly increased relative to just WPC. The AHRR of the coextruded WPC containing HDPE with talc started decreasing after talc contents above 15%. At talc contents in the shell equal to 35 and 50%, AHRR values of the corresponding composites were 215.70 and 192.74 kW/m², respectively. These values are lower than those for just the core.

Time dependence of THR of the manufactured WPCs with different talc contents in the shell is shown in Figure 4(c). Talc addition to shells clearly increased THR. The WPC core had lower THR value (which was equal to 193.23 MJ/m²) than the WPC core layer containing unmodified HDPE (which was equal to 193.24 MJ/m²). THR values for the composites with 5 and 15 wt% of talc were very close to the THR values of the core material during the first 50 seconds. Thus, total heat release was relatively stable with the increase of filler content. However, the THR for composites exhibited a decreasing trend with increased filler content after the talc loading levels reached to 5 wt%. With the increase of talc level, THR values of coextruded WPC decreased from 302.47 MJ/m² (5 wt% of talc) to 262.96 kW/m² (50 wt% of talc).

LOI values of all composites fabricated in this case enhanced as talc loading level in the shell layer enhanced (Figure 5). Before the torch flame was removed, core materials and core-shell structure composite with neat HDPE shells cannot be burned entirely and rapidly. When talc content in the shell layer was higher than 35 wt%, the composites could not be totally burned even in the oxygen-rich combustion probably due to the protective shell layer.

3.5. Surface Char Morphology. Figure 6 shows composite surfaces photographed after the cone calorimetry test. A crisp char residue can be clearly seen for different WPC core composites (Figures 6(a) and 6(b)) with multiple broken pieces. The composite residue aggregated forming multiple cracks and holes. The cracks allowed more heat to penetrate deep into the composite, causing more complete combustion. On the other hand, the char layer, obtained for composites containing 25 wt% of talc in the shell (Figures 6(c) and 6(d)), was continuous with only some holes on the surface. Thus, this

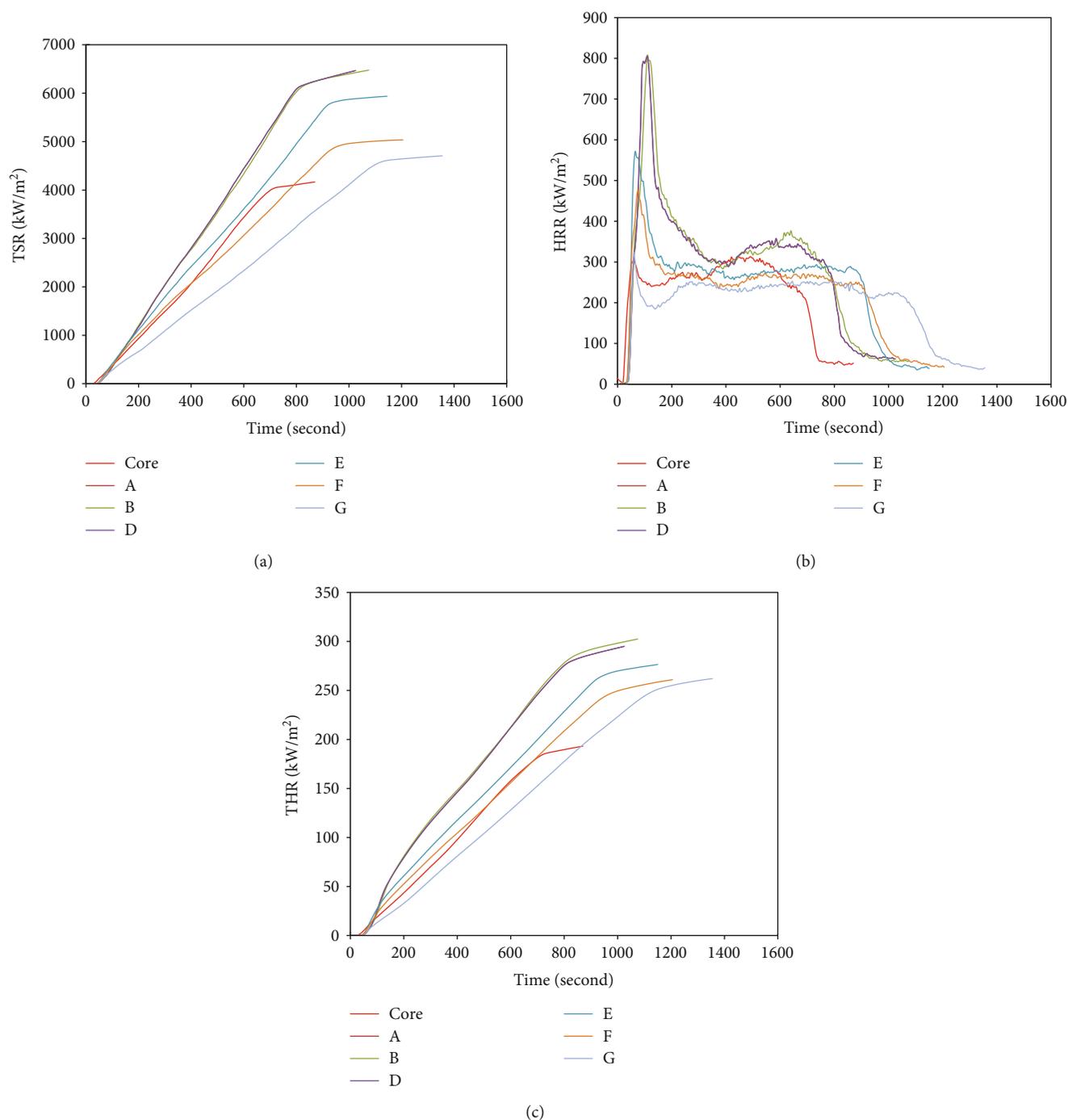


FIGURE 4: (a) Total smoke rate, (b) heat release rate, and (c) total heat release of the composites with different talc contents as a function of time.

cohesive and dense char layer blocked internal combustible gases and protected HDPE from flames. Such stable char provided a heat barrier, which also reduced the HRR and THR values (i.e., heat release).

4. Conclusions

The following conclusions can be drawn from the current study:

- (1) A typical core-shell structured WPC was formed using shell layers filled with various amounts of talc over a regular WPC core
- (2) The use of talc led to increased residual weight during TG analysis
- (3) When talc content exceeded 25 wt%, WPC's total heat release and its rate substantially decreased. PHRR and THR decreased as talc content in the shell

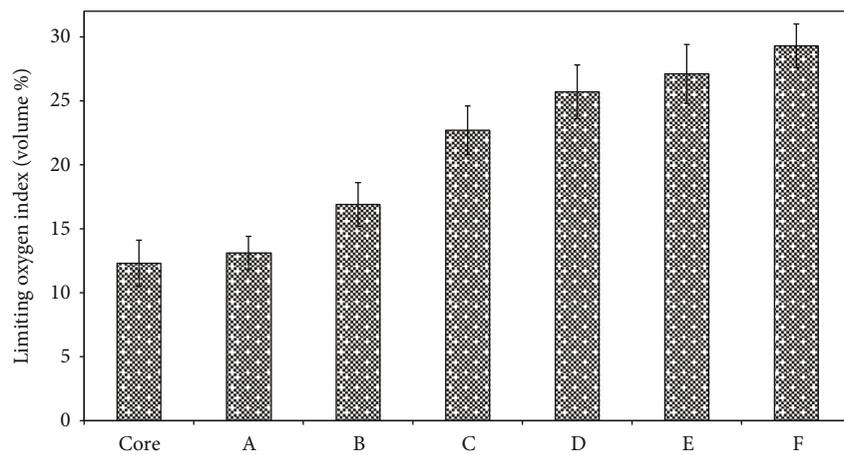


FIGURE 5: LOI of coextruded WPC with a talc-filled shell.

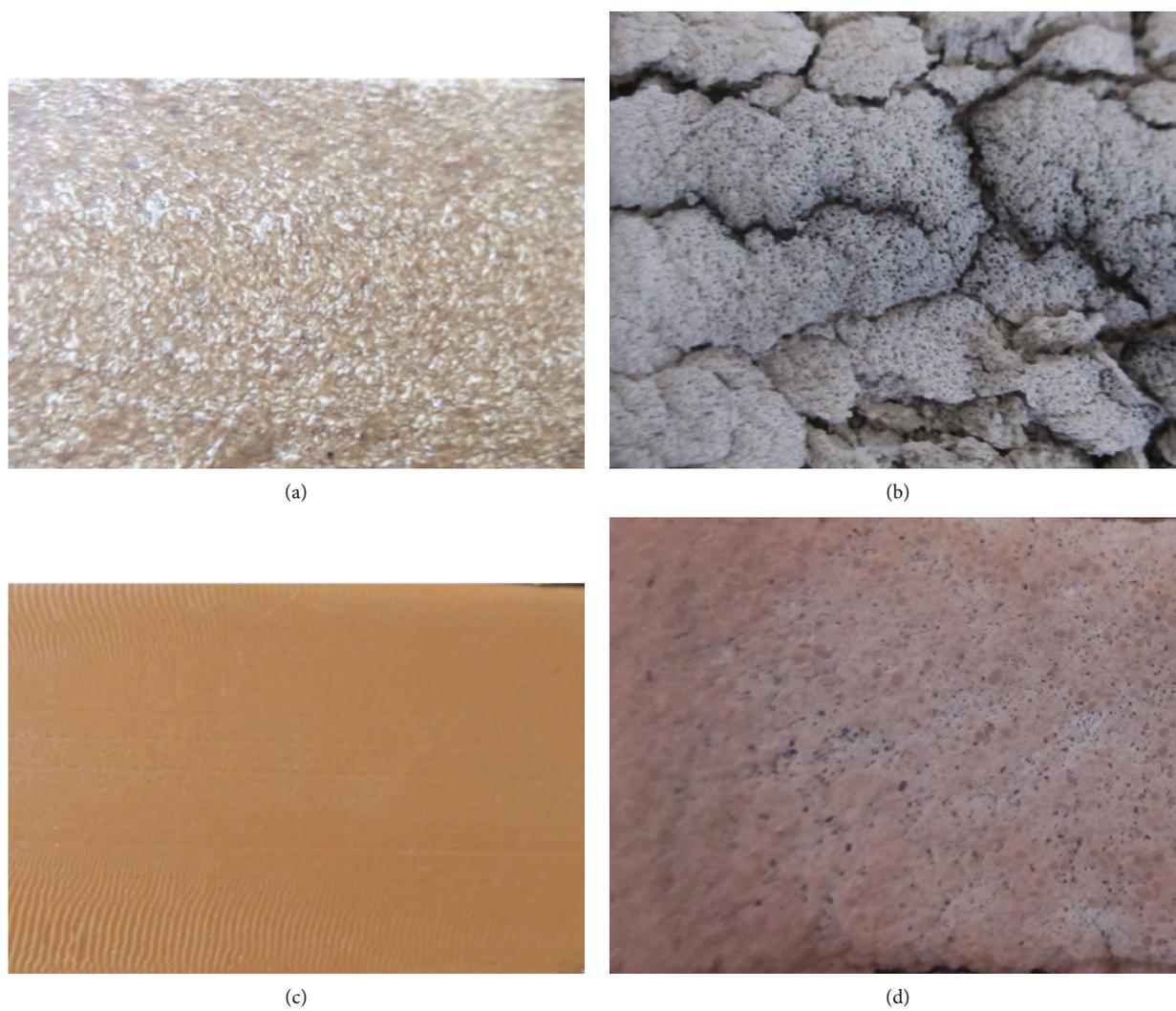


FIGURE 6: Photographs of the char residues on the samples subjected to the cone calorimetry: (a) core, (b) 20 wt% talc-filled shell layer, (c) core, and (d) core-shell structured composites with a HDPE shell.

increased. Thus, combustion of the cure-shell composites was incomplete when talc was present, which is beneficial for their practical applications

- (4) The surface of the composite produced more combustible materials in pyrolysis. With the increase of talc level, THR values of coextruded WPCs decreased from 302.47 MJ/m² (5 wt% of talc content) to 262.14 MJ/m² (50 wt% of talc content)
- (5) Morphological analysis confirmed the formation of a continuous/dense surface char layer with talc addition in the composites which helped further enhance fire resistance of the coextruded WPCs

Data Availability

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

Conflicts of Interest

The authors declare no conflict of interest.

Acknowledgments

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