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Investigation of flexural properties of hexagonal boron nitride added thermoplastic composites

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ABSTRACT

In this study, 0, 0.5 and 2 wt% hexagonal boron nitride (hBN) were added to 30 wt% short glass fiber reinforced polyamide 66 (PA 66) matrix (GF30) to fabricate the thermoplastic composite materials. The hBN additives were applied by coating of granules of GF30 materials. The hBN coated thermoplastic materials were produced by plastic injection method. The effect of various weight percentages of hBNs on the flexural properties of thermoplastic composites were examined in the produced samples. The produced samples were subjected to three-point bending tests. Fourier transform infrared spectroscopy (FTIR), X-ray diffraction (XRD) and Scanning electron microscopy (SEM) were used to analyse structural and physical properties of the composites. The highest enhancement of flexural properties was obtained from the sample of PA 66/GF30/2% hBN. The samples with 2 wt% hBN showed the best flexural properties with an improvement of 85 and 52% flexural modulus and strength, respectively, compared to the samples without hBN (PA 66/GF30). According to the obtained results of this study, as the percentage of the hBN weight contents increased, the flexural strengths and flexural modulus increased significantly. It was concluded that hBN coated thermoplastic composite samples demonstrated high improvements for flexural properties at optimum rates of hBN.

1. Introduction

Hexagonal boron nitride (hBN) nanomaterials have excellent mechanical and thermal properties. Nanomaterials of hBNs can be used in thermoplastic composites in order to increase their mechanical and thermal properties. Mechanical and thermal properties of hBN reinforced polymeric composites have been investigated by several researches as shown in Table 1.

A comparison of injection moulding, powder bed fusion and casting was studied by Andreassen et al. [1]. They found that addition of hBN increased the elastic modulus and strength at high hBN ratios. Effect of hBN on mechanical properties of polyamide 6/glass fibers (5 wt%) nanocomposites was reported by Pramanik et al. [2]. They found the overall mechanical properties of PA6/5GF/hBN nanocomposites at 1 wt% hBN, significantly increased compared to other developed PA6/5GF/hBN composites. Travas et al. [3]. studied tensile performance of high-density polyethylene by addition of expanded graphite and BN. They found that at a high percentage of additives, the elastic modulus increased by 105% and 91%. Producing polyamide/BN nanocomposites with high thermal conductivities and mechanical properties was reported by Park et al. [4]. They showed that enhanced mechanical properties for the composites were obtained by 20 wt% of hBN content. The structural and mechanical performance of boron nanoparticle reinforced nanocomposites and bonded joints exposed to an acid environment were studied by Gültekin et al. [5]. They found that boron nanoparticles reinforced composites showed higher mechanical properties.

Ashrafi et al. studied [6] multifunctional thermoplastic composite materials using BN and carbon nanotubes in different ratios (0.5%, 1%, 2%, 5 wt%). According to the results of the study, the elastic modulus of BN composites had a higher value than that of carbon nanotube composites. Ayrilmis et al. [7] examined the mechanical properties of thermoplastic matrix (high-density polyethylene (HDPE) and polypropylene (PP)) composite materials using poplar wood flour (50 wt%) and BN (2%, 4%, and 6 wt%). With the addition of BN, the flexural strength of poplar/PP composites increased from 28.2 MPa to 43.7 MPa and the flexural strength of poplar/HDPE composites increased from 25.5 MPa to 33 MPa. Cai et al. [8] examined the thermal stability, flame retardancy, and mechanical properties of composite materials with polyurethane matrix using the chemically functionalized form of inert hBN (cetyl-trimethylammonium bromide, 0-4 wt%). According to

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Table 1. Some studies of hBN/polymer composites.

Nanomaterials	Integration Technique	Optimum Loading	Mechanical Properties	Reference
hBN	Coating with PA 66	0, 0.5, 2% wt% hBN	85 % increase in flexural modulus 52% increase in flexural strength	Ozgur et al. [this research]
	Dry mixing with PA 66	0.5, 1, 3 wt% hBN	13.6% increase in hardness, 13.8% increase in tensile strength	Pramanik et al. [2]
	Mixing the components with PA 66 withplasmaassisted mechanochemistry (PMC)	1, 5, 10, 20 wt% hBN	Enhanced mechanical properties for the composites were obtained by 20 wt% of h-BN content	Park et al. [4]
Expanded graphite (EG), BN	Mixing the components with high-density polyethylene (HDPE) in heating	80-5-15, 70-8-22, 70-15-15, 60-25-15, 84-8-8	105% and 91% increase in elastic modulus at a high percentage of additives	Travas et al. [3]
BN and boron carbide (B_4C)	Mixing with epoxy	2 wt% of BN/B_4C	Boron nanoparticles reinforced composites showed higher mechanical properties	Gültekin et al. [5]
BN nanotubes, Carbon nanotubes	Mixing with epoxy	0.5, 1.0, 2.0, 5.0 wt% BNNT and 1.0, 2.0 wt% CNT	2.40 GPa of elastic modulus in epoxy 2 wt% BNNT 2.08 GPa of elastic modulus in epoxy 2 wt% CNT	Ashrafis et al. [6]
Cetyl-trimethylammonium bromide-BN	Solvent blending and co-coagulation of thermoplastic polyurethane-based composites	0, 1, 2, 4 wt% hBN	79.3% increase in tensile strength	Cai et al. [8]
BN	Mixing with vinylester resins	0, 0.5, 1, 1.5, 2% wt% hBN	The ideal values were obtained with the addition of 1% BN	Boztoprak et al. [10]
BN (1-2 μm) **BN (7-10 μm)**	Compression molding with PP	15, 21 and 29 vol% byBN	The thermal conductivity and storage modulus of BN with large particles increased	Cheewawuttipong et al. [16]

the results, the heat dissipation rate of polyurethane composites with nano-filler addition of 4.0% hBN and the maximum value of total heat dissipation decreased by 57.5% and 17.8%, respectively. In addition, the tensile strength of the polyurethane composite increased by 79.3% compared to that of the pure polyurethane composites with the addition of 2.0 wt% hBN. Yu et al. [9] examined the thermal conductivity of epoxy matrix composites by using vertically oriented hBN (10-30 μm, 12-44 vol%). With the increase in hBN content, an increase in thermal conductivity was observed. When 44% hBN was used, the thermal conductivity was 9W/m.K. However, there was a significant decrease in tensile strength when 44% hBN was used. Boztoprak et al. [10] studied the mechanical properties of composite materials with vinyl ester matrix and BN particles. As a result of the research, there was an increase in the hardness of composite samples and the abrasion resistance and impact strengths were improved. Göncü et al. [11] investigated the effect of hBN addition on composite materials with alumina matrix. It was determined that hBN additive reduced the density of composite samples. It was also concluded that hBN additives acted as a lubricant. Owing to having a similar crystal structure to hydroxyapatite, the addition of hBN improves the mechanical properties of the host materials. Such improvements in the contribution of boron compounds in biomedical applications would increase in the future.

Furthermore, Taşdelen et al. [12] investigated the mechanical properties of PA 66 material. It was found that the tensile strength of PA 6 material was 82 MPa. Kaştan et al. [13] examined the mechanical properties of nanoclay-integrated PA thermoplastic composite materials. It was determined a maximum of 30 MPa of the flexural strength for the PA material. Karslı et al. [14] investigated the choice of polymer composite material for light weapons. In their research, it was stated that the tensile strength of PA 66 GF30 material was 155 MPa.

Oz et al. studied the thermal behaviour of hBN in an open atmosphere [15]. It was shown that BN was like the carbon structure, but it showed resistance to higher temperatures compared to similar carbon allotropes. It was mentioned that BN can be used as an additive in polymers and in other industrial applications. It has been explained that the crystal sizes of BNs were different from each other. Cheewawuttipong et al. [16] examined the thermal conductivity of composites with PP matrix using BN in different ratios (15%, 21%, and 29 vol%) and sizes (1-2 µm and 7-10 µm). According to their results, the thermal conductivity of BNs with large particles increased compared to small particles. Isarn et al. [17] examined the thermal conductivity of BN-filled (0%, 10, 20, 30, 40, (6 µm) and 40 wt% (80 µm (batch)) and thiol-epoxy matrix thermoset composites. Composites with a size of 6 µm and containing 40 wt% BN increased thermal conductivity by 400% from 0.2 W/K.m to 0.97 W/K.m, and when a bulk BN containing 40 wt% BN was used, the thermal conductivity values of the composites increased by 775% from 0.2 W/K.m to 1.75 W/K.m. Harrison et al. examined [18] the radiation protection of polyethylene matrix composites with BN (15 vol%) in the application of spacecraft. According to the results, using 2 wt% BN showed high radiation protection compared to the pure PP composite material. Zhou et al. [19] examined the thermal and electrical properties of epoxy matrix composites using intermittent hBN (0.5 µm, 50 wt%). Composites treated with silane-modified BN showed high thermal conductivity compared to the silane-free state. A low dielectric permeability (less than 5.4) and dielectric loss (less than 0.02), high volume resistivity (6.3×10^{14} Ω.cm), high dielectric strength (16 kV/mm) in the frequency range of 10^{-1} to 10^{-7} Hz were obtained with increased boron content (it was added to epoxy by 50 wt%).

Moreover, Madakbas et al. [20] examined the thermal stability and flame retardancy of composites using polyacrylonitrile and 10 wt% hBN. It was found that glass transition temperatures increased with increasing h-BN percentage. The limiting oxygen index (LOI) value reached from 18% to 27%. Muratov et al. [21] studied the thermal conductivity of composites by using two types of hBN (sizes 2-5 µm and 120-140 nm) in the HDPE polymer matrix. hBN was modified with 3-aminopropyl, triethoxysilane, binder (KR TTS and LICA 12) chemicals. The composite materials were fabricated by adding HDPE and hBN in a 1:1 volume ratio. Composite material showed a thermal conductivity of 2 W/m.K without any binding material. Pure hBN showed a thermal conductivity of 0.35 W/m.K. Wang et al. [22] measured the planar thermal conductivity of films with polyvinylidene fluoride (PVDF) matrix by converting hBN (lateral size 7 µm) into boron nitride nanosheet (BNNS) and blending it with chemicals of NaOH, KOH, DMF. According to the results, the planar thermal conductivity of the PVDF/BNNS composite with 4% BNNS reached 4.69 W/mK and the thermal conductivity of the PVDF/BNNS composite increased 2297% compared to pure PVDF. Pan et al. [23] investigated the thermal conductivity of composites with polytetra fluorine ethylene (PTFE) matrix using hBN (diameter 1.5 µm) and aluminum nitride (AlN) particles (diameter 2.5 µm). The thermal conductivity of AlN and hBN-integrated PTFE composites was measured at 1.04 W/mK. This was 3.8 times higher compared than that of pure PTFE.

Currently, 30 wt.% short glass fiber reinforced PA 66 matrix (GF30) thermoplastic composites are used in some parts such as diesel filters and brake pedals in automotive. Especially brake pedals can be subjected to different foot loads during driving of the cars. Therefore, it is important to fabricate brake pedals with increased flexural strength by using hBN additives in the composites. Since increasing of the weight percentages of nanomaterials, agglomerations have increased as well. Hence, the low number of the targeted weight percentages of hBNs (0, 0.5, and 2 wt%) were chosen in this research. The literature search shows that certain rates of hBN material added composites had enhanced mechanical, thermal, and electrical properties compared to the composites without boron additives. The flexural properties of hBN (0, 0.5, and 2 wt%) added GF30 thermoplastic composites have not yet been investigated. During this research, the experiments are designed to answer questions such as which rate of boron material can be used in thermoplastic composite material, and how much the flexural properties of the composite material can be increased by using boron material.

2. Materials and Methods

2.1. Materials

Polyamide thermoplastics are semi-crystalline polymers. Polyamides with excellent mechanical properties can be hard and tight or soft and flexible. The slip and wear properties of polyamides are very good. They could absorb moisture. Polyamides are frequently used in fields such as automotive, electrical, electronics, and textiles. Polyamides are usually produced by plastic injection and extrusion and they can be completely recycled mechanically. Neat PA 66 and 30 wt.% short fiber reinforced PA 66 granules (Mat Polymer, Türkiye) were used as matrix materials (Table 2).

hBN is a type of non-toxic non-porous material of white colour, known for its chemical stability, electrical conductivity, thermal conductivity, and lubricating properties. BN is used in many areas due to its low reactive and superior physical and chemical properties. They are preferred due to their electrical insulation and thermal conductivity properties. hBN was obtained from Boron Research Institute of the Turkish Energy, Nuclear and Mineral Research Agency. The particle sizes of hBN was 250-300 nm (Table 3).

Table 2. Properties of neat PA 66 and 30 wt.% short fiber reinforced PA 66.

Mechanical Properties	PA 66	30 wt.% short fiber reinforced PA 66
Elastic modulus (MPa)	3200	10000
Yield stress (MPa)	85	185
Izod notched impact strength (kJ/m^2)	5	13
Melting temperature (°C)	262	262
Density (Kg/m^3)	1140	1360

Table 3. Properties of hBN.

Parameter	Value
Particle size (nm)	250-300
Form	Powder
Density (g/cm^3)	2.3
Elastic modulus (GPa)	865
Melting point (°C)	3000

2.2. Methods

In this study 0, 0.5, and 2 wt % hBNs were added into thermoplastic composites. hBN nano-scale powders were added to 500 ml of ethanol in the required amounts and the solutions were prepared. After mixing the solutions for 150 min in the magnetic mixer, 30 min in the ultrasonic mixer, and 3 min in the mechanical mixer, homogenization of the solutions was achieved. Homogenized hBN-doped ethanol solutions were mixed with 500 mg PA 66/GF30 granules. In order to mix the solutions in homogeneously, the solutions were divided into equal quantities and each mixture was mixed ultrasonically and mechanically for 10 minutes. Later, they were left to dry for 3 days to remove ethanol in the mixed solutions (Figure 1).

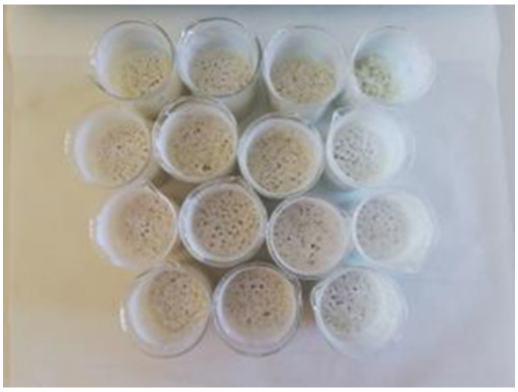

Figure 1. hBN coated PA 66/GF30 granules.

The samples were produced by the plastic injection molding machine (10 Tonnes, Permak Makina, Türkiye) and they were cooled down for three days. Three-point bending tests were applied to the samples that came to the appropriate conditions. The three-point flexural tests were performed on GF30 reinforced hBN added thermoplastic composites with PA 66 matrix in accordance with the ISO 178 Standards. The samples of PA 66, PA 66/GF30, PA 66/GF30/0.5% hBN and PA 66/GF30/2% hBN composites are 80 mm x 15 mm x 4 mm in size. Bending tests were applied on specimens with Instron 5982 100 KN universal test device (USA) at Ondokuz Mayıs University (OMU) KITAM Central Laboratory. The flexural strength (σ_f), modulus (E_B), and strain (ε_f) were calculated using Equ. 1 to 3, where, P is the flexural load (N); L is the support span (mm); b and d are the width (mm) and the thickness (mm) of the sample; m is the slope of the tangent to the initial straight-line portion of the load-deflection curve; D is the maximum deflection of the center of the sample (mm) [24].

$$\sigma_f = \frac{3PL}{2bd^2} \tag{1}$$

$$E_B = \frac{L^3 m}{4bd^3} \tag{2}$$

$$\varepsilon_f = \frac{6Dd}{L^2} \tag{3}$$

Composite void volumes are found by performing burn-out tests according to ASTM D3171 standard. Fourier transform infrared (FTIR, Bruker Tensor 27, Germany) spectroscopy analysis was performed. The specimens were pelletized before FTIR analysis. FTIR spectra were in the range of 650 to 4000 cm^{-1} of wavenumber. Scanning electron microscopy (SEM) images were obtained in an analytical field-emission SEM (JEOL JSM-7001 F, Japan). Before performing SEM, the cross sections of the specimen were coated with gold. X-ray diffraction analysis (XRD, Rigaku Smart Lab, Japan) was done. The Bragg's angle, 2θ, was ranged from 10° to 60° with a scan rate of 1°/min.

3. Results and Discussion

The measured densities and void volumes of the composites with PA 66/GF30/2% hBN were 1.374 g/cm^3 and 0.64 cm^3. These were 1.374 g/cm^3 and 0.18 cm^3 for the PA 66/GF30 specimens.

The measured densities and void volumes of the composites with PA 66/GF30/2% hBN were 1.374 g/cm^3 and 0.64 cm^3. These were 1.374 g/cm^3 and 0.18 cm^3 for the PA 66/GF30 specimens. Figure 2a and Figure 2b depict the Fourier Transform Infrared (FTIR) spectra of the PA 66/GF30/2% hBN composite and hBN powder. The absorption bands at 2934 cm^{-1} and 2859 cm^{-1} attributed to the symmetric and asymmetric C-H stretching vibrations and C-H twisting (Figure 2a). The peaks at 1635 cm^{-1} and 1534 cm^{-1} are attributed to the stretching vibration of the C=O group of amide I and the N-H bending and C-N stretching vibration of amide II. The peak at 1271 cm^{-1} indicates C-N-H coupling vibration of amide III. The peaks at 935 cm^{-1} and 1197 cm^{-1} are attributed to Si-OH and Si-O-Si groups. Typical peaks of hBN at 1353 cm^{-1} and 803 cm^{-1} can be related to in-plane B-N stretching and out-of-plane B-N bending vibrations of hBN (Figure 2b) [2].

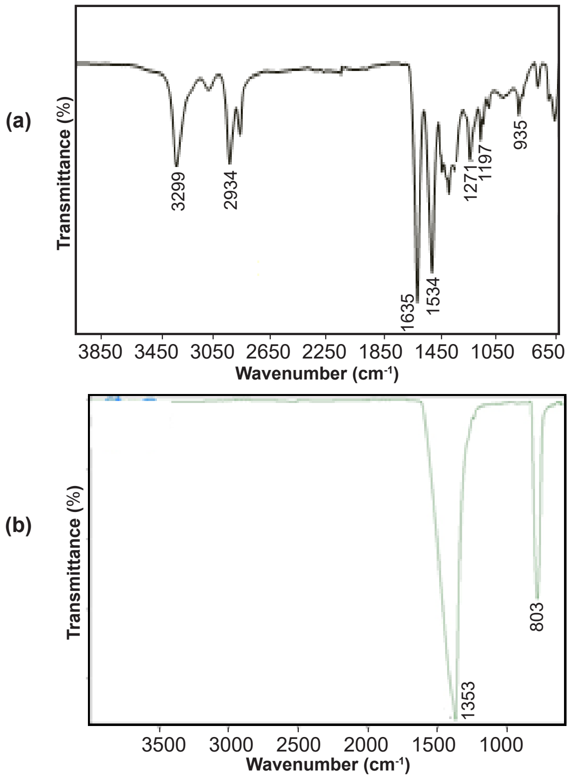

Figure 2. FTIR spectra of the specimens: a) PA 66/GF30/2% hBN composite and b) hBN powder.

Three-point bending test results are demonstrated in Figure 3 and Table 4. According to the results of the flexural tests of the composites with PA 66 matrix (Figure 3 and Table 4), the polymer consisting of pure PA 66 granules showed a ductile behaviour. PA 66/GF30, PA 66/GF30/0.5% hBN and PA 66/GF30/2% hBN composites exhibited higher flexural strength compared to pure PA 66. In the composites, samples with 2wt% hBN showed the best flexural properties with an improvement of 85% and 52% flexural modulus and strength, respectively, compared to the samples without hBN (PA 66/GF30).

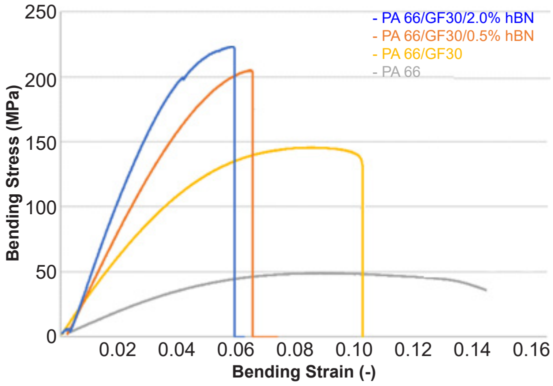

Figure 3. Three-point bending test plots.

Brittle fractures were detected in Figure 4 in hBN-added samples. These brittle fractures are due to the existence of the hBN in the structure of the composites In this study, compared to the literature, hBN-added PA 66 composites with PA 66 matrix showed a significant increase in flexural strength compared to PA 66 and PA 66/GF30 thermoplastics. According to the results, high improvements in bending properties were obtained from hBN particles added PA 66/GF30 thermoplastic composites. The flexural strength of PA 66/GF30/2% hBN increased by 235% when compared to pure PA 66 polymer, by 52% compared to PA 66/GF30 composite, and by 39.5% when compared to PA 66/GF30/0.5% hBN composite. PA 66/GF30/2% hBN samples also showed an increase in bending modulus. The Flexural modulus of PA 66/GF30/2% hBN composite with the addition of hBN achieved the highest value of 5.24 GPa, an increase of 404% compared to pure PA 66 polymer and an increase of 58% compared to PA 66/GF30 composite. Due to the obtained higher mechanical properties from hBN-added composites, which proofs that the coating of the nanomaterials of polymers was homogeneous. When the mechanical properties reduce, in non-uniform coating of nanomaterials into polymers increase. Furthermore, the flexural properties of the composites were compared with the literature. In the work of Pramanik et al. [2], the mechanical (13.6% hardness, 13.8% tensile strength, and 1300% elongation increased) properties of PA 6/5GF/hBN hybrid nanocomposites with 1 wt% hBN were significantly higher than those of PA 6/5GF composites. In the work of Taşdelen et al. [12] tensile strength of PA 6 material was 82 MPa. In the work of Autay et al. [25] flexural strengths of PA 66 and PA 66/GF30 materials were 42.66 MPa and 80.7 MPa, respectively.

Table 4. Bending test results with standard deviations of PA 66 matrix composites.

Sample	Bending Modulus (GPa)	Bending Strength (MPa)
PA66	1.04±0.10	65.20±5.22
PA66/GF30	3.30±0.11	144.00±2.26
PA66/GF30/%0.5 hBN	3.89±0.82	165.00±34.0
PA66/GF30/%2 hBN	5.24±0.21	218.42±8.93

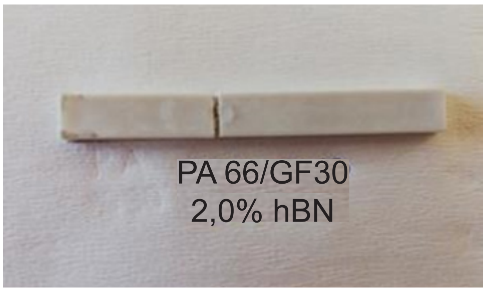

Figure 4. PA 66 matrix composites after flexural test.

Figure 5 represents the SEM image of the surface morphology of the fractured specimen from the bending tests. In that figure, the broken glass fibers and embedded hBN nano-particles into the PA 66 matrix can be seen. There is a certain amount of hBN to absorb the fracture energy and stop the crack propagation [26]

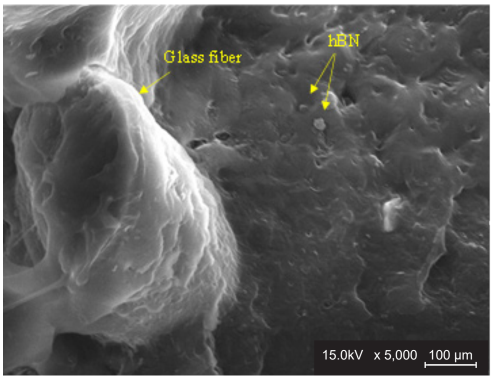

Figure 5. SEM image of hBN particles in fractured specimen from the bending tests.

Figure 6 represents the results of the XRD pattern of the PA 66/GF30 and PA 66/GF30/2% hBN composites. The International Centre for Diffraction Data (ICDD) card number of the hBN particles was 34-421. These XRD results agreed well with the literature. It was observed that PA 66 exhibited strong crystalline diffraction peaks at 2θs = 20.9° and 24.0° [27]. The hBN nanoparticles in the composite exhibited distinct and sharp peaks at 2θ = 26.8° [2]. In the subsequent studies, during the development and fabrication of new designs of composites, the obtained information on different weight contributions of hBN particles in composites will provide the optimization of materials.

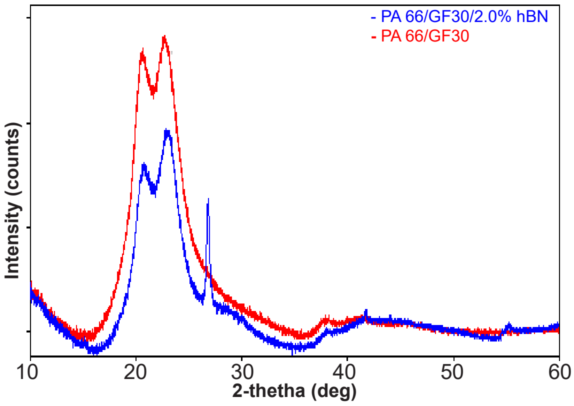

Figure 6. XRD spectra of the PA 66/GF30 and PA 66/GF30/2% hBN composites.

4. Conclusions

The hBN additives were applied by coating granules of PA 66/GF30 materials and the hBN-coated thermoplastic materials were produced successfully by plastic injection method. This study shows that the addition of hBN particles to PA 66/GF30 materials increased the flexural strength and modulus of the composites. The optimum amount of addition of hBN in composites was 2 wt%. Samples with 2 wt% hBN showed the best flexural properties with an improvement of 85 and 52% flexural modulus and strength compared to the samples without hBN (PA 66/GF30). It was concluded that hBN-coated thermoplastic composite samples exhibited very high improvements in flexural strength at low weight rates.

5. Acknowledgements

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