

# Effects of nano-graphene on the physico-mechanical properties of bagasse/polypropylene composites

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**Abstract** This study was aimed at identifying the best approach of incorporating nano-graphene (NG) into bagasse/polypropylene composites to enhance their mechanical and physical properties. The composites with different NG contents were produced by melt compounding in a twin-screw extruder and then by injection molding. The mass ratio of the bagasse flour (BF) to polymer was 15/85 and 30/70 (w/w). Water absorption, thickness swelling, tensile strength, bending characteristics, impact strength and morphological properties of the produced composites were evaluated. In general, applying NG would improve mechanical properties of the composites. The mechanical test results indicated that when only 0.1 wt % of NG was added, tensile and flexural properties reached their maximum values, while the notched impact was slightly decreased. The composites containing 0.1 wt % NG and 30 wt % BF exhibited the highest tensile, flexural and notched impact strength values. Although incorporating NG into the polymer matrix effectively improves mechanical properties, this improvement comes at proper nanofiller loading (0.1 wt %). Addition of NG almost did not change the average water uptake and thickness swelling, compared to the control (without NG) samples. Morphological study confirmed that high contents (0.5–1 wt %) of NG were easily agglomerated. Thermal analysis showed slight increase in thermal stability of WPCs after incorporation of NG particles. In addition, it was found that the effect of BF was notable in material properties of the composites.

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## Introduction

Nanocomposites with exfoliated layered silicate fillers have been investigated as early as 1950, but significant academic and industrial interest in nanocomposites came nearly 40 years later following a report from researchers at Toyota Motor Corporation that demonstrated large mechanical property enhancement using montmorillonite as filler in a Nylon-6 matrix [1]. Polymer nanocomposites show substantial property enhancements at much lower loadings than polymer composites with conventional micron-scale fillers (such as glass or carbon fibers), which ultimately results in lower component weight and can simplify processing. Moreover, the multifunctional property enhancements made possible with nanocomposites may create new applications of polymers [2, 3]. On the other hand, nanoscience and nanotechnology have opened a completely new way to develop wood plastic composites (WPCs) [4]. The interest in producing WPC materials with nano-sized filler or reinforcement, i.e., nanocomposites, has grown tremendously in recent years [5]. Nanotechnology is a very promising field for improving the physical and mechanical properties of WPCs using nano-sized fillers. These improvements include high modulus; increased tensile strength and thermal stability; decreased gas permeability, improved flame retardant properties and decrease in water absorbance; and increased biodegradability of polymers [6, 7]. The effects of the nano-filler on the composite materials depend on its size, aspect ratio, hybrid morphology, and dispersion quality.

Graphene has attracted great attention in recent years owing to its exceptional mechanical, thermal, optical, and electrical properties [8–10]. It is two-dimensional allotrope of carbon, formed by single layer of carbon atoms, bonded by  $sp^2$  orbitals into hexagonal two-dimensional crystal lattice. One of the most promising applications of this material is in polymer nanocomposites, polymer matrix composites which incorporate nano scale filler materials. To date, most work in the area of graphene-based polymer nanocomposites has focused on property modification using graphene [11–14]. This emphasis can be partly attributed to the intrinsic properties of the graphene, which result in remarkable property improvements at low graphene concentrations [9]. The superior properties of graphene compared to polymers are also reflected in graphene/polymer nanocomposites, which show superior mechanical, thermal, gas barrier, electrical, and flame retardant properties compared to the neat polymer [1, 15, 16]. It was also reported that the improvements in mechanical and electrical properties of graphene based polymer nanocomposites are much bigger in comparison to that of nanoclay or other carbon filler-based polymer nanocomposites [17, 18]. Liang et al. [19] demonstrated a solution-processed poly(vinyl alcohol) (PVA)–graphene oxide (GO) nanocomposites and a 76 % increase in tensile strength and 62 % improvement of Young's modulus were achieved at only 0.7 wt % of GO. Rafiee et al. [20] compared reinforcement efficiency of graphene with single- or multi-walled carbon nanotube

(CNT), fully exemplifying the superiority of graphene platelets over CNT at a nanofiller weight fraction of 0.1–0.002 wt %.

This work is aimed at analyzing the influence of using graphene nanoplatelets (NG) on the physico-mechanical properties of bagasse flour (BF) filled composites. In the present work, the fracture mechanism of the tensile fractured samples has also been studied using scanning electron microscopy (SEM).

## Materials and methods

### Materials

#### *Lignocellulosic material*

Raw bagasse stalks were obtained from Khuzestan Cultivation and Industry Co. (Iran). The stalks were depithed and cut into segment of 10 cm length and washed with water to remove soil and dust, and then air-dried. To reduce extractives effects, the lignocellulosic material was treated with water at 50 °C for 48 h. Consequently, the stalks were ground with a Thomas-Wiley miller to fine powder of 40-mesh size, and then oven-dried and stored in sealed plastic bags for subsequent use.

#### *Polymer matrix*

Injection molding grade polypropylene (PP), with trade name V30S, was supplied by Arak Petrochemical Co. (Iran). The PP was in the form of pellets with a melt flow index of 18 g/10 min and density of 0.92 g/cm<sup>3</sup>.

#### *Coupling agent*

The coupling agent used in the preparation of composites was maleic anhydride grafted polypropylene (MAPP), which was supplied by Arkema (France). MAPP was in the form of pellets.

#### *Nanoparticle*

The NG powder (grade AO-4) was obtained from the Graphene Supermarket Co. (USA). The specific surface area was <15 m<sup>2</sup>/g, and the purity was 99.2 wt %. The average thickness and lateral size of NG were about 12 nm and 4.5 μm, respectively.

### Preparation of composites

Formulation of the mixes and the notations used for the respective mixes prepared are given in Table 1. Materials were fed into the co-rotating twin-screw extruder (Collin) using pellet feeder and powder. NG, PP and MAPP were mixed in a beaker and fed into the pellet feeder. Consequently, powder material (BF) was fed into

**Table 1** Formulations of the used composites

Code	BF (wt %)	PP (wt %)	MAPP (wt %)	NG (wt %)
A0	15	82.00	3	0.00
A1	15	81.90	3	0.10
A2	15	81.75	3	0.25
A3	15	81.50	3	0.50
A4	15	81.25	3	0.75
A5	15	81.00	3	1.00
B0	30	67.00	3	0.00
B1	30	66.90	3	0.10
B2	30	66.75	3	0.25
B3	30	66.50	3	0.50
B4	30	66.25	3	0.75
B5	30	66.00	3	1.00

powder feeder. The barrel temperatures of the extruder were controlled at 155, 165, 170, 175, 175, and 180 °C from zones 1 to 6, respectively. The screw speed was 50 rpm and the melt pressure was about ten bars. The extrudates were first cooled down by immersing in a water bath and then grounded. The granules were injection molded using an injection molding machine (model EM80, Aslanian Co., Iran) to produce standard ASTM samples. The temperature used for injection molding was 175 °C from feeding zone to die zone.

### Mechanical property

Specimens were conditioned at a temperature of 23 °C and relative humidity of 50 % for at least 40 h, according to ASTM D618 prior to any test evaluations. ASTM D638 Type I tensile bars were tested for tensile modulus, strength and elongation at break using an Instron universal testing machine (model 4486). The speed of testing was 5 mm/min.

Three-point flexural tests were carried out according to ASTM D790 specification on an Instron (model 4486). The specimens were tested at crosshead rate of 10 mm/min at room temperature. The flexural strength  $\sigma_{fM}$  and the modulus of elasticity in bending  $E_b$  were calculated using the following Equations:

$$\sigma_{fM} = \frac{3PL}{2bd^2} \quad (1)$$

$$E_b = \frac{L^3 m}{4bd^3} \quad (2)$$

where  $P$  is the maximum applied load,  $L$  is the length of the support span,  $m$  is the slope of the tangent of the load to deflection curve, and  $b$  and  $d$  are the width and thickness of the specimen bars, respectively.

Notched Izod impact tests were conducted with a Santam pendulum impact tester (model SIT-20D), that conformed to ASTM D256. Specimen bars were obtained by cutting the flexural specimens in half (to 63.5 mm × 12.7 mm × 10 mm) and then notching. It is to be noted that the notched side was facing the pendulum. All the reported values for the mechanical tests were the average values of five specimens.

### Morphological study

Studies on the morphology of the composites were carried out using a scanning electron microscope (SEM). SEM micrographs of the fracture surfaces of the specimens after tensile test were taken using SEM model WEGA-II TESCAN. The specimen was coated with a thin film (25 nm) of gold to avoid electrical charge accumulation during the examination and then analyzed at an accelerating voltage of 7 kV.

### Dimensional stability tests

The thickness swelling and water absorption tests were conducted in accordance with ASTM D570. Before testing, the weight and thickness of each specimen were measured. Conditioned samples of each composite were soaked in distilled water at room temperature for two different time intervals, namely 2 and 24 h. At predetermined time intervals, the specimens were removed from the distilled water, the surface water was blotted off with paper towels, and their wet mass and thickness were determined. Each value obtained represented the average of five samples.

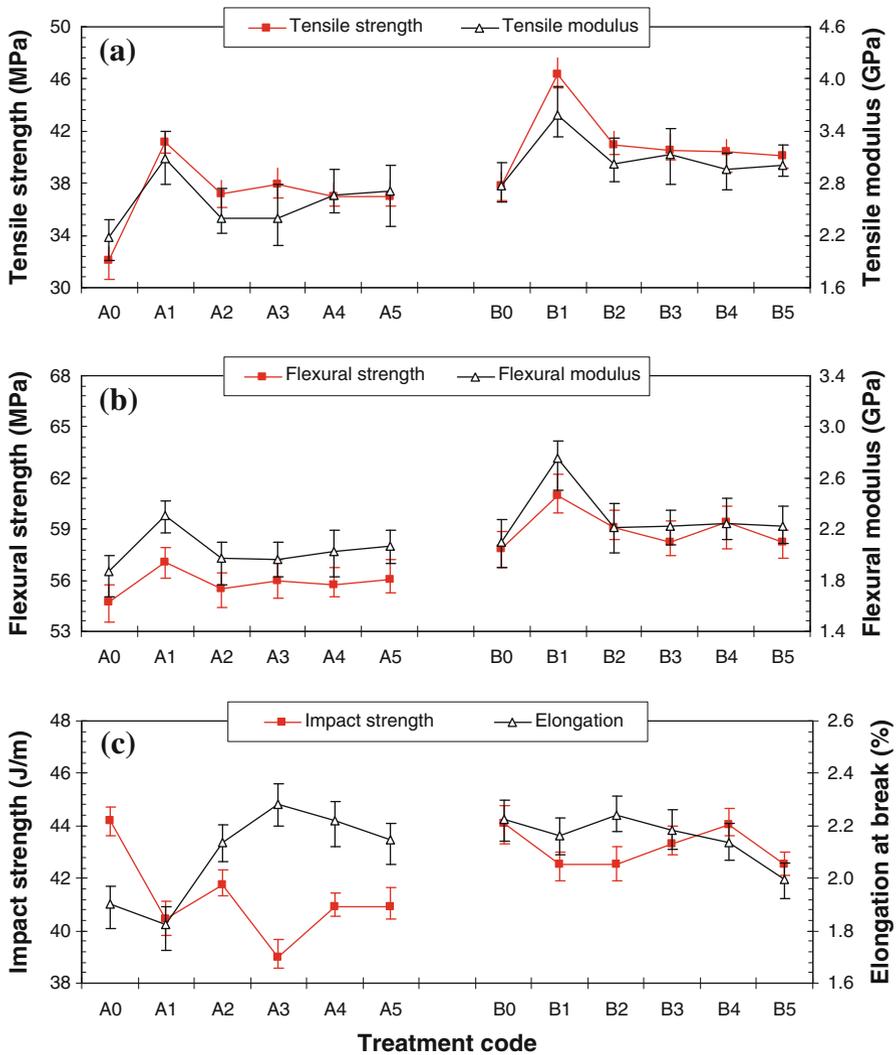
### Thermogravimetric analysis (TGA)

The thermal stability of the graphene filled or unfilled BF/PP composites were tested using a Polymer Laboratories Thermogravimetric Analyzer. Samples were measured in nitrogen atmosphere with a heating rate of 10 °C/min from room temperature to 600 °C.

## Results and discussion

### Tensile properties

The tensile strength and modulus of the composites as functions of the filler type and content are presented in Fig. 1a. It is observed that both tensile strength and modulus of the WPCs increased after incorporation of NG. Samples filled with 0.1 wt % NG showed the highest values among the other types of specimens. The tensile strength values were observed to be 32 and 38 MPa for composites made without NG (A0 and B0), while maximum tensile strength values were approximately 41 and 46.3 MPa for composites filled with 0.1 wt % NG (A1 and B1). Like tensile strength, tensile modulus of the samples increased by 29 to 41 % (Fig. 1a).



**Fig. 1** Comparison of **a** tensile, **b** flexural and **c** impact properties of the composites

This result is consistent with the general observation that the introduction of nano-sized particles into a polymer matrix increases its tensile properties [21]. The enhancement is easily explainable with the large aspect ratio and high interfacial contact area of the NG particles. As mentioned earlier, the NG is much stiffer than polymer matrix and as a result it adds stiffness to the composites. It was also observed that at a fixed level of BF, both tensile and flexural strength increased with increasing NG content up to 0.1 wt %. However, it is clearly illustrated that further addition (more than 0.1 wt %) of NG can not considerably improve tensile properties. This could be explained by the agglomeration of nanoparticles. SEM

study supported the agglomeration of NG nanoparticles. The importance of dispersion and its effect on mechanical properties of composites made with nanoparticles has been discussed in many research works [22, 23]. To achieve an effective reinforcement by adding nanoparticles, NG should be dispersed uniformly into the polymer matrix.

### Flexural properties

The flexural strength and stiffness of the composites measured using 3-point bending tests are shown in Fig. 1b. The flexural strength exhibits a similar trend to the tensile strength, although less variation is observed in the flexural strength with different formulations than the tensile strength. For example, the inclusion of 30 wt % BF improved the flexural strength by almost 6 %, and the addition of NG increased the strength by an additional percentage varying from 4 to 6.8 % depending on the composition. Similarly, composites made with 0.1 wt % NG (A1 and B1) showed the maximum strength and modulus of flexural, whereas composites without NG (A0 and B0) exhibited the lowest properties. The flexural modulus in composites is mainly a function of the modulus of individual component. Flexural modulus of NG is considerably higher than BF and PP, respectively. Increased flexural properties for 0.1 wt % NG loading are attributable to the high stiffness of NG with high aspect ratio. As evident from Fig. 1b, with increase in NG contents from 0.1 to 1 wt %, the flexural properties are moderately decreased. Sheshmani and Amini [10] reported that the modulus of composites at higher nanoparticles loading might not be increased because of the NG agglomerate. One of the most important parameters in producing composites is NG dispersion in the matrix. Nanoparticles aggregation is harmful to physical and mechanical properties of the resultant nanocomposites. Use of a GO is the best method to prevent the NG from aggregating together, and improves good dispersion of NG in nanocomposites [7].

### Izod impact strength

The impact resistance is defined as the energy lost by the pendulum during the breakage of the sample. The impact properties of the various composites are illustrated and compared in Fig. 1c as notched values. With increasing NG contents, Izod impact strength slightly decreased. As shown, the gradual decrease of notched impact strength was due to the incorporation of NG from 0.1 to 1 wt %. It can be easily seen that the samples filled with NG had lower impact strengths in comparison with the control samples (without NG). The lowest increment in impact strength was observed with the addition of 0.5 wt % of NG, where the impact strength decreased by 33 % compared with that of neat PP. This was expected because the presence of NG in the PP matrix provides points of stress concentrations, thus providing sites for crack initiation. Similar results have been reported by Sheshmani et al. [7], who studied the properties of polymer/wood flour/graphene nanocomposites. Their data showed that the impact of a polymeric material is remarkably decreased when nanocomposites are formed with NG.

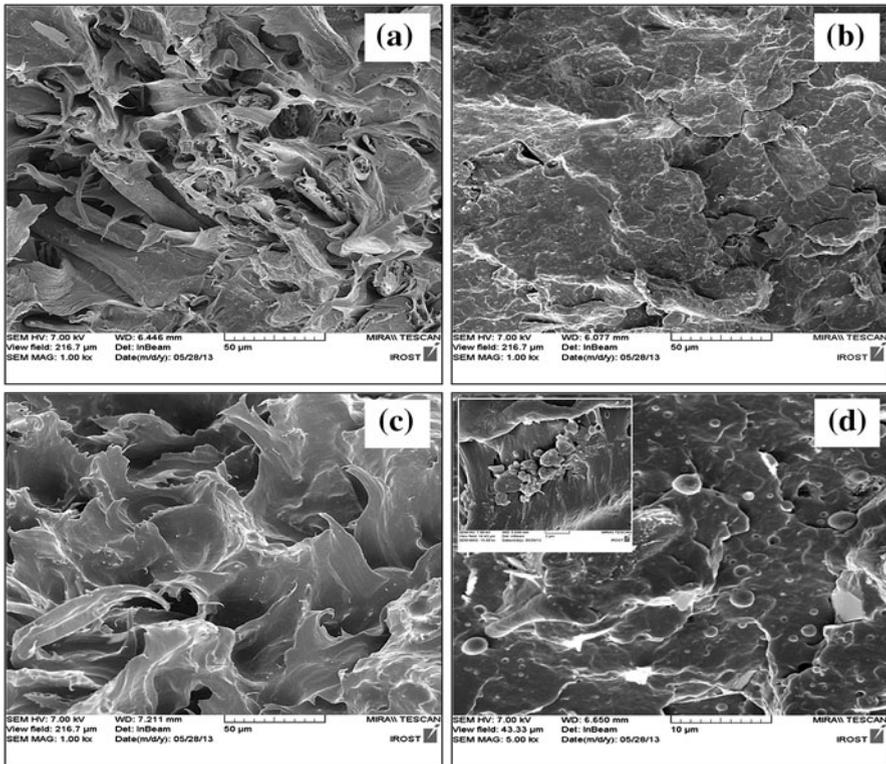
The significant increase in the impact strength of the filled composites, in comparison with those of the unfilled composites (samples A0 and B0), was an indication that the BF filler is stiffer than the PP matrix. The fact that the impact strength decreased with increasing the lignocellulosic material content was in agreement with the trend observed by previous studies [24, 25]. Ashori et al. [24] studied the effect of bagasse fiber loading in the polypropylene matrix. They reported that the Izod impact strength increased with increasing in bagasse loading.

### Elongation at break

The breaking strain of the WPCs is shown in Fig. 1c. Percent elongation at break of the composites increases upon the addition of NG. After the addition of 0.5 wt % of NG, elongation at break of the composites increases by 20 % (from 1.90 to 2.28 %) as compared with the control composite (A0). This was attributed to the high aspect ratio of graphene and the interaction between graphene and the polymer matrix, which restricts the movement of the polymer chain. This is a common tendency that has been reported with GO as well [26]. The addition of 30 wt % BF in the composites increased the stiffness and brittleness, however, reduced the elongation at break.

### Morphological characteristics

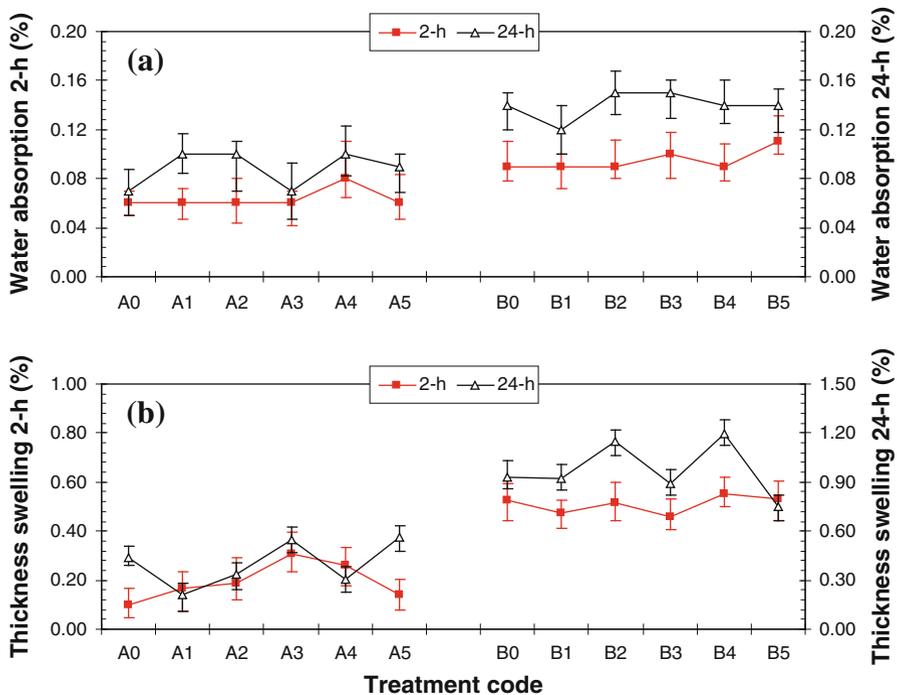
The significant improvements in mechanical properties of the composites with incorporation of NG were further supported by SEM micrographs. Through SEM study, the distribution and compatibility between the fillers and the matrix could be observed. Figure 2a corresponds to WPC without NG (control sample), showing some evidence of fiber pull-out from matrix. Therefore, when stress is applied it causes the fibers to leave the matrix easily and makes micro voids. As seen from Fig. 2b (sample type A1), there is no separation of the fibers from the matrix and a very good interaction between the components can be inferred from the image. The strong adhesion that is observed at the interface has been already discussed in mechanical properties of the composites and is related to encapsulation of fibers in the PP matrix, which causes strong bonding. The surface of composites containing 1 wt % NG and 15 wt % BF (type A5) is depicted in Fig. 2c. There are some cavities in WPC that can absorb water and/or reduce mechanical properties. As illustrated in Fig. 2d (sample type B5), high content of NG was easily agglomerated, which is the characteristic of this nanofiller. In addition, Fig. 2d shows the position of agglomerated nanoparticles in the composites with 1 wt % NG. The presence of these agglomerates results in the generation of flaws and subsequent creation of voids between the filler and the matrix polymer. This causes the mechanical properties of the composites to be reduced, as compared with the composites filled with lower content of NG. On the other side, there are some micro voids and holes, which accelerate the penetration of water into the deeper part of composites, causing higher water absorption and thickness swelling.



**Fig. 2** SEM micrographs of the fractured surfaces of various composite types: **a** A0, **b** A1, **c** A5 and **d** B5

### Water uptake

The results of water absorption and thickness swelling of composites with different percentages of NG and BF loading are shown in Fig. 3a. It is well known that neat PP does not absorb moisture due to its hydrophobic nature, indicating that moisture is absorbed by the hydrophilic woody material in the composite as well as voids and micro-gaps at the interface. The value of water uptake was suddenly increased after the addition of 30 wt % BF to the blend. The hydrophilic nature of BF caused an increase in the water uptake. As BF content was constant (type A or B) in all blends, the different water absorptions among all the produced composites can be attributed to the role of NG. The water uptake was decreased after the addition of NG. WPCs loaded with 1 wt % showed lowest water uptake followed by WPCs with 0.1 wt %. The graphene layers provided tortuous path and increased the barrier property for water transport [26]. According to Das et al. [27], initially, water saturates the cell wall (via porous tubular and lumens) of the fiber, and next, water occupies void spaces. Since composite voids and the lumens of BF were filled with NG, the penetration of water by the capillary action into the deeper parts of composite was prevented. Another reason for less water absorption could be the hydrophobic and



**Fig. 3** Water absorption and thickness swelling of the composites after 2 and 24 h

water repelling nature of NG surface that tends to immobilize some of the moisture, which inhibits the water permeation in the polymer matrix. Both the mentioned mechanisms may suggest that the water absorption has occurred in the surface layer.

### Thickness swelling

Figure 3b depicts thickness swelling of the composites after 2 and 24 h immersion in distilled water. It can be seen that the composite without NG exhibited the highest thickness swelling values among other samples. Like water uptake results, the composites containing NG showed less thickness swelling percentage as compared with those made without it. The trend and explanation of thickness swelling of different samples were similar to those of samples taken for water uptake study. Contrary to previous report [7], low or high content of NG did not significantly decrease the thickness swelling. This conflict could be explained by the agglomeration of NG particles, which was supported using SEM images.

Based on the graph (Fig. 3b), it also can be seen that the thickness swelling values of composites increase with an increased water exposure time. By increasing the exposure time of composites to water, a significant amount of water is absorbed resulting in the swelling of the fiber. The swelling of the fiber places stress on the surrounding matrix and leads to micro cracking which would eventually cause the composite to fail catastrophically. As a consequence, the fiber–matrix adhesion is

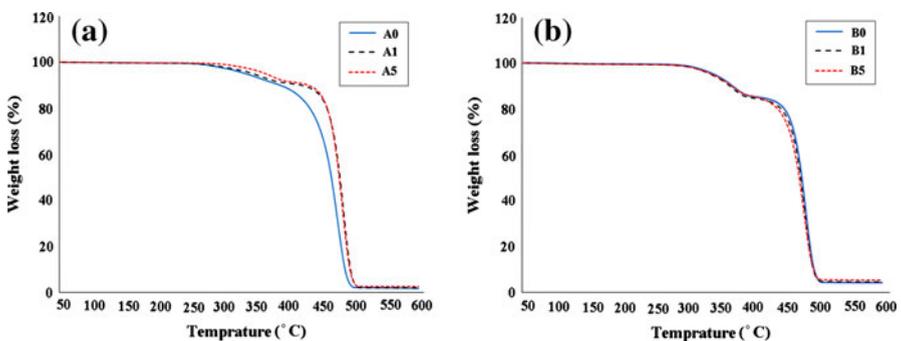
weak and the dimensional stability of composites particularly for outdoor applications will be greatly affected [28].

### Thermal stability

To show the effect of NG on the thermal stabilities of composites, the main TGA results were summarized in Table 2, in which the initial decomposition temperatures at weight loss were displayed. It can be seen from Fig. 4 that the thermal degradation curves can be divided into two stages: the first stage is from room temperature to about 400 °C and the second stage is for above 400 °C. In the first stage, the sharp loss of weight was because of the degradation of the BF; the NG filled composites had lower degradation temperatures both in 15 and 30 wt % BF composites. In the second stage where mainly polymer degradation occurs, the above two composites degraded at a higher temperature, especially sample A5 (composite containing 15 wt % BF and 1 wt % NG) had the highest thermal stability. Additions of the fillers, in general, can improve the thermal stabilities of the resulted polymer composites to some extent [10]. TGA results show that NG filled composites present slightly higher thermal stability than those of the control samples, and incorporations of NG increase the initial decomposition temperatures at 5 % weight loss by about 10–20 °C or even more. Sheshmani et al. [7] observed an increase in thermal stability of WPCs after incorporation of NG particles.

**Table 2** Temperatures of decomposition in °C at different weight loss (%)

Weight loss	5 %	10 %	20 %	40 %	60 %	80 %	97 %
A0	332	383	427	453	465	475	489
A1	344	416	453	468	477	484	497
A5	362	425	453	467	475	483	498
B0	336	363	440	462	471	479	495
B1	332	359	436	460	470	478	553
B5	333	361	431	457	468	477	588



**Fig. 4** TGA curves of the composites **a** with 15 wt % BF and **b** with 30 wt % BF

## Conclusions

In this study, the effects of NG (as reinforcing agent) as well as BF on some physical, mechanical and morphological properties of composites were investigated. The physical and mechanical test results indicated that NG loading and BF agent content have considerable influences on properties of WPCs. The addition of NG had a positive effect on tensile and flexural properties due to the high aspect ratio and large surface area of NG. Compared with the control samples, the composite filled with 0.1 wt % NG and 30 wt % BF (type B1) had 22.5 % higher tensile strength, 29 % higher tensile modulus, 6.8 % higher flexural strength, and 30 % higher flexural modulus, respectively. The elongation at break of the nanocomposites gradually increased as compared to control samples. At higher percentage of NG loading, the effective agglomeration of the nanoparticles resulted in decrease in mechanical properties. The SEM images support the mechanical results of the article. Morphological study also showed that high contents (0.5–1 wt %) of NG were easily agglomerated. This caused the mechanical properties of the composites to be reduced. In addition, there are some distinct cavities between the PP and BF, indicating poor adhesion, but a fewer pulled-out traces on the fracture surfaces of the test samples including 0.1 wt % NG were seen, due to the stronger interfacial bonding. Besides, BF had a significant effect on the studied mechanical and physical properties. In general, it could be concluded that the use of small amount of NG (0.1 wt %) has been an attractive approach for enhancing the mechanical and physical properties of the composites. However, a homogeneous dispersion of nanoparticles in a polymer matrix must be fully accomplished to achieve those improvements. The water absorption of composites increased with an increase in soaking time. Moreover, increasing amounts of BF in composites increased the water absorption percentage of the composites. This is due to the highly hydrophilic nature of the BF owing to the free hydroxyl group present in the cellulose and lignin structures. Despite of high water resistance nature of NG and filling of the composite voids by the nanoparticles, with increase in the amount of NG, water absorption and thickness swelling of the composites were almost unchanged. The thermal degradation behavior of the composites was characterized with TGA. The degradation temperatures slightly shifted to higher values after the addition of NG. However, addition of 30 wt % BF showed more effect on the thermal degradation when compared with NG.

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