Development and Characterization of Cardanol-Based Polymer Reinforced Fibre Bicomposites

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ABSTRACT
The emerging quest for optimal use of naturally obtained resources through advanced techniques and products has escalated research and exploration of naturally oriented renewable materials. In present investigation, cardanol, obtained from cashew nut shell liquid (CNSL), was utilized as base material for a thermosetting polymer (TP) development. Furthermore, rice husks flour (RHF) was utilized as reinforcement for the TP. The fabricated biocomposite was characterized for microstructure, thermal behavior and biodegradability. Results from DSC and TG analysis revealed that the sample filled with 5 wt. % NaOH modified RHF demonstrated most superior thermal stability, and tensile strength. Moreover, results from FESEM revealed uniform dispersion, and bonding to polymer matrix. Enhanced biodegradability was realized from NaOH modified RHF filled biocomposite post 90 days of monitored soil tests. Therefore, the cardanol obtained TP and biocomposites demonstrated good prospects for application in the electronics, automotive and furniture industry.

KEYWORD: Cardanol obtained plastic; thermosetting polymer; bio-fiber; biodegradability; biocomposites

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1. INTRODUCTION
The emerging searches for optimal utilization of natural resources through advanced procedures and products has increased research into new uses of natural renewable materials [1]. CNSL is a notable instance of a renewable resource commonly utilized in polymeric materials synthesis. CNSL is a thick liquid in the soft fleshy part of cashew nut (CN), obtained from cashew tree, or Anacardium occidentale L [2]. The major components obtained from CNSL include cardanol, cardol, anacardic acid and 2-methylcarbol which is a phenolic compound, composed of long side-chain substituted at the meta-position. A large number of polymeric materials with a wide variety of applications can be manufactured using these phenolic compounds. Application of CNSL in place of phenol is an excellent instance of effective usage of a synthetically derived material by product utilization [3]. Technical CNSL (t-CNSL), a commercial type of CNSL, scarcely contains any anacardic acid. This is as a result of the occurrence of decarboxylation during processing, thereby converting anacardic acid to cardanol [4]. Cardanol is composed of compounds, having a variable number of unsaturations, occurring in the fifteenth carbon atom-chains, and situated on the hydroxyl group meta position [5]. Polymers, including polymeric materials synthesized from cardanol are well captured in literature [6], and the theme of developing studies. Included as a CNSL component is cardanol, which is a lucrative alternative in synthesizing polymeric materials. This is as a result of the incentive of belonging to a monoreplaced phenol, which minimizes the challenges of steric obstruction during polymerization reaction. Recently, great interests has been aroused in the formation of composite materials from polymer matrices filled with natural fibers [7]. Ecobien materials formation, and a more purified production procedure, is essential to environmental preservation, and better living conditions for the next generation. Natural biofibers are biopolymers constituting of cellulose, hemicelluloses, and lignin, which individually enhance the composite mechanical strength [8,9]. The application of natural fibers as reinforcements offers minimal density, elevated porosity, effective tensile and impact strength to composite materials apart from increased fissuration control, and improved ductility at break [10-12]. Natural fibers offers benefits such as reduced cost, excellent solvents, and temperature resistance. Additionally, they undergo easy modification by chemicals, and are also non-toxic, and non-abrasive [13, 14]. Lignocellulosic fibers such as coconut fibers [15], jute [16], sisal [17], and banana tree[18], have been versatily researched, and utilized in...
reinforcing polymer matrices. Rice is a cereal grain utilized in producing hull fibers. Recently, corn, oats, wheat, rye and other cereal crops have been utilized in producing fibers and studied as reinforcement for composites [19–21]. Generally, lignocelullosic fibers exhibit hydrophilic tendencies, and hence, exhibit low compatibility with hydrophobic polymer matrices [7]. This deficiency results in poor adherence, which impacts negatively on effective transfer of tensions, thereby compromising the composites mechanical behavior [22,23]. Hence, a pretreatment is essential prior to using these fibers as fillers in polymer matrices. Alkaline modification is a renowned technique for fiber surface modification to enable improved adherence.

Therefore, the motivation for this research is hinged on exploration of the novelty garnered from the development and characterization of a thermosetting polymer obtained from cardanol and filled using pristine and modified rice flour. This thermosetting polymer and biocomposites were studied via techniques including Thermogravimetry (TG), Differential Scanning Calorimetry (DSC), tensile testing, field emission scanning electron microscopy (FESEM) and biodegradative tests in simulated soil.

2. Materials and methods

2.1. Materials

Rice husks were purchased from Abakaliki, Nigeria. Dynamic sodium hydroxide (NaOH - 97%), and a commercial sodium hypochlorite solution composed of 2.0 wt. % active chlorine in NaClO, were utilized in alkaline modification and bleaching of the fibers. The technical CNSL utilized in this study was obtained from fresh cashew-nut gathered from cashew plants (A. occidentale) of Abia state University, Uturu, Abia State, Nigeria. Anhydrous sodium sulfate (Na₂SO₄ - 99%), hexane (C₆H₁₄ - 98.5%), methanol (CH₃OH - 99.8%), ammonium hydroxide (NH₄OH - 28-30%), hydrochloric acid (HCl - 37%), formaldehyde (CH₂O - 36.5-38%), diethylenetriamine (DETA, C₈H₁₄N₃ - 99% - Sigma-Aldrich), toluene (C₆H₅ - 99.5%) were used devoid of any previous modification. Epoxy resin DGEBA (Bisphenol A diglycidyl ether) was commercially purchased.

2.2. Alkaline treatment

The rice husks fibers (RHF) were generally cleaned, and dried. The dried fibers were later ground in a grinding machine and later sieved using a sieving equipment of 150 μ. Sodium hydroxide solutions of concentrations 5% and 10% were used in treating RHF at 65 °C for 4 h. The fibers were subsequently washed with distillate water to remove excess NaOH until neutral pH was attained. The fibers were then further treated with sodium hypochlorite solution containing 1% wt. % of active chlorine at 65 °C for 1 h. Finally, modified-RHF were cleansed with distillate water to eliminate excess NaClO, and dried in an aerated oven at 65 °C.

2.3. Cardanol extraction and polymer synthesis

2.3.1. Cardanol extraction

Cashew nut shell liquid (CNSL) was extracted from Cashew nut (CN) via Soxhlet extraction method. CNSL was dissolved in methyl isobutyl ketone (MIBK), and calculated amounts of barium hydroxide was included with continuous stirring for 10 minutes. The temperature was raised to 60°C and stirring was continued for 3 hrs to obtain anacardic acid. At the end of the reaction, the barium anacardate precipitated underwent filtration and washed with MIBK and dried for 2 hrs. The filtrate obtained was subjected to subsequent isolation of cardol and cardanol. Barium anacardate was modified using distillate water and concentrated HCl at constant stirring at 200 rpm for 30 min. The resultant solution contained petroleum ether. The organic layering was washed with distillate water, dried using anhydrous sodium sulfate, and evaporated to dryness to get anacardic acid. The anacardic acid was decarboxylated to obtain CNSL and subsequently isolated to obtain cardanol using hexane/ethyl acetate [24].

2.3.2. Synthesis of thermosetting polymer

Polymer synthesis was performed in a bench glass reactor containing a reflux-condenser, stirrer, and thermometer. The thermosetting polymer was synthesized via mixing of cardanol, and aqueous solution of formaldehyde, where NH₂OH was used as catalyst. The combination was heated till 80 °C for 4 h via magnetic stirring. Preparation of cardanol/formaldehyde in the molar ratio of 1:2 was undertaken in this synthesis.

2.3.3. TP and biocomposites preparation

In the preparation of the thermosetting polymer, di-ethylene-triamine was utilized as a catalyst, while epoxy resin was utilized as modifying agent. The use of aliphatic amines as catalysts enabled the production of materials under normal conditions of temperature and pressure, thereby facilitating their processing. The TP was prepared through blending of thermosetting polymer, epoxy resin and di-ethylene-triamine in the proportion of 0.95:1:0.15 (wt. %), respectively. The blend was then moved to a metallic mold of 180 mm height, 160 mm width and 5 mm thickness, with subsequent curing for 48 h, at room temperature in a dispenser, while post-curing occurred in the oven at 100 °C for 4 h. The mold releasing agent used was camaba wax. The resin obtained from cardanol was utilized as the polymer matrix, while the biocomposites were prepared using modified and unmodified RHF fibers as reinforcement phase with matrix-fiber weight ratio of 85:15. The processing conditions used for the biocomposites were the same process utilized for thermosetting plastics.

2.3.4. Cross-linking degree determination

ASTM D2765 [25] gravimetric method was applied in ascertaining the degree of cross-linking (DoC). The TP specimens were weighed (W₁), and subsequently immersed in toluene for 24 h to extract the soluble content and determine the DoC. When immersion was finished, the specimens were dried at room temperature to constant weight and weighing repeated (W₂). The experimentation were performed thrice for the TP which underwent curing at room temperature for 48 h, in addition to the post-cured at 60, 80, and 100 °C for 2 h. At the end of curing, another experiment was carried out, using a fixed temperature, while the DoC was measured at post-cure ranges of 30, 60, 120, 180, 240, 300, and 360 min. The DoC was calculated using Equation 1.

\[
DGC = \frac{W_1}{W_2} \tag{1}
\]

2.4. Characterization

2.4.1. Thermogravimetry (TG)

Perkin Elmer TG STA 6000 equipment was used in achieving TG under N₂ atmosphere with a flow-rate of 50 ml/min, and 10 °C/min heating-rate, within a temperature range of 30 and 600 °C to analyze TP, and biocomposites thermal stability. Platinum-pan was utilized, while the weight of each specimen was about 5 mg.
2.4.2. Differential scanning calorimetry (DSC)

A DSC Q-20 equipment (TA Instruments) was used in obtaining DSC curves under N\textsubscript{2} atmosphere at a flow rate of 50 ml/min, and heating-rate of 10 °C/min, within temperature range of 30 °C and 400 °C used in studying the DOC, and investigate TP and biocomposites heat capacity variation with temperature. A hermetic aluminum-pan was utilized, while the weight of each specimen was about 2.5 mg.

2.4.3. Tensile testing

ASTM D638 [26], and ASTM D3039 [27] were applied in conducting the tensile tests on the specimen. A DL 10000/700 tensile machine with test speed of 5 mm/min, and a load-cell of 100 kN was used in performing the tensile test on the materials. Sample dimensions of height 175 mm, width 25 mm, and thickness of 5 mm thick were used for the testing. Five samples were used in testing each material, while the mean value was calculated.

2.4.4. Field emission scanning electron microscopy (FESEM)

For FESEM analysis, the materials were coated with a mild layering of metallic gold in a sputter-coater Q-150R-ES. A Shimadzu-SSX-550 electron microscope was utilized in achieving micrographs from the fractured surface of the samples after tensile testing at an acceleration voltage of 15 kV.

2.4.5. Biodegradability analysis

The specimens for biodegradation analysis were subjected to prepared soil analysis. They were initially weighed, and buried at about ambient temperature. A combination in (wt/wt) of loamy-silt (23%), cow manure (23%), sand (23%), and distilled water (31%) were used in preparing the soil. For about 90 days, biodegradative activity was closely monitored through measurement of changes in mass of the buried samples. After this period, the buried samples were exhumed, and distilled water utilized in drying them at room temperature, until there was zero weight changes, after which the materials were weighed. Subsequently, the samples were re-buried. These investigation were conducted four times.

2.4.6. Fourier transform infrared spectroscopy (FT-IR)

A BOMEM - MB 100 spectrometer (4000 to 400 cm\textsuperscript{-1}) was used in recording the FTIR spectra of the specimens.

3. Results and Discussion

The physicochemical characterization of raw cashew nut shell liquid (CNSL) and cardanol were determined using international standard methods. Technical CNSL composed majorly of cardanol and cardol. Proximate analysis showed that cardanol contained 0.1% moisture, 4.9mgKOH/g acid value, 37.22mg/100g iodine value, 0.92kg/m\textsuperscript{3} density, 1.12g/cm\textsuperscript{3} specific gravity and insignificant refractive index of 0.0001m/s. The cardanol maintained all the major components of an oil after preparation. The peroxide value of the cardanol was observed to be higher than that of raw CNSL. There was an observed decrease in specific gravity with the cardanol which may be attributed to the elimination of hydrogen bond during decarboxylation and isolation. This decrease in specific gravity enhances bulk properties without adverse effect on the material properties.

Rice husk flour was modified using already established techniques [28, 29]. Generally, chemical modification involved the use of equal concentration of NaClO (1 wt. %). Hence, in this report, increasing concentration of NaOH, (5 and 10 wt. %), was discussed. Themosetting polymer and biocomposites filled with untreated rice husks flour (URHF) and those filled with modified rice husks fiber (MRHF) treated using 5 and 10 wt. % NaOH were designated as TP, URHF, MRHFS, and MRHF10, respectively. It is vital to investigate the degree of cross-linking (DoC) of thermosetting materials because it is directly related to the behavior of the end product. Figure 1 reveals TP DoC results obtained via gravimetric method.

![Figure 1 DoC at varying ranges of post-curing temperature of the materials](image-url)

TP is represented by 0 °C, and underwent curing for 48 h at ambient temperature devoid of any after-curing procedure. Thus, the material demonstrated degree of cross-linking of 95% ± 0.2 after the curing process. Apart from undergoing curing for 48h at ambient temperature, TP specimen also underwent post-curing for 2 h at increasing temperatures of 60, 80, and 100 °C where the degrees of crosslinking were 98 ± 0.9, 98 ± 0.7, and 99.5% ± 0.4, respectively. Additionally, temperatures of over 100 °C were also used in further testing, however material deformation were observed. Hence, post-curing temperature was fixed at 100 °C, and demonstrated the highest degree of cross-linking at the investigated time duration. However, the DoC was studied for varying time ranges, as shown in Figure 2 below.

![Figure 2 DoC at varying time ranges of post-curing temperature of the materials](image-url)
The degree of cross-linking to post-cure time ranges of 30, 60, 120, 180, 240, 300, and 360 min were 96 ± 0.4, 98±0.1, 97±0.2, 98.06 ± 0.3, 99.5 ± 0.1, 98.1 ± 0.2, and 99.9% ± 1, respectively. The DoC increased with increasing temperature and no significant changes were observed from 240 minutes.

Investigative results reveal that the condition of curing and post-curing of TP were situated at 48 h at room temperature in the distribution hood and 4 h at 100 °C in the oven, respectively. Same conditions were also attributed to biocomposites, while the curing time of 48 h in the distribution hood was to enhance total release of the catalyst used (DETA). The results from the degree of cross-linking were confirmed via DSC. This is a major technique used in studying curing of TP materials. Figure 3 represents DSC curve obtained from the curing procedure of TP.

The curing procedure revealed a large exothermic reaction at 88 °C and total heat of 93 J/g at 136 °C ascribed to dehydration.

From the result, all the samples demonstrated a change in behavior about 60 °C. This change was ascribed to glass transition temperature (Tg), determined using the technique presented in ASTM D3418 [30]. This is similar to the result obtained in a recent study involving biocomposites fabricated using a matrix synthesized from cardanol and filled using natural fibers. Here, Tg of the samples was depicted around 56 °C [31].

Results emanating from DSC revealed that zero exothermic interaction occurred as regards the residual heat released from the TP or any of the biocomposites. It revealed that the curing parameters used for the TP were also good for the biocomposites, enabling full conversion and subsequently, total curing of all samples. The samples DSC curing results confirmed the results obtained from DoC experiments as shown in Figure 4. A brief endothermic activity was observed at 200, 180, and 175 °C for MRHF10, and MRHF5, and URHF respectively.
This event was attributed to dehydration. This trend was not observed in TP because of its hydrophobicity. However, significant endothermic activities were observed at 350, 285, 300, and 325 °C ascribed to the degradation of TP, URHF, MRHF5, and MRHF10 respectively. Moreover, results demonstrate that TP is slightly more thermally stable when compared with the biocomposites. However, chemical modification improved the biocomposites thermal stability, especially MRHF10. This occurred because chemical modification significantly eliminated the lignin and hemicellulose and initiated degradation at lower temperatures. The chemical modification removed waxes on the surface, thereby enhancing improved bonding between biocomposite interface. Hence, the more improved the interaction between fiber and matrix, the more elevated the initial thermal degradation temperature. Here, the fiber-matrix interface is a two-dimensional line between the surfaces of fiber and matrix [31]. Fiber/matrix interface enables control of composite properties as fiber/matrix interactions take place at the interface. These interactions are postulated to occur via surface energies and secondary bonding between the fiber and the matrix, as the fibers act as heat sink during curing of TP, via endothermic cross-linking reaction. This is posited to create a thin three-dimensional interfacial region encompassing the fiber, and exhibiting properties different from the TP. Additionally, the interfacial region is created as a result of special adsorption of the TP at the fiber surface, resulting in a region of higher or lower cross-linking TP, from interpretation of DoC results. From the DSC results, the interphase is the transcrystallinity region on the surface of the fiber developed during cooling [31]. This is also notable in FESEM and TEM images.

Thermogravimetry (TG), was used in studying materials weight changes relative to temperature. Figure 5 shows the TG and DTG (Differential Thermogravimetry) curves of the TP and biocomposites.

**Figure 4 DSC curves of samples**

**Figure 5. TG and DTG curves of TP, URHF, MRHF5, and MRHF10**

TG curves in Figure 5 show a minor loss in weight from ambient temperature to 180 °C of 2.8, 2.9, and 3.9% for URHF, MRHF5 and MRHF10, respectively. This trend relates to water loss, and was not observed in TP due to its hydrophobicity, as also confirmed in DSC curves. The biocomposites water content increased with increasing concentration of NaOH. This happened because alkaline modification exposed the fiber’s hydroxyl groups, which escalated its hydrophobic behavior. A study has revealed that alkaline modification of fiber escalated moisture uptake [32]. DTG curves revealed two stages of weight loss. Initially, thermal stability of TP, MRHF10, MRHF5, and URHF were maintained, with maximum temperature peak at about 495 and 400, 490 and 395, 450 and 360, 430 and 359 °C, respectively. This trend occurred due to lignin, hemicellulose, and cellulose decomposition [33-35]. In addition to degradation of polymer matrix soft segments. At the second stage, deterioration was attributed to carbonaceous residues degradation. At the end of both stages, TP, MRHF10, MRHF5, and URHF showed 16, 18, 15, and 14 % residues, respectively. Both TG and DTG data confirm the DSC results. The mechanical behavior of the
biocomposites revealed dependence on synergy between the reinforcing agent and the matrix as shown in Figures 6 and 7. Figure 6 (a) depicts the tensile strength, while 6(b) depicts the Young’s Modulus. Figure 7 (a) represents the breaking load, while 7 (b) depicts the deformation behavior of the samples.

Overall, the mechanical behavior depicts an increasing trend respectively. From the Figures, all the specimens were investigated to ascertain the effect of RHF as reinforcement, in addition to the effects of chemical modification on biocomposites mechanical behavior. Significant variations were observed. Tensile strength values of URHF, MRHF5, and MRHF10 increased by 99, 91, and 150% respectively, in comparison to TP. Also, values of the Young modulus and breaking load of URHF, MRHF5 and MRHF10 were increased by 129 and 120, 125 and 106, 140 and 150 % respectively, in comparison with TP. All the biocomposites samples exhibited higher mechanical behavior, in comparison with TP, especially MRHF 10. This is because, lignocellulosic fibers can be effective reinforcement for polymeric matrices. These results demonstrate that RHF modified with 10 wt. % NaOH exhibited improved interaction with polymer matrix, enabling greater stiffness, improved tension distribution and therefore, a higher tensile tests results. Both DSC and TG data also corroborated these results.

FESEM and TEM were used in obtaining micrographs from fractured surface of samples to enable investigation of the microstructure and interaction between polymer matrix and RHF s. FESEM and TEM of TP and biocomposites are shown in Figures 8 and 9 respectively.
The images revealed crevices on TP fractured surface (Figure 8a). This may be ascribed to exothermic curing, air inclusion or water availability. The surface of URHF (Figure 8b) and MRHF5 (Figure 8c) revealed bonding fracture, demonstrated by interfacial de-adherence between fiber and matrix as a result of reduced interaction and inferior adhesion. However, on the surface of MRHF 10 (Figure 8d), no voids or debonding were observed, implying effective compatibility between RHF and matrix, resulting in enhanced tension distribution. The fractured area dwells more on debonding in URHF, and improved adherence and greater flattened fracture in MRHF 10 at macroscopic level as revealed in Figure 8d. These are confirmed by TEM images (9a, b, c, and d) respectively. It is well established that pre-modification can initiate improved adherence between matrix and fiber [36, 37]. Hence, micrographic and photographic results revealed that alkaline modification enabled improved interface between RHF and cardanol thermosetting polymer, especially MRHF 10. These results demonstrate agreement with DSC, TG, and Tensile tests results.

Nowadays, there is increasing demand for ecobenign and more durable materials. Results of TP and biocomposites behavior in simulated soil after 90 days are shown in Figure 10.

Initially, increased weight notable in TP, URHF, and MRHF5 were ascribed to water absorption or ad-sorption by samples, which demonstrated resistance to hydrolysis [38], in comparison with MRHF 10. TP demonstrated the highest water inclusion due to inherent voids as observed by FESEM. Observable increase in material weight was ascribed to development of micro-organisms involved in polymer degradation. Also, during the 90 day duration of analysis, a weight loss of 10 ± 0.5, 41 ± 0.2, 65 ± 0.3 and 66% ± 0.3% were observed in TP, URHF, MRHF5, and MRHF 10, respectively. Research has shown that polymers undergo biological degradation via oxidation and hydrolysis. Hence, initial stage of aging in simulated-soil is equivalent to abiotic phase, were macromolecules undergo hydrolyzation and formation of smaller molecules such as monomers and oligomers [39]. A study has demonstrated that lignin protects the fiber from activities of micro-organisms. Hence, it becomes feasible to imply that alkaline modification was responsible for the highest weight reduction observed in MRHF5, and MRHF10, due to lignin elimination, which caused the modified fibers and their biocomposites to become more prone to biodegradation.
The FTIR spectra of cardanol revealed the presence of notable functional groups. Alkynes bend (C–H bending) was observed at 693 cm⁻¹ with a doublet at 823 cm⁻¹. The ester group (C–O stretch) was identified at 909 cm⁻¹, 1156 cm⁻¹, and 1207 cm⁻¹. The presence of aromatic amines (C–N stretch) was confirmed at 3318 cm⁻¹. The peaks corresponding to the presence of alkenes (–C=C– stretch) was confirmed at 1453 cm⁻¹ with a doublet at 3012 cm⁻¹. The alkanes (C–H stretch) group was identified at 2855 cm⁻¹ - 2926 cm⁻¹ with CH₂ symmetric stretch. A lower peak observed on the aliphatic C–H stretching in CH₃ which was identified at 1362 cm⁻¹ was reported [40].

The FTIR spectra of specimens are elucidated in Figure 11.

4. Conclusions
This study investigated efficient usage of cardanol as basis for the development of a TP matrix filled RHF biocomposite. Combined curing at room temperature and post-curing were simultaneously conducted to elevated handling duration, with enhanced matrix-polymer interaction, in addition to optimal degree of cross linking and complete curing. Gravimetric technique utilized in determining TP DoC revealed that DETA has capability of giving room temperature DoC thereby presenting a good alternative as a polymer system indexing tool, especially as a result of its inexpensiveness and efficient performance. However, results from DSC affirmed complete conversion of groups linked in the crosslinking, while also demonstrating that curing and post-curing parameters applied to the TP and biocomposites were appropriate. DSC curves revealed a Tg around 60 °C of the TP and biocomposites, implying that the RHF did not impact on Tg. Despite the TP exhibiting superior thermal stability when compared with biocomposites, RHF seemed a viable reinforcement, as demonstrated by the high enhancement in mechanical behavior of the biocomposites. Results emanating from DSC and TG revealed that MRHF10 exhibited the highest thermal stability. The most superior tensile behavior was exhibited by MRHF10, and ascribed to good inclusion, distribution and adherence to the polymer matrix, as demonstrated by FESEM. The presence of modified fiber enabled improved biocomposites biodegradability behavior as demonstrated after 90 days in simulated-soil. Thus, the present paper offers vital information, while exposing a set of experimental results on the synthesis, and characterization of a cardanol obtained TP and RHF biocomposites using a cheap mode of fabrication. The materials demonstrate good potential for applications in several sectors such as electronics, furniture, articles, and automobile industries.

CRediT authorship contribution statement
Iheoma C. Nwuzor: Conceptualization, Methodology, Formal analysis, Writing - review & editing, Funding acquisition.
Jeremiah L. Chukwunweke: Validation, Formal analysis.
Chinomso M. Ewulonu: Investigation, Writing - original draft.
Paul C. Okolie: Supervision.

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Conflict of interests
The Authors report no conflict of interests.

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