

Dimensional and Thermal Reliability of Multi-Walled Carbon Nanotube Filled Natural Rubber Nanocomposites

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Abstract. The exceptional mechanical and thermal properties, conceivable with carbon nanotubes (CNTs) make a distinction of them as excellent choices for natural rubber nanocomposite reinforcement. Nigeria, in recent years, has been awash with foreign prostheses, many of which fail to meet the criteria of the International Society of Prosthetics and Orthotics (ISPO) for developing countries. However, there are major marked gaps that make them susceptible to premature failure and dimensional instability, caused by a high rate of water absorption in a humid environment; owing to the dynamic nature of human gait. This paper critically examines the effect of water absorption, thermogravimetric and dynamic mechanical properties, on carbon nanotube-reinforced natural rubber nanocomposite (NC) for prosthetic foot application. CNTs were synthesised via catalytic chemical vapour deposition (CCVD) technique and the NCs were produced by using an electrically heated hydraulic press. Thermogravimetric analysis (TGA), water absorption rate and dynamic mechanical analysis (DMA) of the nanocomposites were carried out with a view to comparing the various compositions used in this paper. Of the five NCs developed (NR/MWCNT-0, NR/MWCNT-3, NR/MWCNT-6, NR/MWCNT-9, NR/MWCNT-12 and NR/MWCNT-15), NR/MWCNT-3 (3 g of MWCNT in 100 g of natural rubber) showed the highest thermal stability of 260 °C, optimal water absorption rate of 0.1% and highest quality energy storage and dissipation capacity, as indicated by 2.239 DMA loss factor curve amplitude, hence giving it a comfortable edge over its existing counterparts. The results of the various analyses carried out indicated, therefore, that reinforcing natural rubber with multi-walled carbon nanotube offers a reliable alternative material for the prosthetic industry.

1. Introduction

Other relevant properties of interest notwithstanding, specific modulus and specific strength both play important roles in determining the good attributes of composite materials over other competing conventional materials for a wide range of applications [1]. Reinforcement contributes significantly to the mechanical, dimensional and thermal properties of fibre reinforced composites. Over the years, the need to have polymer matrices reinforced with high-strength fibres in order to provide

strength and rigidity for load-bearing purposes, has received the attention of researchers [2]. One of such high-strength reinforcing filler is carbon nanotube (CNT). Low weight percent CNT, incorporated into polymer matrices has been proven to cause a vital improvement to the mechanical properties of its host matrix for biomedical engineering applications [3, 4]. By exhibiting a Young's modulus value of approximately 1000 GPa and a tensile strength 50 times that of steel (approximately 63 GPa), informed the conclusion of CNTs being the best reinforcing filler for a variety of applications.

The attention of the research world, as well as the industry, has been drawn to CNT-reinforced natural rubber nanocomposite (NC), owing to the superior thermal and mechanical properties it offers [5]. This material has created a secure position for itself in the tyre and sports industries; providing reduced weight and energy storage and dissipation in addition to enhanced air retention to the applied products. Reinforcement is said to play a key role in enhancing the fatigue, impermeability, tear and abrasion resistance, while at the same time, increasing modulus and elongation-at-break of elastomers. This dual nature justifies the capacity of reinforced rubber to provide excellent material properties and applications in many fields, one of which is the prosthetic and orthotic industry. A major technical challenge, however, associated with the production of CNT-reinforced natural rubber NC, is achieving homogeneous mixture of the constituent parts [6]. According to Endo et al [7], accomplishing this using low temperature roll mill, produces dependable materials capable of operating at extremely high temperatures and pressures. This problem has been reported to limit the application of elastomeric polymer because of its high viscosity. In their report, Khalid et al [5] correctly enthused that practical applications of this NC remains industrially viable.

Harnessing the large surface area available in CNTs and getting this interface intimately with polymer matrix, is such a huge task. Like carbon black (CB), but to a higher degree, CNTs coalesce into aggregates and agglomerate, thereby making dispersion extremely difficult, especially when higher wt% of fillers is involved [8]. Dispersion process is largely expected to cause a disentanglement of the agglomerates and homogeneously distribute them throughout the matrix. This is because the strength of CNTs is better exploited if they exist as single entities [9]. Scanning electron microscopy (SEM) and transmission electron microscopy (TEM) micrographs of the purified CNTs used in this work are illustrated in Fig. 1. Khalid et al [5] identified the inherent van der Waals' attraction existing between individual tubes as the reason behind the non-uniformity in filler dispersion, often encountered during rubber NC production. In a similar study, Wernik and Meguid [10], in addition to van der Waals' force, identified the high aspect ratios of CNTs as another obstacle to even distribution of fillers in matrix.

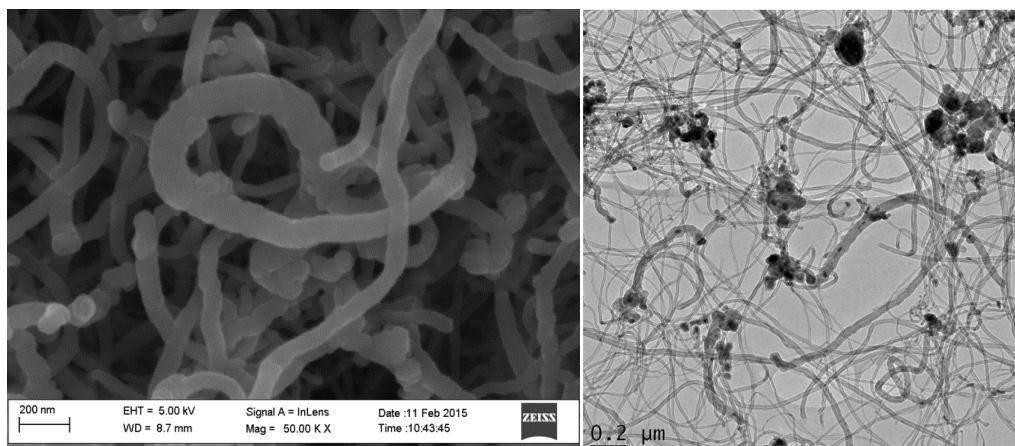


Fig. 1: SEM (left) and TEM (right) Micrographs of Purified CNTs

While many researchers consider the application of high shear stress, which is a viable technique to overpower the electrostatic and van der Waals attraction binding tube to tube, this in itself may only last for a moment since re-agglomeration in the composites is practically unavoidable without

a chemical treatment of the CNTs [11]. One of these mechanical techniques employed is ultrasonication, which operates on the principle of inertial cavitation with quick formation and violent collapse of bubbles in the liquid, thereby producing extreme shear forces [5, 12]. These intensely collapsed bubbles cause a forced separation of the tubes. Covalent and non-covalent functionalisations utilize chemicals to induce the desired separation. Whereas the functional groups produced by this method prevent the individual CNTs from attracting one another and hence, check the formation of agglomerates [13], and fragmentation of the CNTs occurs in the process, thereby disrupting π -electron system in the nanotubes; particularly with covalent functionalisation. Therefore, in order to avoid the difficulty associated with using either chemical and mechanical methods to disperse CNTs in polymers, both methods are suggested and are currently being used with less concentrated acids and better results.

This study, critically, examines the effect of water absorption, thermogravimetric analysis and dynamic mechanical analysis on multi-walled carbon nanotube-reinforced natural rubber NC for prosthetic foot application.

2. Materials and Methods

2.1. Materials

The characteristics of the natural rubber used in this study and supplied by Tony West Rubber Factory, Agboju, Lagos, Nigeria, are tabulated in Table 1:

Table 1: Characteristics of Natural Rubber Used

Parameter	Values
Dirt content	$\leq 0.05\%$
Volatile content	$\leq 1.00\%$
Nitrogen content	$\leq 0.70\%$
Ash content	$\leq 0.60\%$
Initial plasticity	≥ 30
Plasticity retention index	≥ 60
Colour	Dark brown

Multi-walled carbon nanotube (MWCNT) was synthesised by using VACUTEC Chemical Vapour Deposition equipment (model: XD-1200NT, USA) at the Centre for Genetic Engineering and Biotechnology, Federal University of Technology, Minna, Nigeria. Stearic acid, sulphur, zinc oxide (ZnO), 2-2-dithiobis benzothiazole (MBTS), and 2,2,4-Trimethyl-1,2-dihydroquinoline polymer (TMQ) were received from the Nigeria Institute of Leather Science and Technology, Zaria, Nigeria, while sodium dodecylbenzene sulfonate ($C_{18}H_{29}NaO_3S$), nickel (II) nitrate hexahydrate, $Ni(NO_3)_2 \cdot 6H_2O$, iron (III) nitrate nonahydrate, $Fe(NO_3)_3 \cdot 9H_2O$ and aluminium oxide, Al_2O_3 were supplied by Sigma-Aldrich with a percentage purity in the range of between 95.0 – 99.9%.

2.2. Experimental procedures

MWCNTs were synthesised in a cylindrical fixed tube reactor of length 1010 mm with internal and external diameters of 52 mm and 60 mm, respectively. The schematic diagram of the reactor is shown in Fig. 2. In these conditions, 1.0 g of Fe-Ni supported by Al_2O_3 was spread evenly on a quartz boat, gently placed at the centre of the reactor at room temperature. Argon flow was fluxed at 30 mL/min for 55 minutes, as the furnace temperature was raised from ambient to 750 °C, prior to the growth, in order to purge the system of any trapped air. Acetylene was then introduced at a rate of 100 mL/min with the flow rate of argon raised to 200 mL/min for the reaction time of 45 minutes, after which the flow rate of argon was returned to 30 mL/min and acetylene flow was stopped. This condition was sustained for 30 minutes and the reactor was cooled to room

temperature. The cylinder, at the end of MWCNT synthesis, was opened in order to harvest the MWCNTs for purification and functionalisation.

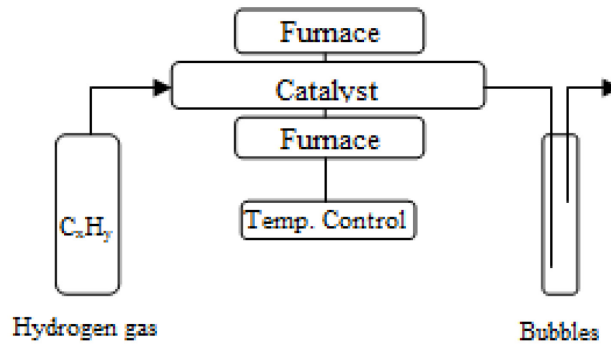


Fig. 2: Schematic diagram of chemical vapour deposition

About 10 mg of MWCNT was added to 4 mL HNO₃ 30% in a beaker. This was then placed in the ultrasonic cleaner with the temperature set to 45 °C and run for 180 minutes. After the completion of ultrasonication process, the suspension was washed repeatedly in distilled water until a neutral pH was achieved. The MWCNTs were afterwards dried in the furnace for 12 hours at 70 °C. A solution of 1.2 mM C₁₈H₂₉NaO₃S in 50 cm³ distilled water was prepared in the laboratory and 20 mg MWCNTs was added and ultrasonicated for 30 minutes at ambient temperature. The MWCNTs were filtered, washed thoroughly and dried again for 12 hours at 70 °C. The treatment with C₁₈H₂₉NaO₃S was to ensure a homogeneous dispersion of the filler in the matrix.

Compound formulation employed followed the procedure described by Hemkaew et al. [14]. Natural rubber compounds were prepared at 65±2 °C in a two-roll open mill (5183, USA) with the speeds of the front and rear rolls adjusted to 30 rpm and 18 rpm, respectively.

The formulations for preparing the rubber nanocomposites are illustrated in Table 2. Mixing was performed according to ASTM D-3182 standard on a two-roll mill with 300 mm working distance and outside diameters of 115 mm with a gear ratio of 1. The vulcanisation process was accelerated by using MBTS in order to reduce the vulcanisation time [15-17]. Zinc oxide (ZnO) and stearic acid were used as activators in order to increase the effectiveness of the accelerator [18]. Combining ZnO with stearic acid helped to reduce the time of vulcanisation and also to improve wear resistance of the base elastomer, and in consequence, the nanocomposites [19]. TMQ served as the antioxidant in the system. Table 3 shows the sequence of addition of vulcanisation ingredients. MBTS and sulphur were the last ingredients added with the former coming earlier in order to accelerate the process.

The different compounded formulations were left for 24 hours at ambient conditions in order to allow for full interaction of the ingredients and were cured at 150±2 °C for 10 minutes in an electrically heated hydraulic press. Test samples were cooled under a curing pressure of 0.2 MPa at room temperature and were neatly packed for analyses.

Table 2: Formulation of NR/MWCNTs Composites

Ingredient	Sample Code					
	NR/MWCNT-0	NR/MWCNT-3	NR/MWCNT-6	NR/MWCNT-9	NR/MWCNT-12	NR/MWCNT-15
	phr ^a					
NR	100	100	100	100	100	100
ZnO	5.0	5.0	5.0	5.0	5.0	5.0
Stearic Acid	2.0	2.0	2.0	2.0	2.0	2.0
TMQ	1.0	1.0	1.0	1.0	1.0	1.0
MWCNTs	0.0	3.0	6.0	9.0	12.0	15.0
MBTS	1.0	1.0	1.0	1.0	1.0	1.0
Sulphur	2.5	2.5	2.5	2.5	2.5	2.5
Total	111.5	114.5	117.5	120.5	123.5	126.5

where

a: parts per hundred parts of rubber

Table 3: Mixing procedure of NR/MWCNTs on two-roll mill

Mixing order	Mixing time (minutes)
Natural rubber	4.0
ZnO, Stearic acid, TMQ	3.0
MWCNT	5.0
MBTS	1.5
Sulphur	1.5
Total time (minutes)	15.0

2.3. Water absorption

Five specimens for each of the NC formulation were dried in an oven at 70 °C for 6 hours in order to drain the moisture in them, in preparation for water absorption test carried out according to ASTM D-7031-04 standard. The weight of each specimen was determined to a precision of 0.001 g and immersed in distilled water at room temperature according to a method adopted by Stelescu et al. [20]. Wet weights of the samples immersed in water were measured within 6 months soaking period at room temperature and at regular intervals of 30 days. Water absorption was determined according to the relationship presented in Equation 1.

$$MC_t = \frac{M_t - M_o}{M_o} \times 100 \quad (1)$$

where MC_t is water absorption at time t (%), M_t is wet weight at time t (g) and M_o is the dried weight (g).

2.4. Thermogravimetric analysis

Thermal degradation profiles of the nanocomposites were run in line with global mass loss by using Perkin Elmer TGA. About 25 mg of the sample was spread on a 7.4 mm diameter open sample pan and 4.2 mm deep. The temperature change was controlled from 30 °C to 950 °C at a heating rate of 10 °C/min. The sampling segment was set as 0.5 second per point. Nitrogen was constantly run through the furnace at a flow rate of 50 mL/min at room temperature and atmospheric pressure.

2.5. Dynamic mechanical analysis

The samples' response to external cyclic stress was observed by using TA Instruments, DMA Q800. They were subjected to temperature ramp from -90 °C to +100 °C at a rate of 3 K/min and frequency of 1 Hz. The operation was carried out in the dual cantilever bend mode with a dynamic force of 2.5 N and an amplitude of 15.0 μm. Storage modulus and mechanical damping of the nanocomposites were measured as a function of the temperature in the DMA test. Following the mounting and cooling of the samples with liquid nitrogen for about 30 minutes to a temperature of -90 °C, the storage and loss moduli as well as damping curves were generated and recorded.

3. Results and Discussion

3.1. Water absorption

Water uptake is a complex phenomenon that can be influenced by various factors among which are: free volume, crosslinking degree of the polymeric matrix, morphology and hydrophobicity [21]. This explains the reasons behind the twofold increase in the water resistance by MWCNT-reinforced polymer NCs, as reported by Prolongo et al. [22]. First, nanofillers operate as a capable obstruction against water absorption as a result of the tortuous nature of water molecules dispersion in the matrix, and the restriction of the intermolecular movement of fibres in the matrix being the second reason. Figures 3 – 8 illustrate the moisture absorption curves obtained for neat rubber vulcanisate and MWCNT-reinforced rubber NCs. In the first 30 days of the experiment, water

uptake curves presented a linear growth trend, after which the nanocomposites gradually approached their saturation points as a result of a reduced rate of water absorption.

Interestingly, NR/MWCNT-3 offered the most outstanding resistance to the phenomenon of water absorption by manifesting an approximately 0.064% moisture uptake, overall. Apart from the fact that the NC enjoyed a relatively improved fibre dispersion in the matrix [23], its consequential enhancement of specific surface area and aspect ratio, explains why it presented the highest barrier properties [22] and, hence, the optimal mechanical properties when compared to other compositions. The unfilled vulcanisate absorbs the most moisture (0.078%) when compared to the MWCNTs reinforced rubber vulcanisate. Earlier reports indicated that the high aspect ratio of the reinforcing filler is responsible for this decrease [22]. Owing to its least water absorption, it can be concluded that NR/MWCNT-3 is the most dimensionally stable of all the other compositions; the unfilled rubber vulcanisate inclusive. A decrease in diffusivity rate, induced by MWCNTs addition, has also been identified as a major reason behind the optimal dimensional stability of NR/MWCNT-3.

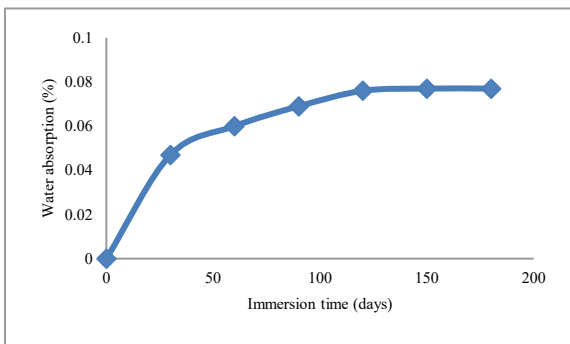


Fig. 3: Absorption – time curve of NR/MWCNT-0 NC

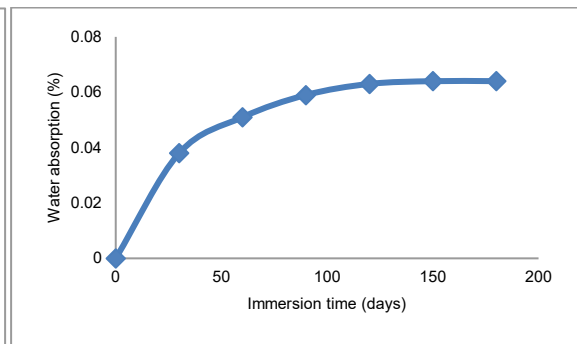


Fig. 4: Absorption – time curve of NR/MWCNT-3 NC

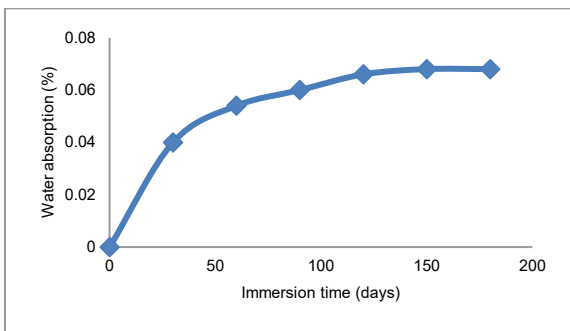


Fig. 5: Absorption – time curve of NR/MWCNT-6 NC

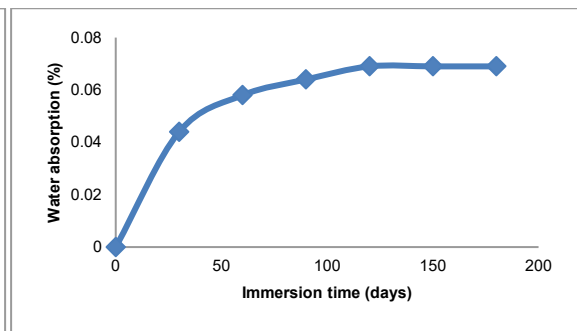


Fig. 6: Absorption – time curve of NR/MWCNT-9 NC

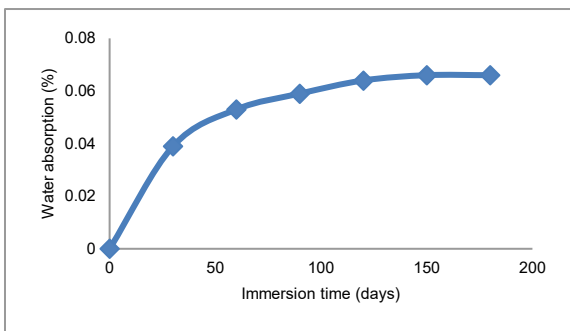


Fig. 7: Absorption – time curve of NR/MWCNT-12 NC

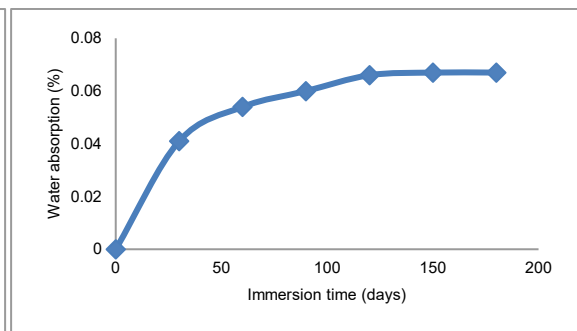


Fig. 8: Absorption – time curve of NR/MWCNT-15 NC

3.2. Thermogravimetric analysis

The TGA and DTG curves of the unfilled natural rubber and MWCNTs reinforced nanocomposites are plotted in Figures 9 – 14. The weight loss rate of the nanocomposites is considerably lower than that of the neat natural rubber vulcanizate; the highest of them (NR/MWCNT-6) being 20.49% less. The initial weight loss attributed to temperature-induced moisture evaporation for all the nanocomposites took place between 60 °C and 250 °C [24]. These slight percentage weight drops are obvious on the TGA curves with NR/MWCNT-3 having the least. This is in synchrony with the water absorption analysis results because its water absorption rate was also the least as reported earlier. While the unfilled vulcanizate and NR/MWCNT-15 both lost more than 4.00% moisture according to the thermal analysis data, NR/MWCNT-3 lost less than 2.50% moisture, making it more thermally stable than other formulations. This agrees with the reports of Sep et al. [25] who posited that MWCNTs reinforcement accounts for improved thermal stability of CNT reinforced nanocomposites. The temperature range within which constituent decomposition occurred for all the nanocomposites lies between 250 °C and 600 °C. Beyond 600 °C, constant weight was achieved. Above 700 °C, the residual weight of the nanocomposites were: 5.46%, 7.35%, 8.64%, 9.96% and 11.67 % for the 3 phr, 6 phr, 9 phr, 12 phr and 15 phr MWCNTs filler, respectively. For the unfilled vulcanizate, only 2.86% residue was left after the thermal analysis (Table 4).

It is obvious that the initial decomposition temperature (T_{onset}) (Table 4), which pointed toward thermal stability of the materials, witnessed significant improvements with the incorporation of MWCNTs into the natural rubber vulcanizate. The enhancement was more in the case of NR/MWCNT-3 and NR/MWCNT-12 with their T_{onset} values were above 260 °C. This improvement was reported to be as a result of the resistance effect of nanofillers and nano-confinement imposed by them. Maximum pyrolysis temperatures for the nanocomposites were also illustrated in Table 3. The first steps of the decomposition were due to the continuous phase of the composites, while the second decomposition peaks were linked to the discontinuous fillers. The values of the pyrolysis temperatures for both stages were found to shift towards higher temperature when MWCNTs were added to the natural rubber vulcanizate. A shift from this trend was, however, observed with NR/MWCNT-6, where the temperature dropped when compared to the 3 phr filled NC. This could be attributed to the inhomogeneous dispersion of the fillers often in the matrix, which is a difficult challenge with nanofillers [23].

The temperature at which half of the samples degraded (T_{50}) is averagely 309 °C for all the materials, as shown in Table 4. Furthermore, it is clearly shown that the temperature at which maximum degradation occurred (T_p) for the unfilled NR, dropped following the incorporation of nanofillers into the matrix. This was observed to decrease considerably over the values obtained for the unfilled vulcanizate (320.91%). This is not unexpected because of the presence of the ingredients used during the vulcanisation procedure, which may have also acted as resistance to polymeric chain mobility, like MWCNTs [11].

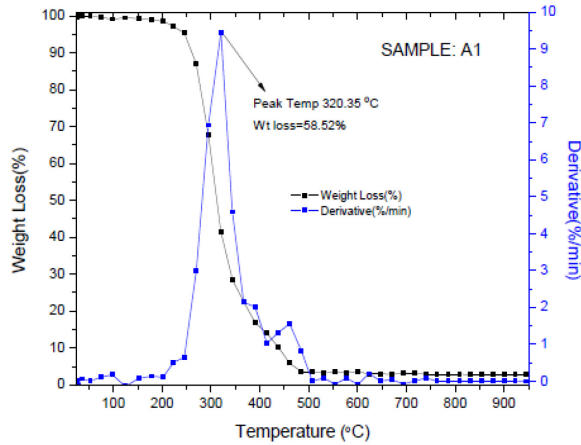


Fig. 9: TGA/DTG thermographs for NR/MWCNT-0

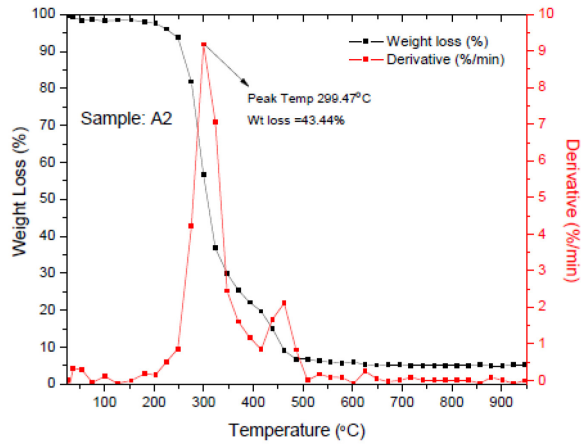


Fig. 10: TGA/DTG thermographs for NR/MWCNT-3

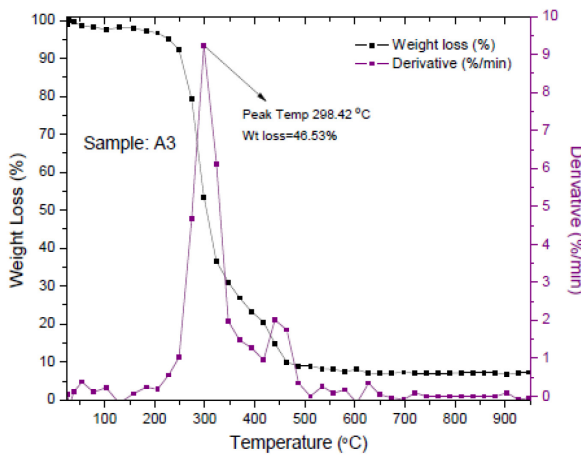


Fig. 11: TGA/DTG thermographs for NR/MWCNT-6

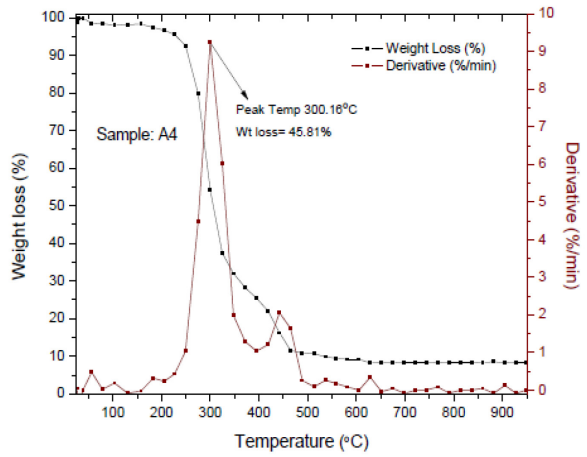


Fig. 12: TGA/DTG thermographs for NR/MWCNT-9

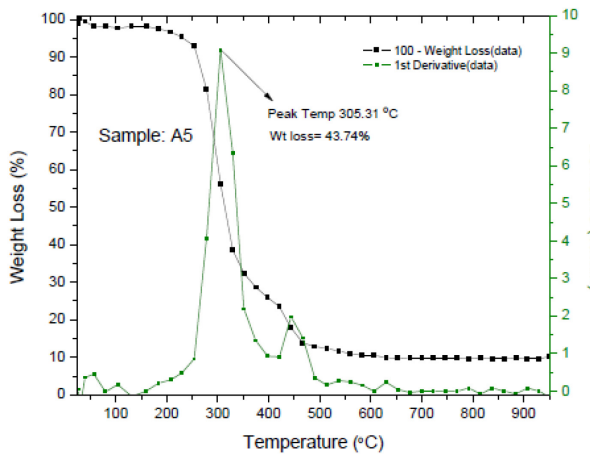


Fig. 13: TGA/DTG thermographs for NR/MWCNT-12

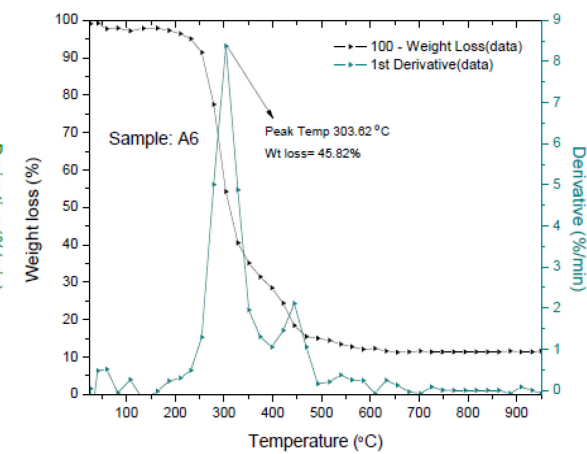


Fig. 14: TGA/DTG thermographs for NR/MWCNT-15

Table 4: Thermal parameters of unfilled vulcanisate and NCs from TGA and DTG curves

Samples	T_p (°C)	T_{50} (°C)	T_{onset} (°C)	Pyrolysis Temp. (°C)	Residue (%)
NR/MWCNT-0	320.35	310.91	252.12	202.40-623.06	2.86
NR/MWCNT-3	299.47	307.77	260.01	202.18-416.72 416.72-648.02	5.46
NR/MWCNT-6	298.42	304.39	258.34	205.23-416.65 416.65-626.20	7.35
NR/MWCNT-9	300.16	306.58	255.70	162.48-396.07 396.07-675.17	8.64
NR/MWCNT-12	305.31	313.91	260.99	184.69-399.56 399.56-655.00	9.96
NR/MWCNT-15	303.62	310.17	258.34	187.14-396.75 396.75-726.34	11.67

3.3. Dynamic mechanical analysis

Dynamic mechanical analysis (DMA) was carried out in order to investigate the dynamic response of the NCs following the incorporation of MWCNTs. DMA demonstrated that the storage modulus (E'), loss modulus (E'') and loss factor ($\tan \delta$) of both the low and higher MWCNTs content in natural rubber were somewhat similar, as illustrated in Figures 15 to 20.

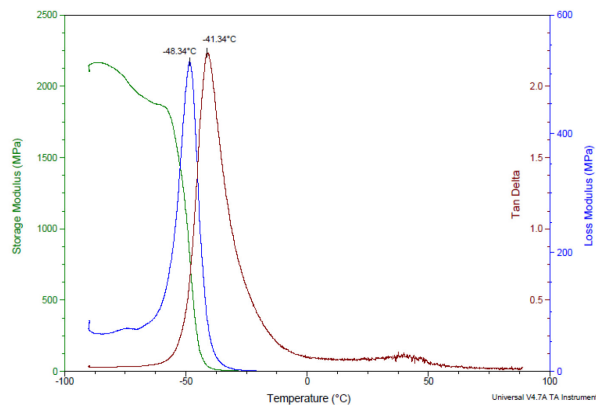


Fig. 15: DMA thermograph of NR/MWCNT-0

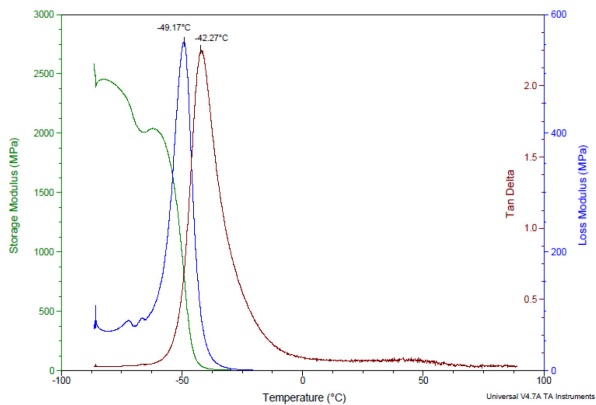


Fig. 16: DMA thermograph of NR/MWCNT-3

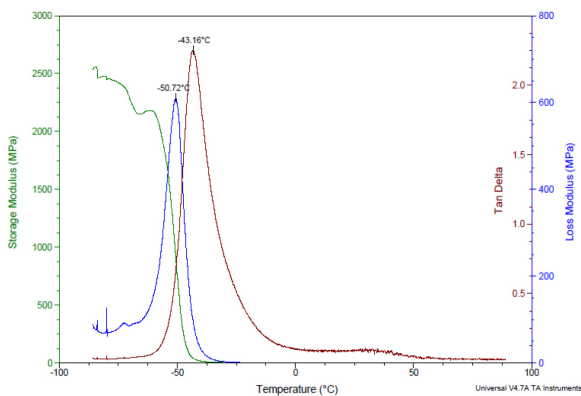


Fig. 17: DMA thermograph of NR/MWCNT-6

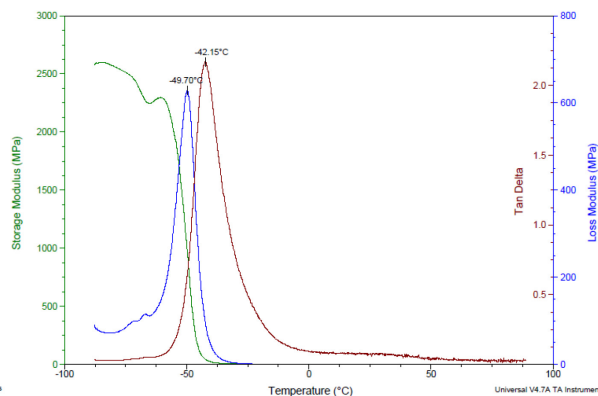


Fig. 18: DMA thermograph of NR/MWCNT-9

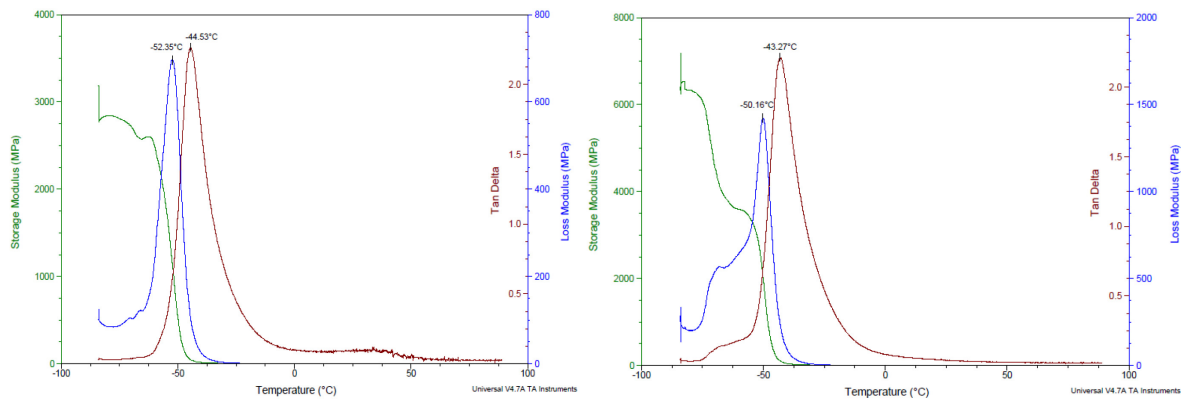


Fig. 19: DMA thermograph of NR/MWCNT-12 Fig. 20: DMA thermograph of NR/MWCNT-15

All the figures showed the dependence of E' , E'' and $\tan \delta$ as a function of temperature with comparable glassy, transition and rubbery plateau regions. The curves also illustrated steep decline of E' values at the temperature range of between -90°C and -60°C (glassy regions) and -60°C and -40°C (transition regions) followed by the rubbery plateau regions. Information about the interfacial bonding that exists between the reinforcing and polymer phases as well as the crosslinking density are explained by the dynamic parameters [26].

Storage modulus value was observably raised significantly with the incorporation of MWCNTs in the natural rubber vulcanisate. Approximately 6391 MPa of 15 phr MWCNT reinforced NC represents a whopping 67% increase to the E' when compared to the unfilled vulcanised rubber. Evidently, however, the incorporation of 3 phr, 6 phr, 9 phr and 12 phr MWCNT, resulted in less than 20% increment in the values of E' . This trend is consistent with earlier reports by Pongpilapruet and Magaraphan [27]. The high values of E' in the glassy region are attributed to the highly compact and frozen nature of the NC components. This is observed to decrease considerably beyond the glass transition regions, owing to the mobility of the polymer chain above the glass transition temperatures (T_g). Therefore, stiffness and the capacity of the NCs to store up energy during a stress perturbation for future use are greatly improved by MWCNT reinforcement. At room temperatures and slightly above, stiffness is seen to increase by as much as 600% when comparing NR/MWCNT-15 to other NCs.

A measure of the dissipation of energy stored as heat per cycle under deformation experienced in a viscoelastic material is described as loss modulus [26, 28]. The effect of temperature on E'' for the NCs is illustrated in Figures 13 to 18. For all the samples, E'' curves reached their highest values for maximum dissipation of mechanical energy and decreased as temperatures increased, while the polymer chains were allowed free movement. Like the storage modulus, loss modulus also increased with the incorporation of MWCNTs in the vulcanised rubber, as illustrated in Table 4, which represents the results obtained from the curves.

Loss modulus peak height of the vulcanised rubber NCs was improved by the addition of MWCNTs as shown in Table 5. These peak heights represent the glass transition temperature (T_g) values of the NC systems. Evidently, T_g of the NCs from both the $\tan \delta$ and E'' decreased with filler reinforcement, consistent with earlier reports [26, 29]. The obvious closeness of T_g for each of the materials, indicates that reinforcement has little or no effect on the T_g of elastomeric NCs. Furthermore, the narrow peaks of $\tan \delta$ curves substantiated the reinforcing efficiency of the MWCNTs and show complete crosslink process of the NR matrix formulations [30, 31]. The narrow peaks observed for the different samples further validate the production route adopted for the manufacture of the NCs because broad peaks are believed to be indicative of incomplete crosslink process of the compounds and hence casts doubts on the functionality of the NCs in the field.

Table 5: DMA data of NCs and control test samples

Nanocomposites	Peak height of $\tan \delta$ curve	T_g from $\tan \delta_{max}$ (°C)	Peak height of E'' curve	T_g from E''_{max} (°C)
NR/MWCNT-0	2.219	-41.61	516.11	-48.91
NR/MWCNT-3	2.239	-42.42	550.50	-49.57
NR/MWCNT-6	2.229	-43.95	607.10	-51.01
NR/MWCNT-9	2.150	-42.98	624.40	-50.08
NR/MWCNT-12	2.243	-45.15	691.50	-52.82
NR/MWCNT-15	2.187	-43.78	1413.00	-50.43

In another study by Padal *et al.* [32], the height of the $\tan \delta$ curves was believed to be directly related to the materials' capacity to dissipate energy through segmental motion. They reported that composite systems with higher $\tan \delta$ peaks have higher ratios of energy absorption, viscous motions and are normally tougher than those with lower $\tan \delta$ amplitudes. It is against this background that 3 phr and 12 phr loadings remain the optimum filler loading for the NC system studied, as evidenced in Table 5.

4. Conclusion

The results published in this paper indicate that thermal and mechanical analyses are foolproof approaches in the investigation of the physical, thermal and mechanical performances of MWCNT reinforced natural rubber NC for prosthetic foot use. Generally, while crosslink density increased as a result of increased MWCNT loading into the rubber vulcanisate, polymer chain mobility of the NCs was appreciably decreased. Having been subjected to the same process conditions during production, NR/MWCNT-3 demonstrated the highest level of thermal stability at 260 °C. Its optimal water intake rate of approximately 0.1% is also higher than any of the other NCs. The viscoelastic analysis of the materials via DMA also indicated a high quality energy storage and dissipation capacity of the NCs with NR/MWCNT-3, having an edge over its counterparts studied, with a peak height of tangent delta curve of 2.239. It is expected, therefore, that this work will benefit a large number of below-knee amputees across Africa and open up several possibilities that can lead to increased research efforts, directed towards the area of elastomeric matrix NCs for the prosthetic industry.

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