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Effect of Chemical Treatment and Titanium Dioxide Nanoparticles on the Impact Strength of Miscanthus Fiber-Reinforced Polypropylene Composites Tailored for Helmet Applications

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ABSTRACT

The quest for the use of natural fibers in the reinforcement of polymers has risen due to various advantages of natural fibers over their synthetic counterparts. This study investigated the effects of chemical treatment and titanium dioxide nanoparticles in the impact strength of *miscanthus* fiber-reinforced polypropylene composites tailored for helmet application. Water retting was employed to extract the fibers from the stems of *miscanthus* grass. The extracted fibers were washed, chemically treated, oven-dried, pulverized, and sieved to a fine particle size. Titanium dioxide nanoparticle contents of 5 wt% to 20 wt%, an interval of 5%, were used. *Miscanthus* fiber-reinforced polypropylene composite samples, with or without titanium dioxide nanoparticles, were produced via the injection molding process. The impact test on the set of composite samples was conducted by ISO 179 standards. The results indicated that the chemically treated *miscanthus* fiber-reinforced polypropylene composite had a better impact strength than the untreated *miscanthus* fiber-reinforced polypropylene composite. The addition of titanium dioxide nanoparticles enhanced the impact strength of the composite. This study concludes that chemical treatment of *miscanthus* fibers and the addition of titanium dioxide nanoparticles are needed to enhance the mechanical properties of *miscanthus* fiber-reinforced polypropylene composite tailored for helmet application.

1. Introduction

The quest for environmentally friendly, available, renewable, and low-cost materials for industrial applications has renewed the interests of researchers on natural fibers over the last two decades [1-8]. Natural fibers being explored are fibers obtained from natural sources such as plants, animals, and minerals [3].

Natural fiber-reinforced polymer composites are obtained by reinforcing polymeric materials with natural fibers. In such composites, natural fibers are the reinforcing materials, and the polymers are utilized as the matrix materials. Many natural fiber-reinforced composites have been developed and are being utilized as possible replacements or alternative materials for non-renewable and environmentally unfriendly materials being used in various manufacturing industries today [4, 5, 9, 10]. The quest for the use of natural fibers in the reinforcement of polymers has risen due to various advantages of natural fibers over their synthetic counterparts, which include low density, good mechanical properties, low cost, abundance, and biodegradability [3, 11-13].

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Many plant fibers have been investigated by researchers for their possible use in polymer composites for various industrial applications. Some of the natural fibers that have been investigated for use in polymer composites include fibers obtained from jute [14], ramie [7], rice straw [15], bamboo [8, 16], hazelnut shells [17], hemp [18, 19], kenaf [20], sisal [1], plantain [4, 5, 21], coconut [6], okra [22], fax [23], oil palm, and wood [24].

The main drawback in the use of natural fibers in polymer composites, which has been identified by researchers, is the hydrophilic nature of natural fibers and poor fiber-matrix adhesion [25]. This hydrophilic nature affects the mechanical properties of the composite, durability, and their applications in various endeavors [3, 10]. The drawbacks are overcome through surface modification (chemical treatment) of the natural fibers [25]. Chemical treatments of natural fibers such as mercerization, acetylation, and salinization, among others, have been found to enhance fiber-matrix interaction, improve wettability, reduce moisture absorption, and increase the mechanical properties of the composites [26-28].

Many researchers have examined the effects of chemical treatments on the mechanical properties of various natural fiber-reinforced polymer composites developed for various industrial applications [3, 12, 13, 29-35]. For instance, James et al. [12] examined the effects of chemical treatment (alkaline-saline treatment) on the physical and mechanical properties of coir fiber-reinforced polypropylene composites and reported superior performance of the composites as a result of the treatment. Narayana et al. [31] investigated the effect of chemical treatment on the mechanical properties of fiber-reinforced composites. The fibers examined were fibers obtained from Thespesia lampas and Hibiscus cannabinus plants. Feng et al. [29] examined the effect of alkali and silane treatments on exemplary mechanical properties of kenaf and pineapple leaf fiber-reinforced polypropylene composites and found that chemical treatment enhanced the mechanical properties of the composites.

Impact strength is one of the major mechanical properties needed to enhance natural-fiber reinforced polymer composites. According to Navaranjan and Neitzert [36], impact resistance is the ability of a material to withstand a shock loading or an applied stress at high speed. Inorganic nanoparticles such as titanium dioxide have been reported to increase the mechanical properties (impact strength inclusive) of natural fiber-reinforced polymer composites [37, 38]. Ihueze et al. [39] determined the optimum *miscanthus* fiber content that optimized the quality characteristic (impact strength) of *miscanthus* fiber-reinforced polypropylene composites. However, the study did not consider the influence of inorganic nanofillers on the composite. There is a need to study the influence of nanoparticles, such as titanium dioxide nanoparticles, on the impact strength of *miscanthus* fiber-reinforced polypropylene composite tailored for helmet application.

Some studies have investigated the effects of titanium dioxide nanoparticles on natural fiber reinforced polymer composites [37, 38, 40-50]. For instance, Awang et al. [38] studied the effects of the addition of titanium dioxide on the mechanical properties of rice husk reinforced polypropylene composites produced through the injection moulding process. The results showed an improvement in the mechanical strength of the composite with titanium dioxide when compared with pure polypropylene and the composite without titanium dioxide.

Masoudifar et al. [37] studied the effects of surface treatment and titanium dioxide nanoparticles on the mechanical and morphological properties of wood flour/polypropylene nanocomposites produced using the injection moulding method. The results showed that the mechanical properties of the composites increased when the TiO_2 nanoparticle levels were between 1 and 3 parts per hundred of the compound (phc), and decreased above 3 phc. Suresha et al. [40] studied the effect of TiO_2 filler loading on the physico-mechanical properties and abrasion of jute fabric reinforced epoxy composites prepared using the hand lay-up method. The results showed an improvement in the mechanical properties up to 7.5 wt% TiO_2 loading. Iyer et al. [46] investigated the influence of TiO_2 nanoparticle modification on the mechanical properties of basalt-reinforced epoxy composites produced using a combination of hand lay-up and compression moulding techniques. The study showed significant improvement in the mechanical properties of the composites with nanoparticles compared with the composites without TiO_2 up to 4 wt% filler loading, and a decrease in properties above that.

Kaymakci [50] investigated the effect of titanium dioxide on some mechanical, thermal, and surface properties of wood-plastic nanoparticles produced using the injection moulding process with TiO_2 contents of 1–5 wt%. The results indicated that the mechanical properties of the composite increased with an increase in nanoparticle content. Gopal et al. [43] investigated the influence of TiO_2 nanoparticles on the mechanical and thermal behaviour of sisal/jute fiber-reinforced interpenetrating polymer network (epoxy/polyurethane) composites produced through the compression moulding process. The results indicated that the incorporation of TiO_2 increased the mechanical properties of the composites compared with the composites without the nanoparticles.

Though many studies have investigated the effects of nanoparticles or nanofillers on natural fiber-reinforced polymer composites, none was found, to the authors' best of knowledge, on the effect of titanium dioxide nanoparticles on *miscanthus* fiber-reinforced polypropylene composite. Hence, this is a research gap that needs to be filled. This study is significant as chemical modification of *miscanthus* fibers and introduction of titanium dioxide nanoparticles in the composite will enhance the impact strength of the composite and increase its usability in helmet applications.

Hence, this study is aimed at the investigation of the effects of chemical treatment and titanium dioxide nanoparticles on *miscanthus* fiber-reinforced polypropylene composite tailored for helmet applications. The following objectives were pursued: (1) to determine the optimum titanium dioxide nanoparticle content for optimum impact strength of *miscanthus* fiber-reinforced polypropylene composite; (2) to assess the effect of titanium dioxide nanoparticles on the impact strength of *miscanthus* fiber-reinforced polypropylene composite; and (3) to investigate the effect of chemical treatment on the impact strength of the composite.

2. Materials and Methods

2.1 Materials

The materials used were *miscanthus* fiber particles, polypropylene, maleic anhydride grafted polypropylene (MAPP), and titanium dioxide nanoparticles. *Miscanthus* fibers, polypropylene, and titanium dioxide nanoparticles were obtained from Anambra State, Nigeria. The maleic anhydride grafted polypropylene used in this study was purchased from Hebei Jintian Plastic New Material Company Limited, China. The polypropylene used in this study was SASOl HRV 140. The typical properties of SASOL HRV 140 polypropylene as obtained from SASOL Data Sheet are: melt flow index (230^0C/ 2.16 kg) of 20 g/10 min (ISO 1133) and density of 0.905 g/cm^3 (ISO 1183-1), and impact strength of 3 kJ/m^2 determined using ISO 179-2 standard. Table 1 shows the properties of *miscanthus* fiber and polypropylene used in this study.

Table 1: Mechanical Properties of SASOL HRV 140 Polypropylene (Source: Company's Product Data Sheet) and *Miscanthus* Fiber [3]

Material	**Property**	**Value (SI)**	**Method**	**Source**
Propylene	Flexural modulus	1550 MPa	ISO 178	SASOL Product Data Sheet
	Tensile modulus of elasticity	1600 MPa	ISO 527-2	
	Tensile stress at yield	34 MPa	ISO 527-2	
	Tensile strain at yields	8.0%	ISO 527-2	
	Charpy notched impact strength (23^{o}C)	3.0 kJ/m^2	ISO 179-1	
	Ball indentation hardness – HB	73 N/mm^2	ISO 2039-1	
Miscanthus	Tensile strength (NaOH treatment only)	627 MPa	ASTM D638	[3]
	Tensile strength (NaOH + Acetic Anhydride treatment)	1262 MPa	ASTM D638	
	Tensile Modulus of Elasticity	33.9 GPa	ASTM D638	
	Elongation at peak	3.7%	ASTM D638	

The following reagents with 99% purity: sodium hydroxide (NaOH), acetic acid and acetic anhydride used in the chemical treatments of the extracted fibers were obtained from Anambra State, Nigeria.

2.2 Fiber Extraction and Treatment

Miscanthus fibers were extracted from *miscanthus* giganteus stem (Fig. 1), after retting in water for 30 days and washed in clean water. The extracted *miscanthus* fibers were treated with 3.2% NaOH solution, neutralized with 10% acetic acid, and washed in distilled water. The treated fibers were further treated with 10% acetic anhydride solution. The treated fibers were washed, dried at room temperature for 48 hours, and further subjected to oven drying at 80^{o}C for 12 hours using Lab-Tech Oven. The dried *miscanthus* fibers were ground into powder and sieved to fine particles following ASTM D100 standards.

Fig. 1: *Miscanthus* Giganteus Grass [3]

2.3 Preparation of *Miscanthus* Fiber-Reinforced Polypropylene Composite

First, *miscanthus* fiber-reinforced polypropylene samples were prepared using 25 wt% fiber loading and 75 wt% polypropylene, and labeled 75PPMF. To ascertain the effects of inorganic titanium dioxide nanoparticles on MFRPP composites, matrix-fiber-filler composite mixtures were prepared as shown in Table 2. The *miscanthus* fiber content in all the samples was kept constant throughout the experiments at 25 wt%. The polypropylene contents in the composites ranged from 55 wt% to 75 wt%, and filler contents ranged from 0 wt% to 20 wt%. MAPP was applied at 1.5 wt%. The samples were coded 75PPMF, 70PPMF5T, 65PPMF10T, 60PPMF15T, and 55PPMF20T, as shown in Table 2. PP, MF, and T represent polypropylene, *miscanthus* fiber, and titanium dioxide, respectively. 70PPMF5T implies 70 wt% of polypropylene, 25 wt% of *miscanthus* fiber, and 5 wt% of titanium dioxide. The mixtures were thoroughly mixed using a mechanical stirrer, and the composite samples were produced using the injection moulding process. The fiber content was kept constant throughout the experiments at the optimum material and process parameter settings already established in Ihueze et al. [39]. A melt temperature of 200°C, an injection speed of 150 rpm, a fiber content of 25 wt%, and a cooling time of 10 seconds were used in the injection moulding of the samples. Fig. 2 shows some of the samples produced through the injection moulding process.

Table 2 – Experimental Setup for matrix-fiber-filler composite Mixtures

S/N	Sample	PP Content (wt%)	*Miscanthus* Fiber Content (wt%)	Titanium Dioxide (wt%)
1	75PPMF	75	25	0
2	70PPMF5T	70	25	5
3	65PPMF10T	65	25	10
4	60PPMF15T	60	25	15
5	55PPMF20T	55	25	20

PP = Polypropylene; MF = *Miscanthus* Fiber, T = Titanium dioxide

Fig. 2 – Samples of MFRPP Composite for Impact Test

2.4 Impact Strength Test

The impact strength is directly related to the overall toughness of the material. The impact strength is a fundamental material property, as the suitability of a composite for certain applications is determined not only by usual design parameters but also by its impact or energy-absorbing properties. The impact strengths of the composite samples produced were tested using the XJJC-50 Computerized Impact Testing Machine, as shown in Figure 3. The impact strength was performed on unnotched samples with dimensions 63.3 mm X 12.3 mm X 5 mm. The impact strength tests on the MFRPP composite samples were carried out at the Strength of Materials Lab of Nnamdi Azikiwe University, Awka, by the requirements of ISO 179. The pendulum energy and the impact velocity used during the tests were 7.5 J and 3.8 m/s, respectively. Three measurements of each sample were taken, and the mean value in kJ/m^2 was presented. The test samples were conditioned at room temperature and 50% relative humidity.

Fig. 3 – XJJC Computerized Impact Testing Machine

3. Results and Discussion

3.1 Impact Strength of Miscanthus Fiber-Reinforced Polypropylene Composite Filled with Titanium Dioxide Nanoparticle

Table 3 shows the impact strengths of *miscanthus* fiber-reinforced polypropylene composites filled with titanium dioxide nanoparticles at three replications. Samples – 75PPMF with 0 wt% titanium dioxide has mean impact strength of 10.893 kJ/m^2, 70PPMF5T with 5wt% titanium dioxide nanoparticle has mean impact strength of 11.276 kJ/m^2, 65PPMF10T has mean impact strength of 12.917 kJ/m^2, 60PPMF15T has mean impact strength of 12.959 kJ/m^2, and 55PPMF20T has mean impact strength of 13.067 kJ/m^2.

Table 3: Impact Strengths of MFRPP Composite further Reinforced with Titanium Dioxide Nanoparticles

S/N	Sample	Impact Strength (kJ/m^2)			
		Response 1	Response 2	Response 3	Mean
1	75PPMF0T	10.344	10.450	11.887	10.893
2	70PPMF5T	10.426	12.565	10.838	11.276
3	65PPMF10T	12.421	12.973	13.356	12.917
4	60PPMF15T	14.372	11.677	12.827	12.959
5	55PPMF20T	12.219	13.002	13.981	13.067

Fig. 4 vividly shows the mean impact strengths of the MFRPP composite at different titanium dioxide nanoparticle loadings and fixed fiber content. From the figure, it can be observed that there is an upward trend in the impact strengths of the MFRPP composites. The impact strength began to increase from 0% titanium dioxide loading, which has the lowest impact strength of 10.893 kJ/m^2, up to 20% titanium dioxide nanoparticle weight content, which has the highest impact of 13.067 kJ/m^2. This suggests that the addition of inorganic nanoparticles on *miscanthus* fiber-reinforced polypropylene composite improves the energy absorption capacity of the composite and the impact strength. This is in line with the findings of previous studies [37, 38, 40, 51]. Al Mahmood et al. [51] recorded improved mechanical properties – tensile, impact, and flexural when titanium dioxide was added as a filler material in glass fiber reinforced epoxy composites. The result also found that the composite filled with 20 wt% of titanium dioxide gave the maximum impact strength. Suresha et al. [40] recorded an improvement in the physico-mechanical properties of jute fabric reinforced epoxy composites improvement in the mechanical properties up to 7.5 wt% TiO_2 loading. Awang et al. [38] recorded an increase in the mechanical properties of rice husk reinforced polypropylene composites on the addition of titanium dioxide. Masoudifar et al. [37] reported improved mechanical and morphological properties of wood flour/polypropylene nanocomposites with the addition of titanium dioxide nanoparticles.

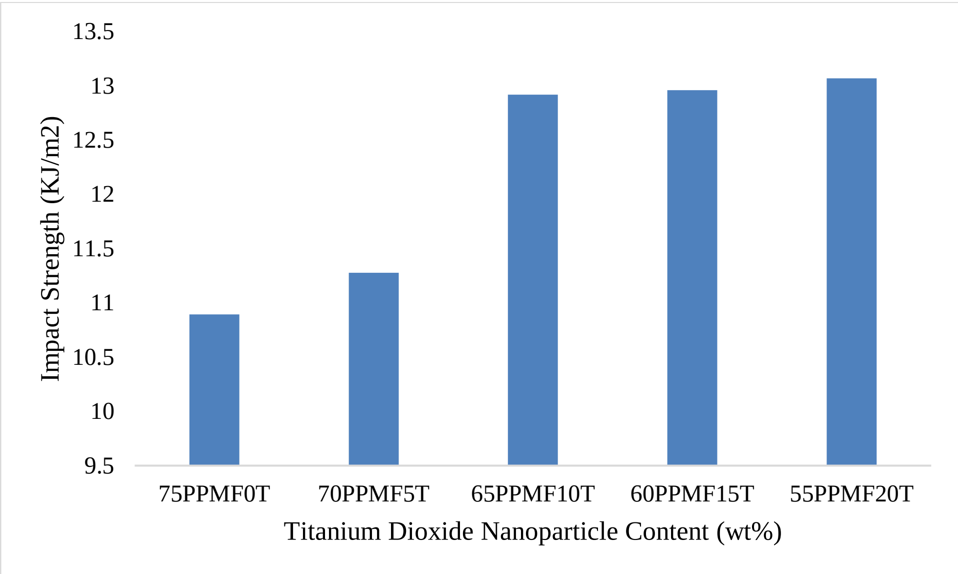

Fig. 4 – Mean Impact Strengths of MFRPP Composite at Different Loadings of Titanium Dioxide Nanoparticle

3.2 Effect of Titanium Dioxide Nanoparticle on the Impact Strength of MFRPP Composite

The effects of the addition of titanium dioxide nanoparticles on MFRPP composites were examined as shown in Fig. 5 using 75PPMF as the reference point for comparison purposes. From the figure, filling the optimum *miscanthus* fiber-reinforced polypropylene composite with 5% titanium dioxide nanoparticle (70PPMF5T) improved the impact strength of the MFRPP composite by 3.52%; 10% titanium dioxide nanoparticle (65PPMF10T) improved the impact strength by 10.58%; 15% titanium dioxide nanoparticle (60PPMF15T) improved the impact strength by 18.97%; and 20% titanium dioxide nanoparticle (55PPMF20T) improved the impact strength of the MFRPP composite by 19.96%. The figure shows that the optimal MFRPP composite treated with 20% titanium dioxide nanoparticles has the highest impact on the impact strength of the MFRPP composite, and 5% titanium dioxide nanoparticles had the least positive impact on the impact strength of the MFRPP composite. Hence, for optimum improvement of the impact strength of *miscanthus* fiber-reinforced polypropylene composite, the titanium dioxide content in the composite should be up to 20 wt%. The findings of this study are in line with previous studies on fiber-reinforced composites [51].

Also, there is a great improvement in the impact strength of all the *miscanthus* fiber-reinforced composites with or without titanium dioxide nanoparticles compared to the unreinforced or neat PP (matrix) with an impact strength of 3 kJ/m^2. This shows the positive impact of the reinforcement of the polymer matrix with natural fibers.

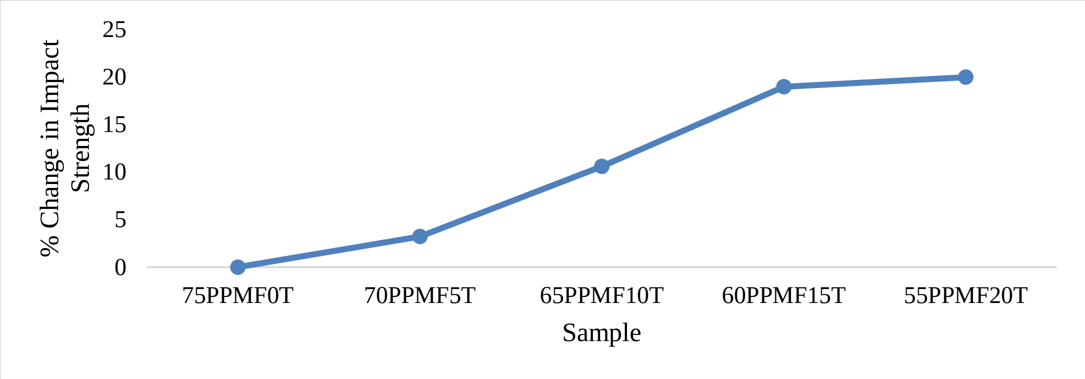

Fig. 5: Effects of Titanium Dioxide Nanoparticles on MFRPP Composite

3.3 Effect of Chemical Treatment on the Impact Strength of *Miscanthus* Fiber-Reinforced Polypropylene Composite

The effect of chemical treatment on the impact strength of *miscanthus* fiber-reinforced composite was examined by producing samples of composites with the untreated fibers using the already established optimal parameter settings and comparing the results with the results obtained with the treated fibers at the same settings. Table 4 shows the three replications of the impact strength of the untreated *miscanthus* fiber-reinforced polypropylene composites manufactured with the optimal settings (melt temperature = 200^{o}C, injection speed = 150 rpm, fiber content = 25 wt%, and cooling time = 10 seconds).

Table 4: Impact Strength of Untreated *Miscanthus* Fiber-Reinforced Polypropylene Composite

S/N	**Impact Strength (kJ/m^2)**
1	6.168
2	5.7076
3	9.742
Mean	**7.206**

Comparing the three sets of composites – untreated *miscanthus* fiber-reinforced composite (UMFRPPC), treated *miscanthus* fiber-reinforced composite (TMFRPPC), and treated *miscanthus* fiber-reinforced composite with filler – titanium dioxide nanoparticle (TMFRPP+filler), as can be seen in Fig. 6, shows that the untreated fiber-reinforced polypropylene composite has the least impact strength (7.206 kJ/m^2), followed by the treated fiber-reinforced composite (10.893 kJ/m^2), and the treated fiber-reinforced composite filled with 20% titanium dioxide nanoparticle has the highest impact strength (13.067 kJ/m^2) among the composites. The treated *miscanthus* fiber-reinforced polypropylene composite has a 51.16% improvement in impact strength compared with the untreated fiber-reinforced composite. This agreed with the previous studies that compared the impact strength of the treated and untreated natural fiber polypropylene composites [12, 29. 33]. Feng et al. [29] found that chemical treatment enhanced the impact strength of kenaf- and pineapple leaf fiber-reinforced polypropylene composites. Jacob et al. [33] recorded a great improvement in the mechanical properties of African fan palm powder-reinforced polypropylene due to chemical treatment. James et al. [12] found a great improvement in the mechanical properties of coir fiber-reinforced polypropylene composite due to chemical treatment.

The treated fiber-reinforced composite with 20% titanium dioxide nanoparticle has 81.33% improvement on the impact strength of the composite compared with the untreated *miscanthus* fiber-reinforced polypropylene composite, and 19.96% improvement compared with the treated *miscanthus* fiber-reinforced polypropylene composite without titanium dioxide nanoparticle.

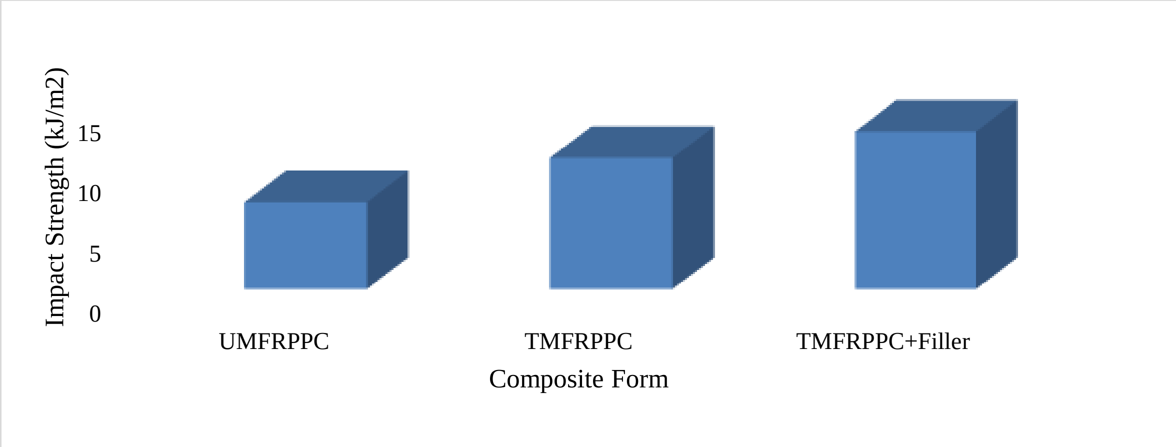

Fig. 6 – Comparison of the Impact Strengths of the Three Forms of MFRPP Composites

4. Conclusion

In this study, the effects of chemical treatments and titanium dioxide nanoparticles on the impact strengths of *miscanthus* fiber-reinforced polypropylene composites were studied. The results revealed the following:

a. Addition of titanium dioxide nanoparticles improved the impact strength of *miscanthus* fiber-reinforced polypropylene composites with the optimum result occurring at 20 wt% titanium dioxide nanoparticle content. 19.96% improvement over the MFRPP composite without titanium dioxide filler was recorded.
b. Chemical treatment of *miscanthus* fibers improved the impact strength of the MFRPP composite. The treated MFRPP composite (without filler) improved the impact strength of MFRPP by 51.16% when compared with the untreated MFRPP composite. The treated MFRPP composite with titanium dioxide nanoparticle improved the impact strength of MFRPP by 81.33% when compared with the untreated MFRPP composite.
c. There is an improvement in the impact strength of the *miscanthus* fiber-reinforced polypropylene composites with or without titanium dioxide nanoparticle when compared with the unreinforced or neat polypropylene. This shows the positive impact of reinforcement of polymer matrix with natural fibers.
d. Chemical treatment and titanium dioxide nanoparticles have a strong positive effect on the impact strength of *miscanthus* fiber-reinforced polypropylene tailored for helmet applications.

This study concludes that *miscanthus* fiber is a good reinforcement material for polymer composite applications, as shown by the high impact strength of *miscanthus* fiber-reinforced PP composite in this study. The chemical treatment of the fiber and the addition of titanium dioxide nanoparticles are needed in order to enhance the impact strength of the composite. The developed MFRPP composite filled with titanium dioxide nanoparticles can be considered for helmet applications.

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