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Influence of Chemical Surface Modifications on Mechanical Properties of *Combretum dolichopetalum* Fibre - High Density Polyethylene (HDPE) Composites

Azeez Taofik Oladimeji[a]*, Onukwuli Okechukwu Dominic[b], Walter Peter Echeng[b] and Menkiti Mathew Chukwudi[b]

[a]Biomedical Technology Department, Federal University of Technology, P. M. B. 1526, Owerri, Imo State, Nigeria

[b]Chemical Engineering Department, Nnamdi Azikiwe University, P. M. B. 5025, Awka, Anambra State, Nigeria

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Abstract. Maximizing the use of natural fibres as ecofriendly materials in polymer composite applications reduces its threat posed to human through increased biomass in the environment. In this study, the effect of chemical surface modifications using acetic anhydride and sodium hydroxide solution on the mechanical properties of *Combretum dolichopetalum* fibre-HDPE composites was aimed to be investigated. Fibres were treated with 6 % acetic anhydride and 12 % NaOH solutions for 30 minutes at room temperature based on optimum treatment conditions after water retting extraction process, then, the composites were prepared. The mechanical properties (tensile strength, tensile modulus, flexural strength, flexural modulus, hardness and impact strength) of the *C. dolichopetalum* fibre reinforced HDPE matrix composites and scanning electron microscope analysis were studied. *C. dolichopetalum* fibre was not only effective as reinforcement of HDPE matrix but mercerization and acetylation of C. *dolichopetalum* fibre ultimately enhanced the mechanical properties of HDPE composites. Scanning electron microscope analysis revealed that HDPE matrix possess better adhesive interaction with acetylated and mercerized *C. dolichopetalum* fibre compared with untreated *C. dolichopetalum* fibre at ultimate tensile strength.

Keywords: *Combretum dolichopetalum,* fibre, mechanical properties, HDPE matrix, mercerization, acetylation

Introduction

Combretum dolichopetalum plant is commonly known as *sun birds wine* plant which belongs to the genus *Combretum* that comprises of about 20 genera and 600 species distributed in Africa and Asia. The extract of *C. dolichopetalum* species are extensively applied in traditional medicine in southern part of Nigeria. After extraction of the active ingredients, the crystalline fibre of *C. dolichopetalum* usually disposed to the environment, thus increasing biomass in the environment. The gradual depletion of petroleum resources worldwide and the enactment of new rules and regulations on environmental preservation and management have triggered the demand for new materials that are ecofriendly (Hashim *et al*., 2012; Srinivasa and Bharat, 2011; Sinha and Rout, 2009). Products made from synthetic fibre reinforced composites are non-recyclable and constitute a threat to the environment at the end of their useful life, since they cannot be conveniently disposed (Saira *et al*., 2007). Therefore, the use of natural cellulosic fibres as reinforcement for polymeric matrix has become an attractive venture. For three thousand years now, natural fibres have been used to reinforce materials (Ashik and Sharma, 2015; Sakthivei and Romesh, 2013). Currently, natural fibre have been employed in combination with plastics. Composites made by reinforcing natural fibres are less dense, ecofriendly, and improved electrical resistance, high strength to weight ratio and corrosion resistance (Azeez and Onukwuli, 2017; Thompson, 2013; Ishak *et al.*, 2009). These composites reduce wear of processing equipment and devoid of health implication during processing, application and upon disposal. However, the inclusion of lignocellulosic fibres into thermoplastic or thermosetting polymer is often associated with poor fibre dispersion due to the large differences in polarity between the fibre and polymer, strong intermolecular and hydrogen bond between the fibres and matrix (Sanjay *et al*., 2016; Shah *et al*., 2010 Siregar *et al*., 2010). These bottlenecks have been overcome by suitable physical, chemical and enzymatic treatments (Osorio *et al*., 2012). The chemical treatment

*Author for correspondence;
E-mail: taofikoladimeji@gmail.com

may be used to improve hydrophilic in nature of natural fibre, interfacial bonding between matrix and fibre, surface roughness, wettability and decreased moisture absorption, thermal and electrical properties (Beckermann and Pickering, 2008; Noranizan and Ahmad, 2012; Raj *et al*., 2011). Many researchers have applied mercerization and acetic anhydride treatment with remarkable improvement in the mechanical properties of both treated fibres and/or composites at optimum conditions (Hossain *et al*., 2014; Punyamurthy *et al*., 2014; Singha and Thakur, 2014; Tlijania *et al*., 2014; Noorunnisa *et al*., 2011; Zhong *et al*., 2010). Higher concentration of alkali solution may also lead to excess delignification of fibre which weakens and damages the fibre (Li *et al*., 2007). Sampathkumar *et al.* (2012) and Arsene *et al.* (2005) reported decline in properties after alkali treatment of areca fibre, sugar cane bagasse and banana tree trunk fibres respectively. However, the investigation was not only pioneer the use of *C. dolichopetalum* fibre as reinforcement of HDPE matrix which will reduce the threat posed by biomass of *C. dolichopetalum* fibre but influence of chemical surface modifications to improve the mechanical properties of *C. dolichopetalum* fibre-HDPE composites was aimed to be investigated.

Materials and Methods

Combretum dolichopetalum plant was obtained from Bayaoje in Surulere Local Government Area of Oyo state, Nigeria. HDPE matrix obtained from Eleme Petrochemical Company, Port Harcourt in River State, Nigeria was used with tensile strength, tensile modulus, flexural strength, flexural modulus, hardness and impact strength of 24.619 MPa, 836.25 MPa, 27.114 MPa, 1390.7 MPa, 21 HR and 859.3 kPa, respectively. Sodium hydroxide and acetic anhydride used for fibre modifications were collected from Rovert Scientific Limited (RC-627785), Benin City in Edo State, Nigeria.

Fibre extraction. The *C. dolichopetalum* fibres were extracted from the plant stem using water retting extraction process in accordance with the method described by Nguyen *et al.* (2012). 30kg of plant stem was retted in deionized water for 21 days, washed at 3 days interval until fibre was produced, then sun dried for 7 days and later dried at a temperature of 60 °C for 2 h.

Mercerization and acetylation of *C. dolichopetalum* fibre. Strands of *C. dolichopetalum* fibres (average length of 150 mm was cut into 10 mm) mercerized with 12% NaOH solution (mCDF) and acetylated with 6% acetic anhydride (aCDF) having tensile strength of 71.267 and 99.282 MPa, respectively, at room temperature for 30 min as optimum treatment conditions reported by Walter *et al*. (2016), then washed severally with deionized water to ensure neutral pH. The fibres were finally dried in an air oven at 60 °C for 2 h.

Composite preparation. The untreated and treated *C. dolichopetalum* fibres of 10 mm length were mixed with high density polyethylene pellets (HDPE) in different proportions (0:100, 2.5:97.5, 3.75:96.25, 5:95, 6.25: 93.75 and 7.5:92.5). The fibre - HDPE mixtures were processed by injection moulding method. Rectangular test specimens having dimensions of 150 x 25 x 3 mm^3 were cut from the composites according to ASTM 638-90 standard.

Characterization of composites. ***Tensile testing.*** Tensile test using tenstometer machine (Model: M500-25KN, OL11 1NR, England) was carried out at Foundary Department, Federal Institute of Industrial Research, Osodi, Lagos in accordance with BS EN ISO 903: 1998 on a rectangular shape of CDF - HDPE laminates having dimensions of 80 mm (span) × 25 mm (width) × 3mm (thickness) with a constant rate of transverse of the moving grip of 40 mm /min was used in evaluating the tensile properties.

Flexural testing. 3 - point flexural test using tenstometer (Model: M500-25KN, OL11 1NR, England) was carried out at Foundary Department, Federal Institute of Industrial Research, Osodi, Lagos in accordance with BS EN ISO 903: 1998 on a rectangular shape of CDF-HDPE composites having dimensions of 80 mm (span) × 25mm (width) × 3mm (thickness) with a constant rate of 40 mm/min.

Impact testing. Unnotched Izod impact test using cantilevered beam configuration with tenstometer (Model: M500-25KN, OL11 1NR, England) was carried out at Foundary Department, Federal Institute of Industrial Research, Oshodi, Lagos in accordance with BS EN ISO 903: 1998 on a rectangular shape of CDF-HDPE laminates with dimensions of 80mm (span) × 25mm (width) × 3mm (thickness) for a constant rate of 40 mm /min.

Hardness testing. A standard Rockwell tester (model Testor HT 1a, Otto Wolpert-Werke, Germany) was used with steel indenter to measure the hardness of the test specimen. The hardness test was carried out at Material

and Metallurgical Department, Federal University of Technology, Owerri, Nigeria. A load of 150kgf was applied for each measurement on the specimen with parallel flat surfaces of the avail of the apparatus and minor load (15kg$_f$) was applied by lowering the steel ball onto the surface of the specimen. The dial was adjusted to zero on the scale under minor load and the major load was immediately applied by releasing the trip lever. After 15sec, the major load was removed and Rockwell hardness was recorded.

Scanning electron microscope (SEM). The SEM micrograph of tensile strength fractured surface of CDF - HDPE composites were taken using Scanning Electron Microscope (Model Phenom-Prox of Eindloven Netherlands) at Ahmadu Bello University, Zaria, Nigeria. The samples were sputter coated with gold within 24 h in a SEM coating unit. The fractured surfaces of gold coated samples were stored in desiccators till SEM observation was made.

Results and Discussion

Tensile properties. Figure 1(a) shows the effect of fibre loading on the tensile strength behaviour of untreated and treated *C. dolichopetalum* fibre - HDPE composites. The tensile strength of the composite of both untreated and treated *C. dolichopetalum* fibre - HDPE composites varies with increased fibre loading. The ultimate tensile strengths of uCDF, mCDF and aCDF - HDPE composites were obtained at fibre weight fraction of 2.5, 3.75 and 5.0% percent, respectively. This shows that the ultimate tensile strength of mCDF and aCDF - HDPE composite increased by 7.64 and 18.78% of the uCDF - HDPE composite, respectively. This may be attributed to increased interfacial adhesion between the fibre and matrix, hydrophilic nature of the fibres and the presence of strong hydrogen bonding as well as improved stress transfer between the matrix and fibre, thereby, maximizes utilization of the fibre in the composite as also reported by many researchers (Arfin *et al*., 2012; Ramanaiah *et al*., 2012; Zhong *et al*., 2007; Yang *et al*., 2004). Figure 1(b) shows the tensile modulus of untreated and treated *C. dolichopetalum* fibre - HDPE composites with varying fibre loading. The ultimate tensile modulus for untreated, mercerized and acetic anhydride treated HDPE composites was obtained at fibre loading of 2.5, 3.75 and 2.5 %, respectively. The mercerized and acetylated fibre loading increased the tensile modulus by 4.83 and 129.84%, respectively, as compared to that of untreated composite. This indicated

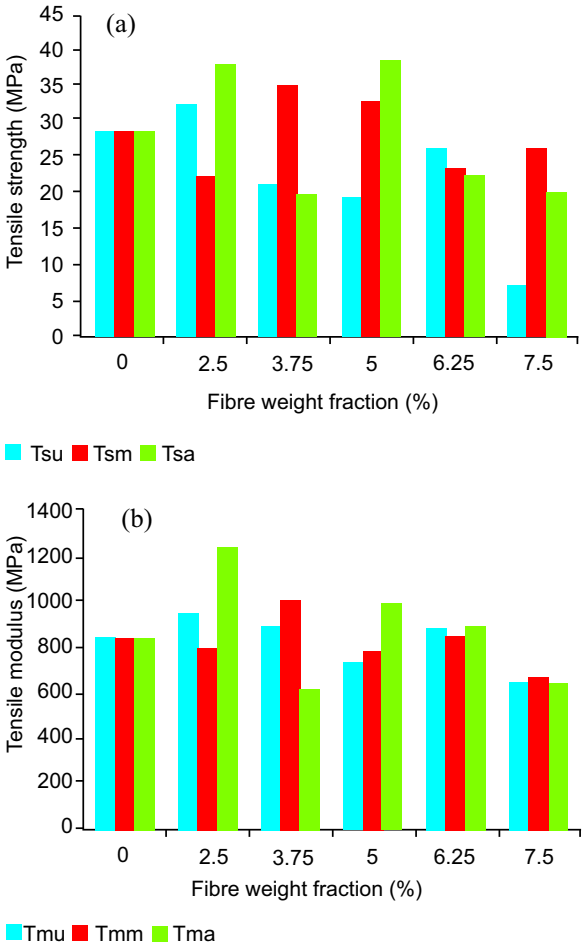
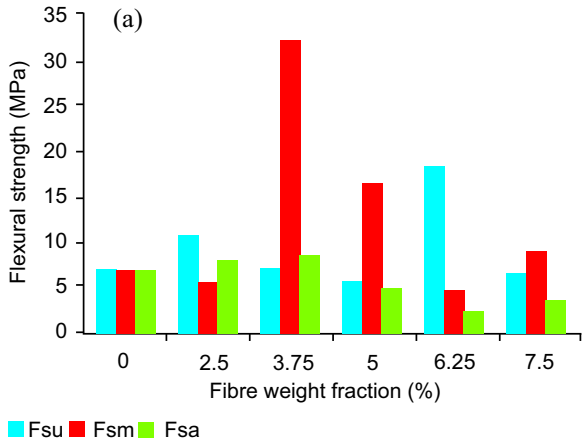


Fig. 1. Chemical surface modification of CDF - HDPE composite on (a) tensile strength (b) tensile modulus.
Tsu, Tsm and Tsa, represent the tensile strength of untreated, mercerized and acetic anhydride treated fibre-HDPE composites, respectively. Tmu, Tmm and Tma means tensile modulus of untreated, mercerized and acetic anhydride treated fibre-HDPE composites, respectively.

that mercerization and acetylation of fibres increases the fibre distribution which increases the stiffness of the CDF - HDPE composites. This is in agreement with the report of Arfin *et al.* (2012).

Flexural properties. Flexural strength of CDF - HDPE composites as illustrated in Fig. 2(a). It was observed that the flexural strength of uCDF and mCDF - HDPE composites initially decreases which may be attributed to non- uniform distribution of the fibre in the matrix and then, increases which may be due to good



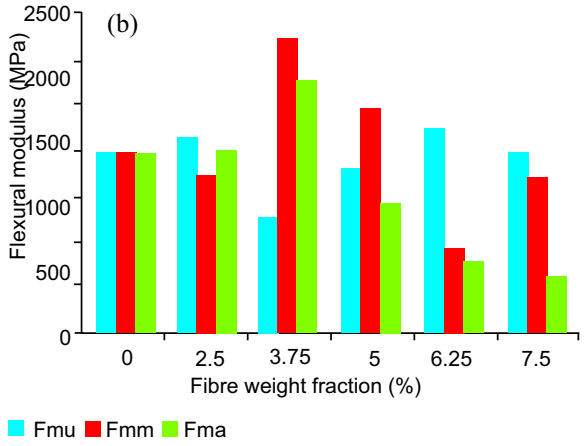


Fig. 2. Chemical surface modification of CDF - HDPE composite on (a) flexural strength (b) flexural modulus Fmu. Fsu, Fsm and Fsa means flexural strength of untreated, mercerized and acetic anhydride treated fibre - HDPE composites, respectivily. Fmm and Fma means Flexural modulus of untreated, mercerized and acetic anhydride treated fibre-HDPE composites, respectively.

compatibility between the fibre and matrix with increased fibre loading. This shows that the ultimate flexural strength of mCDF - HDPE composite increased by 76.19% and that of an acetylated one reduces by 53.24% of the uCDF - HDPE composite at fibre loading of 3.75%. However, the ultimate flexural modulus of mCDF and aCDF - HDPE composites increased by 45.22 and 23.99%, respectively, at fibre loading of 3.75% compared with uCDF - HDPE composite at 6.25% of the fibre loading as shown in Fig. 2(b). This may be attributed to the increased interfacial adhesion,

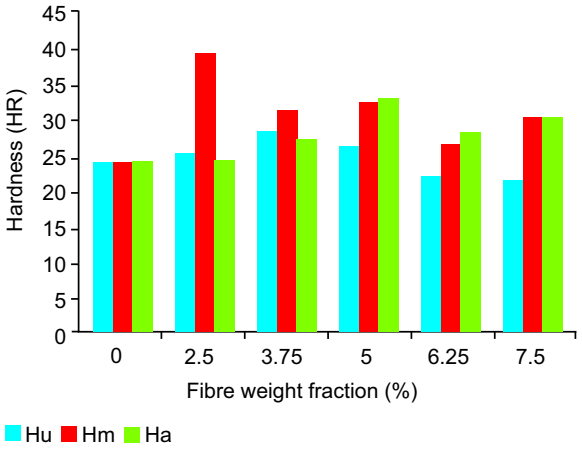


Fig. 3. Chemical surface modification of CDF - HDPE composite on hardness property. Hu, Hm and Ha means Hardness of untreated, mercerized and acetic anhydride treated fibre-HDPE composites, respectively.

dispersion of fibre in the matrix and stabilization of molecular orientation of fibre (Chandramohan and Marimuthu, 2011).

Hardness. The hardness of untreated and treated of *C. dolichopetalum* fibre - HDPE composites were initially increased with increasing fibre loading and later declined as shown in Fig. 3. The ultimate hardness of mCDF and aCDF - HDPE composites increased by 39.29 and

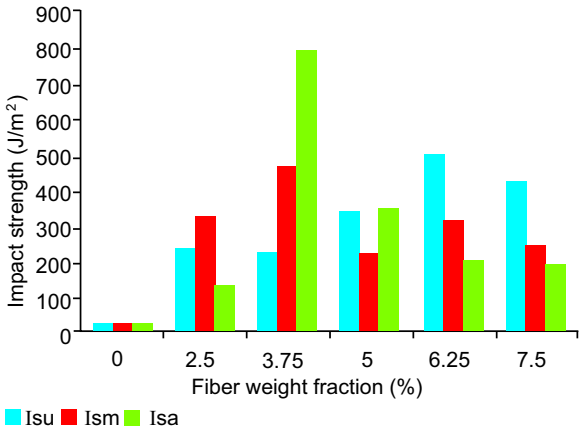


Fig. 4. Chemical surface modification of CDF-HDPE composite on impact strength. Isu, Ism and Isa means Impact strength of untreated, mercerized and acetic anhydride treated fibre-HDPE composites, respectively.

17.85% at fibre loadings of 2.5 and 5.0%, respectively, compared with that of uCDF - HDPE composite at fibre loading of 3.75%. This indicated that mercerization and acetylation of *C. dolichopetalum* fibre improved the hardness of the HDPE composite which is in agreement with the report of researchers (Aldousiri *et al*., 2011; Ishidi *et al*., 2011).

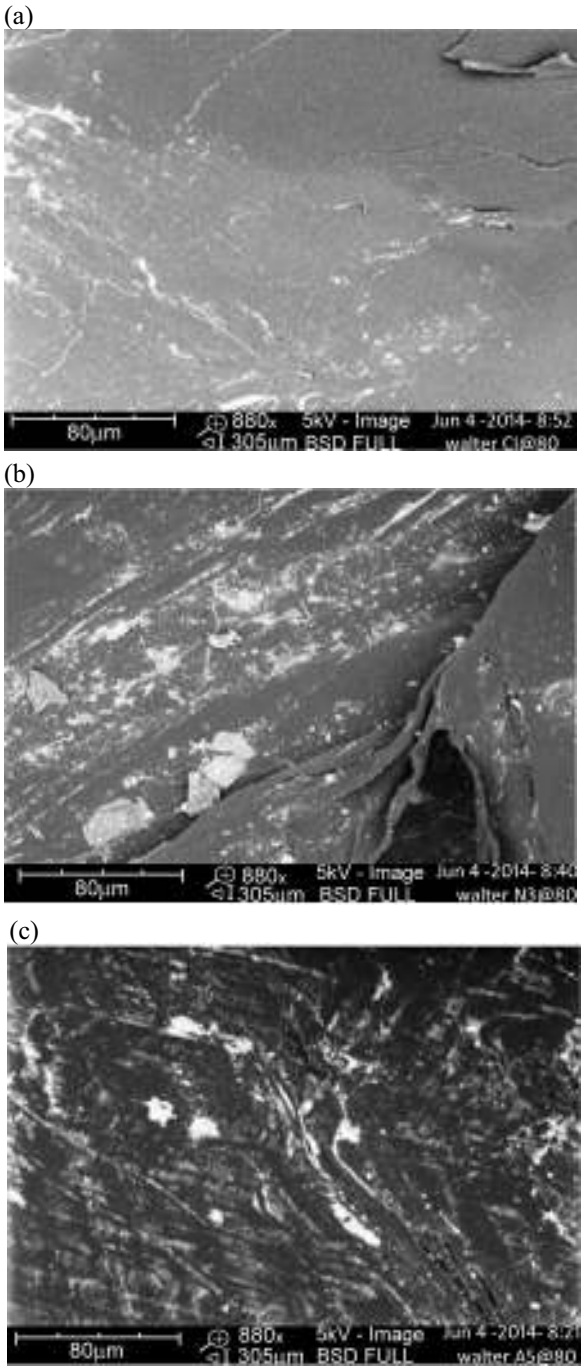


Fig. 5. SEM micrographs of CDF - HDPE composites (2.50% v/v) with magnifications of x 880 for (a) untreated (b) mercerized and (c) acetylated.

Impact strength. Figure 4 shows that the impact strength of the uCDF, mCDF and aCDF - HDPE composites increases with increasing fibre loading for up to 3.75%. Ultimate impact strength was obtained at 3.75% for mCDF and aCDF - HDPE composites while that of uCDF - HDPE composite reaches at fibre loading of 6.25%. It can be deduced that the ultimate impact strength of mCDF - HDPE composite reduced by 5.33% while that of aCDF - HDPE composite increased by 58.73% of uCDF - HDPE composite.

Scanning electron microscope analysis. The changes in the topography and morphology of *C. dolichopetalum* fibre - HDPE composites were studied by SEM. It can be observed that *C. dolichopetalum* fibre distribution into the HDPE matrix is reasonably good with minimal voids found in the composites. It seems that the uCDF were not evenly distributed in HDPE matrix as observed in Fig. 5a compared with mCDF and aCDF as shown in Fig. 5b and 5c, respectively. However, acetylated *C. dolichopetalum* fibres are more evenly distributed than mercerized fibres. This indicates mercerization and acetylation treatments of *C. dolichopetalum* fibres in the HDPE composite has a profound effect in creating a reasonably good dispersion and better interfacial adhesion between the components, which have been confirmed with the mechanical studies. Though, acetylated fibre shows superiority when compared with mercerized fibre in reinforcement of HDPE composites. This is similar to the report of Muhammed *et al.* (2015) and Favaro *et al.* (2010).

Conclusion

The influence of chemical surface modifications on the mechanical properties of *C. dolichopetalum* fibre reinforced HDPE composites was studied for fibre process conditions of 12% NaOH and 6% acetic anhydride solutions for 30 min, respectively, at room temperature. HDPE composites with acetylated *C. dolichopetalum* fibres showed superior improvement in tensile strength, tensile modulus and impact strength compared to untreated and mercerized composites due to improved fibre distribution, fibre - matrix interaction and mechanical interlocking facilitated by fibre surface

modification. Mercerized *C. dolichopetalum* fibre proved to have the best improvement in flexural properties and hardness of HDPE composites.

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