



Effect of Nanoalumina on Physical and Thermal Properties of Polypropylene-Glass Fiber Reinforced Nanocomposites

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The present study examines the effect of nanoalumina on the mechanical and thermal properties of glass fiber and elastomer reinforced PP/EPDM/GF/Al₂O₃ with an aim to develop suitable composite material for orthotics. In present study, PP/GF, PP/EPDM/GF composite and PP/EPDM/GF/Al₂O₃ nanocomposites with specific composition were fabricated using melt-blending technique. Prepared composites were characterized through scanning electron microscopy (SEM), X-ray diffraction (XRD), thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC). The mechanical characteristics including tensile strength, tensile modulus, notched Izod impact strength and elongation at break of the prepared composite were studied in accordance with ASTM guidelines. PP/EPDM/GF/Al₂O₃ composites showed the mechanical properties of PP/EPDM/GF/Al₂O₃ composites showed 62%, 145%, 15% and 165% enhancement in tensile strength, tensile modulus, notched Izod impact strength and elongation at break, respectively as compared to PP/GF composites. The mechanical properties and thermal properties of PP/EPDM/GF/Al₂O₃ were also compared with PP/EPDM/GF composites. The use of nanoalumina significantly and brilliantly balances the complimentary tensile and elongation properties in PP/EPDM/GF/Al₂O₃ composite, which is an essential requirement for fabricating composite material capable of being thermoformed to orthotic devices.

Keywords: Polypropylene, Nanocomposites, Glass fiber, Nanoalumina, Ethylene propylene diene monomer, Mechanical properties.

INTRODUCTION

Polypropylene (PP) is an extremely popular material for the production of orthotic devices due to its ability to provide excellent comfort and support, while still being lightweight and durable [1]. It exhibits thermal and mechanical properties suitable for orthotics due to its low weight, strong recyclability and affordable production, polypropylene can be a good choice for use in the field of orthopaedic applications [2,3]. As a thermoplastic material, polypropylene can be easily molded and adjusted to fit the needs of the patient. Its strong flexibility allows it to fit snugly around the foot and alleviate any discomfort or pain. Additionally, polypropylene is highly resistant to water, dust and other elements that could damage orthotics, making them an ideal choice for orthotic aid. However, due to its lack of rigidity, mechanical property imbalance in terms of tensile, impact and elongation have restricted some of its applications [4]. Mixing polypropylene with other polymers or nanoscale materials could further enhance its mechanical properties [5,6].

In an effort to improve the thermal and mechanical properties of polypropylene, several inorganic fibres have been used as micro-fillers [7,8]. Glass fibres due to their many benefits, have recently become a popular reinforcement method for composite materials [9]. Benefits of glass fibres include their high temperature strength, corrosion stability, good dimensional durability, exceptionally low weight, support of continuous production process of automotive sector and nearly no moisture absorption [10-12]. However, the molecular mobility of the polymer chains is greatly reduced in composites due to the incorporation of glass fibres into the polymer matrix, which increases the composite's strength but significantly decreases its elongation at break and impact strength.

Combining ethylene propylene diene terpolymer (EPDM) elastomer with polypropylene can certainly improve the impact strength and elongation at break [13-15]. Numerous studies exploring the mechanical characteristics of PP/EPDM blends have recently been reported [16-18]. According to Hajibabazadeh *et al.* [19], addition of EPDM resulted in a modest increase in

impact strength at the cost of tensile strength which decreases with the inclusion of elastomer into the polypropylene matrix.

Adding nanoparticles as nanofillers is another viable option for strengthening polymeric materials. In comparison to conventional fillers, nanocomposites demonstrated higher mechanical performance and improved barrier characteristics at significantly lower loading levels. To create a nanocomposite with high durability, stiffness and superior mechanical properties depends on the homogeneous distribution of the nanoparticles in the matrix [20,21]. Agglomeration of nanoparticles can reduce the effectiveness of reinforcement and lead to a decrease in mechanical properties. Therefore, a proper dispersion of nanoparticles in the matrix is important to ensure the effective reinforcement and improvement of mechanical properties. This suggests that combining optimized concentration of rigid reinforcing nanoparticles as nanofillers within the polymer/elastomer matrix with strong glass fibres could be an efficient strategy for achieving balanced toughening and strengthening of fibre-reinforced polypropylene composite.

As far, no prior studies have focused on manufacturing and mechanical characterization of PP/EPDM/GF/Al₂O₃ nanocomposite materials for use in orthotics. This study seeks to investigate the mechanical properties of a PP/EPDM/GF/Al₂O₃ composite that could be used in orthotic devices. The SEM, tensile, elongation, impact strength, thermal analysis and crystallization behaviour of PP/GF, PP/EPDM/GF and PP/EPDM/GF/Al₂O₃ composites are examined.

EXPERIMENTAL

Reliance polymers polypropylene (PP) grade Repol CO15EG with melt flow index of 1.8 g/10 min at 230 °C/2.16 kg was used. As reinforcing microfibers, 3 mm long silanized glass fibres from Vaani Fiber Glass in Delhi were used. Polypropylene grafted maleic anhydride (PP-*g*-MA), made by Pluss Advanced Technologies Pvt. Ltd. Gurgaon, India, was used as a compatibilizer. Arihant Oil and Chemicals provided ethylene propylene diene rubber (EPDM) in pellet form under the brand name EPDM-8450. 99% Pure alumina (Al₂O₃) nanoparticles (50-80 nm) was procured from Nanolabs, Jamshedpur, India.

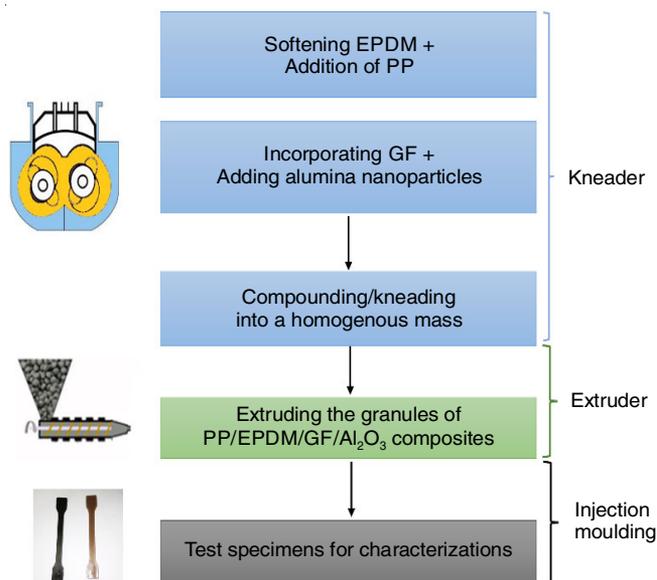
Preparation of polypropylene glass fiber (PP/GF) composite: Polypropylene glass fiber (PP/GF) composite master batch was manufactured by combining glass fibre, polypropylene granules and PP-*g*-MA as a compatibilizer in a counter-rotating twin-screw extruder (Thermoscientific Haake poly rheocord RC-90). This created the pellet form of the PP-GF composite master batch. In order to provide a strong interfacial binding strength between the fibre and the polymer, compatibilizer was employed to make hydrophobic polymers and hydrophilic reinforcing glass fibres compatible. Compatibilizers that possess both hydrophilic and hydrophobic qualities make it easier to wet the hydrophobic polymer on one side while also assisting with the coupling with hydrophilic fibres on the other at 175-205 °C and 25-50 rpm were employed. Silanized glass fibres were loaded at a concentration of 15 phr with 5% compatibilizer (PP-*g*-MA) in composites.

Injection moulding was utilized to construct tensile test specimens with the geometry specified in ASTM Standard

D-638, type 1 [22] using the master batch of PP/GF pellets. In order to facilitate a point-by-point evaluation, pristine polypropylene samples were also manufactured *via* extrusion and injection moulding using the same parameters.

Preparation of PP/EPDM/GF composite: The PP/EPDM/GF composites were made using two step procedures. The batch of PP/EPDM/GF was manufactured by using a kneader manufactured by SM Engineering Works, Ghaziabad, India. This kneader had a mixing capacity of 15 L, a primary motor with 10 HP, a tilting angle of 130° and a 2 HP air compressor. By changing the temperature, the EPDM was given time to soften in the kneader before polypropylene was introduced and just 15 min of blending was done there. In order to finish the dynamic vulcanization, dicumyl peroxide (DCP) was added and the mixing process continued for an additional 10 min. The final batch was prepared by adding glass fiber with compatibilizer and blending for 30 min. Following the same procedure as detailed in the fabrication of PP/GF composite, the aforementioned master batch of PP/EPDM/GF was subsequently injection moulded into dumbbell-shaped specimens for the mechanical testing.

Preparation of PP/EPDM/GF/Al₂O₃ composite: A small amount of alumina (Al₂O₃) nanoparticles can significantly enhance the tensile characteristics of polymeric nanocomposites since alumina (Al₂O₃) is used as reinforcement inside the matrix of polymers [17,18]. The PP/EPDM/GF/Al₂O₃ nanocomposite batch was prepared using the same procedure that was utilized to make the PP/EPDM/GF composites. Following the addition of EPDM for mixing, 1 phr of alumina nanoparticles were added to the mixer as illustrated in **Scheme-I**. Lower concentrations of the alumina particles are utilized since higher concentrations could lead to the formation of agglomerates, which would have an adverse impact on the mechanical properties of composite [23,24]. Following the same procedure as with the PP/GF composites, the PP/EPDM/GF/Al₂O₃ nanocomposite batch was molded into the dumbbell-shaped samples.



Scheme-I: Schematic flow diagram for the preparation of the PP/GF, PP/EPDM/GF and PP/EPDM/GF/Al₂O₃ nanocomposite

Characterizations

Scanning electron microscopy (SEM): Quanta 200 FEG scanning electron microscopy (SEM) was used to examine the fracture surface of PP/EPDM/GF/Al₂O₃ composites for morphological properties. Coating the composite samples with an Au coating made the surface conductive enough for SEM analysis.

Mechanical characterization: The tensile and impact properties of the polypropylene composites were evaluated with the use of the ASTM D638 and D256 standards, respectively. When assessing the tensile properties of reinforced and unreinforced plastics using standard dumbbell-shaped test specimens in a controlled environment for pretreatment, temperature and humidity as well as testing machine speed, the ASTM D638 standard outlines the technique that must be adhered to. The Instron-3382 equipment was used to measure the tensile characteristics at room temperature. For the purpose of determining the tensile characteristics, a cross head speed of 5 mm/min was utilized. The impact resistance was evaluated with a TMI digital Impact testing Machine that had a 6.779 Nm pendulum as the measuring instrument.

Thermogravimetric analysis (TGA): The samples were heated from room temperature to 600 °C at a rate of 100 °C/min using a Perkin-Elmer thermal analysis instrument having temperature range from 15-1000 °C.

Differential scanning calorimetry (DSC): A Perkin- Elmer differential scanning calorimeter was utilized to investigate the samples for thermal characteristics and crystallization behaviour. The crystallization features were studied by heating the samples to 230 °C, holding them there for a few minutes and then letting them return to room temperature. The thermograms were acquired while the heating and cooling rates were held constant at 10 °C/min. The temperature at which a material melts, is known as its melting point (T_m). It is determined by the highest temperature reached during an endothermic process.

The amount of heat required to transform a substance from a solid to a liquid at its melting point is referred to as the heat of fusion (ΔH_m) and measured in terms of heat per unit mass. The temperature at which a liquid undergoes the exothermic transformation into a solid is known as the crystallization temperature (T_c). The degree to which the polymer chains are aligned with one another in crystallized regions is referred to as the crystallinity of polymer (X_c). To study the crystallization behaviour, these parameters are to be selected. The standard heat of fusion of polypropylene composites can be calculated using the following formula to reveal their crystallinity:

$$X_c = \frac{\Delta H_m}{\Delta H^*} \times 100$$

where H_m and H* are the heat of fusion of pure polypropylene and its composite, respectively. The value of H* for a 100% crystalline polypropylene is reported in the literature [25] to be 209 (J/g) and this value is employed in the above equation.

X-ray diffraction: The X-ray diffraction (XRD) patterns for the synthesized PP/GF, PP/EPDM/GF and PP/EPDM/GF/Al₂O₃ composites were taken using a PANalytical X'Pert PRO powder X-ray diffractometer. The composite samples scanned over the interval of 2θ 10-80° at 40 kV and 30 mA.

FTIR analysis: A Perkin-Elmer spectrophotometer spectrum 2 (wavenumber range: 4000-400 cm⁻¹) was used to characterize the chemical structures of PP/GF, PP/EPDM/GF and PP/EPDM/GF/Al₂O₃ nanocomposites.

RESULTS AND DISCUSSION

Mechanical properties: The parameters of the polymer composites were evaluated in accordance with ASTM D638, ASTM D790 and ASTM D256. Specifically, the tensile strength, tensile modulus, impact and percentage elongation at break were assessed. At room temperature, measurements of tensile and elongation properties were carried out utilizing equipment model Instron-3382, with a cross head speed of 5 mm/min. The impact resistance of a Notched Izod was tested with a TMI digital Impact testing machine equipped with a 6.779 Nm pendulum. Each composite composition were subjected to five separate tests and the mean was then determined and the average values of tensile strength, modulus, impact strength and elongation at break were calculated. The obtained results for tensile strength, tensile modulus, impact strength and percentage elongation at break has been tabulated in Table-1.

TABLE-1
TENSILE STRENGTH, MODULUS, IMPACT STRENGTH
AND PERCENTAGE ELONGATION @ BREAK OF PP/GF,
PP/EPDM/GF AND PP/EPDM/GF/Al₂O₃

Samples	Tensile strength (MPa)	Tensile mod (MPa)	Impact strength (J/m)	Elongation at break (%)
PP	32	1000	130	60
PP/GF	38	2100	105	52
PP/EPDM/GF	30	2150	165	180
PP/EPDM/GF/Al ₂ O ₃	52	2454	149	159

In Fig. 1, it is evident that in PP/GF composites with 15% glass fibers, tensile strength is increased by 20% (from 32 to 38 MPa). On adding EPDM as a modulus modifier to PP/GF composites, the tensile strength of the resulting PP/EPDM/GF composites deteriorates back to that of polypropylene. However, it imparts the required elongation to the composite to facilitate their thermoforming property to orthotic aid and enhance the durability of the material respectively.

To enhance the tensile strength of the impact modified PP/EPDM/GF composites, one option is to increase the amount of glass fiber, but increasing glass fiber content is not feasible as it causes tearing of composites during thermoforming. The use of alumina as nanofiller to develop PP/EPDM/GF/Al₂O₃ composites, manage to balance the tensile as well as toughness property of the composites. The addition of nanoalumina particles in PP/EPDM/GF/Al₂O₃ composites significantly enhances the tensile strength to 52 MPa, an increase of 37% and 73% over PP/GF and PP/EPDM/GF composites without significantly decreasing the impact strength and elongation at break. The composite maintains the impact strength at 149 J/m and percentage elongation at break at 159%. The reason for obtaining balanced tensile and toughness properties can be attributed to the enhanced mechano-reinforcement of the matrix of composite with dispersed alumina nanoparticles. The results show that

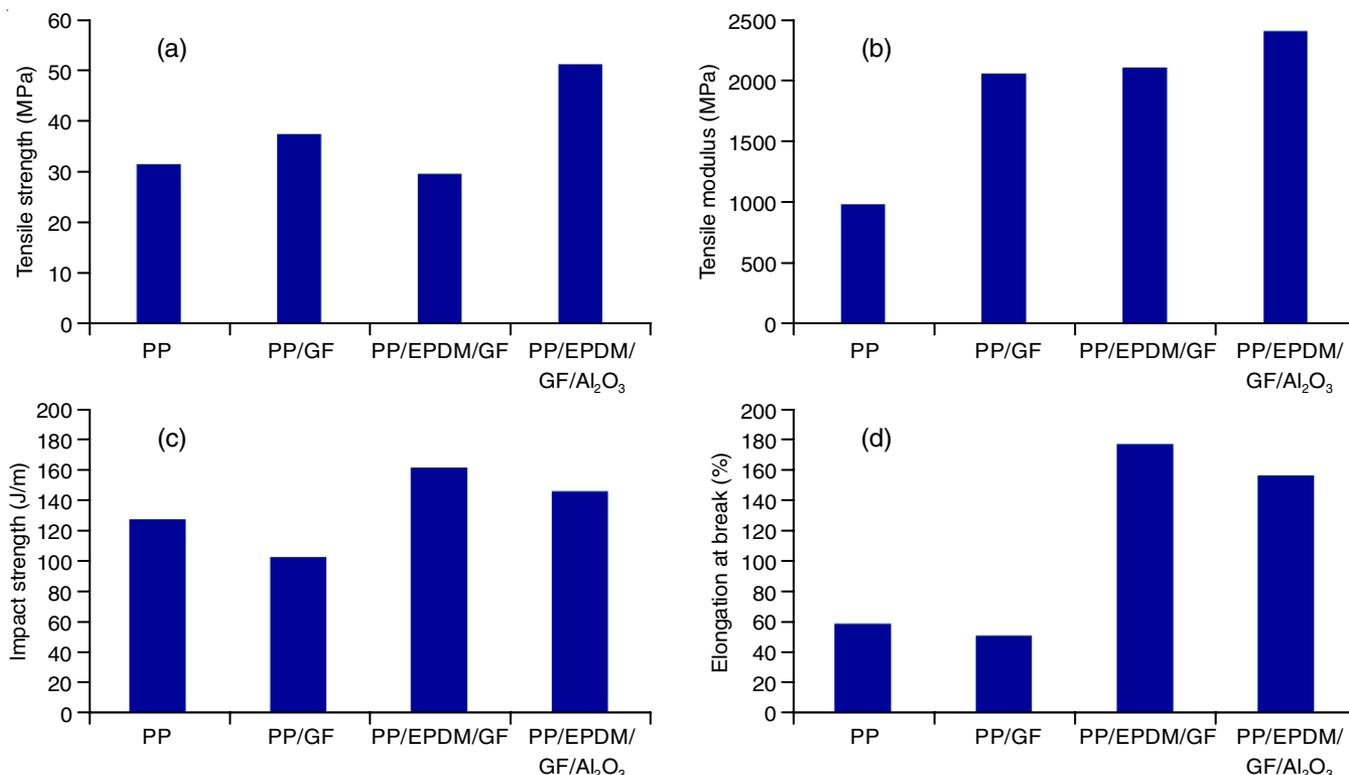


Fig. 1. Compared mechanical characteristics of PP/GF, PP/EPDM/GF and PP/EPDM/GF/Al₂O₃ with (a,b) tensile strength and modulus; (c) impact strength and (d) % elongation @ break

alumina nanoparticles when used along with glass fiber and EPDM in PP/EPDM/GF/Al₂O₃ composites improve the tensile strength more effectively. Adding nanoalumina leads to an overall increment in the tensile properties and sustain the impact strength and elongation property making it a good material for orthotics.

XRD studies: Fig. 2 depicts the XRD spectrum of the PP/GF, PP/EPDM/GF and PP/EPDM/GF/Al₂O₃ composites. Four distinct peaks 2θ at 14.2°, 16.9°, 18.5° and 21.8° corresponding to the (110), (040), (130) and (131) planes of α -crystals of polypropylene are observed in XRD patterns of PP/GF, PP/EPDM/GF and PP/EPDM/GF/Al₂O₃. The XRD spectra show that the characteristic diffraction peak of polypropylene comp-

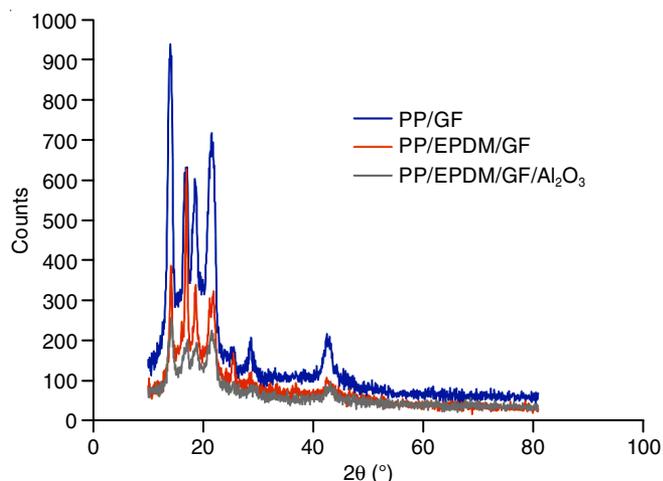


Fig. 2. XRD patterns of PP/GF, PP/EPDM/GF and PP/EPDM/GF/Al₂O₃

osites is almost similar for all composites. Incorporation of Al₂O₃ nanoparticles only affects the diffraction peak's width and intensity; the diffraction peak with characteristic angle is unaffected and identical [26]. It is observed that the XRD peaks suppress in presence of nano-Al₂O₃ in PP/EPDM/GF/Al₂O₃ composites that could be due to nanoparticle's ability to absorb X-ray radiations. Further, with the inclusion of nanoalumina, the reduction in the intensity of peaks may be due to the increased interaction between polypropylene and nanofillers, deteriorating the crystalline orientation as reported [27]. No separate peak is observed for nano alumina, which suggests that nanoalumina is uniformly distributed in the polymer matrix and there is no evident agglomeration of nanoparticles [28].

SEM studies: The SEM images of a fractured PP/EPDM/GF/Al₂O₃ composite surface are shown in Fig. 3a-d. It can be observed from Fig. 3c that glass fiber and Al₂O₃ are uniformly distributed along the polymer matrix without agglomeration. Interfacial adhesion is crucial for effective stress transfer and mechanical properties in composites [29,30]. The figure elucidates the morphology of interfacial region between the glass fibre and PP/EPDM matrix. Resulting in composites with no visible void space. One possible explanation for the improved mechanical characteristics of PP/EPDM/GF/Al₂O₃ composites is the dispersion and distribution of the alumina nanoparticles within the polymer matrix wherein strong interface between the nanoparticles and the polypropylene matrix leads to an effective stress transfer to provide enhanced mechanical properties.

FTIR studies: Polypropylene can be identified by the presence of spectral peaks at 2950 cm⁻¹ (asymmetric -CH₃ *str.*), 2918 cm⁻¹ (asymmetric -CH₂ *str.*), 2867 cm⁻¹ (symmetric -CH₃

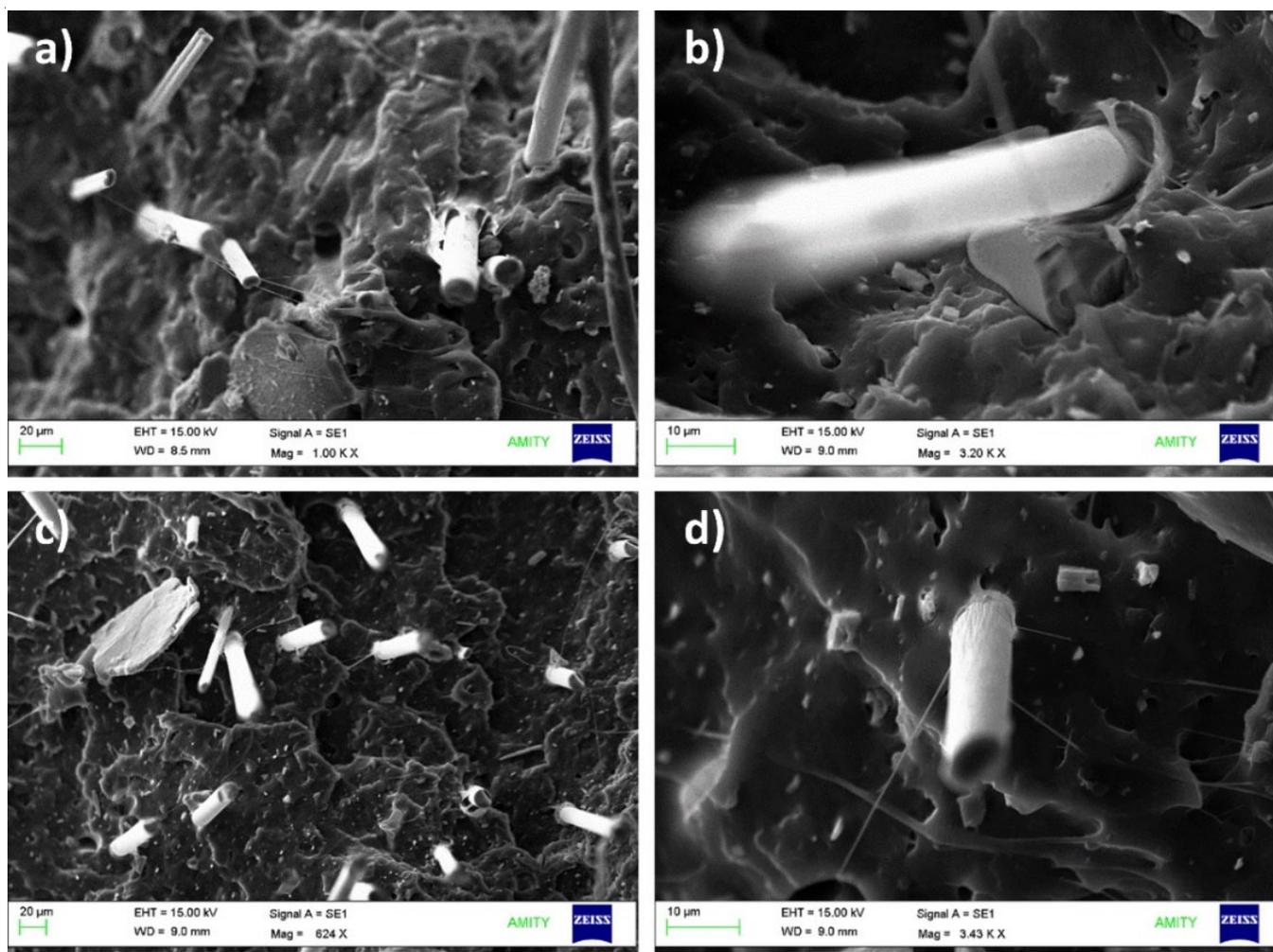


Fig. 3. SEM images of PP/GF/EPDM/Al₂O₃ nanocomposite at (a) 1.00 KX (b) 3.2 KX (c) 624 KX (d) 3.43 KX

str.), 2840 cm⁻¹ (symmetric -CH₂ *str.*), 1455 cm⁻¹ (is due to the overlapping of the asymmetric bending mode of methyl and the methylene scissoring mode), 1375 cm⁻¹ (symmetric -CH₃ *def.*) and 716 cm⁻¹ (rocking vibrations of methylene groups) [17,19]. The stretching of C-O and Si-O is responsible for the minor peak near 1060 cm⁻¹. The spectra of PP/EPDM/GF/Al₂O₃ nanocomposites show the signature Al-O bond at 568 cm⁻¹ caused by the nanoalumina particle as shown in Fig. 4. The presence of this prominent band indicates the occurrence of Al₂O₃ cluster formation within the polypropylene pores. As a result of the uniform distribution of nanoalumina filler in the matrix, the intensity of the peaks in the range of 1000-500 cm⁻¹ is somewhat enhanced.

DSC analysis: Crystallization and fusion parameters for the polypropylene composites are listed in Table-2. Addition of nanoalumina to a polymer matrix increases the melting point (T_m) of the nanocomposite by a small amount as compared to PP/GF composite. Fig. 5 shows DSC heating and cooling curves of PP/GF, PP/EPDM/GF and PP/EPDM/GF/Al₂O₃. A nominal increase in crystallization (T_c) and melting (T_m) temperatures of PP/EPDM/GF over PP/GF can be attributed to the mutual interactions between rubber crosslinks, polymer chains and micro-fillers, which impede the movement and rigidity of the

TABLE-2
CRYSTALLIZATION AND FUSION PARAMETERS OF
PP/GF, PP/EPDM/GF AND PP/EPDM/GF/Al₂O₃

Sample	T_m (°C)	ΔH_m (J/g)	T_c (°C)	X_c (%)
PP/GF	145.48	61.703	119.94	29.52
PP/EPDM/GF	163.14	55.123	120.11	26.37
PP/EPDM/GF/Al ₂ O ₃	147.69	65.646	116.92	31.41

polymer chains. Reduced polypropylene chain mobility is also a result of the vulcanizing system's increased viscosity [31], even though addition of Al₂O₃ does lower the T_m value, as compared with PP/EPDM/GF composite.

The heat of fusion of polypropylene (ΔH_m) increases in the PP/EPDM/GF/Al₂O₃ composites that contain nanoalumina-additives due to the increment of the degree of crystallinity (X_c). This improvement is attributed to the nucleation effect of nanoalumina. The increase in degree of crystallinity (X_c) on addition of Al₂O₃ nanoparticles in PP/EPDM/GF matrix may be attributed to heterogenous nucleation effect of nano-Al₂O₃ particles. These results indicate that Al₂O₃ nanoparticles dispersed well without significant agglomeration in PP/EPDM matrix at 1 phr concentration and thus leads to increase in nucleating effect which inturn increases X_c [32,33]. This rise

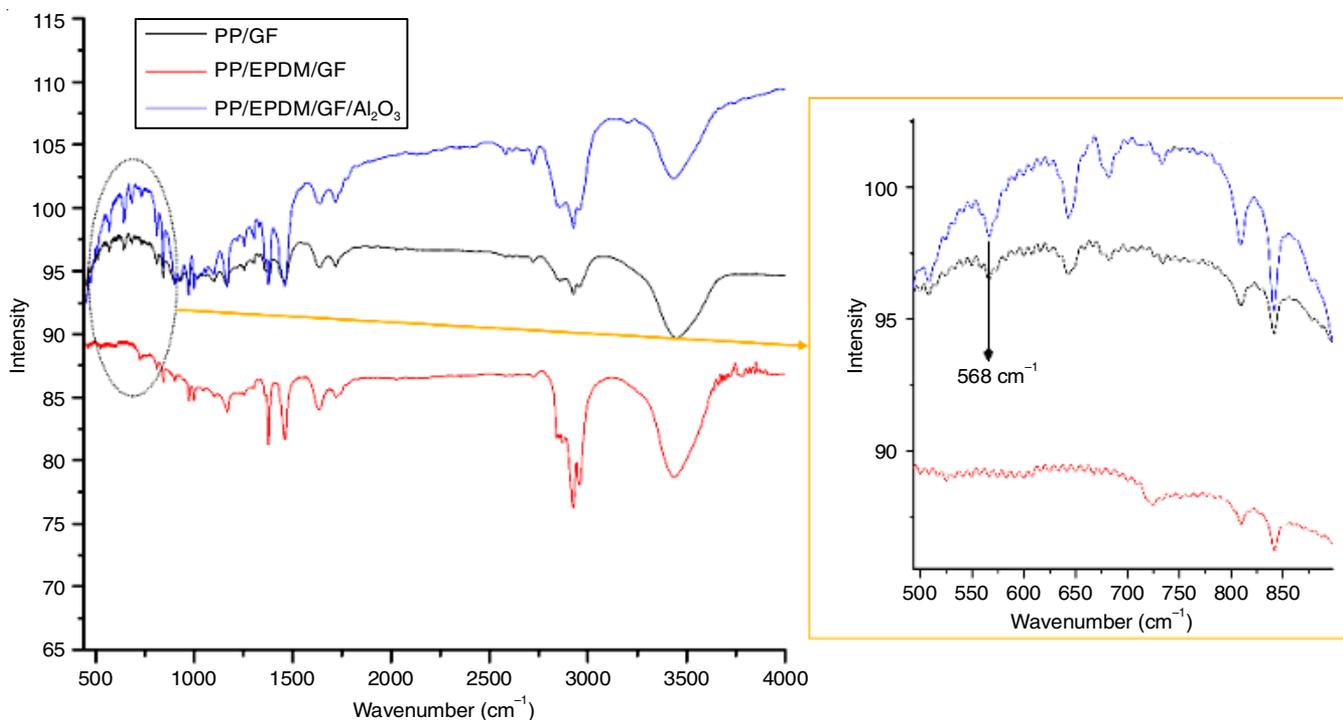


Fig. 4. FTIR spectra of PP/GF, PP/EPDM/GF and PP/EPDM/GF/Al₂O₃ composites

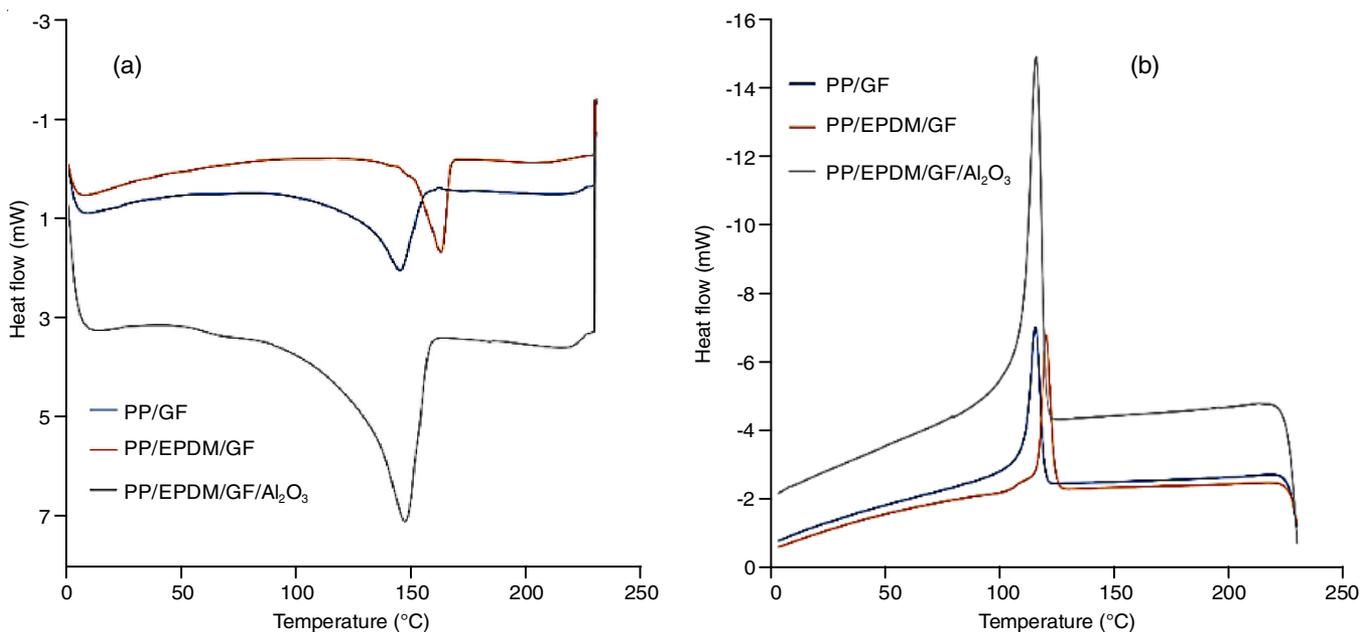


Fig. 5. DSC comparison with (a) heating and (b) cooling curves of PP/GF, PP/EPDM/GF and PP/EPDM/GF/Al₂O₃ composite

in the heat of fusion in polypropylene composites can result in increased stiffness, strength and heat resistance and exhibit improved impact resistance, as well as enhanced dimensional stability and resistance to creep. This is because the high heat of fusion indicates a high degree of crystallinity, which provides a more ordered and dense molecular structure, leading to improved mechanical properties.

TGA analysis: The thermal stabilities of the composites and the degradation characteristics were analyzed. Fig. 6 shows the thermogravimetric curves of PP/GF, PP/EPDM/GF and PP/EPDM/GF/Al₂O₃ composites, which demonstrates the temp-

erature dependent thermogravimetric analysis curves for the polypropylene composites and polypropylene nanocomposite between 20 and 600 °C. The initial degradation temperature, final degradation temperature and total weight loss for the PP/GF composite were 257 °C, 415 °C and 92.2%, respectively, as compared to 265 °C, 425 °C and 91.5% for PP/EPDM/GF composite. However, the thermal behaviour of PP/EPDM/GF/Al₂O₃ composites was shown to be significantly impacted by the incorporation of nanoalumina, with a 270 °C initial degradation temperature, a 445 °C degradation temperature and a 90.13% total weight loss. As can be seen, the degradation

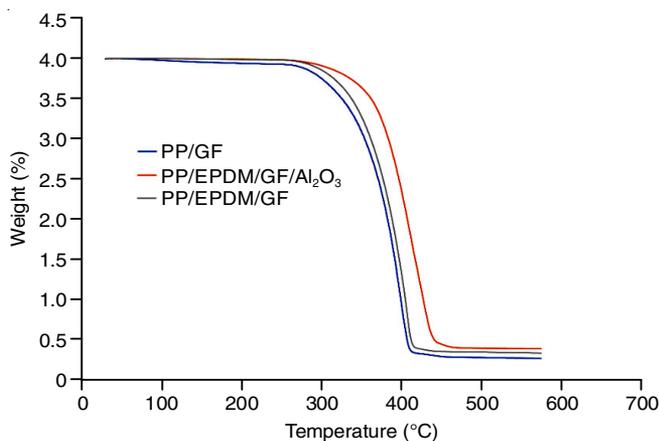


Fig. 6. TGA curves of PP/GF, PP/EPDM/GF and PP/EPDM/GF/Al₂O₃

temperature of PP/EPDM/GF/Al₂O₃ nanocomposites increased as compared to other composites. An increased thermal stability can also be attributed to the polymer-nanofiller interactions due to high thermal insulating characteristics of nanoalumina homogeneous dispersion and strong interaction of alumina nanoparticles in polypropylene matrix [34,35]. To maintain tensile strength, we incorporated nanoalumina to PP/EPDM/GF composites, which reduced the mobility of polymer chains because of the intense contact between the polymer and the filler at the interface, which is required to maintain the tensile properties of the composite. An increased degradation temperature result in improved resistance to chemical degradation, such as oxidation and UV degradation, which can lead to improved long-term durability.

Conclusion

The work investigated the effect of nanoalumina on the mechanical, morphological and thermal properties of PP/EPDM/GF/Al₂O₃ nanocomposites, to evaluate their suitability for use in orthotics. The mechanical properties such as tensile strength, tensile modulus, notched Izod impact strength and elongation at break shows remarkable increment of 62%, 145%, 15% and 165%, respectively in PP/EPDM/GF/Al₂O₃ nanocomposites as compared to PP/GF composites. The obtained results highlight the efficient use of Al₂O₃ nanoparticles in enhancing the overall mechanical properties of PP/EPDM/GF/Al₂O₃ composites including both tensile and elongation. The SEM images of PP/EPDM/GF/Al₂O₃ composites show that alumina nanoparticles are homogeneously dispersed in the polymer composite matrix, which may be the most probable reason for the improved mechanical properties. The enhanced thermal properties of PP/EPDM/GF/Al₂O₃ composites seems to be favourable for the thermoforming of these composites for orthotic device fabrication. The TGA analysis revealed a good thermal stability of PP/EPDM/GF/Al₂O₃ composite and DSC analysis shows high heat of fusion responsible for increased stiffness, strength and heat resistance and enhanced dimensional stability.

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CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

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