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MECHANICAL AND THERMAL PROPERTIES OF WOOD PLASTIC COMPOSITES REINFORCED WITH HEXAGONAL BORON NITRIDE

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Abstract:

Mechanical and thermal properties of injection molded wood plastic composites (WPCs) were prepared from poplar wood flour (50 wt%), thermoplastics (HDPE or polypropylene) with coupling agent (3 wt%), and hegzagonal boron nitride (h-BN, 2, 4, or 6 wt%) nano powder were investigated. The flexural and tensile properties of WPCs significantly improved with increasing content of the h-BN. Unlike the tensile and flexural properties, the notched izod impact strength of WPCs decreased with increasing content of h-BN but it was higher than that of WPCs without the h-BN. The WPCs containing h-BN were stiffer than ones containing wood flour alone i.e., the partial replacement of h-BN with polymer had a synergistic reinforcing effect. The tensile elongation at break values of WPCs increased with the addition of h-BN. The differential scanning calorimetry (DSC) analysis showed that the crystallinity, melting and crystallization enthalpy of WPCs increased with increasing content of the h-BN. The increase in the crystallization peak temperature of WPCs indicated that h-BN was the efficient nucleating agent for on thermoplastic composites to increase the crystallization rate. Based on the findings obtained from the present study, it could be said that the optimum content of h-BN in the WPC for structural applications was 4 wt%.

Key words: *wood plastic composite; hegzagonal boron nitride; mechanical properties; thermal properties.*

INTRODUCTION

The major drawback of wood plastic composites (WPCs) is lower flexural and tensile properties than plywood and oriented strandboard in load bearing structural applications. Enhancing the flexural and tensile properties of WPCs can expand their acceptance in structural applications. There are two major ways to improve mechanical properties of the WPCs. One way for improving the mechanical properties of WPCs is the treatment with coupling agents such as maleic anhydride grafted polyolefins and silanes (Lu *et al.* 2000). Another way is the incorporation of nanoscale reinforcing fillers, such as nano-clay and carbon nanotube (Yeh *et al.* 2005, Kordkheili *et al.* 2013). A possible solution to enhance the mechanical properties of natural fiber reinforced composites (including WPCs), can be represented by the hybridization with fillers. Wood flour and boron nitride (h-BN) reinforcing fillers can play an important role in the manufacture of high performance WPC for structural applications.

Boron nitride is a ceramic material that is isoelectronic with carbon (Daga 2008). Much like carbon, it exists in multiple allotropic forms. The most common structures of boron nitride are the hexagonal form (h-BN, analogous to graphite) and the cubic form (cBN, analogous to diamond. The h-BN is a layered material with a graphite-like structure in which planar networks of h-BN hexagons are regularly stacked, is a promising dielectric. It has strong chemical and thermal stability, low thermal expansion, easily machined-non abrasive and lubricious, low reactivity with metals, easy machinability, excellent thermal shock resistance, improved the wear resistance of polymers and non toxic (Ertug 2013, Raman and Meneghetti 2008, Ziaei *et al.* 2011). Apart from carbon nanotubes, silica, alumina, aluminum nitride, zinc oxide, and organoclay, the h-BN is a soft, lubricious material that can be compounded into plastics with minimal impact on processing equipment (Ziaei *et al.* 2011). Another significant advantage of the h-BN over competing carbon-based fillers is its white colour (Raman and Meneghetti 2008). Consequently, it looks cleaner in a manufacturing environment and also allows considerable freedom to introduce colour in the plastics. Graphite and carbon limit the colour space since filled thermoplastic composites with these materials are invariably black or dark grey.

The studies on polymer/nano filler composites are very common in the literature and being studied frequently. There are a plenty of study on the effect of nanofillers such as nanoclay, SiO_2, and carbon nanotube on the mechanical performance of WPCs (Kordkheili *et al.* 2013, Deka and Maji 2013, Faruk and Matuana 2008). However, the studies on thermoplastics composites containing h-BN are very limited in the literature (Hy *et al.* 2005, Demir 2010). The h-BNs have excellent elastic modulus of 1.22 TPa (Lahiri *et al.* 2010) (similar to CNTs) and are thus a potential candidate as reinforcement for WPCs. Based on an extensive literature review, there is no scientific evidence for the use of h-BN in the WPC. The main goal of present study was to investigate the effect of nano h-BN powder as well as its loading level on the mechanical and thermal properties of wood/PP (polypropylene) and wood/HDPE (high density polyethylene) composites.

EXPERIMENTAL

Materials

Poplar wood (*populus euramericana* I-214) was used as lignocellulosic filler in the thermoplastic composites. Poplar wood having a moisture content of 20-30% based on the oven-dry weight of the wood was processed by a rotary grinder and retained on a 60-mesh screen. The wood flour was dried in a laboratory oven at 100°C for 24 h to moisture content of 1% before the compounding process. Nano h-BN powder was supplied from Bortek Boron Technologies and Mechatronics Inc. Eskisehir, Central Turkey. The h-BN has a specific gravity of 2.27g/cm^3 and melting range of 2700-3000°C. It consists thin plates, which have an average diameter of about 200nm and a thickness of 30nm.

Two types of polymer matrix, PP (MFI/230°C/2.16kg = 5.5g/10min) and HDPE (MFI/230°C/2.16kg = 5.0g/10min), were supplied by a Petrochemical Company in Ukraine and Petkim Petrochemical Corporation in Izmir, Turkey, respectively. Two types of coupling agent were maleic anhydride-grafted PP (MAPP, Optim®P-425, MFI/190°C, 2.16kg = 120g/10min) and maleic anhydride-grafted polyethylene (MAPE, Optim-E156, MAH content: 1.2 wt.%, MFI 190°C/2.16kg = 4.5g/10min, density = 0.95g/cm^3) were supplied by Pluss Polymers Pvt. Ltd. in India.

Preparation of injection molded composites

The wood flour, h-BN nano powder, and plastic granulates with and without coupling agent were processed in a 30 mm co-rotating twin-screw extruder with a length-to-diameter (L/D) ratio of 30:1. The barrel temperatures of the extruder were controlled at 175-190°C. The temperature of the extruder die was held at 200°C. The extruded strand passed through a water bath and was subsequently pelletized. These pellets were stored in a sealed container and then dried for about 3-4 h before being injection molded. The temperature used

for injection molded specimens was 170-190°C from feed zone to die zone. The specimens were injected at injection pressure between 5 and 6 MPa with cooling time about 30s. Finally, the specimens were conditioned at a temperature of 23°C and relative humidity of 50% according to ASTM D 618 (2008). The formulations of the filled thermoplastic composites are presented in Table 1.

Table 1

Composition of the injected molded WPC composite types

WPC type	WPC compositon					
	Wood flour	Plastic type		Coupling agent type		h-BN
		PP	HDPE	(MAPP)	MAPE	
	(wt %)	(wt %)	(wt %)	(wt %)	(wt %)	(wt %)
A	50	47	0	3	0	0
B	50	45	0	3	0	2
C	50	43	0	3	0	4
D	50	41	0	3	0	6
E	50	0	47	0	3	0
F	50	0	45	0	3	2
G	50	0	43	0	3	4
H	50	0	41	0	3	6

PP: Polypropylene. HDPE: High density polyethylene. h-BN: Hexagonal boron nitride.

Property testing

Determination of mechanical properties

The flexural tests were conducted in accordance with ASTM D 790 (2010) using a Lloyd testing machine at a rate of 1.3mm/min crosshead speed. Dimensions of the test specimens were 3.5mm x 13mm x 128mm. The tensile tests were conducted according to the ASTM D 638 (2010). Tensile specimens (dogbone shape (type III)) were tested with a crosshead speed of 5mm/min in accordance with ASTM D638. Seven specimens were tested for the tensile and flexural properties of each composite formulation. The notched izod impact resistance were performed according to ASTM D 256 (2010) using an impact test equipment with 5 Joule hammer. Eight specimens were tested for the izod pendulum impact resistance of each WPC formulation.

Differential scanning calorimetry (DSC) analysis

Melting and crystallization behavior of the composites were studied in a heat-flux type differential scanning calorimeter (DSC, Perkin Elmer DSC 8000) according to ASTM D3418 (2008). Temperature and heat flow calibration of the instrument were performed with high purity indium (In), tin (Sn) and zinc (Zn) metals. The test specimens weighing about 9-10mg in an aluminum crucible were heated up to 200°C with the heating rate of 10°C/min and kept at this temperature for 2min to remove thermal history. Then the specimens were cooled down to 0°C with the cooling rate of 10°C/min by an electrical cooling device to allow the sample crystallize dynamically and kept at this temperature for 2min. Subsequently, the non-isothermally crystallized specimens were re-heated up to 200°C with the heating rate of 10°C/min. All heating-cooling runs in melting and crystallization studies were carried out under nitrogen (N_2) atmosphere at a flow rate of 50ml/min to prevent oxidation of the specimens. Degree of crystallinity (X_c %) was determined from the second melting enthalpy values using the following equation:

$$X_c = \frac{\Delta H_m}{(1-\alpha)\Delta H_m^o} x100 \quad (1)$$

Where, ΔH_m is melting enthalpy of the specimens (J/g), ΔH_m^o is the enthalpy value of melting of a 100% crystalline form of PP (209 J/g) and (1-α) is the weight fraction of polymer into the composite material.

Interfacial morphological analysis

The morphology of tensile fracture surfaces was studied by means of scanning electron microscopy (SEM, JEOL Neo Scope JSM-5000) under an acceleration voltage of 10KV. The test specimens were attached

to an aluminum stub and sputtered with gold to eliminate the electron charging effects. The objective was to get information regarding the h-BN filler dispersion and bonding quality between wood flour and polymer matrix.

Statistical analysis

Analysis of variance (ANOVA) ($p<0.01$) was used to determine the effect of h-BN content on the mechanical and thermal properties of the wood/PP and wood/HDPE composites using SPSS statistical package program. Significant differences among the average values of the composite types were determined using Duncan's multiple range tests.

RESULTS AND DISCUSSION

Mechanical properties

The flexural properties of the WPCs as function of the h-BN content are presented in Table 2. The flexural strength and modulus of the WPCs significantly improved with increasing content of the h-BN. The WPCs containing the h-BN were stiffer than ones containing wood flour alone i.e., the partial replacement of h-BN with polymer had a synergistic reinforcing effect (Table 2). The h-BN more increased the flexural strength than flexural modulus. For example, as 6 wt% h-BN was incorporated into the wood/HDPE, the flexural strength increased by 55% while the flexural modulus increased by 15%. The flexural properties of wood/PP composites were better than those of the wood/HDPE composites. The flexural modulus of wood/PP composites increased from 5260 to 6314MPa as the h-BN content was increased to 6 wt% while it increased from 4978 to 5711MPa for the wood/HDPE composites. Significant differences ($p<0.01$) between some group averages for the flexural strength and modulus values are presented in Table 2.

The flexural modulus in WPCs is mainly function of the modulus of individual component. The incorporation of rigid and stiff reinforcement into the composition significantly enhanced the flexural modulus and strength of the WPCs. The flexural modulus of h-BN (1.22TPa (Lahiri *et al.* 2010)) was considerably higher than wood flour, polyproylene, and HDPE, respectively. Hence, the wood/PP and wood/HDPE with h-BN had higher flexural modulus values than the WPCs without the h-BN. The flexural modulus in WPCs is mainly function of the modulus of individual component. The incorporation of rigid and stiff reinforcement into the composition significantly enhanced the flexural modulus and strength of the WPCs.

Table 2

Mechanical Properties of Unfilled and h-BN Filled WPCs

WPC type[1]	Density (g/cm^3)	Mechanical properties					
		Flexural strength (MPa)	Flexural modulus (MPa)	Tensile strength (MPa)	Tensile modulus (MPa)	Elongation at break (%)	Notched izot impact (J/m)
A	1.06 (0.002)	47.1 (3.5)a[2]	5260 321)ab	28.2 (3.0)ad	5435 (443)a	1.48 0.19)ab	20.4 (0.8)a
B	1.06 (0.008)	58.9 (5.6)be	5543 (610)bc	36.9 (4.2)b	5647 (521)a	2.05 0.22)de	29.0 (1.1)e
C	1.07 (0.005)	69.4 (3.3)c	6058 (204)de	42.9 (2.0)c	6491 639)cd	2.26 (0.17)ef	24.5 (1.2)bc
D	1.08 (0.007)	73.9 (3.0)d	6314 (140)e	43.7 (1.5)c	6557 (302)d	2.34 (0.37)f	23.1 (0.9)b
E	1.07 (0.003)	44.1 (2.6)a	4978 (233)a	25.5 (2.4)a	5255 (128)a	1.23 (0.08)a	20.8 (1.1)a
F	1.08 (0.003)	55.3 (2.0)b	5204 (578)ab	30.9 (2.4)de	6184 (792)c	1.55 (0.30)b	28.0 (0.5)ef
G	1.09 (0.007)	62.7 (3.7)e	5480 (362)bc	31.5 (1.9)de	6539 (360)d	1.75 (0.20)bc	26.3 (0.4)df
H	1.12 (0.004)	68.4 (3.1)c	5711 (308)cd	33.0 (2.4)e	6682 (275)d	1.89 (0.09)cd	25.7 (1.1)cd

[1] See Table 1 for WPC formulation.
[2] Groups with same letters in column indicate that there is no statistical difference ($p<0.01$) between the specimens according to Duncan's multiply range test. The values in the parentheses are standard deviations.

The flexural modulus of h-BN (1.22TPa) was considerably higher than wood flour, polypropylene, and HDPE, respectively. Hence, the wood/PP and wood/HDPE with h-BN had higher flexural modulus values than the WPCs without the h-BN. These results were good consistent with previous studies. For example, Kordkheili *et al.* (2013) reported that flexural modulus of wood flour/LDPE composites increased from 1900 to 4500MPa as 3 wt% carbon nanotubes were incorporated into the WPC. Adding fillers to a polymer restrains the movements of its chains, thus increasing the stiffness. Similar results have been reported by Deka and Maji (2013), who studied the properties of polymer/wood flour/nanoclay/SiO_2 composites. They reported that the properties were further improved after the incorporation of clay and SiO_2. In other study, Faruk and Matuana (2008) reported that

mechanical properties of WPCs improved with the incorporation of nanoclay. However, in our study the increment ratios in the flexural strength and modulus of the wood/PP and wood/HDPE composites decreased with increasing content of the h-BN. This result was also observed in previous studies (Deka and Maji 2013, Tabari and Khademieslam 2012) and explained by the fact that the agglomeration of the nanoparticles resulted in a decrease in mechanical properties of the composites.

The tensile modulus and strength of the WPCs were significantly improved by the incorporation of h-BN. The tensile strength of wood/PP composites increased from 28.2 to 43.7MPa as the 6 wt% was added into the WPC while it increased from 25.5 to 33.0MPa for the wood/HDPE composites. The increment ratio in the tensile strength values of the WPCs was found better than that in the tensile modulus of wood/HDPE composites. This showed the h-BN more increased the tensile strength than the modulus. The incorporation of 6 wt% h-BN into the wood/PP and wood/HDPE composites resulted in the tensile modulus increments of 20.6% and 27.2%, respectively. This behaviour has been described in similar studies and explained by the reduction of polymer chains mobility in the presence of the filler (Ayrilmis and Kaymakci 2013, Ayrilmis *et al.* 2013). The improvement in tensile properties of WPCs was also due to the improved adhesion between the wood flour and polymer. In the WPCs made with compatibilizing agent, the interfacial bonding between the filler and matrix polymer is strong, and the fracture occurred in the filler itself. This means that the stress is well propagated between the filler and the matrix polymer in the composite incorporating the compatibilizing agent, causing it to have a higher tensile strength and modulus. As shown in the SEM micrograph of the tensile fracture surface of the WPC specimen (Fig. 1), the h-BN nano particles were good dispersed in the polymer matrix, suggesting that the tensile strength of WPCs is high. The tensile modulus of wood/PP and wood/HDPE composites significantly increased as the content of h-BN increased up to 4 wt%, but further increment in the h-BN content increased little. This result revealed that the effectiveness of the coupling agent on the tensile modulus decreased with increasing content of the h-BN as well as agglomeration of the h-BN. The tensile elongation at break values of the wood/PP and wood/HDPE composites increased with the addition of the h-BN. This was attributed to the h-BN's soft and lubricious structure. The elongation at break values was significantly increased by addition of the 2 wt% h-BN into the WPC but it slightly increased beyond 2 wt% h-BN.

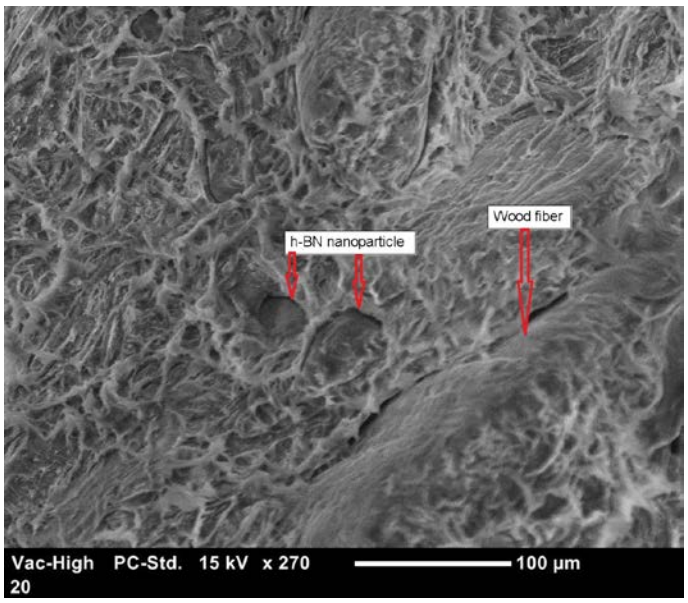

Fig. 1

The SEM micrograph of wood/polypropylene composite containing 2 wt% h-BN nano particle.

The impact strength of WPCs with h-BN was higher than that of the WPCs without the h-BN which. However, the impact strength of the WPCs with the h-BN decreased with increasing h-BN content). The impact strength of wood/HDPE composites with the h-BN were higher than that of wood/PP composites with the h-BN, expect for the addition of 2 wt% h-BN. Unlike the tensile and flexural modulus, the izod impact strength of WPCs decreased with the increase in the h-BN content. The results showed that the optimum content of h-BN for the highest impact strength of wood/PP and wood/HDPE composites was 2 wt%. The decreases in impact strength at higher h-BN content levels were probably due to the formation of h-BN agglomeration and the presence of un-exfoliated aggregates.

DSC analysis

The second melting enthalpy (ΔH_m) and melting peak temperature (T_m) of unfilled HDPE and PP composites decreased with the addition of wood flour (Fig. 2). However, the ΔH_m and T_m values of WPCs increased with the incorporation of h-BN. For example, the T_m of wood/PP composites increased from 164.3°C 166.2 as 6 wt% h-BN was added into the WPC composition. A similar increment in the T_m value was also observed for the wood/HDPE composites (128.4 to 130.3°C). The crystallization peak temperature (T_c) of the WPCs with the h-BN was higher than that of ones without h-BN, indicating that the h-BN which was a conductive fillers could act as nucleating agents (Fig. 3). The h-BN more increased the peak T_c of wood/PP composites than the wood/HDPE composites (Table 3). The increase in the peak T_c showed faster crystallization of polymer chains upon cooling. Similar results were reported in previous studies (Liu *et al.* 2002, Wang *et al.* 2009). The addition of nucleating agents provides sites for the initiation of crystallization, which increases the crystallization temperature (Maier and Calafut 1998). It could be said that the h-BN functioned in a similar manner with nucleating agents.

Table 3

The Results of Differantial Scanning Calorimetery (DSC) Analysis

WPC type[1]	Second melting enthalpy (ΔH_m)	Second melting peak temperature (T_m) (°C)	Crystallization peak temperature T_c (°C)	Crystallization enthalpy (ΔH_c)	X_c Degree of crystallinity (%)
Unfilled PP composite	79.2	166.9	121.7	91.8	37.9
A	35.6	164.3	121.4	37.5	34.1
B	44.2	164.9	126.5	46.9	44.1
C	45.7	165.6	127.4	47.0	47.5
D	46.2	166.2	128.3	58.9	50.2
Unfilled HDPE composite	128.1	130.8	119.7	105.3	43.7
E	60.8	128.4	118.8	59.5	41.5
F	64.5	128.9	119.9	60.7	45.9
G	66.6	129.5	120.7	68.7	49.4
H	69.3	130.3	121.4	70.1	53.3

[1]See Table 1 for WPC formulation.

The crystallization enthalpy (ΔH_c) of unfilled PP and HDPE composites is significantly decreased with the addition of wood flour (Table 3). The ΔH_c is the required energy to freze a material. The lower ΔH_c of WPCs without h-BN as compared to the unfilled PP and HDPE composites shows that wood filler acts as nucleating agents and allows chains packed easily around the particles. However, the ΔH_c of the WPCs increased with increasing h-BN content. This indicated that the h-BN induced the formation of crystals as heterogeneous nucleation sites, thus, promoting the crystallization rate and degree of crystallinity in the WPCs. The crystallinity (X_c) of the unfilled PP (37.9%) and wood/HDPE (43.7%) composites decreased with the addition of the wood flour. The X_c values of wood/PP and wood/HDPE were lower than that of the unfilled PP and HDPE, which were 34.1% and 41.5%, respectively. However, the addition of h-BN significantly increased the X_c of the WPCs. For example, as the content of h-BN increased to 6 wt% in the wood/PP composite, the X_c increased to 50.2%. The highest increment ratio in the X_c was found for the wood/polyproylene and wood/HDPE composites with 2 wt% h-BN (Table 3). The X_c influences the mechanical properties of polymer composites (Nurul and Mariatti 2009) because the crystalline region acts as a physical crosslink that enhances the tensile strength of the filled thermoplastic composite. This was also confirmed by the results of tensile strength and modulus.

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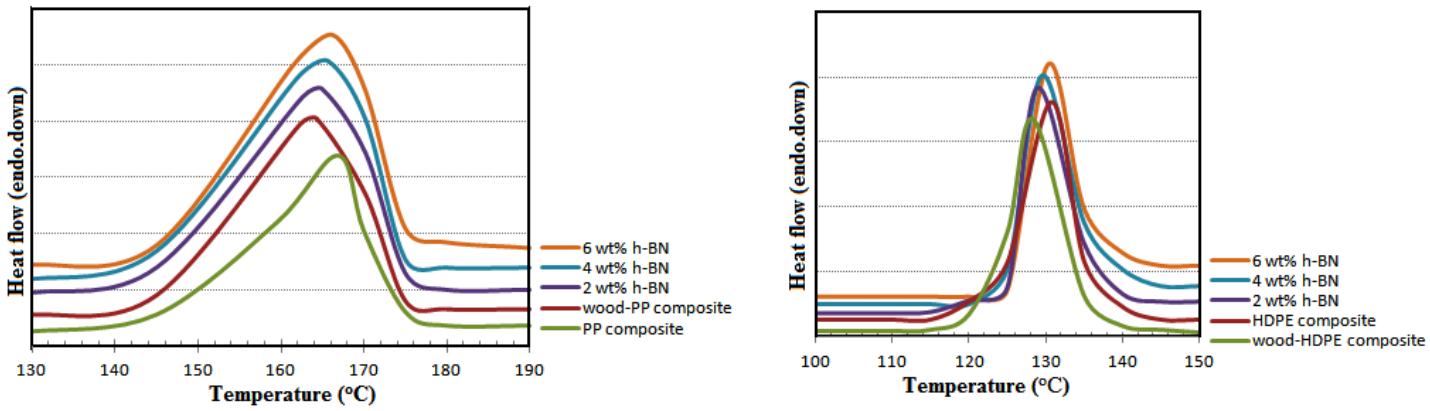

Fig. 2

The second melting (peak) temperatures of unfilled thermoplastics and WPCs containing nano h-BN powder (see Table 1 for formulation).

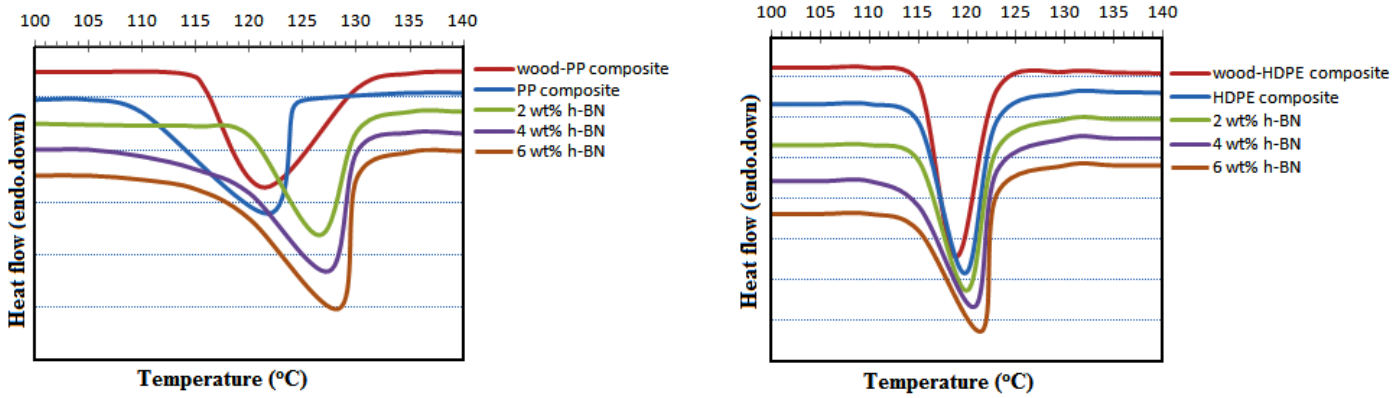

Fig. 3

The crystallization peak temperatures of of unfilled thermoplastics and WPCs containing nano h-BN powder (see Table 1 for formulation).

CONCLUSIONS

The results of this study showed that the flexural and tensile properties of WPCs significantly improved with increasing content of the h-BN nano powder. Unlike the tensile and flexural properties, the notched izod impact strength of WPCs decreased with increasing content of h-BN but it was higher than that of WPCs without the h-BN. The WPCs containing the h-BN were stiffer than that containing wood flour alone i.e., the partial replacement of h-BN with polymer has a synergistic reinforcing effect. The tensile elongation at break values of WPCs increased with the addition of h-BN. The DSC analysis showed that the melt crystallization enthalpies of the WPCs increased with increasing h-BN content. The increase in the T_c indicated that h-BN was the efficient nucleating agent for on thermoplastic composites to increase the crystallization rate. Based on the findings obtained from the present study, it could be said that the optimum content of h-BN in the WPC for structural applications was 4wt%.

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