

# The Experimental Investigation of the Effect of CNFs on the Thermal and Mechanical Properties of LDPE and HDPE

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**Abstract**—The impact of inserting various nanofillers on the mechanical, thermal, chemical, and other properties of polymers is widely investigated to attain optimal properties for several industrial applications such as in automotive, aerospace, pipelines, and others. This study investigates the effect of adding a 2% weight fraction of Carbon Nanofibers (CNFs) on the thermal, rheological, and mechanical properties of Low-Density Polyethylene (LDPE) and High-Density Polyethylene (HDPE) processed through compression molding. The produced polymer nanocomposite samples were prepared for the Thermogravimetric Analysis (TGA), rheology analysis, and tensile test. The thermogravimetric results obtained showed that CNF has no major influence on the thermal stability of LDPE and HDPE. Although the rheological properties of LDPE and HDPE improved with the addition of 2 wt% CNF in terms of storage modulus, loss modulus, and complex viscosity at various angular frequencies. The tensile test results obtained showed that 2 wt% of CNF can improve the tensile strength of LDPE by 36% and HDPE by 22% as well as their specific strength by 33% and 13% respectively. The CNF proved to be a good reinforcing agent for mechanical and rheological properties of the polymer nanocomposites however not for their thermal stability improvement.

**Keywords**—carbon nanofiber, polymer nanocomposites, specific strength, tensile strength, thermal stability

## I. INTRODUCTION

Carbon Nanofibers (CNFs) are very special nanofillers with very good mechanical properties even though they are thermally conductive [1]. CNFs are part of the Carbon Nanotubes (CNTs) family together with Multiwall Carbon Nanotubes (MWCNTs) and Single-Walled Carbon Nanotubes (SWCNTs). Although they are part of the same family, most research on carbon nanofiber-reinforced polymers has been mainly focused on CNT, MWCNT, and SWCNT-filled nanocomposites. This is owing to their remarkable mechanical properties. The CNTs are found to contain fewer microstructural defects compared to CNFs, hence they turn out to be better polymer reinforcing agents among their groups. However, CNFs have similar properties, and they are less expensive, moreover, they can be manufactured in large quantities [1]. This study focuses on the investigation of the effect of CNF nanofillers on the thermal stability, rheological, and mechanical properties (specific strength) of Low-Density Polyethylene (LDPE) and High-Density Polyethylene (HDPE). The influence of various nanofillers on Polyethylene (PE) mechanical, thermal, as well as electrical properties has been experimentally, theoretically, and numerically studied and several observations together with the conclusions were made.

Tebet *et al.* [2] conducted an experimental and numerical study on the influence of SWCNT weight fraction on the elastic properties of HDPE considering the effect of processing techniques. Their results showed that an increase in the weight fraction of SWCNT within the HDPE improved Young's modulus of the HDPE/SWCNT nanocomposites. Tebeta *et al.* [3] led an experimental investigation of the compression load effect on the elastic properties of HDPE/SWCNT nanocomposites. The obtained results demonstrated that the compression load of 3000 N provides better elastic properties of HDPE/SWCNT nanocomposites relative to the compression load of 2000 N. Fattahi *et al.* [4] developed a FEM model to analyze the elastic behavior of PE/SWCNT nanocomposites. Their model predicted that increasing the weight fraction of SWCNT enhances the elastic properties of nanocomposites. The studies conducted on CNF-based nanocomposites are looking at electrically conductive composites. This is because CNFs are electrically conductive by nature, and they can produce electrically conductive nanocomposites when added to a polymer matrix [5]. However, previous studies showed that CNF can enhance the mechanical, thermal, and electrical properties of various polymer matrices at relatively low weight fractions [6–9]. Tibbetts *et al.* [10] proved that adding 1 wt% of CNFs into an epoxy matrix improves its thermal conductivity by 45%. Further studies on how CNF influences the mechanical and thermal properties of various polymer matrices such as epoxy, PE, Polycarbonates (PC), Polypropylene (PP), Polystyrene (PS), and Poly (methyl methacrylate) (PMMA) were conducted by Choi *et al.* [11, 12], Yang *et al.* [13], Higgins and Brittain [14], Lozano and Barrera [15], Xu *et al.* [16], Jimenez and Jana [17], and Zeng *et al.* [18]. The overall result shows that CNF has a great ability to enhance the mechanical and thermal properties of the stated polymer matrices. The mentioned studies show that CNFs can be used as polymer reinforcing members for various industrial applications and there are more research studies to be conducted for a better understanding of the CNF/polymer nanocomposites. Hence the present study focuses on the influence of adding 2 wt% CNFs on the thermal stability, rheological behavior, and the specific strength of LDPE and HDPE polymer matrices.

## II. MATERIALS AND METHODS

### A. Materials and Procedures

The Carbon Nanofiber (CNF) used in this work is graphitized iron-free and composed of conical fibres of 100 nm in diameter and roughly 200  $\mu$ m in length. The LDPE,

HDPE, and polyester block (acting as intermediate phase), together with the CNF were provided by Sigma-Aldrich Chemistry, South Africa. The LDPE/2wt% CNF and HDPE/2wt% CNF nanocomposites were processed through compression moulding according to the flow diagram in Fig. 1. Before compression moulding the Scanning Electron Microscope (SEM) was performed to examine the structure and the orientation of the CNF according to Fig. 2. The processing of the pure LDPE and pure HDPE was carried out by first grinding the LDPE and HDPE pellets and then weighing 150 g of the ground LDPE and HDPE respectively. The measured LDPE and HDPE granules were dried for 16 hours at 60 °C before being extruded with the twin screw extruder at the screw speed of 120 rpm and 100 rpm at the melting temperature of 160 °C and 190 °C respectively. The extruded LDPE and HDPE stripes were then ground into pellets and dried overnight in the oven at 60 °C. The mixing of the LDPE with CNF and that of HDPE with CNF was performed by first weighing 141.6 g of grounded LDPE and HDPE pellets in two separate beakers, then adding 3 g of CNF and 5 ml of polyester block in each beaker and stirrer to achieve a better mixture of LDPE/2wt% CNF and HDPE/2wt% CNF. The two mixtures were then dried and extruded according to the procedure used for pure LDPE and pure HDPE. All pure LDPE, pure HDPE, LDPE/2wt% CNF, and HDPE/2wt% CNF pellets were then proceeded to the next step for sample preparations.

### B. Samples Preparations

The production of the rheology and the tensile test samples was performed using the compression moulding processing techniques as stated. The dried pellets for pure LDPE, pure HDPE, LDPE/2wt% CNF, and HDPE/2wt% CNF were then processed at the following compression parameters: The pure LDPE and LDPE/2wt% CNF were processed at compression temperature of 160 °C whereas pure HDPE and HDPE/2wt% CNF was processed at 190 °C, the heating was set to 4 minutes and the compression mass of 2 tons was imposed for 4 minutes to produce the rheology and tensile test samples conforming to the ASTM standard D 638-02a Type V<sup>C,D</sup>. Fig. 1 summarises the stated process using LDPE/2wt% CNF as an example.

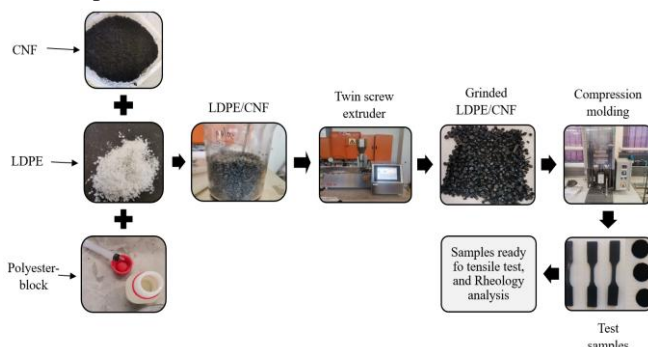


Fig. 1. Material processing flow diagram.

## III. RESULTS AND DISCUSSION

### A. CNF SEM Characterisation

Scanning Electron Microscopy (SEM) was used to analyse the structure of pure Carbon Nanofibers (CNF) used. According to AL-Saleh and Sundararaj [19], the

Vapor-Grown Carbon Fibers (VGCNFs) are entangled and agglomerated when viewed under a microscope (SEM micrograph). This is influenced by the way they are produced. VGCNF is normally produced through the pyrolysis of carbon monoxide on a metal catalyst like iron or a hydrocarbon feedstock. This processing technique is cost-effective and the most promising for the mass production of the VGCNF [20, 21]. Hence the SEM analysis was conducted to examine the structure of the CNF as presented in Fig. 2.

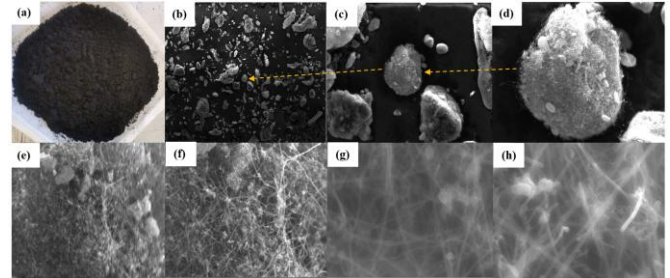


Fig. 2. The SEM images of the CNF at various magnifications.

Fig. 2 (a) is the CNF, (b) is the SEM image of the CNF at the magnification of 24 × with a scale of 200 μm, (c) CNF magnified to 150 × at a scale of 100 μm, (d) CNF magnified to 389 × at a scale of 20 μm, (e) CNF magnified to 1.06 k × at a scale of 10 μm, (f) CNF magnified to 2.06 k × at a scale of 2 μm, (g) CNF magnified to 9.75 k × at a scale of 1 μm, and (h) CNF magnified to 12.81 k × at a scale of 1 μm. The SEM images in Fig. 2 were obtained to analyze the structure of CNF at a micro level. Based on the images of higher magnifications, it shows that CNF is made of nano strings/nanofibers entangled together to form micro balls (aggregates) that look like steel wool. However, these micro balls look like black particles when looked at with the naked eye as presented in Fig. 2(a). These observations correspond to the characteristics of VGCNF; therefore, it confirms that the CNF used in this study is VGCNF.

### B. Thermal Stability

The thermal stabilization process of polymer-based nanocomposites is usually assessed through the Thermogravimetric Analysis (TGA) instrument by monitoring the weight loss as a function of temperature [22]. Hence, in this study, the thermal stability analysis of the pure LDPE, pure HDPE, LDPE/2wt% CNF, and HDPE/2wt% CNF were conducted according to ASTM E1131/ISO 11358 standards using Thermogravimetric Analysis (TGA) model Q500. Figs. 3 and 4 show the Thermogravimetric (TG) and Differential Thermogravimetric (DTG) curves of the pure LDPE, pure HDPE, LDPE/2wt% CNF, and HDPE/2wt% CNF heated at the rate of 10 °C/min from 25 °C to 700 °C. From Fig. 3 it is noticed that pure LDPE, pure HDPE, LDPE/2wt% CNF, and HDPE/2wt% CNF are thermally stable from 25 °C to roughly 200 °C with no mass loss. However, from 200 °C to roughly 360 °C, LDPE/2wt% CNF, and HDPE/2wt% CNF start to degrade whereas, pure LDPE and pure HDPE are still thermally stable. This might be due to the breaking of the bonds (intermediate phase) between the polyethylene matrices and the CNFs as the temperature increases. From 360 °C to about 480 °C, all polymers and nanocomposites undergo decomposition which is illustrated

by the drastic weight loss of the material as the temperature increases. However, LDPE/2wt% CNF and HDPE/2wt% CNF show to decompose at slightly higher temperatures compared to pure LDPE and pure HDPE respectively. The maximum peak temperature ( $T_{max}$ ) of each weight loss % of pure and reinforced polyethylene is presented in Fig. 4.

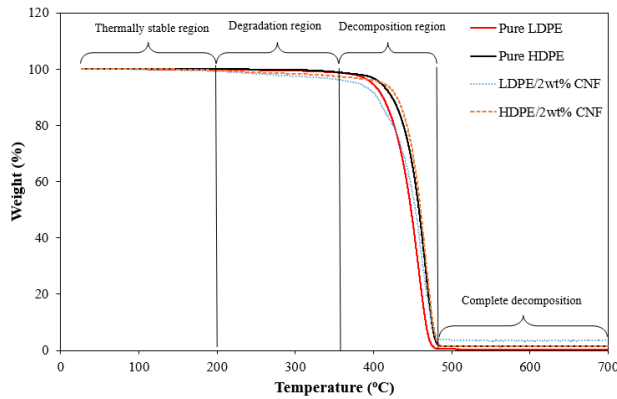


Fig. 3. The TG curves for pure LDPE, pure HDPE, LDPE/2wt% CNF, and HDPE/2wt% CNF.

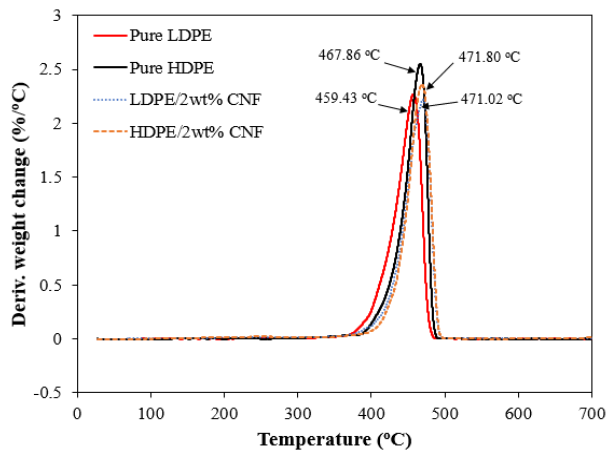


Fig. 4. The DTG curves for pure LDPE, pure HDPE, LDPE/2wt% CNF, and HDPE/2wt% CNF.

From Fig. 4 it can be observed that the  $T_{max}$  for pure LDPE, pure HDPE, LDPE/2wt% CNF, and HDPE/2wt% CNF are 459.43 °C, 467.86 °C, 471.02 °C, and 471.80 °C respectively. In general, the pure and reinforced LDPE and HDPE nanocomposites are thermally stable at temperatures ranging from 25 °C to roughly 200 °C. This indicates that the best processing temperatures for pure LDPE and LDPE/2wt% CNF are between 110 °C and 200 °C, and for pure HDPE, and HDPE/2wt% CNF are between 130 °C and 200 °C, where the minimum temperature are the melting point for each polymer matrix and the maximum temperature is the acceptable processing temperature before the thermal degradation of the polymers.

### C. Rheological Properties

Rheological analysis is an effective technique used to predict the processing behaviour of polymer-based nanocomposites as well as the dispersion of nanofillers and their adhesion between polymer matrices [23, 24]. The rheological behaviour of polymer-based nanocomposites is shown by the transition of polymer from liquid-like to pseudo-solid-like or solid-like when reinforced with the nanofiller. This transition is normally presented by proportionality of the storage modulus ( $G'$ ), loss modulus

( $G''$ ), and the complex viscosity ( $\eta^*$ ) to angular frequency (rad/s) [25, 26]. In this investigation, the rheological behaviour of pure LDPE, pure HDPE, LDPE/2wt% CNF, and HDPE/2wt% CNF were analysed according to the ASTM D4440-15/ISO 6721, part 10 standards using Modular Compact Rheometer 302e. Figs. 5(a) and (b) present the relationship of the storage modulus ( $G'$ ) and loss modulus ( $G''$ ) against angular frequency ranging from 0.1 rad/s to 100 rad/s in the respective manner. The trend for both graphs shows that the storage modulus and loss modulus for pure LDPE and pure HDPE slightly increase with the addition of 2 wt% of CNF. The curves display a monotonic increase with pure HDPE and HDPE/2wt% CNF retaining higher storage modulus and loss modulus at all angular frequencies. This illustrates that a good interaction among polymer matrices and CNF fillers has been established which resulted in the improvement of the viscous properties of the proceed nanocomposites.

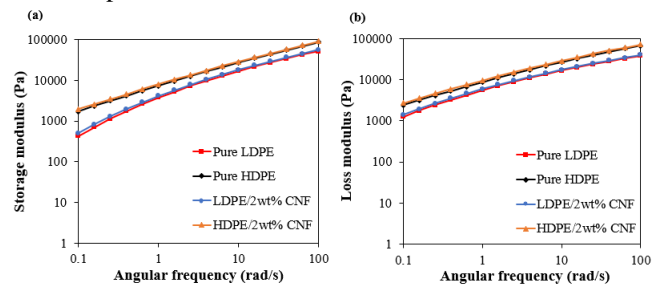


Fig. 5. (a) storage modulus and (b) loss modulus against angular frequency for Pure LDPE, Pure HDPE, LDPE/2wt% CNF, and HDPE/2wt% CNF at 190 °C.

Fig. 6 shows the angular frequency dependence of the complex viscosity ( $\eta^*$ ) for pure LDPE, pure HDPE, and their corresponding CNF nanocomposites. The graph demonstrates a small increase in the complex viscosity of the constituent polymer matrices with the addition of 2 wt% CNFs. However, the complex viscosities of the polymers and their respective nanocomposites are high at low angular frequency (0.1 rad/s) and decrease with an increase in angular frequency even though pure HDPE and HDPE/2wt% CNF show to have higher complex viscosity at all angular frequencies compared to pure LDPE and LDPE/2wt% CNF respectively. This proves that a 2% weight fraction of CNF has improved the viscosity of the pure HDPE and pure LDPE and all frequencies. Moreover, pure HDPE and HDPE/2wt% CNF proved to be more viscous compared to pure LDPE and LDPE/2wt% CNF at all frequencies.

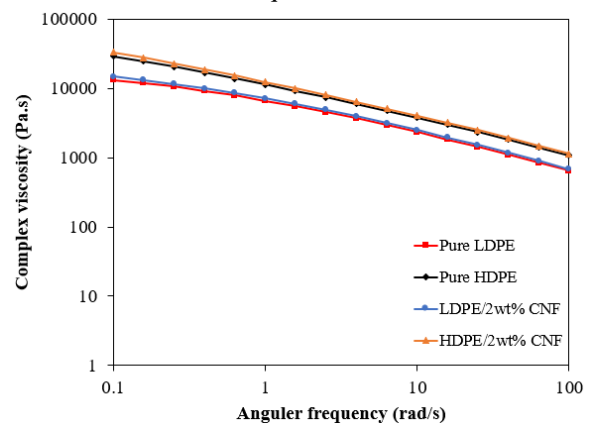


Fig. 6. The plot of complex viscosity versus angular frequency of pure LDPE, pure HDPE, LDPE/2wt% CNF, and HDPE/2wt% CNF at 190 °C.

#### D. Tensile Properties

The tensile tests of the pure LDPE, pure HDPE, LDPE/2wt% CNF, and HDPE/2wt% CNF samples were conducted according to the ASTM D 638-02a standard using an Instron 1195 tensile testing machine. The tensile tests were then used to determine the specific strength of the pure LDPE, pure HDPE, LDPE/2wt% CNF, and HDPE/2wt% CNF, utilizing their ultimate tensile strength obtained from the resulting stress strain curves. This was conducted to analyse the effect of a 2% weight fraction of CNF on the specific strength of the pure LDPE and pure HDPE. To determine the specific strength of the mentioned polymers and nanocomposites, the produced rheology specimens were weighed (three samples for each polymer and nanocomposites) according to Table 1, and then the average mass was considered. The obtained average mass per sample was then divided by the measured volume (0.687 cm<sup>3</sup>) of the specimen to calculate the density of the samples as presented in column three of Table 3.

Table 1. The average masses of the rheology samples for polymer matrices and the respective composites

Nanocomposites	Sample Masses [g]			Average Mass [g]
	Mass 1	Mass 2	Mass 3	
Pure LDPE	0.637	0.639	0.632	0.636
Pure HDPE	0.652	0.647	0.650	0.650
LDPE/2wt% CNF	0.659	0.660	0.658	0.659
HDPE/2wt% CNF	0.665	0.669	0.663	0.666

The tensile test results are presented on the stress strain curve in Fig. 7, showing that pure HDPE had higher average ultimate tensile strength compared to pure LDPE. The same trend is illustrated by the comparison of the HDPE/2wt% CNF and LDPE/2wt% CNF with the results summarised in Table 2. Statistically, the addition of 2 wt% of CNFs improves the average ultimate tensile of pure LDPE by 36% and that of pure HDPE by 22%. This shows that CNFs have significantly improved the average tensile strength of the reinforced polymers.

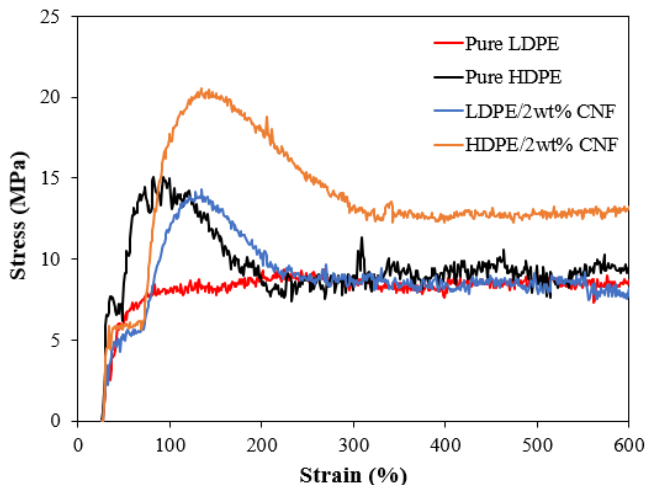


Fig. 7. The stress-strain graph of pure LDPE, pure HDPE, LDPE/2wt% CNF, and HDPE/2wt% CNF.

Table 2. The average ultimate tensile strength for pure LDPE, pure HDPE, LDPE/2wt% CNF, and HDPE/2wt% CNF

Nanocomposites	Tensile Strength [MPa]			Average Tensile Strength [MPa]	Improvement %
	Test 1	Test 2	Test 3		
Pure LDPE	10.260	09.347	10.267	09.958	--
Pure HDPE	17.710	15.081	16.640	16.477	--
LDPE/2wt% CNF	13.987	16.400	16.173	15.520	35.838
HDPE/2wt% CNF	20.466	18.642	20.304	19.804	21.632

The data in Table 2 was used to calculate the specific strength for pure LDPE, pure HDPE, LDPE/2wt% CNF, and HDPE/2wt% CNF by dividing the average ultimate tensile strength of each polymer and composite by the calculated density as shown in Table 3.

Table 3. The specific strength for pure LDPE, pure HDPE, LDPE/2wt% CNF, and HDPE/2wt% CNF

Nanocomposites	Tensile Strength [MPa]	Density [g/cm <sup>3</sup> ]	Specific Strength [kN.m/kg]	Improvement %
Pure LDPE	09.958	0.925	10.765	--
Pure HDPE	16.477	0.946	17.765	--
LDPE/2wt% CNF	15.520	0.959	16.184	33.484
HDPE/2wt% CNF	19.804	0.969	20.438	13.079

The data in Table 3 shows that CNFs do not only enhance the tensile strength of pure LDPE and pure HDPE, but they also improve their densities by 4% and 2% respectively. The results also show the specific strength of the pure LDPE and pure HDPE were improved by 33% and 13% after the addition of 2 wt% of CNF respectively. The overall results show that the addition of 2 wt% of CNF into LDPE and HDPE improved their tensile strength, density, and specific strength.

#### E. Other Recommendations

Investigate the influence of various weight fractions of CNFs on the thermal, and rheological properties, and the specific strength of LDPE.

Study of the effect of CNFs on the thermal and mechanical properties of various polymer matrices such as Polypropylene (PP), and Polyamide 6 (PA6).

#### IV. CONCLUSION

This experimental study characterized Carbon Nanofiber (CNF) nanofillers through Scanning electron microscopy (SEM) and investigated the effect of adding 2% weight fractions of CNF on the thermal stability, rheological behavior, and the specific strength of Low-Density Polyethylene (LDPE) and High-Density Polyethylene (HDPE) matrixes. Twin-screw extruder and compression moulding were used to process the Thermogravimetric (TG), Rheology, and tensile test samples. The obtained results from the analysed samples are as follows:

- The SEM characterisation revealed that the used CNF is infests agglomerated aggregates of Vapor Grown Carbon Fibers (VGCNFs) that can be viewed at a scale of 1 μm when magnified to 12.81 k X.
- The thermal stability of the pure LDPE and pure HDPE was established between 26 °C and 200 °C. The addition of 2 wt% of CNF did not improve the thermal stability

of the presented polymer matrices.

- The rheological results showed that the addition of 2 wt% of CNF improves the viscous properties of the pure LDPE and pure HDPE.
- The tensile strength of pure LDPE and pure HDPE improved by 36% and 22% after the addition of a 2% weight fraction of CNF respectively.
- The addition of a 2% weight fraction of CNF improved the specific strength of pure LDPE and pure HDPE by 33% and 13% respectively.
- The obtained results show that CNF has great potential to improve the mechanical properties of LDPE and HDPE.

#### CONFLICT OF INTEREST

The authors declare no conflict of interest.

#### AUTHOR CONTRIBUTIONS

All authors contributed to the study's conception and design. Ronny T. Tebeta performed material preparation and data collection; Ronny T. Tebeta and Daniel D. Madyira performed data analysis was performed; Ronny T. Tebeta wrote the first draft of the manuscript; Daniel M. Madyira provided Resources and financial support; Ronny T. Tebeta, Daniel M. Madyira, and Harry M. Ngwangwa revised the original manuscript to the current version; all authors read and approved the final version of the manuscript.

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