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Article

## Effect of Chemically Modified *Cissus Populnea* Fibers on Mechanical, Microstructural and Physical Properties of *Cissus populnea*/High Density Polyethylene Composites

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**Abstract.** The effect of chemically modified *Cissus populnea* (*C. populnea*) fiber using sodium hydroxide (NaOH) and sodium lauryl sulphate (SLS) on mechanical, morphological and physical (density and water absorption behaviour) properties of *C. populnea* fiber/recycled HDPE composites was aimed to be investigated. The composites of unmodified and modified *C. populnea* fiber/HDPE were prepared using injection molding machine. The mechanical properties (tensile strength and modulus, flexural strength and modulus, hardness and impact strength), interfacial shear stress, density, water absorption behaviour and microstructural properties using scanning electron microscope (SEM) and fourier transform infrared (FTIR) spectroscope were characterized. The results shows that *C. populnea* fiber increased the mechanical properties of HDPE matrix with reduced impact strength of the composites. The NaOH and SLS treatments, respectively, improved the mechanical properties of *C. populnea* fiber/HDPE composites, although NaOH treated *C. populnea* fiber reduced the tensile modulus. The change in morphology and functional group, respectively, due to the modification was observed in SEM and FTIR. The density and water absorption of the composites, respectively, reduced when SLS modified *C. populnea* fiber was used compared to untreated *C. populnea* fiber/HDPE composites. The SLS treated *C. populnea* fiber prove to be superior for reinforcement, stiffness and light weight material.

**Keywords:** *C. populnea* fiber, optimization, mechanical properties, SEM, physical properties.

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## 1. Introduction

Polyethylene (PE) is the most widely used kind of synthetic thermoplastic polymers today because of its low cost and its easy processability. Researchers reported that its restriction on application attributed to poor mechanical properties such as tensile strength, tensile modulus and flexural strength [1]. Fibers are considered to be effective reinforcing materials for PE. Various kinds of fibers (natural-fiber, glass fiber, keratin feather fiber, metallic fibers) have been widely used to improve the mechanical properties of PE [1–5]. Natural fiber exhibits a high hydrophilicity due to interaction between the hydroxyl groups of fiber components and water molecules originated from non-crystalline region of the fiber [6]. This initiated poor interfacial adhesion and resistance to water absorption. Many surface modifications (such as acetylation, methylation, cyanoethylation/mercerization, benzylation, permanganate treatment, acrylation,  $\text{Cr}_2(\text{SO}_4)_3 \cdot 12\text{H}_2\text{O}$ , and  $\text{CrSO}_4$  and  $\text{NaHCO}_3$  etc) have been used to improve the mechanical properties of fiber, density and water absorption for effective use in composites applications [6–9]. The effectiveness of various surface modifications depends on the types of fiber, composition, climatic conditions, source etc [7]. It has been reported that acetylation increased tensile and flexural properties with reduced impact strength of dolichopetalum fiber/HDPE composites [8], as well as flax fiber/polypropylene composites [6, 8]. Carbonization also improved the mechanical properties of saw dust/HDPE composites [10]. Mercerization had been reported to be a good modification techniques and improved the water absorption, tensile, flexural, thermal properties with reduced impact strength and density through improved interfacial adhesion of natural fiber/HDPE composites [11–16], henequen fiber/HDPE composites [14], palm kernel nut shell/HDPE composites [15], hemp fiber/HDPE composites [16]. Although, sodium lauryl sulphate modification has been reported to be a better modification of banana and kenaf fiber/unsaturated polyester matrix composites for improvement of mechanical properties compared with NaOH treatment [17] but with no report on other natural fibers/HDPE composites. The objective of this work was to study the effect of chemically modified *Cissus populnea* fiber on the mechanical, microstructural (using SEM and FTIR) and physical properties of composites of recycled high density polyethylene (PE).

## 2. Materials and Methods

### 2.1. *C. populnea* Fiber Extraction and Modifications

*C. populnea* fiber was extracted by water retting technique from its plant obtained from Gbana, Oriire Local Government Area of Oyo state, Nigeria. Its proximate composition using gravimetric method described by Hänninen et al. [18]: moisture content (3.94 %), water soluble (2.33 %), ash (1.59 %), wax (2.94 %), pectin (1.14 %), lignin (11.52 %), hemicelluloses (14.74 %) and cellulose (61.8 %). *C. populnea* were chopped into short length of 10mm and modified at optimal modifications of NaOH (15 % for 20.94 mins) and sodium lauryl sulphate (SLS) (5.84% for 20.22 mins), washed with deionized water, then oven dried at 60°C for 2hours. Sodium hydroxide and sodium lauryl sulphate are analytical grade chemicals obtained from Rovert scientific limited, Benin city in Edo state, Nigeria. The aspect ratio of untreated, NaOH and SLS treated *C. populnea* fibers, respectively, was obtained to be 909.1, 961.5 and 1052.63.

### 2.2. Composite Preparation

The *C. populnea* fibers were mixed with waste HDPE of density and melting point of 0.9425g/cm<sup>3</sup> and 195°C. *C. populnea* fiber/HDPE matrix were processed by injection moulding machine at temperature range of 180°C – 210°C. The developed CCD matrix of RSM using Design of Experiment (DoE) software version 6.0.8 (2002 East Hennepin Ave., Suite 480 Minneapolis, MN 55413, Stat-Ease, Inc.) for two factors (weight fraction of fiber and matrix) with six responses (tensile strength and modulus, flexural strength and modulus, hardness and impact strength) are presented in Table 1–3. The fiber weight fraction of 1.4645, 2.5, 5, 7.5 and 8.5355%. The fiber weight fraction was determined by 100% conversion technique (ratio of weight fraction of fiber to summation of the mixed fibers and HDPE matrix, then multiply by 100%). The choice of using range of fiber weight fraction was based on preliminary experiment conducted so as to avoid difficulty in processing of the waste HDPE and fibers with the use of injection molding machine due to agglomeration and uneven distribution of fibers. The DoE with ANOVA was used to obtain and justify optimum responses at optimum composition (factors), observed interaction between the fibers and waste HDPE composites and avoid waste of materials.

Table 1. CCD matrix of untreated *C. populnea* fiber/HDPE composites with mechanical properties.

$W_f$ (wt %)	$W_p$ (wt %)	$T_s$ (MPa)	$T_m$ (MPa)	$F_s$ (MPa)	$F_m$ (MPa)	$H$ (HR)	$I_s$ (J/mm <sup>2</sup> )
7.5	97.5	28.293*	743.51	36.524	1425.2	33	0.14536
5	95	27.647	924.44	34.321	1297.8	32	0.11799736
2.5	97.5	26.757	690.45	35.837	1549.1*	31	0.17289855*
2.5	92.5	26.324	731.87	36.319	1421.5	31	0.153
8.5355	95	8.3173	261.73	36.712*	1400.1	35*	0.15524
7.5	92.5	3.6576	257.6	36.693	1375.9	33	0.14704875
5	91.4645	27.956	927.53*	35.641	1395.4	33	0.1573
5	95	27.647	924.44	34.321	1297.8	32	0.117997
5	95	27.647	924.44	34.321	1297.8	32	0.117997
5	98.5355	26.843	819.62	35.001	1368.2	32	0.13615
5	95	27.647	924.44	34.321	1297.8	32	0.117997
5	95	27.647	924.44	34.321	1297.8	32	0.117997
1.4645	95	25.483	591.81	31.304	1411.3	28	0.15214

Note:  $W_f$ ,  $W_p$ ,  $T_s$ ,  $T_m$ ,  $F_s$ ,  $F_m$ ,  $H$ , and  $I_s$ , are weight fraction of fiber, HDPE weight fraction, tensile strength, tensile modulus, Flexural strength, flexural modulus, hardness and impact strength respectively. Superscript (\*) indicates ultimate property.

Table 2. CCD matrix for NaOH treated *C. populnea* fiber-HDPE composites with mechanical properties.

$W_f$ (wt %)	$W_p$ (wt %)	$T_s$ (MPa)	$T_m$ (MPa)	$F_s$ (MPa)	$F_m$ (MPa)	$H$ (HR)	$I_s$ (J/mm <sup>2</sup> )
8.5355	95	28.921	780.14	38.134	1791.3*	36*	0.338421321
5	95	26.231	708.27	30.501	993.33	36*	0.230693333
5	95	26.231	708.27	30.501	993.33	36*	0.230693
5	95	26.231	708.27	30.501	993.33	36*	0.230693
5	95	26.231	708.27	30.501	993.33	36*	0.230693
2.5	97.5	21.431	592.73	19.104	658.04	34	0.241933333
5	91.4645	23.257	712.41	30.703	1041.3	34	0.47351
7.5	92.5	29.703*	781.49*	39.328*	1783.1	35	0.398413333
1.4645	95	20.862	730.37	23.921	984.2	29	0.43215
2.5	92.5	22.415	600.13	27.821	1426.8	32	0.2536333
5	98.5355	25.001	641.89	28.05	1000.5	34	0.0242431
7.5	97.5	26.153	653.12	33.031	1573.3	34	0.42653333*
5	95	26.231	708.27	30.501	993.33	36*	0.230693

Note:  $W_f$ ,  $W_p$ ,  $T_s$ ,  $T_m$ ,  $F_s$ ,  $F_m$ ,  $H$ , and  $I_s$ , are weight fraction of fiber, HDPE weight fraction, tensile strength, tensile modulus, Flexural strength, flexural modulus, hardness and impact strength respectively. Superscript (\*) indicates ultimate property.

Table 3. CCD matrix of SLS treated *C. populnea* fiber-HDPE composites with mechanical properties.

$W_f$ (wt %)	$W_p$ (wt %)	$T_s$ (MPa)	$T_m$ (MPa)	$F_s$ (MPa)	$F_m$ (MPa)	$H$ (HR)	$I_s$ (J/mm <sup>2</sup> )
2.5	92.5	22.146	613.23	41.007	1645	34	0.341326
5	95	27.853	844.52	38.076	1420.7	39*	0.33797333
5	91.4645	27.981	872.71	41.562	1582.3	36	0.36217
5	95	27.853	844.52	38.076	1420.7	39*	0.337973
7.5	92.5	33.84	836.97	34.321	1336.7	37	0.49772*
5	95	27.853	844.52	38.076	1420.7	39*	0.337973
1.4645	95	29.148	801.24	30.013	2014.5	31	0.02954
7.5	97.5	31.833	795.32	39.513	1491.6	39*	0.4951932
5	95	27.853	844.52	38.076	1420.7	39*	0.337973
5	98.5355	26.417	861.5	39.53	1440.1	34	0.345373
8.5355	95	34.23	847.13	32.153	1345.1	39*	0.51231
5	95	27.853	844.52	38.076	1420.7	39*	0.337973
2.5	97.5	36.453*	932.17*	66.206*	2710.4*	38	0.32906667

Note:  $W_f$ ,  $W_p$ ,  $T_s$ ,  $T_m$ ,  $F_s$ ,  $F_m$ ,  $H$ , and  $I_s$ , are weight fraction of fiber, HDPE weight fraction, tensile strength, tensile modulus, Flexural strength, flexural modulus, hardness and impact strength respectively. Superscript (\*) indicates ultimate property.

### 2.3. Mechanical Properties

Tensile test with 10kN load cell on a sample of 150mm x 25mm x 3mm with a gauge length of 100mm, 3-point flexural test on a 80mm (span) x 25mm (width) x 3mm (thickness) and unnotched impact properties on a 80mm (span) x 25mm (width) x 3mm (thickness) were determined on a rectangular shape of *C. populnea* fiber/HDPE laminates using tensometer machine Model: M500-25KN, OL11 1NR at a constant rate of 40 mm /min of moving grip. A standard Rockwell tester (model Testor HT 1a, Otto Wolpert-Werke) was used with steel indenter to measure the hardness of the test specimen according to ASTM E – 18. Load of 150kgf was applied for each measurement on the specimen with parallel flat surfaces of the avail of the apparatus and minor load (15kgf) 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 (150kgf) was immediately applied by releasing the trip lever. After 15 second the major load was removed and Rockwell hardness was recorded. Each mechanical test was conducted on five test specimens.

### 2.4. Microstructural Analysis

#### 2.4.1. Scanning electron microscope analysis

High resolution scanning electron microscope (SEM) of ASPEX 3020 model was used to study the morphology of surfaces of the *C. populnea* fiber/HDPE at optimal conditions. The surfaces of the fiber was examined directly by scanning electron microscope (SEM) ASPEX 3020 model at 20 KeV and  $5.0 \times 10^{-5}$  torr. *C. populnea* fiber/HDPE composites sample was mounted on stubs with silver paste. To enhance the conductivity of the *C. populnea* fiber/HDPE composites, a thin film of platinum is vacuum-evaporated before the photomicrographs or spectrum were taken.

#### 2.4.2. Fourier transform infrared (FTIR) analysis

4.0 g of *C. populnea* fiber/HDPE composites at optimal properties was crushed into pellets. A Buck Scientific M500 Infrared Spectrophotometer was used for the analysis. A total of 10 scans was taken for each fiber sample on KBr cell covering the range of wave number of  $600 - 4000 \text{ cm}^{-1}$  with a resolution of  $4 \text{ cm}^{-1}$  for 20 seconds. The crushed powder sample (0.1g) was mixed with dry KBr (0.4g) and transferred to sample compartment of the Buck Scientific M500 Infrared Spectrophotometer. The spectrophotometer was set at 100% transmittance with pure KBr pellet and the transmittance reading was obtained and stored.

### 2.5. Physical Properties

#### 2.5.1. Density of *C. populnea* fiber/HDPE composites

*C. populnea* fiber/HDPE samples were selected and bound into a bundle and its mass measured on a digital weighing balance with resolution 0.001 g. The volume of this fixed mass of *C. populnea* fiber/HDPE composites. The density was calculated using Eq. (1):

$$\rho_f = \frac{M}{V} \quad (1)$$

where  $\rho_f$  is density of *C. populnea* fiber/HDPE composites measured in grams per cubic centimeters,  $M$  is the *C. populnea* fiber/HDPE composites quantity immersed in deionized water in grams and  $V$  is the volume water displaced by the composites.

#### 2.5.2. Water absorption of *C. populnea* fiber/HDPE composites

The test was carried out in accordance with ASTM D – 570. Prior to testing, the *C. populnea* fiber/HDPE composites were dried in an oven at  $60^\circ\text{C}$  for 24 hours. The *C. populnea* fiber/HDPE composites with a dimension of 30mm x 25mm x 3mm were then soaked in deionized water for 24 hours at room temperature. The fibers were removed, rid of surface water and immediately weighed. The process was

continued until equilibrium was attained. The water absorption was determined by percentage mass gain using Eq. (2) as given by Isa et al. [19], Singha and Rana [20];

$$\text{Water absorption (\%)} = \frac{M_t - M_0}{M_0} \times 100\% \quad (2)$$

where  $M_t$  is the mass of the sample after conditioning in grams (wet weight),  $M_0$  is the mass of the sample before conditioning in grams (dry weight).

### 2.5.3. Kinetics of water absorption and diffusion behaviours of composites

The water diffusion phenomenon was studied through water absorption method. The kinetics of water absorption and water diffusion coefficient  $D_c$ , respectively, using power law expression (Eq. (3)) and Fickian diffusion model (Eq. (4)) were evaluated as reported by Gierszewska - Drużyńska and Ostrowska-Czubenko [21];

$$\frac{M_t}{M_m} = kt^n \quad (3)$$

$$D_c = \pi \left[ \frac{h}{4M_m} \right]^2 [S]^2 \quad (4)$$

where  $M_t$  and  $M_m$  are the water content at specific time  $t$  and the equilibrium water content (EMC),  $k$  and  $n$  are constants as an intercept and slope of respectively of  $M_t/M_m$  versus  $t$  in the  $\ln - \ln$  plot of water absorption with time. Where  $S = \frac{M_2 - M_1}{\sqrt{t_2} - \sqrt{t_1}}$ ,  $M_m$  is the maximum percentage of water content,  $h$  is the fiber thickness,  $M_1$  and  $M_2$  are percentage of water content at respective time  $t_1$  and  $t_2$  selected in the linear portion of the plot of water sorption ( $M_t$ ) versus  $\sqrt{t}$ .  $S$  was evaluated as gradient plot of  $M_t$  against  $\sqrt{t}$  based on Eq. (4).

## 2.6. Fiber–Matrix Adhesion Test

The fiber pull-out method described by Herrera-Franco and Valadez-Gonzalez [14] was used to determine the interfacial shear strength between the fiber and HDPE matrix. One end of the fiber was embedded in the middle plane of a plate made from the resin. The fibers were first aligned on a plate and a second end one was used to complete the assembly. The samples were made using compression molding at a pressure of 1 ton. The force was applied by holding one end of the resin plate fixed and pulling from the free end of the fiber using tensometer machine Model: M500-25KN, OL11 1NR at a constant rate of 0.4 mm /min of moving grip, equipped with a 100 kg load cell, after conditioning at 25°C. The load and displacements were monitored continuously and upon fiber pull-out, the debonding load (P) was converted into an average interfacial shear strength (IFSS) using Eq. (5):

$$\text{IFSS} = \frac{P}{\pi dL} \quad (5)$$

where  $d$  is the fiber diameter,  $L$  is the embedded length of the fiber.

## 3. Results and Discussion

### 3.1. Mechanical Properties

The mechanical properties of the waste HDPE used include: tensile strength (27.628MPa), tensile modulus (792.59MPa), flexural strength (34.519MPa), flexural modulus (1390.7MPa), hardness (24HR) and impact strength (0.9628J/mm<sup>2</sup>). The CCD matrix of untreated, NaOH and SLS treated *C. populnea* fiber/HDPE composites with mechanical properties as responses are presented in Table 1–3, respectively. For untreated and treated *C. populnea* fiber/HDPE composites, the ultimate mechanical properties were obtained at different composition. The variation in ultimate properties with composition may be misleading in composites application because of uncertainty in composition. This means that there is need to determine the optimum properties of *C. populnea* fiber/HDPE composites at optimum composition. The RSM with CCD design helps in obtain the optimum composition of *C. populnea* fiber/HDPE composites with optimum properties.

The response quadratic models using RSM with CCD of tensile strengths for untreated, NaOH and SLS treated *C. populnea* fiber/HDPE composites are presented in Eq. (6)–(8), respectively. ANOVA of the response model of tensile strengths are presented in Table 4, it can be deduced that tensile strength models for untreated, NaOH and SLS treated *C. populnea* fiber/HDPE composites were significant since  $p < 0.05$ . The results of the experimental data for untreated, NaOH and SLS treated *C. populnea* fiber/HDPE composites, respectively, with  $R^2$  of 0.8832, 0.9232 and 0.9928 explains 88.32, 92.32 and 99.28 % of the observed variability in tensile strength as a result of weight fraction of fiber and matrix. It can be deduced that 11.68, 7.68 and 0.72 % represent residue of untreated, NaOH and SLS *C. populnea* fiber/HDPE composites, respectively, which cannot be explained but may be due to nature, source, diameter of food gum *C. populnea* fiber and other factors that did not considered for composites production. The observed variability may be attributed to good intermolecular structure, interfacial adhesion bond between *C. populnea* fiber and HDPE matrix. Adj.  $R^2$  indicates that the variables (weight fractions of fiber and HDPE matrix) were fit with tensile strength and closer to  $R^2$ . Moreover,  $R^2$  is judged by the adequacy precision  $> 4$  for a model to be considered adequate. Thus, with adequacy precision of 9.7951, 32.8212 and 44.1510 indicated that the quadratic models for tensile strength of untreated, NaOH and SLS treated *C. populnea* fiber/HDPE composites, respectively, were adequate and may be used for design applications. The significance of model terms were judged based on the  $p < 0.05$  of 95% confidence interval. The significant variables obtained for tensile strength of HDPE composites with *C. populnea* fiber: untreated ( $W_f, W_f^2$  and  $W_f W_m$ ), treated with NaOH ( $W_f, W_m, W_f^2$  and  $W_f W_m$ ), and treated with SLS ( $W_f, W_m, W_f^2, W_m^2$  and  $W_f W_m$ ) since  $p < 0.05$ .

$$T_s = -135.82 - 85.285W_f + 6.8749W_m - 0.89543W_f^2 - 0.055477W_m^2 + 0.9681W_fW_m \quad (6)$$

$$T_s = -320.248 + 12.212W_f + 6.9658W_m - 0.1291W_f^2 - 0.03594W_m^2 - 0.10264W_fW_m \quad (7)$$

$$T_s = 2165.473 + 59.8885W_f - 49.3333W_m + 0.2818W_f^2 + 0.2828W_m^2 - 0.65256W_fW_m \quad (8)$$

Table 4. ANOVA for response surface quadratic model of tensile strength of *C. populnea* fiber/HDPE composites.

Source	Model Coefficient	Sum of Squares	DF	Mean Square	F Value	Prob > F	$R^2$	Adj $R^2$	Adeq Precision
<i>HDPE+D</i>									
Model	-135.819665	692.07	5.00	138.41	10.5827	0.0037	0.8832	0.7997	9.7951
$W_f$	-85.28518829	257.72	1.00	257.72	19.7042	0.0030			
$W_m$	6.874869015	69.00	1.00	69.00	5.2754	0.0552			
$W_f^2$	-0.895425	218.08	1.00	218.08	16.6736	0.0047			
$W_m^2$	-0.055477	0.84	1.00	0.84	0.0639	0.8076			
$W_f W_m$	0.968096	146.44	1.00	146.44	11.1962	0.0123			
<i>HDPE+D<sub>NaOH</sub></i>									
Model	-320.2481444	79.34	5.00	15.87	100.3878	< 0.0001	0.9882	0.9783	32.8212
$W_f$	12.2120255	68.49	1.00	68.49	433.2685	< 0.0001			
$W_m$	6.965838841	4.63	1.00	4.63	29.2956	0.0016			
$W_f^2$	-0.129086814	4.37	1.00	4.37	27.6170	0.0019			
$W_m^2$	-0.035939557	0.21	1.00	0.21	1.3440	0.2904			
$W_f W_m$	-0.10264	1.65	1.00	1.65	10.4137	0.0180			
<i>HDPE+D<sub>SLS</sub></i>									
Model	2165.472699	163.82	5.00	32.76	166.1578	< 0.0001	0.9928	0.9869	44.1510
$W_f$	59.88847923	25.42	1.00	25.42	128.9236	< 0.0001			
$W_m$	-49.3333226	36.31	1.00	36.31	184.1432	< 0.0001			
$W_f^2$	0.281777244	22.40	1.00	22.40	113.5727	< 0.0001			
$W_m^2$	0.282828268	13.16	1.00	13.16	66.7210	0.0002			
$W_f W_m$	-0.65256	66.54	1.00	66.54	337.4283	< 0.0001			

Note: *D* is the *C. populnea* fiber. Subscript NaOH and SLS are treatment.

From Table 5, the ANOVA of the quadratic response model for tensile modulus of untreated, NaOH and SLS treated *C. populnea* fiber/HDPE composites, respectively, are presented in Eq. (9)–(11) and significant since  $p < 0.05$ .

$$T_m = -43983 - 1632.4W_f + 1019.1W_m - 41.586W_f^2 - 5.8418W_m^2 + 21.093W_fW_m \quad (9)$$

$$T_m = -30149.4 + 520.714W_f + 632.59W_m - 3.4115W_f^2 - 3.2641W_m^2 - 4.8388W_fW_m \quad (10)$$

$$T_m = 10801.5 + 117.185W_f - 212.18W_m + 4.6255W_f^2 + 1.1543W_m^2 - 1.9334W_fW_m \quad (11)$$

The results of the experimental data for untreated, NaOH and SLS treated *C. populnea* fiber/HDPE composites, respectively, with  $R^2$  of 0.9301, 0.9677 and 0.9210 explains 93.01, 96.77 and 92.03 % of the observed variability in tensile modulus as a result of weight fraction of *C. populnea* fiber and HDPE matrix. It can be deduced that 6.99, 3.23 and 7.97 % represent residue of untreated, NaOH and SLS *C. populnea* fiber/HDPE composites, respectively, which cannot be explained but may be due to source of fiber, diameter of food gum fiber and other factors that did not considered for composites production. The change in observed variability of properties may be attributed to improve in intermolecular structure, interfacial adhesion bond between *C. populnea* fiber and HDPE, and void formation in the composites.

Table 5. ANOVA for response surface quadratic model of tensile modulus of food gum fiber-HDPE composites.

Source	Model Coefficient	Sum of Squares	DF	Mean Square	F Value	Prob > F	$R^2$	Adj $R^2$	Adeq Precision
<i>HDPE+D</i>									
Model	-43982.6271	648736.77	5.00	129747.35	18.6292	0.00064438	0.9301	0.8802	11.9374
$W_f$	-1632.39268	98571.02	1.00	98571.02	14.1529	0.0070561			
$W_m$	1019.070111	10649.40	1.00	10649.40	1.5290	0.25613			
$W_f^2$	-41.5862	469948.92	1.00	469948.92	67.4754	< 0.0001			
$W_m^2$	-5.8418	9273.54	1.00	9273.54	1.3315	0.28641			
$W_fW_m$	21.0932	69519.23	1.00	69519.23	9.9816	0.015942			
<i>HDPE+D<sub>NaOH</sub></i>									
Model	-30149.4026	38547.58	5.00	7709.52	35.9440	0.0002	0.9677	0.9408	18.8348
$W_f$	520.7136938	23024.91	1.00	23024.91	107.3489	< 0.0001			
$W_m$	632.5877681	6932.55	1.00	6932.55	32.3216	0.0013			
$W_f^2$	-3.41146129	2232.19	1.00	2232.19	10.4071	0.0180			
$W_m^2$	-3.26404624	2699.49	1.00	2699.49	12.5858	0.0121			
$W_fW_m$	-4.8388	3658.44	1.00	3658.44	17.0567	0.0061			
<i>HDPE+D<sub>SLS</sub></i>									
Model	10801.49498	9843.13	5.00	1968.63	11.6616	0.0087	0.9210	0.8420	13.0593
$W_f$	117.185134	5582.99	1.00	5582.99	33.0720	0.0022			
$W_m$	-212.18249	61.45	1.00	61.45	0.3640	0.5726			
$W_f^2$	4.625474909	3623.01	1.00	3623.01	21.4617	0.0057			
$W_m^2$	1.154254395	373.71	1.00	373.71	2.2138	0.1969			
$W_fW_m$	-1.93336191	201.97	1.00	201.97	1.1964	0.3239			

Note: *D* is the *C. populnea* fiber. Subscript NaOH and SLS are treatment.

The closeness of  $R^2$  and Adj.  $R^2$  indicated that the variables (weight fractions of fiber and HDPE matrix) were fit with tensile modulus. With adequacy precision of 11.9374, 18.8348 and 13.0593 (adequacy precision > 4), respectively, the quadratic model are adequate for tensile modulus response model for untreated, NaOH and SLS *C. populnea* fiber/HDPE composites and may be used for design applications. The model terms obtained for tensile modulus of HDPE composites with *C. populnea* fiber: untreated ( $W_f$ ,  $W_f^2$  and  $W_fW_m$ ), treated with NaOH ( $W_f$ ,  $W_m$ ,  $W_f^2$ ,  $W_m^2$  and  $W_fW_m$ ) and treated with SLS ( $W_f$  and  $W_f^2$ ) were significant since  $p < 0.05$ . This shows that the tensile modulus is a function of weight fraction of food gum fiber, quadratic of weight fraction of food gum fiber and /or interaction of weight fraction of food gum fiber and HDPE matrix.

The ANOVA of flexural strength and modulus response models for *C. populnea* fiber/HDPE composites are presented in Table 6 and 7, respectively. The Flexural strength and modulus response models, respectively, for untreated, NaOH and SLS treated *C. populnea* fiber/HDPE composites are represented by Eq. (12)–(14) and (15)–(17) were significantly fit and may be used to navigate design applications since  $p < 0.05$  with high value of observed variability and adequacy precision > 4.

$$F_s = 901.15 - 5.235W_f - 17.871W_m + 0.1812W_f^2 + 0.0925W_m^2 + 0.0373W_fW_m \quad (12)$$

$$F_s = -998.36 + 40.95.1881W_f + 19.696W_m - 0.4417W_f^2 - 0.0966W_m^2 - 0.3459W_fW_m \quad (13)$$

$$F_s = 2230.94 - 47.626W_f - 43.349W_m - 0.4391W_f^2 + 0.2121W_m^2 - 0.54738W_fW_m \quad (14)$$

$$F_m = 65229.45 - 506.02W_f - 1316.8W_m + 8.7132W_f^2 + 6.8014W_m^2 + 4.4110W_fW_m \quad (15)$$

$$F_m = 93094 - 2381.3W_f - 1768.2W_m + 37.793W_f^2 + 8.445W_m^2 + 22.358W_fW_m \quad (16)$$

$$F_m = 76811.62 - 1900.31W_f - 1466.73W_m - 3.4013W_f^2 + 7.0821W_m^2 + 20.251W_fW_m \quad (17)$$

From Table 6, the model terms obtained for flexural strength of HDPE composites with *C. populnea* fiber: untreated ( $W_f, W_m, W_f^2$  and  $W_m^2$ ), treated with NaOH ( $W_f, W_m, W_f^2, W_m^2$  and  $W_fW_m$ ) and treated with SLS ( $W_f, W_m, W_fW_m, W_f^2$  and  $W_m^2$ ) were significant since  $p < 0.05$ . The flexural modulus response models (Eq. (14)–(16)) for *C. populnea* fiber/HDPE composites were fit based on Table 7. The model terms of tensile modulus of HDPE composites with *C. populnea* fiber: untreated ( $W_m, W_f^2, W_m^2$  and  $W_fW_m$ ) treated with NaOH ( $W_f, W_m$  and  $W_f^2$ ) and treated with SLS ( $W_f, W_m, W_f^2, W_m^2$  and  $W_fW_m$ ) were significant since  $p < 0.05$ . This reveals the stress transfer between *C. populnea* fiber and matrix content. The ANOVA for hardness response models of the composites are presented in Table 8. More so, the hardness response models of untreated, NaOH and SLS treated *C. populnea* fiber/HDPE composites represented by Eq. (18)–(20), respectively, were significantly fit and may be used to navigate design applications since  $p < 0.05$  with high value of coefficient of determination ( $R^2$ ) and adequacy precision  $> 4$ . The model terms for hardness response models of HDPE composites with *C. populnea* fiber: untreated ( $W_f, W_m, W_f^2, W_m^2$  and  $W_fW_m$ ), treated with NaOH ( $W_f, W_f^2, W_m^2$  and  $W_fW_m$ ) and SLS treated ( $W_f, W_f^2, W_m^2$  and  $W_fW_m$ ) and EDTA ( $W_f$ ) were significant since  $p < 0.05$ .

$$H = 478.87 - 5.9762W_f - 8.9506W_m - 0.1357W_f^2 - 0.044W_m^2 + 0.08375W_fW_m \quad (18)$$

$$H = -1648.35 + 13.1921W_f + 34.6963W_m - 0.1424W_f^2 - 0.1792W_m^2 - 0.12W_fW_m \quad (19)$$

$$H = -2261.11 - 7.3843W_f + 48.7793W_m - 0.3W_f^2 - 0.26W_m^2 + 0.12W_fW_m \quad (20)$$

Table 6. ANOVA for response surface quadratic model of flexural strength of *C. populnea* fiber/HDPE composites.

Source	Model Coefficient	Sum of Squares	DF	Mean Square	F Value	Prob > F	R <sup>2</sup>	Adj R <sup>2</sup>	Adeq Precision
<i>HDPE+D</i>									
Model	901.1491	11.16	5.00	2.23	35.9956	0.0006	0.9730	0.9459	14.7039
$W_f$	-5.2351	2.82	1.00	2.82	45.5388	0.0011			
$W_m$	-17.8712	1.41	1.00	1.41	22.7379	0.0050			
$W_f^2$	0.1812	4.63	1.00	4.63	74.6885	0.0003			
$W_m^2$	0.0925	2.22	1.00	2.22	35.7976	0.0019			
$W_fW_m$	0.0373	0.08	1.00	0.08	1.2150	0.3206			
<i>HDPE+ D<sub>NaOH</sub></i>									
Model	-998.3605	275.84	5.00	55.17	3167.7452	< 0.0001	0.9997	0.9994	208.2062
$W_f$	40.9527	234.49	1.00	234.49	13464.4760	< 0.0001			
$W_m$	19.6964	26.09	1.00	26.09	1498.2602	< 0.0001			
$W_f^2$	-0.4417	6.94	1.00	6.94	398.5466	< 0.0001			
$W_m^2$	-0.0966	1.85	1.00	1.85	106.1917	0.0001			
$W_fW_m$	-0.3459	6.47	1.00	6.47	371.2514	< 0.0001			
<i>HDPE+ D<sub>SLS</sub></i>									
Model	2230.9365	73.67	5.00	14.73	176.1468	< 0.0001	0.9944	0.9887	43.5506
$W_f$	-47.6255	41.04	1.00	41.04	490.5901	< 0.0001			
$W_m$	-43.3492	1.41	1.00	1.41	16.8945	0.0093			
$W_f^2$	-0.4391	1.71	1.00	1.71	20.4687	0.0063			
$W_m^2$	0.2121	13.32	1.00	13.32	159.2828	< 0.0001			
$W_fW_m$	0.5473	16.19	1.00	16.19	193.4981	< 0.0001			

Note: D is the *C. populnea* fiber. Subscript NaOH and SLS are treatment.



Table 7. ANOVA for response surface quadratic models of flexural modulus of *C. populnea* fiber/HDPE composites.

Source	Model Coefficient	Sum of Squares	DF	Mean Square	F Value	Prob > F	R <sup>2</sup>	Adj R <sup>2</sup>	Adeq Precision
<i>HDPE+D</i>									
<i>Model</i>	65229.44721	32847.09	5.00	6569.42	157.4451	< 0.0001	0.9924	0.9861	28.5487
$W_f$	-506.01865	6.19	1.00	6.19	0.1484	0.7134			
$W_m$	-1316.79825	439.83	1.00	439.83	10.5411	0.0175			
$W_f^2$	8.713241943	16917.69	1.00	16917.69	405.4556	< 0.0001			
$W_m^2$	6.801241943	13659.26	1.00	13659.26	327.3629	< 0.0001			
$W_f W_m$	4.411032227	1824.11	1.00	1824.11	43.7174	0.0006			
<i>HDPE+D<sub>NaOH</sub></i>									
<i>Model</i>	93093.51165	1331686.87	5.00	266337.37	11.8917	0.0026	0.8947	0.8194	11.6147
$W_f$	-2381.32941	727804.09	1.00	727804.09	32.4958	0.0007			
$W_m$	-1768.155	134229.33	1.00	134229.33	5.9932	0.0442			
$W_f^2$	37.793	372164.49	1.00	372164.49	16.6168	0.0047			
$W_m^2$	8.445	19379.90	1.00	19379.90	0.8653	0.3832			
$W_f W_m$	22.3584	78109.07	1.00	78109.07	3.4875	0.1041			
<i>HDPE+D<sub>SLS</sub></i>									
<i>Model</i>	76811.61484	85437.83	5.00	17087.57	1728.4754	< 0.0001	0.9994	0.9988	131.5635
$W_f$	-1900.30994	38305.36	1.00	38305.36	3874.7400	< 0.0001			
$W_m$	-1466.73071	2523.86	1.00	2523.86	255.2983	< 0.0001			
$W_f^2$	-3.40130379	7352.60	1.00	7352.60	743.7452	< 0.0001			
$W_m^2$	7.082087633	15097.24	1.00	15097.24	1527.1457	< 0.0001			
$W_f W_m$	20.2510409	22158.77	1.00	22158.77	2241.4477	< 0.0001			

Note: *D* is the *C. populnea* fiber. Subscript NaOH and SLS are treatment.

Table 8. ANOVA for response surface quadratic models of hardness of *C. populnea* fiber/HDPE composites.

Source	Model Coefficient	Sum of Squares	DF	Mean Square	F Value	Prob > F	R <sup>2</sup>	Adj R <sup>2</sup>	Adeq Precision
<i>HDPE+D</i>									
<i>Model</i>	478.86618	19.55	5.00	3.91	223.5346	< 0.0001	0.9955	0.9911	51.9107
$W_f$	-5.976150274	14.77	1.00	14.77	844.7143	< 0.0001			
$W_m$	-8.950585969	0.75	1.00	0.75	43.1605	0.0012			
$W_f^2$	-0.135696867	2.67	1.00	2.67	152.9359	< 0.0001			
$W_m^2$	0.043981138	0.69	1.00	0.69	39.5540	0.0015			
$W_f W_m$	0.083753453	0.65	1.00	0.65	37.3083	0.0017			
<i>HDPE+D<sub>NaOH</sub></i>									
<i>Model</i>	-1648.350475	18.10	5.00	3.62	26.6334	0.0005	0.9569	0.9210	14.5944
$W_f$	13.1921152	3.52	1.00	3.52	25.9040	0.0022			
$W_m$	34.69632756	0.12	1.00	0.12	0.9196	0.3746			
$W_f^2$	-0.142426407	4.07	1.00	4.07	29.9363	0.0016			
$W_m^2$	-0.179191198	8.14	1.00	8.14	59.8542	0.0002			
$W_f W_m$	-0.12	2.25	1.00	2.25	16.5529	0.0066			
<i>HDPE+D<sub>SLS</sub></i>									
<i>Model</i>	-2261.110913	91.79	5.00	18.36	67.7198	< 0.0001	0.9797	0.965278	20.75319
$W_f$	-7.384314575	51.58	1.00	51.58	190.2641	< 0.0001			
$W_m$	48.77928932	0.02	1.00	0.02	0.0791	0.7866			
$W_f^2$	-0.3	19.57	1.00	19.57	72.1971	< 0.0001			
$W_m^2$	-0.26	18.37	1.00	18.37	67.7590	< 0.0001			
$W_f W_m$	0.12	2.25	1.00	2.25	8.2995	0.0236			

Note: *D* is the *C. populnea* fiber. Subscript NaOH and SLS are treatment.

Based on the ANOVA presented in Table 9, it can be observed that the impact strength response model of untreated, NaOH and SLS treated *C. populnea* fiber composites, respectively, represented by Eq. (21)–(23) were significant, adequate and may be used for design application since  $p < 0.05$  and adequacy

$precision > 4$ . The model variables for impact strength of HDPE composites with *C. populnea* fiber: untreated ( $W_m, W_f^2$  and  $W_m^2$ ), treated with NaOH ( $W_f, W_m, W_f^2, W_m^2$  and  $W_f W_m$ ) and SLS treated ( $W_f$  and  $W_f^2$ ) were significant since  $p < 0.05$ . This indicated that impact strength is a quadratic function of weight fraction of fiber and matrix. This is in support with the report of researchers that varying the weight fraction of fiber and matrix to improve the hardness and impact strength of the composites, although the quadratic of model terms were not considered with the interaction between the weight fraction of fiber and matrix [20, 21].

$$I_s = 19.855 - 0.07562W_f - 0.4092W_m + 0.00269W_f^2 + 0.00213W_m^2 + 0.000516W_fW_m \quad (21)$$

$$I_s = -86.3441 + 1.336W_f + 1.789W_m + 0.01097W_f^2 - 0.009198W_m^2 - 0.01534W_fW_m \quad (22)$$

$$I_s = 25.486 - 0.07303W_f - 0.5253W_m + 0.00675W_f^2 + 0.00274W_m^2 - 0.000389W_fW_m \quad (23)$$

Table 9. ANOVA for response surface quadratic model of impact strength of *C. populnea* fiber/HDPE composites.

Source	Model Coefficient	Sum of Squares	DF	Mean Square	F Value	Prob > F	R <sup>2</sup>	Adj R <sup>2</sup>	Adeq Precision
<i>HDPE+D</i>									
<i>Model</i>	19.85455059	0.00314	5.00	0.00063	61.9670	< 0.0001	0.9810	0.9652	17.0598
$W_f$	-0.075614856	0.00001	1.00	0.00001	0.7896	0.4084			
$W_m$	-0.409231257	0.00036	1.00	0.00036	35.6940	0.0010			
$W_f^2$	0.002685311	0.00151	1.00	0.00151	148.8512	< 0.0001			
$W_m^2$	0.002128111	0.00124	1.00	0.00124	122.0373	< 0.0001			
$W_fW_m$	0.000516116	0.00002	1.00	0.00002	2.4631	0.1676			
<i>HDPE+D<sub>NaOH</sub></i>									
<i>Model</i>	-86.34413235	0.17097	5.00	0.03419	53.3766	< 0.0001	0.9780	0.9597	23.3849
$W_f$	1.336032084	0.00641	1.00	0.00641	10.0040	0.0195			
$W_m$	1.788652563	0.08591	1.00	0.08591	134.0986	< 0.0001			
$W_f^2$	0.01097088	0.02798	1.00	0.02798	43.6821	0.0006			
$W_m^2$	-0.009198481	0.01392	1.00	0.01392	21.7232	0.0035			
$W_fW_m$	-0.015337499	0.03676	1.00	0.03676	57.3752	0.0003			
<i>HDPE+D<sub>SLS</sub></i>									
<i>Model</i>	25.48565746	0.06305	5.00	0.01261	31.7414	0.0001	0.9578	0.9276	16.4300
$W_f$	-0.073030568	0.04950	1.00	0.04950	124.5892	< 0.0001			
$W_m$	-0.52531018	0.00019	1.00	0.00019	0.4673	0.5162			
$W_f^2$	0.006751083	0.01130	1.00	0.01130	28.4391	0.0011			
$W_m^2$	0.002744403	0.00205	1.00	0.00205	5.1515	0.0575			
$W_fW_m$	0.000389301	0.00002	1.00	0.00002	0.0596	0.8141			

Note: *D* is the *C. populnea* fiber. Subscript NaOH and SLS are treatment.

The significant of the model terms especially, interaction shows that the effect of fiber treatments on composites. The optimal properties of *C. populnea* fiber/HDPE composites are presented in Table 10. It can be observed that untreated *C. populnea* fiber increased the tensile strength, tensile modulus, flexural strength, flexural modulus and hardness by 10.33, 5.88, 4.84, 0.98 and 2.17% of the HDPE matrix, respectively, with reduced impact strength by 84.27% of HDPE matrix. It can be observed that the model predicted values by RSM for mechanical properties are close to the experimental values based on the error generated. However, the choice of optimal values of mechanical properties for adequately fit model is based on high desirability as shown in Fig. 1. This shows that the incorporation of *C. populnea* fiber may be used to reinforce HDPE matrix with increased stiffness and SLS treatment improved the tensile strength, tensile modulus, flexural modulus, hardness and impact strength, respectively by 3.18, 7.51, 0.046, 41.38, 16.13 and 91.97% of untreated *C. populnea* fiber/HDPE composites based on the experimental data. Although, NaOH treated *C. populnea* fiber reinforced HDPE matrix but reduced the tensile strength of the composites when compared with untreated *C. populnea* fiber. It can also be observed that NaOH treated *C. populnea* fiber/HDPE composites improved the flexural strength than SLS treated *C. populnea* fiber. It can be deduced that SLS treatment of *C. populnea* fiber seems to be favourable than NaOH treatment for improving mechanical properties of the *C. populnea* fiber/HDPE composites.

Table 10. Optimal data of mechanical properties of *C. populnea* fiber/HDPE composites.

Composite Sample	HDPE +D	HDPE +D <sub>NaOH</sub>	HDPE +D <sub>SLS</sub>
$W_f$	2.5	7.5	6.13
$W_m$	92.5	92.5	97.5
$T_{sa}$	30.4827	29.6903	31.8013*
$T_{sp}$	30.4935	29.7067	31.7961
$T_{ma}$	839.022*	793.05	823.245
$T_{mp}$	834.364	793.511	822.979
$F_{sa}$	36.1904	39.3962	39.586*
$F_{sp}$	36.2274	39.4452	39.5926
$F_{ma}$	1425.89	1568.44*	1455.68
$F_{mp}$	1428.2	1573.73	1456.27
$H_a$	30	35	38*
$H_p$	30.8765	35.5345	38.4263
$I_a$	0.155795	0.396983*	0.394683
$I_p$	0.156407	0.398692	0.395543

Note:  $W_f$ ,  $W_m$ ,  $T_s$ ,  $T_m$ ,  $F_s$ ,  $F_m$ ,  $H$ , and  $I_s$ , are weight fraction of fiber, HDPE weight fraction, tensile strength, tensile modulus, Flexural strength, flexural modulus, hardness and impact strength, respectively. Subscript a and p indicate actual and predicted values. Superscript (\*) indicates maximum property based on experimental value.

Moreover, Table 11 reveals the change in interfacial shear strength IFSS of HDPE composites with untreated and treated *C. populnea* fiber. It can be observed that composites formed with NaOH and SLS treated *C. populnea* fiber increased the IFSS may be due to removal of amorphous constituents, hence reduced the diameter of the fiber, although composites formed with SLS treatment gave highest IFSS. This indicated that superior mechanical properties of SLS treated *C. populnea* fiber/HDPE composites may be attributed to improved IFSS between the fibers and HDPE matrix. Hence increased the interfacial adhesion region between fibers with polymer matrix in the composites, thereby, increases the compatibility of the fiber – polymer matrix. This is in agreement with the report of Herrera-Franco & Valadez-Gonzalez, (2005).

Table 11. Evaluated interfacial shear stress of fiber-HDPE composites.

Composite Sample	$F$ (N)	$d_f$ (mm)	IFSS (N/mm <sup>2</sup> )
HDPE + D	1282.00	0.110	3708.264
HDPE + D <sub>NaOH</sub>	2227.70	0.104	6815.516
HDPE + D <sub>SLS</sub>	2538.00	0.095	8500.478

Note: D is the *C. populnea* fiber. Subscript NaOH and SLS are treatments.

### 3.2. Microstructural Analysis

#### 3.2.1. SEM analysis

Figure 1 shows the morphology of *C. populnea* fiber/HDPE composites at fracture surface of tensile test. It reveals the uniform void formation in HDPE matrix. From Fig. 1(b), uneven distribution of the *C. populnea* fiber with pullout and *C. populnea* fiber debondings caused by poor interface adhesion between fibers and HDPE matrix was observed. This influenced the mechanical properties of the composites due to poor interaction between the fiber surface and HDPE matrix. However, better distribution of NaOH and SLS treated *C. populnea* fiber and fiber tearing with small void formation were observed in Figs. 1(c) and (d), respectively, compared with Fig. 1(b). The fiber tearing indicated improvement in interfacial adhesion between the *C. populnea* fiber and HDPE matrix caused by NaOH and SLS treatment. The failure of the composites by fiber tearing at the fracture surface indicated that fibers are not pulled out directly from the matrix and fibers are compatible with the matrix.

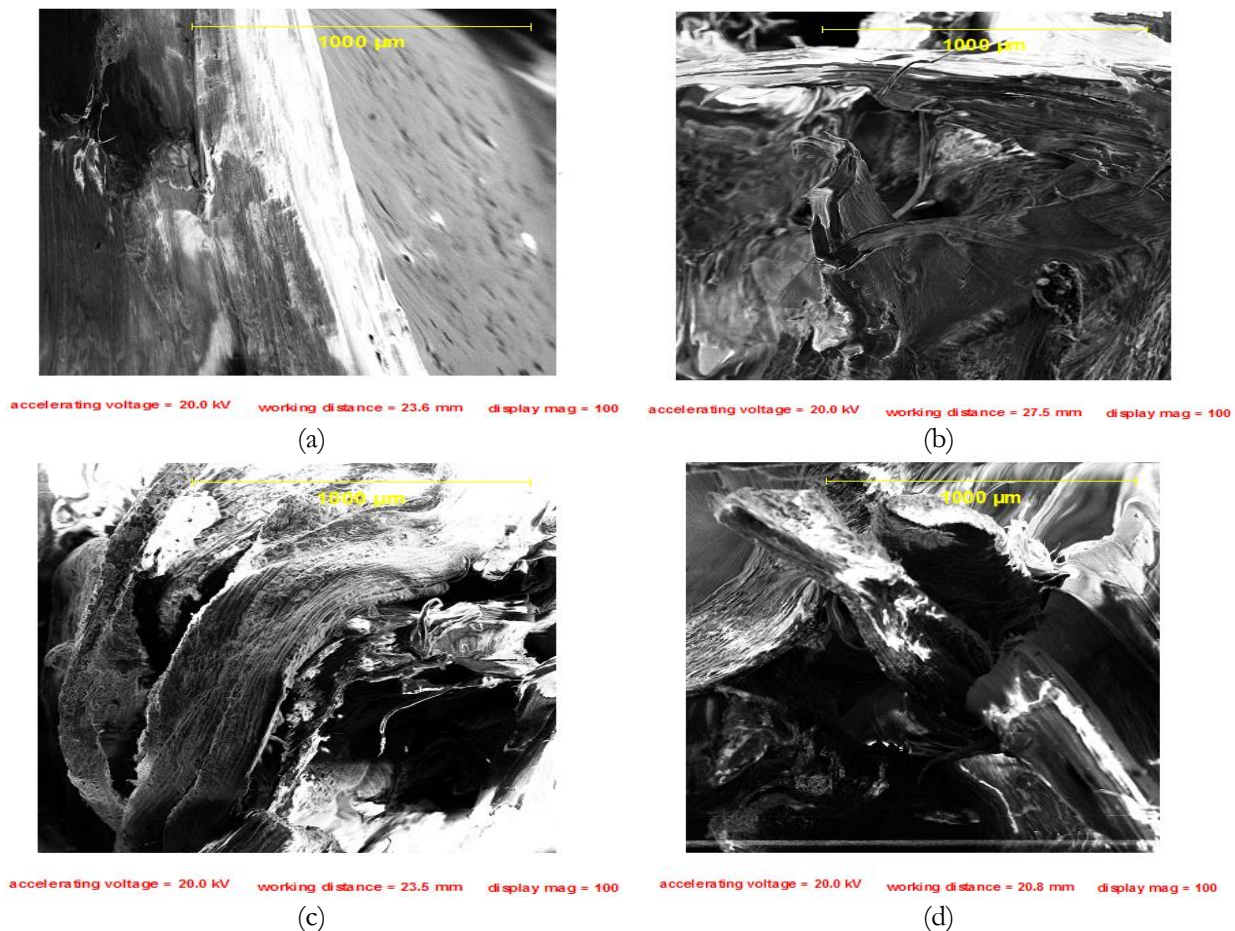
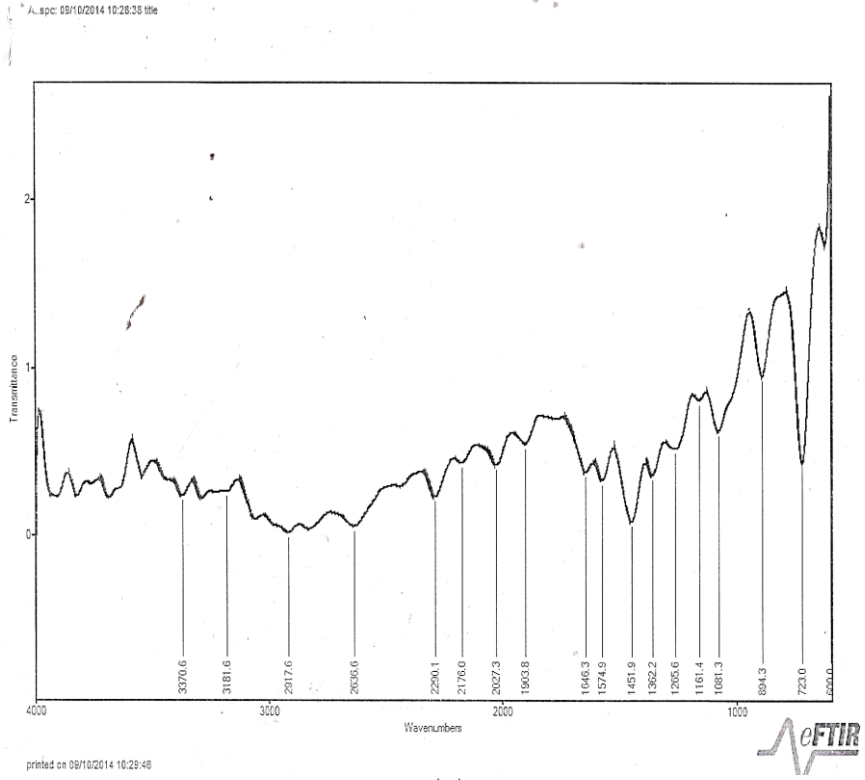


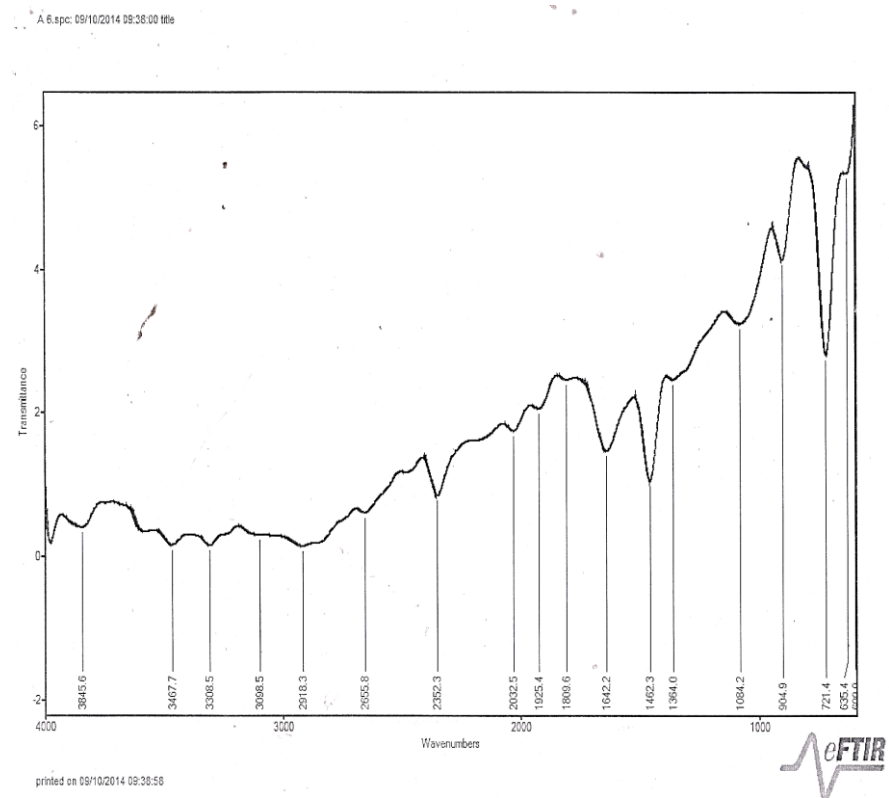
Fig. 1. SEM micrographs of food gum fiber-HDPE composites: (a) HDPE matrix; (b) untreated food gum fiber-HDPE; (c) NaOH treated food gum fiber-HDPE; and (d) SLS treated food gum fiber-HDPE.

### 3.2.2. FTIR

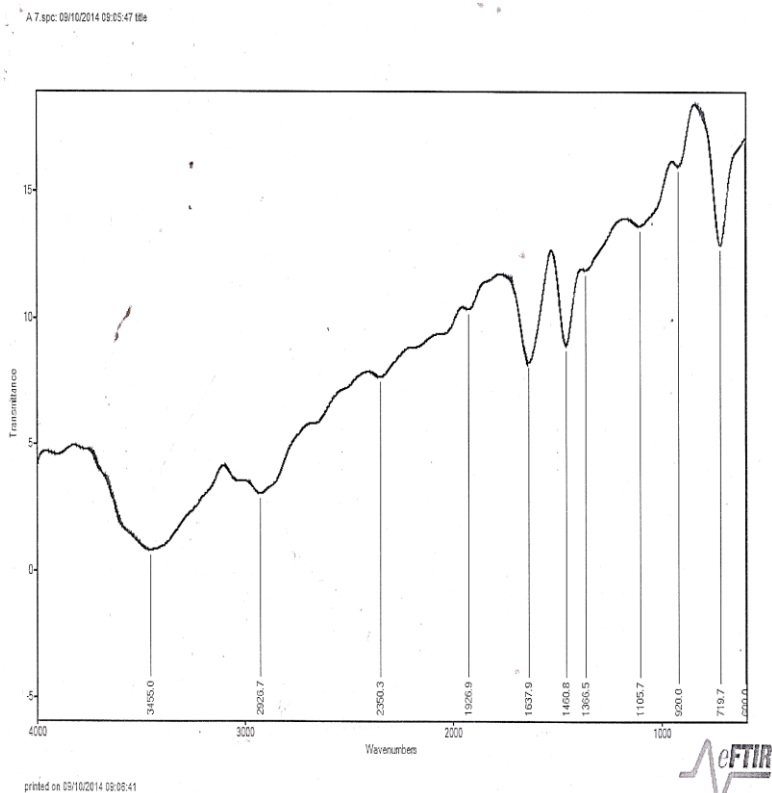
The FTIR spectra of recycled polyethylene matrix, untreated, NaOH and SLS treated *C. populnea* fiber/HDPE composites are presented in Figs. 2(a), (b), (c) and (d), respectively. The shift in absorbance peak from 1903.8 and 894.3  $\text{cm}^{-1}$ , respectively to 1925.4 and 902.3  $\text{cm}^{-1}$  which corroborates with (C = C - H) and (C - H) when HDPE reinforced with untreated *C. populnea* fiber as observed in Fig. 2(b). The absorption peak of 1817.4  $\text{cm}^{-1}$  corresponds to formation of (C = O) bonds between the fiber and matrix. It can be observed from Fig. 2(c) that treated *C. populnea* fiber with NaOH caused disappearance of (O - H) weak bonds in the composites. There is shift in absorption peaks of all the functional groups of the composites as shown in Fig. 2(d) compared with Fig. 2(b). The formation and shift in absorption peaks with change in intensity caused change in strengths, modulus and hardness of the composites.



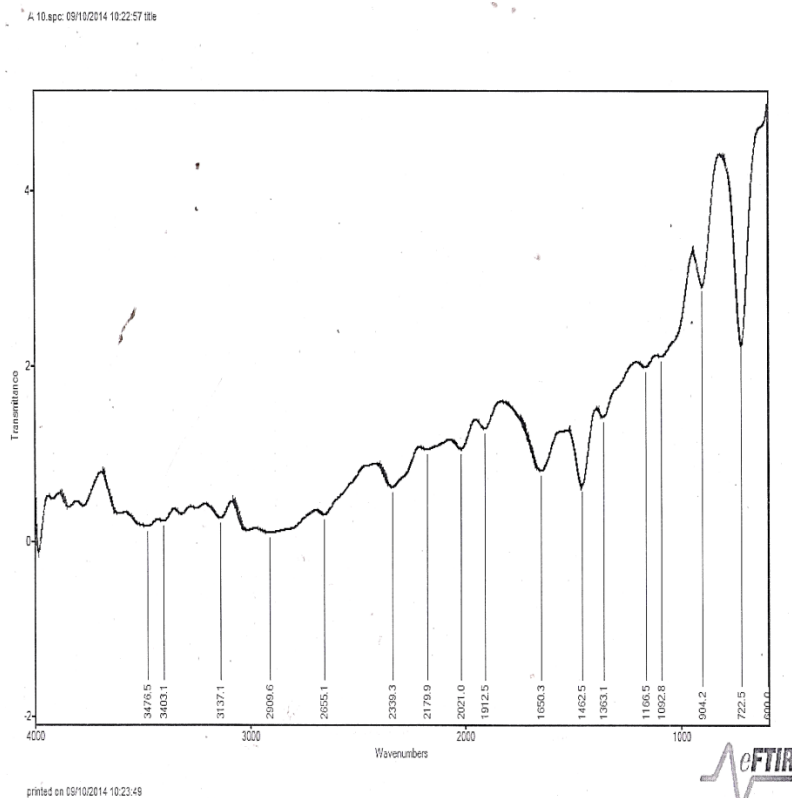
(a)



(b)



(c)



(d)

Fig. 2. FTIR spectra of *C. populnea* fiber/HDPE composites: (a) HDPE matrix; (b) untreated *C. populnea* fiber/HDPE; (c) NaOH treated *C. populnea* fiber/HDPE; and (d) SLS treated *C. populnea* fiber/HDPE.

### 3.3. Physical Properties of the Composites

#### 3.3.1. Density

Form Table 12, it can be observed that the density of untreated *C. populnea* fiber/HDPE composites lower than the density of HDPE matrix by 0.65%. This makes the *C. populnea* fiber/HDPE composites to be lighter than matrix. At optimum treatment conditions of *C. populnea* fiber with SLS, the density of *C. populnea* fiber/HDPE composites reduced by 1.1 % of untreated *C. populnea* fiber/HDPE composites which may be attributed to reduction in lignin, hemicellulose and other impurities as reported by Thiruchitrambalam [24]. NaOH treated *C. populnea* fiber /HDPE composites is higher by 10.2 % of untreated *C. populnea* fiber HDPE composites which may be due to substitution of sodium ion ( $\text{Na}^+$ ) with ( $\text{H}^+$ ) of the hydroxyl ions. This is in agreement with the report of Salim & Sorya [25].

#### 3.3.2. Water absorption

The raw data for water absorption of *C. populnea* fiber/HDPE composites with increased time are presented in Fig. 3. It can be observed that the maximum water absorption or saturation point of HDPE matrix, untreated, NaOH and SLS treated *C. populnea* fiber/HDPE composites, respectively, obtained with a value of 0.813, 3.659, 1.507 and 2.033 % at 30, 100, 60 and 80 mins. It can be deduced that fiber increased the maximum water absorption or saturation of the matrix, while NaOH and SLS treatment, respectively, reduced the absorption time of the composites. The water absorption of *C. populnea* fiber/HDPE composites is higher than HDPE matrix due to hydrophilic nature of the fibers. It can be observed that the modification of *C. populnea* fiber using NaOH and SLS, respectively, reduced the water absorption of *C. populnea* fiber/HDPE composites by 58.82 and 44.45% of *C. populnea* fiber/HDPE composites at saturation point which indicated good water resistance.

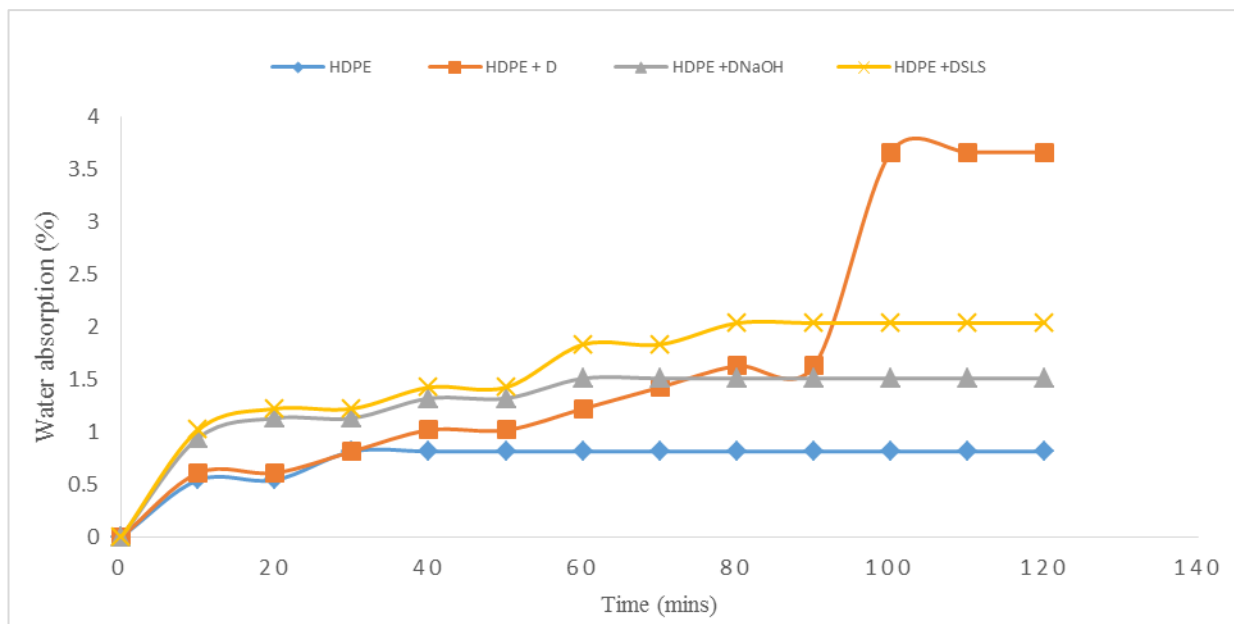


Fig. 3. Water absorption of fiber/composites.

Figure 4 reveals the kinetics of water absorption of *C. populnea* fiber/HDPE composites. It can be deduced that water diffusion behaviour of HDPE matrix is less Fickian since  $n < 0.5$ , which indicated that the water penetration rate is much below the polymer relaxation rate. The untreated *C. populnea* fiber/HDPE composites is non – Fickian diffusion behaviour since  $0.5 < n < 1$ . This is anomalous condition because water diffusion rate is comparable with polymer relaxation rate due to hydrophilic nature of fibers. Treatment of *C. populnea* fiber with NaOH and SLS, respectively, reduced the water absorption behaviour of *C. populnea* fiber/HDPE composites to less Fickian since  $n < 0.5$ . This implies that NaOH and SLS treatment improved hydrophobicity of the *C. populnea* fiber/HDPE composites. The water absorption

rate constant ( $k$ ) for HDPE, untreated, NaOH and SLS treated *C. populnea* fiber/HDPE composites are presented in Table 12. It can be observed that the value of  $k$  for untreated *C. populnea* fiber/HDPE composites is higher than HDPE matrix but reduced when treated with NaOH and SLS. The reduction in value of  $k$  may be attributed to shrinkage in fiber, dispersion, hemicellulose and lignin content. The value of  $R^2$  for HDPE, untreated, NaOH and SLS treated *C. populnea* fiber/HDPE composites, respectively are 0.6645, 0.7497, 0.9222 and 0.9273 which explains 66.45, 74.97, 92.22 and 92.73 % of observed variability in the water absorption with time. Table 12 also presented the value of  $S$  obtained from Fig. 5. It can be observed that the water diffusion coefficient of untreated *C. populnea* fiber/HDPE composites higher than HDPE matrix but reduced when NaOH and SLS treated *C. populnea* fiber, respectively, was used for composites. This reduction in diffusion coefficient using NaOH and SLS treatment of *C. populnea* fiber may be due to reduction in pores formation.

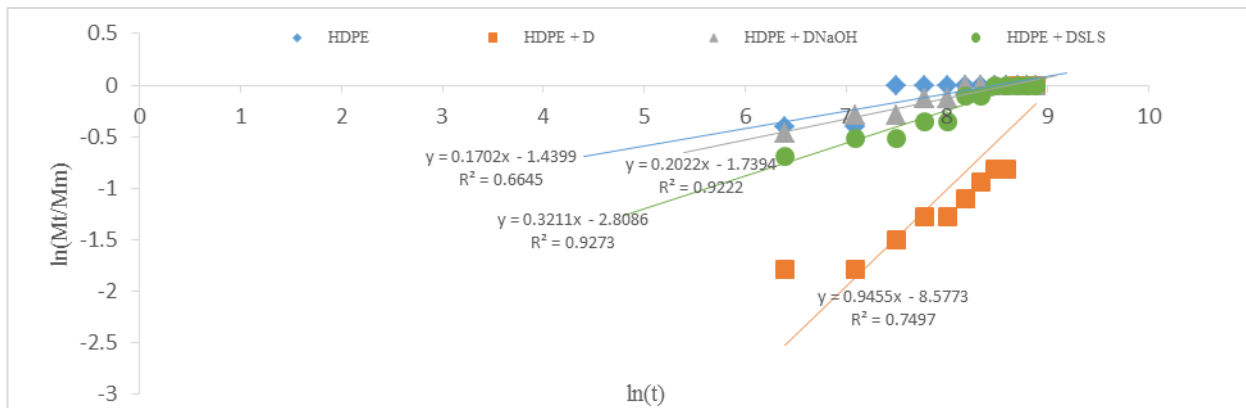


Fig. 4. Evaluation of water diffusion parameters  $n$  and  $k$  for *C. populnea* fiber/HDPE composites at room temperature.

Table 12. Density, water sorption and diffusivity of fiber-HDPE composites at optimal conditions.

Sample	$W_f$	$W_m$	$\rho$ (g/cm <sup>3</sup> )	$h$ (mm)	$n$ (s <sup>-1</sup> )	$k$	$M_m$ (%)	$S$ (s <sup>-1/2</sup> )	$D_{vc}$ (mm/s)
HDPE	0.0	100	0.9425	3.0	0.1702	0.237	0.813	0.0041	4.49418E-05
HDPE + D	7.5	97.5	0.9364	3.0	0.9455	0.0002	3.65854	0.0533	0.00037507
HDPE + D <sub>NaOH</sub>	7.5	92.5	1.032	3.0	0.2022	0.1756	1.50659	0.0096	7.17503E-05
HDPE + D <sub>SLS</sub>	2.5	97.5	0.9261	3.0	0.3211	0.0603	2.03252	0.0195	0.000162657

Note:  $D$  is the *C. populnea* fiber,  $h$  is the thickness of the sample, subscript NaOH and SLS are treatments.

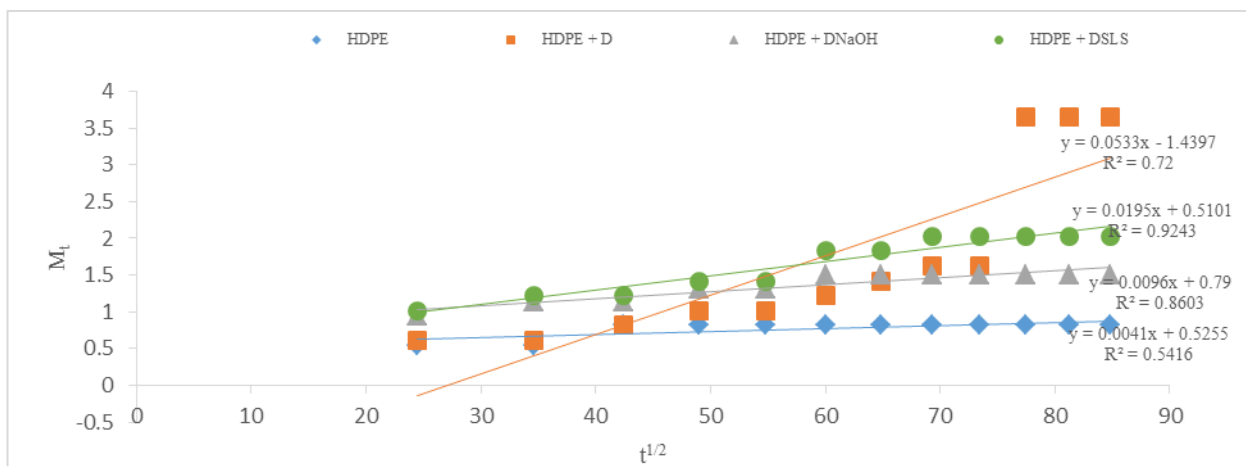


Fig. 5. Estimation of  $S$  (min<sup>-1/2</sup>) for untreated and treated food gum fiber-HDPE composites.



#### 4. Conclusion

The present study has shown that recycled HDPE composites using *C. populnea* fiber can be produced with improved properties such as tensile strength, high stiffness, flexural strength, flexural modulus, density and water absorption properties. The following conclusions could be drawn from the results of the present study:

- (1) The reinforcement, stiffness, flexural strength, flexural modulus and hardness of *C. populnea* fiber/HDPE composite improved with SLS treated fiber and superior over NaOH treated fiber. NaOH *C. populnea* fiber may not be used for reinforced HDPE in composites application but better for bending design applications with high flexural and impact strengths.
- (2) The superiority of SLS treatment for improvement in mechanical properties of fiber may be attributed to the increased interfacial adhesion as observed in SEM micrographs and FTIR spectra.
- (3) By lowering overall weight of the composites, the use of *C. populnea* fiber may reduce domestic vehicle fuel consumption. In addition to removal of impurities, SLS treatment for *C. populnea* fiber surface modification has been shown to provide good HDPE matrix interfacial adhesion, hence provide good compatibility between *C. populnea* fiber and HDPE.
- (4) The water absorption at saturation of the matrix increased markedly by *C. populnea* fiber in the composites. NaOH and SLS treatment, respectively, reduced the water absorption and controlled by water penetration rate. NaOH treated *C. populnea* fiber/HDPE composites showed better water absorption compared to SLS treated *C. populnea* fiber/HDPE composites and untreated *C. populnea* fiber/HDPE composites.

#### Competing Interests

The authors declare that they have no competing interests regarding the publication of this paper.

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