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Mechanical and Physical Characteristics of Nanofiller- and Nanofiber-Reinforced Polyethylene Hybrid Nano composites

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ABSTRACT: The effect of various combinations of filler materials on the performance of Polyethylene (PE)-based composites was investigated. PE in particulate form was used as the matrix. Milled short carbon fiber (SCF) micro-size, graphite nano-platelet (GNP), and titanium dioxide nanoparticles (TiO₂) were used as fillers. These fillers were incorporated in the polymer matrix to produce mono-filler (PE/SCF and PE/nanofiller) and hybrid composites. Hybrid composites consist of PE/5SCF/GNP, PE/5SCF/TiO₂, and PE/10SCF/GNP/TiO₂. The effect of the addition of SCF, GNP, and TiO₂ on PE-based composites was investigated by analysing their morphological, mechanical, and physical properties. The addition of mono-filler to the PE matrix improved the mechanical properties of the composites when compared to the neat PE. The ultimate tensile strength (UTS), flexural modulus, flexural strength, and impact toughness of the hybrid composites with 10 wt % total loading of fillers were determined. When compared to the PE/15SCF composite, the hybrid composite containing 10% SCF and an extra 2.5% GNP and 2.5% TiO₂ showed a maximum 20% increase in flexural modulus. Comparing the 10.5 wt% SCF reinforced PE to the PE/10SCF composite; the addition of 2.5 wt% TiO₂ increased the strain at break by 15%. When compared to PE/SCF alone, a scanning electron microscopy image of the PE/10SCF composite with GNP added showed that the interfacial bonding between PE and SCF was improved. For every composition, a decline in the melt flow index (MFI) was noted. On the other hand, hybrid composites displayed a greater MFI decline.

KEYWORDS: Polyethylene, hybrid, composite, short carbon fiber, GNP and TiO₂ nanoparticles.

I.INTRODUCTION

Thermoplastics are materials that are widely utilized in everyday life, like plastic bottles, containers, and bags. The comparatively poor mechanical and physical qualities of thermoplastic materials limit their use. The impacts of the environment and loading circumstances also cause thermoplastics to perform less well over time [1]. Because of their lightweight, processability, and recyclability, thermoplastic materials are therefore widely utilized in non-load-bearing applications, such as the automotive sector (e.g., car bumper, door panel, engine cover, dashboard, etc.) [2]. The improvement of thermoplastic polymer materials' mechanical and physical qualities has been the subject of numerous studies. Polymer composites, which consist of fibers or fillers, are frequently utilized to enhance and fortify the characteristics of polymers. Some of the compounding strategies used to improve the properties of polymers are continuous fiber reinforced polymer composites [3], long fiber reinforced polymer composites [4-6], short-fiber reinforced polymer composites [5, 7], nanofiller-reinforced polymer composites [8-12], and hybrid composites, which are a combination of these composites [13-15]. There are restrictions on the use of continuous fiber and thermoplastic together, in contrast to thermoset plastics, which can be coupled with continuous fiber with ease to create massive pieces [16]. Thermoplastic composites bonded with both long and short fibers are commonly utilized. A fiber is referred to as short-fiber when its length to diameter ratio falls between 100 and 17 [17]. When combined with thermoplastic polymers, the advantage of short fibers over continuous or lengthy fibers is their processability. However, the mechanical properties of short-fiber reinforced polymer composites are weak. Fillers are inorganic and organic solid particles of various shapes that are added to polymers to change their composition. Fillers can be used to change attributes such as processing, electrical, magnetic, mechanical, fire retardant, and surface. In addition, fillers can be employed as gas barriers, warpage reducers, and to improve degradability and antiaging [18].



The overall characteristics of thermoplastic composites are influenced by fillers' structural, morphological, surface, mechanical, and physical characteristics [19]. Research indicates that the incorporation of spherical particles, like silicon nanoparticles, enhances toughness and elasticity [20]. Conversely, the incorporation of comparable particles in the fiber shape augments the modulus of elasticity and strength [16]. The use of fillers with both micro- and nano-scales has drawn a lot of attention lately. It is anticipated that the application of a limited quantity of nanofillers will raise the interfacial shear stress between the matrix and macro-filler, improving the composites' mechanical and physical characteristics [13]. Arao et al. [13] mixed various nanofillers, including alumina, carbon nanotube (CNT), silica, and nano-clay, with short carbon fiber/polypropylene composites. They came to the conclusion that, with the exception of nano-clay, the addition of maleic anhydride and nanofillers can boost the interfacial shear strength between carbon fibers and polypropylene by up to 60%. Additionally, the matrix's strength, modulus, and crack resistance are all enhanced by the use of nanofillers. The automobile industry uses a lot of thermoplastic polymers, including nylon, polypropylene, polyamide 6, polyamide 66, polyethylene, polycarbonate, and Acrylonitrile Butadiene Styrene (ABS). Thermoplastic composites can be filled with nanofillers such graphite nano-platelet (GNP) [21], graphene oxide [22], carbon nanotube (CNT) [23], SiO₂ nano-sphere [20, 24, 25], and TiO₂ nano-sphere [26], as well as micro-scale materials like short carbon fiber (SCF) and glass fiber [7]. Researchers are also interested in the integration of nanofiber into polymers [27, 28].

Thermoplastic composites reinforced with micro- and nanofiller were processed using standard techniques. To distribute the filler throughout the thermoplastic matrix, pre-mixed materials containing microfiller, nanofiller, and thermoplastic are added in varying proportions. Using a twin-screw extruder, thermoplastic composites with different filler materials can be melt-mixed to create pellets of composite material, which can then be injection molded to create a thermoplastic composite component. The investigation of the synergistic reactions between the binary and ternary filler materials in Polyethylene (PE)-matrix composites is the innovative aspect of this work. In order to do this, PE was reinforced with TiO₂ nanoparticles, graphite nano-platelets, and short carbon fiber (SCF) to create hybrid polymer Nano composites.

II.MATERIALS AND METHODS

2.1. Materials

The PE homopolymer in the form of particles, which was used as the matrix and subjected to tensile testing, flexural tests, and impact studies. At 230 °C and 2.16 kg of load, the MFI of the PE was 22.7 g/10 min. Instead of using pellets, particulate PE was utilized to improve the filler dispersion in the matrix through mechanical mixing. The particle PE has an average diameter size of 560 μm. An SCF measuring 9-10 μm in diameter and 150 μm on average length, which has a surface area of 325–375 m²/g, diameter <1 μm, thickness of ~3 nm, and a true density of 2.16 g/cm³. TiO₂ nanoparticles (TiO₂) with an average diameter of 30 nm. Table 1 lists every additional characteristic of the raw materials used in this investigation.

Table.1 Properties of the raw materials

Properties	Unit	PE	SCF	GNP	TiO ₂
Grade	-	Homopolymer isotactic	PAN-based	Asbury Nano307	Rutile
Density	g/cc	0.9	1.73–1.79	2.16	4.23
Tensile strength	MPa	27.6	2000–3800	-	-
Young's modulus	GPa	1.5	180–240	-	-
Shape	-	Particulate	Fiber	Sheet/plate	Sphere

PE: polyethylene; SCF: short carbon fiber; GNP: graphite nano-platelet; TiO₂: Titanium dioxide.

The composition mentioned in the Table.2 was followed in the weighing of the PE/SCF, PE/nanofillers, and PE/SCF/nanofillers. To disseminate the carbon fiber and/or nanofillers into the PE matrix, they were first pre-mixed for 15 minutes at room temperature using a mechanical mixer set to 500 rpm. They were next shown the twin-extruder apparatus. The pre-mixed raw ingredients were put into a pelletizer to create pellets after being extruded at a predetermined temperature with ten distinct zones between 160 and 210 °C and a speed of 50 rpm to create a strand. The twin-screw extruder has a 40 L/D (length to diameter ratio) and a 16 mm screw diameter. To create testing samples of the composites, the pellets of composite were subsequently placed into an injection molding machine with a barrel temperature range of 180, 195, and 220°C.

Table.2 Composition of the polyethylene composites

Sample Name	Compositions	wt % SCF	wt % GNP	wt % TiO ₂
S1	Neat PE	0	0	0
S2	PE/5SCF	5	0	0
S3	PE/10SCF	10	0	0
S4	PE/15SCF	15	0	0
S5	PE/20SCF	20	0	0
S6	PE/1GNP	-	1	-
S7	PE/2.5GNP	-	2.5	-
S8	PE/5GNP	-	5	-
S9	PE/1nTiO ₂	-	-	1
S10	PE/2.5nTiO ₂	-	-	2.5
S11	PE/5nTiO ₂	-	-	5
S12	PE/10SCF/1GNP	10	1	-
S13	PE/10SCF/2.5GNP	10	2.5	-
S14	PE/10SCF/5GNP	10	5	-
S15	PE/10SCF/1nTiO ₂	10	-	1
S16	PE/10SCF/2.5nTiO ₂	10	-	2.5
S17	PE/10SCF/5nTiO ₂	10	-	5
S18	PE/4GNP/1nTiO ₂	-	4	1
S19	PE/2.5nTiO ₂ /2.5GNP	-	2.5	2.5
S20	PE/10SCF/4GNP/1nTiO ₂	10	4	1
S21	PE/10SCF/2.5GNP/2.5nTiO ₂	10	2.5	2.5

III. RESULTS AND DISCUSSION

3.1 Fillers Characterization

Figure 1a, 1b illustrates the SCF morphology. The majority of the fibres, as seen in Figure 1a, are longer than 100 μm . An enlarged image of Figure 1b reveals that the fiber's diameter was approximately 7 μm and its surface was unsized. The manufacturer said that the average length of the fiber measured was 150 μm . Figure 2c depicts the GNP morphology. It is clear that the GNP's diameter was less than 1 μm . In the meantime, the GNP's thickness was at the nanoscale. The TiO₂ morphology is displayed in Figure 1d. According to the manufacturer, the majority of the particles had a diameter of less than 30 nm. The characterization of the as-received filler was in agreement with the manufacturer specifications.

The surface area of the fillers is shown in Table 2. The filler's known dimensions were used to calculate the surface areas of SCF and TiO₂. The dimensions of the SCF were 7 μm in diameter, 150 μm in length, and 1.79 g/cm³. In contrast, the TiO₂ was spherical in shape, with a diameter of 30 nm and a density of 4.23 g/cm³. The supplier datasheet was used to determine the weight and surface area of GNP. In the meantime, the surface area/weight was multiplied by its density (2.16 g/cm³) to determine the surface area/volume. Of the three fillers, it was found that the GNPs had the largest surface area.

Table.2 The surface area of the three fillers

Filler	Surface Area/Weight (m ² /g)	Surface Area/Volume (m ² /cm ³)
SCF	0.33	0.59
GNP	350	756
nTiO ₂	48	200

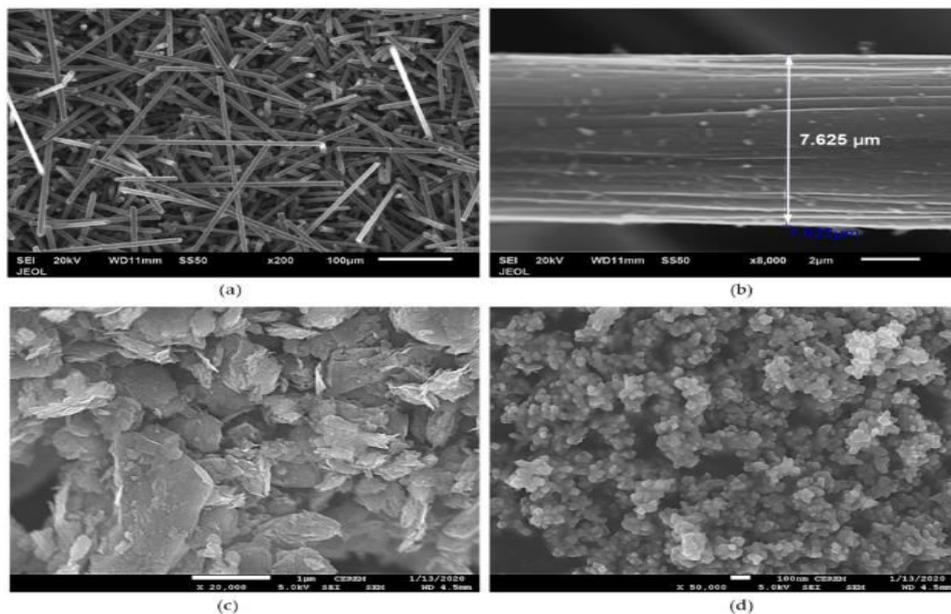


Fig.1 SEM images of raw materials (a,b) short carbon fiber at different magnifications, (c) graphite nano-platelet, (d) titanium dioxide nanoparticles.

A comparison of the fiber lengths of the PE/SCF composite and PE/10SCF/Nanofiller hybrid composites as-received and after processing is shown in Figure 4. As received, the SCF had an average fiber length of 150 μm, which was in line with the manufacturer's standard. All composites showed, on average, a reduction in fiber length as compared to the SCF that was received. Fiber breakage may result from production procedures such mixing, extrusion, and injection molding [37]. The hybrid composites PE/10SCF/5GNP and PE/10SCF/2.5GNP/2.5TiO₂ show a higher average fiber length reduction than the PE/10SCF composite. In contrast to PE/10SCF, virtually no decrease in fiber length was seen for PE/10SCF/5TiO₂. It was shown that the average fiber length after processing decreased as a result of the addition of GNPs. This might be the result of GNP addition-induced increases in viscosity or MFI decreases. Shear forces during processing may have increased as a result of the viscosity rise. The increase in fiber breaking may be explained by higher shear forces [38].

3.2 Microstructures

The composite with a 15wt% SCF loading is shown in Figure 2a. Red circles indicate the fibers that have already begun to clump together. When adding nanofillers to the SCF loading at 10 wt% (Figure 2b–d), very little agglomeration was seen. The longitudinal section of the same composites is displayed in Figure 2. The arrow at the bottom of the picture indicates the direction of the injection molding. Different skin and core structures are visible in the microstructure photographs. The fibers in the samples' skin are oriented perpendicular to the injection direction, whereas the fibers in the samples' cores tend to align with the direction of the injection molding for all microstructures. Others have previously reported on the various architectures seen in the skin and core of the injected molding fiber reinforced polymer.

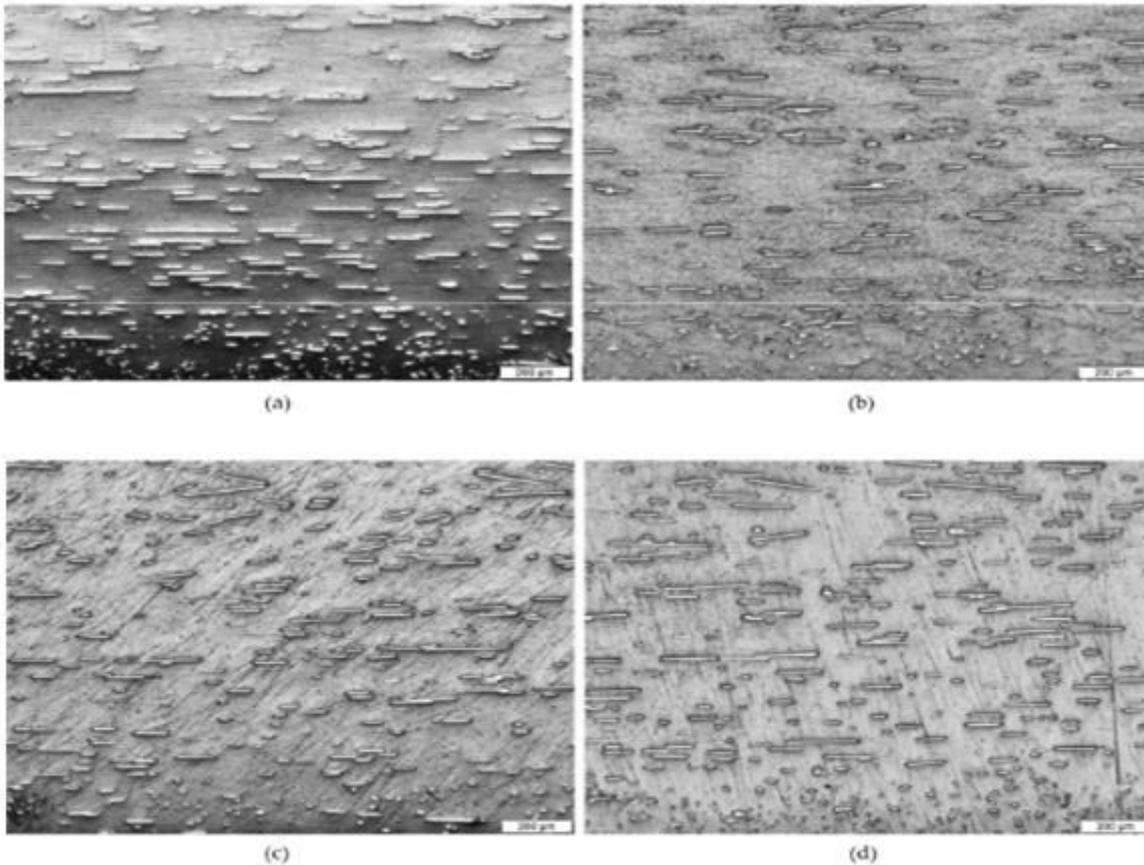


Fig.2 Optical microscope images of longitudinal section surfaces

3.3 Tensile Test

The mechanical characteristics obtained from the tensile experiments. For every combination taken into consideration in this investigation, the tensile modulus and UTS of the composites rose with the addition of carbon fiber or nanofillers. The fact that all filler types' tensile strengths have increased suggests that the fiber and nanofiller material were successfully dispersed throughout the PE matrix through the usage of particulate PE in conjunction with a twin-extruder and mechanical stirrer. The mechanical characteristics of PE/SCF composites are depicted in Figure 9. The tensile modulus of PE/SCF composites rose to 6314 MPa for loadings up to 20 wt% SCF, or 365% more than that of clean PE. In a similar vein, the UTS rose from 27.6 to 46.9 MPa by 70%. At break, the strain dropped from 845% to just 5.3%. Previously, other studies had documented similar behavior [44, 45]. The load transfer from the PE matrix to SCF may be the cause of an increase in the tensile strength and modulus. This is due to the extremely high strength and modulus as well as the extremely low ductility of carbon fiber. Because of the shear stress created by the screws on the extrusion machine and the interaction between the fibers, during the manufacturing process, fibers may break into shorter, shorter fibers [7, 37]. This condition is verified by the study's results. More fiber ends are produced by a shorter fiber. The failure path would preferentially stop where these fibers finish and fiber perpendicular to the direction of tension loading. Carbon fibers are near to one another at 10 wt% loading, and in certain areas, they begin to form agglomerations. As previously discussed in the subsection on the tensile test fracture surface, these agglomerations can lead to a concentration of stress at the ends of the fibers and the production of voids that may be responsible for reducing the strain at break. Figure 10a–c compares the strain at break, UTS, and modulus of elasticity for composites made with various fillers as a function of filler weight fraction. The greatest tensile modulus for the PE/GNP and PE/TiO₂ composite systems was demonstrated by PE/5GNP up to a five wt% loading of nanofillers. The strain at break remained at 514%, while the tensile modulus increased by 58% and the UTS increased from 27.6 to 33.6 MPa. Figure 10b compares the three types of fillers at a 5 wt% loading and reveals that GNP has a greater potential to influence the UTS than SCF and TiO₂. Of the three fillers, it is clear from the filler characterization (Table 3) that GNP has the biggest surface area. The interaction between the GNP and PE chain molecules is enhanced by the larger surface area.

PE chain molecule mobility and stress transfer from the matrix to GNP are subsequently inhibited as a result of this interaction. According to earlier reports, GNPs can also raise the polymer's degree of crystallinity [46], which suggests that they can also raise the polymer's strength.

In the meantime, the characteristics of PE/GNP/TiO₂ composites at 5 wt% total loading are logically in between those of PE/5GNP and PE/5TiO₂. The strain at break, UTS, and modulus of elasticity for PE/10SCF/nanofiller for various nanofillers at varying weight percentage loading are shown in Figure 3. These composites showed the same tendencies as the PE/nanofiller. When compared to the PE/10SCF composite, the maximum tensile modulus and UTS of the PE/10SCF/GNP composites rose by approximately 27% and 20%, respectively, at 5 wt% of GNP (Figure 11a, b). Additionally, the PE/10SCF composite's strain at break decreased from 7.8% to 8.7% for the PE/10SCF/2.5GNP composite (Figure.3). The bridging effect of GNP produced a plausibly increased ductility that was perpendicular to the direction of the tensile stress [21]. In the meantime, the addition of GNP resulted in a loss in ductility because of the material's increased agglomeration and orientation parallel to the direction of the tensile load [47]. In addition, PE/10SCF/TiO₂ composites demonstrated a tensile modulus increase of up to 18.5% at 5 wt% TiO₂ and a UTS increase of up to 10% at 2.5 wt% TiO₂ in comparison to the PE/10SCF composite. The UTS then dropped at 5 wt% of TiO₂. Agglomeration of TiO₂ was identified as the cause of the UTS drop at 5 wt% of TiO₂. As shown in Figure 3, strain at break increased from 7.8% for the PE/10SCF composite to 9% for the PE/10SCF/2.5TiO₂ composite. A spherical nanoparticle's aggregation may result in crack shrinking [48] or the debonding of nanoparticle agglomerates, which promotes the formation of plastic voids [49, 50]. While the presence of spherical TiO₂ is more effective in improving the strain at break, the addition of GNP generally improves the tensile modulus and UTS of the composites. Rasana and Jayanarayana [51] and Rasana et al. [52] have also reported on the enhancement of toughness and ductility on fiber-reinforced polymer by the addition of spherical shape nanoparticles at specific content.

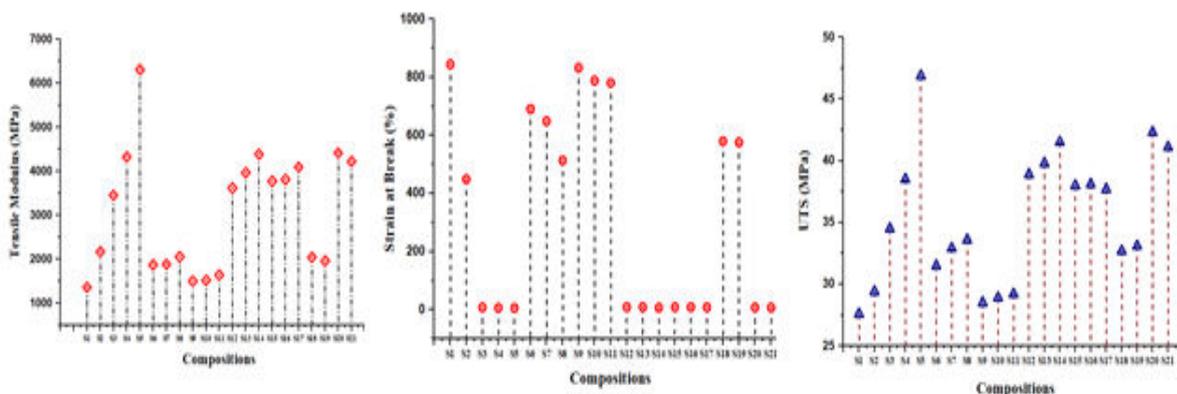


Fig.3 various composites tested Tensile Modulus, strain brake and UTS

The composites' tensile modulus, UTS, and strain at break values at 15% loading with various filler combinations. When compared to other compositions at the same weight percentage loading, the PE/10SCF/4GNP/1TiO₂ composite showed superior properties. The characteristics remained below those of 20 wt% SCF. As seen in Figure 3, the addition of various fillers raised the tensile modulus and UTS relative to the clean PE by 226% and 53%, respectively. On the other hand, when comparing the PE/10SCF/4GNP/1TiO₂ composite with PE/15SCF, the hybrid composite's UTS increased by about 10%, while its tensile modulus increased by nearly none. The improved filler dispersion caused by the presence of several filler types was identified as the cause of the strength enhancement. In order to enhance the dispersion of nanofillers, Yang et al. [29] and Chatterjee et al. [30] also combined two distinct nanofillers. In contrast to the PE/15SCF composite, the strain at break increased to 7.7% of strain, a 20% increase.

3.4 Flexural Test

The flexural results of the tensile and flexural tests concurred. Flexural strength and modulus for the PE/SCF composite system increased to 240% and 100%, respectively, with an increase in SCF loading up to 20 wt%. But when compared to the composite's increased UTS, the flexural strength increase was greater. This happened as a result of the flexural test's non-uniform stress distribution, which happened over a smaller volume as opposed to the tensile test's uniform stress distribution over a greater volume. Din and Hashemi [53] reported the same behaviour.

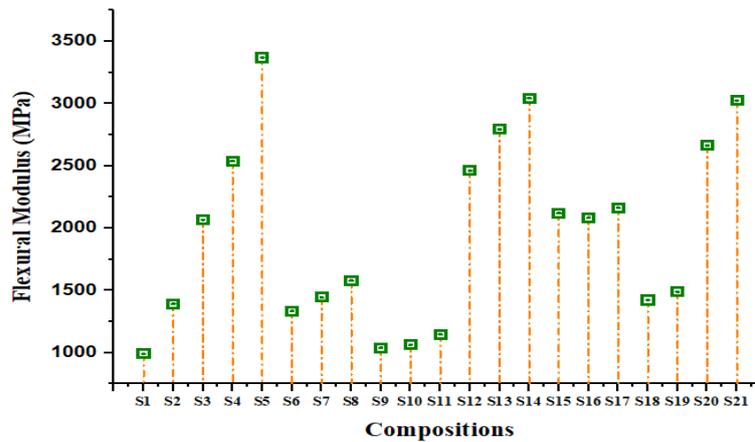


Fig.4 various composites tested Flexural Modulus

3.9. Impact Test

Impact Test that adding carbon fiber or nanofillers improved the composites' impact toughness. At 20 wt% loading of SCF, the highest impact toughness was 3.6 KJ/m², which is a 75% increase over the impact toughness of neat PE. As previously mentioned in the impact fracture surface section, the addition of SCF may have improved impact toughness by causing crack deflection. Furthermore, the fracture process, which includes fiber pull-out and fiber fracture, may potentially be the cause of this improvement [54]. The impact toughness of the PE/GNP composite system is increased by GNP from 2 KJ/m² for neat PE to 2.7 KJ/m² for 1 and 2.5 wt% loading. However, the impact toughness of the composite decreases with increased loading up to 5 wt%. The bridging effect of GNP [21] and the decrease brought on by agglomeration and GNP orientated parallel to the impact fracture surface [47] may also be responsible for the increase in impact toughness. On the other hand, the impact toughness of the PE/TiO₂ composite increased to 2.6 KJ/m² when the TiO₂ loading was increased to 5%. The impact toughness of the composite is enhanced by the TiO₂ through the processes of crack blunting [48] and plastic void formation [49, 50]. In comparison to PE/GNP and PE/TiO₂ alone, the combination of GNP and TiO₂ at 5 wt% total loading in PE/GNP/TiO₂ composites can increase the impact toughness for PE/2.5GNP/2.5TiO₂ composites by up to 3 KJ/m².

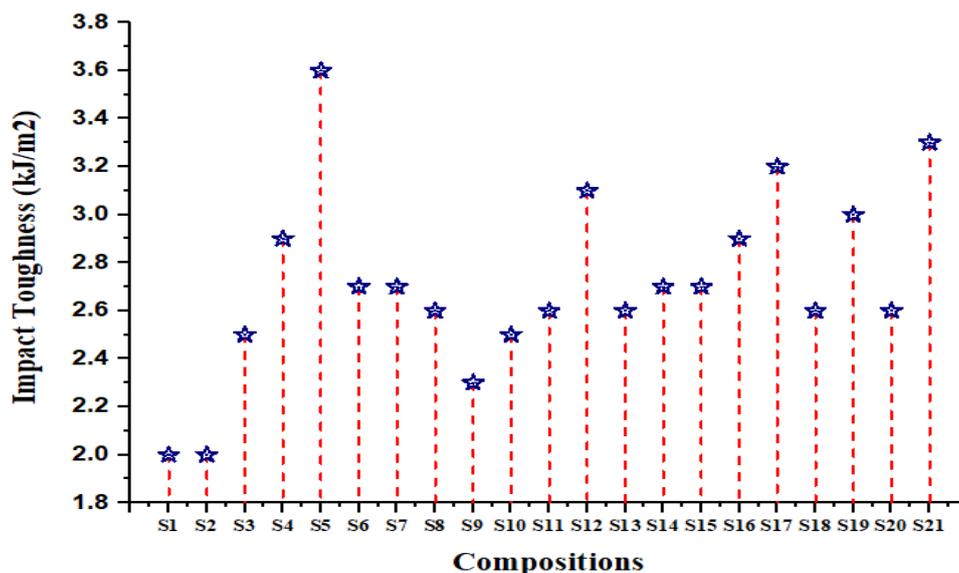


Fig.5 various composites tested Impact Toughness

Impact toughness can be increased by combining different nanofillers to increase the dispersion of nanofiller in the composite [30]. A maximum impact toughness of 3.1 KJ/m² was observed for PE/10SCF/GNP composite systems at 1

wt% of GNP, and it began to decrease with the addition of more GNP. The PE/GNP system likewise showed this decline. The addition of TiO₂ to PE/10SCF/TiO₂ composites results in a progressive increase in impact toughness up to 5 wt% and 3.2 KJ/m², which was similarly noted in PE/TiO₂ composites. Similar to PE/nanofillers composites, PE/SCF composites' impact toughness was enhanced by the inclusion of nanofillers. The comparison of notch Izod impact toughness at various composites with 15% total loading is shown in Figure 5. The results indicate that the PE/10CF/5TiO₂ composite and the PE/10CF/2.5GNP/2.5TiO₂ composite had the maximum impact toughness, with a value of 3.2 KJ/m². These composites outperformed PE/15SCF by 10% and increased by 60% in comparison to neat PE. The impact hardness may be increased by combining several nanofiller types.

IV. CONCLUSIONS

Combinations of SCF, GNP, and TiO₂ were used to create PE-based composites. When SCF, GNP, and TiO₂ were added, as well as when they were combined, the MFI dropped. Nonetheless, compared to SCF and TiO₂ fillers, GNP had a greater impact on MFI. The addition of SCF and nanofillers enhanced the density of the composites when compared to clean PE. The theoretical density and the measured density agreed. The addition of weight percentage of SCF or nanofillers resulted in an increase in the modulus of elasticity and UTS of the PE/SCF, PE/nanofiller, and PE/SCF/nanofiller composites. The ductility of PE/SCF and PE/10SCF/nanofillers tended to decrease dramatically. PE composites at 15 wt% loading with various fillers, PE/10SCF/4GNP/1TiO₂ and PE/10SCF/2.5GNP/2.5TiO₂, exhibited superior tensile modulus and improved UTS and strain at break in comparison to the PE/15SCF composite. In comparison to the PE/15SCF composite, the PE/10CF/2.5GNP/2.5TiO₂ hybrid composite exhibited a 13% and 20% increase in flexural modulus and flexural strength, respectively. In comparison to the composites made of PE/10SCF and PE/15SCF, the strain at break for PE/10CF/2.5TiO₂ was 15% and 41% higher, respectively. When compared to neat PE, the notch Izod impact toughness generally increased with the addition of SCF, GNP, and TiO₂ and their combinations. When compared to neat PE and the PE/15SCF composite, the hybrid composite PE/10CF/2.5GNP/2.5TiO₂ improved by 65% and 14%, respectively, at maximum filler loading weight percentage of 15%.

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