

Enhancement of the Mechanical Properties and Dimensional Stability of Oil Palm Empty Fruit Bunch-Kenaf Core and Oil Palm Mesocarp-Kenaf Core Hybrid Fiber-Reinforced Poly(lactic acid) Biocomposites by Borax Decahydrate Modification of Fibers

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The surfaces of kenaf core fiber (KCF), oil palm empty fruit bunch fiber (EFBF), and oil palm mesocarp fiber (OPMF), were chemically modified using 5 wt.% aqueous sodium tetraborate decahydrate (borax) solution to enhance their hybrid fiber interface bonding with a polylactic acid (PLA) matrix. The untreated fibers (KCF, EFBF, and OPMF) and treated fibers (BXKCF, BXEFBF, and BXOPMF), were examined using chemical analysis, Fourier transform infrared (FTIR) spectroscopy, and scanning electron microscopy (SEM). The treatment caused minimal removal of lignin and significant elimination of hemicellulose and waxy substances. The treated and untreated KCF (5%), as a secondary fiber, was randomly mixed, respectively, with treated and untreated EFBF and OPMF (55%), melt-blended with PLA (40%), and subsequently compression-molded to form hybrid fiber-PLA biocomposites. The resulting composite is aimed to exhibit improvements in its mechanical properties and dimensional stability. The optimum results for tensile and flexural properties, as well as water uptake and thickness swelling, were observed for the borax-treated fibers in comparison with the untreated fibers. The BXEFBF-BXKCF-PLA biocomposites exhibited the best results. This work demonstrated that aqueous borax modification of natural fibers could offer a possible option to the most common mercerization method.

Keywords: Borax decahydrate; Oil palm empty fruit bunch fiber; Oil palm mesocarp fiber; Kenaf core fiber; Poly(lactic acid); Hybrid biocomposite

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INTRODUCTION

Natural fibers have attained a key position in the area of polymer composite technology, being used in a wide variety of applications, ranging from relatively low strength (e.g., packaging materials) (Leão *et al.* 2008; Then *et al.* 2013) to relatively high strength (e.g., automobiles) (Deka *et al.* 2013; Jawaid *et al.* 2015). This is largely attributed to their attractive properties, such as abundance, eco-friendliness, environmentally-benign, renewability, low density profile, *etc.* (Eichhorn *et al.* 2001; Kim and Kim 2005; Mukherjee and Kao 2011; Hashim *et al.* 2015). Both hardwood- and softwood-based

natural fibers have been reported, including banana (Thiruchitrabalam *et al.* 2009), pineapple (Arib *et al.* 2006; Shih *et al.* 2014), hemp (Aziz and Ansell 2004), sisal (Jacob *et al.* 2004), kenaf (Ibrahim *et al.* 2010; Fiore *et al.* 2014), oil palm (Khalil *et al.* 2010a; Eng *et al.* 2014; Then *et al.* 2014a, 2015), and bamboo (Li 2004; Okubo *et al.* 2005).

Current research in the area of polymer composites has experienced increasing attention in the fabrication of natural fiber-based hybrid systems aiming to achieve synergistic performance, complement material properties, and a sustainable supply chain of fiber resources (Jacob *et al.* 2004; Ghasemi *et al.* 2008; Thiruchitrabalam *et al.* 2009; Jawaid *et al.* 2010; Othman *et al.* 2016). Recently, the hybridization of cellulose-rich oil palm fibers (OPFs) and lignin-rich kenaf core fiber (KCF) into the poly(lactic acid) PLA matrix has been reported by our research group, presenting enhanced mechanical properties and dimensional stability relative to single fiber biocomposites (Birnin-Yauri *et al.* 2016)

However, optimum synergism in material properties of hybrid natural fiber-based thermoplastic composites is realizable with modification of the natural fibers (Thiruchitrabalam *et al.* 2009). This is because, even though the natural fibers possess green attributes, the major bottleneck associated with their use as a reinforcement in polymer composites is their relatively high polarity, which hampers their bonding ability with non-polar polymer matrixes (Okubo *et al.* 2005; Then *et al.* 2014a). To enhance the bonding capacity of natural fibers with the non-polar polymer matrix, many attempts have been reported, such as chemical mercerization (Aziz and Ansell 2004; Liu *et al.* 2004; Fiore *et al.* 2014; Then *et al.* 2015), physical applications (corona and plasma) (Faruk *et al.* 2012), and biological enzymatic (Kalia *et al.* 2013) approaches.

The mercerization method has been widely accepted as an efficient chemical method of fiber modification (Sreekala *et al.* 1997; Razak and Kalam 2012; Atiqah *et al.* 2014; Fiore *et al.* 2014); however, the major bottleneck associated with the use of sodium hydroxide is its corrosive nature (Then *et al.* 2014a). To find an alternative chemical method that is less harmful, previous work has reported the use of boric acid and borax mixtures for the modification of lignocellulosic fiber (Widiarto 2005; Baysal *et al.* 2007; Özalp 2010). Borax has a long history of application in cellulosic products and coatings since the 1980s, and it is an attractive additive because it is relatively non-toxic, eco-friendly, and possesses a high thermal and biological resistance. A recent study reported the effect of boric acid and borax on the mechanical, fire, and thermal properties of wood flour-high density polyethylene composites and observed that the borax-impregnated samples exhibited better mechanical properties and a 50% decrease in the burning speed of the composite (Cavdar *et al.* 2015).

Most of the previous research focused on the use of borax as a filler or in the impregnated form. However, borax decahydrate is hygroscopic boron salt, and its application as a filler or impregnated state can adversely affect the dimensional stability and mechanical strength of composites, possibly, due to high concentration of borax in the composites (Baysal *et al.* 2007). Therefore, a novel approach involving fiber soaked in aqueous borax solution, followed by a water-washing procedure, will be the focus of this study. This study is built from our previous work, which demonstrated that the incorporation of KCF into EFBF and OPMF provided a positive hybridization effect (Birnin-Yauri *et al.* 2016). Unlike our previous work, the present study aims to investigate the treatment of EFBF, OPMF, and KCF using an aqueous borax solution, with the objective of achieving enhanced synergistic performance regarding mechanical properties and dimensional stability.

EXPERIMENTAL

Materials

Poly(lactic acid) was purchased from NatureWorks LLC (Minnetonka, MN) under the trade name, polylactide resin 3052D. The melting point ranged between 170 and 190 °C, and the density ranged from 1.4 to 1.5 gcm⁻³. The oil palm fibers, *i.e.*, EFBF and OPMF, were obtained from Sabutek (M) Sdn. Bhd. (Malaysia), and FELDA Serting Hilir Oil Palm mill (Malaysia), respectively. The KCF was supplied by the Lembaga Kenaf dan Tembakau (Malaysia). The sodium tetraborate decahydrate (borax) was purchased from R&M Chemicals (Malaysia).

Methods

Fiber purification

To remove impurities from the EFBF, OPMF, and KCF, they were physically treated by sorting and soaking in distilled water for 24 h at 25 °C, washed with heated water at 60 °C, cleaned with acetone (analytical grade, 99.5% purity), and oven-dried at 60 °C for 24 h. The oven-dried fibers were then ground into smaller particles, followed by sieving into sizes ranging from 300 to 400 µm. The purified fibers were then stored in sealed plastic bags at 25 °C for subsequent investigations.

Modification of natural fibers using aqueous borax solutions

The treatment was performed in accordance with the previous method (Cavdar *et al.* 2015), followed by some modifications involving a water-washing procedure in order to reduce borax concentration in the modified natural fibers. The purified natural fibers, EFBF, OPMF, and KCF, were chemically treated by soaking in an aqueous borax solution (5 wt. % concentration). The pH of the solution was 9.1. The treatment was performed at 25 °C for 24 h. Subsequently, the treated fibers were thoroughly washed with distilled water with pH monitoring intermittently until a neutral pH was obtained. Thereafter, the treated fibers were oven-dried at 60 °C for 24 h. The dried fibers were then stored in sealed plastic bags at 25 °C for subsequent investigations.

Determination of chemical components of treated and untreated fibers

To determine the holocellulose content, *i.e.*, the cellulose and hemicellulose contents, the borax-treated and untreated EFBF, OPMF, and KCF were reacted with acidified aqueous sodium chlorite (NaClO₂) solution to delignify the fibers, as previously reported in the literature (Abe *et al.* 2007; Then *et al.* 2015; Yue *et al.* 2015). The NaClO₂ solution was acidified with a H₂SO₄ solution until a pH of 4 was reached. The fibers were soaked in 5 wt. % NaClO₂ solution at 70 °C for 1 h, at a weight ratio of 1:20 fiber-to-NaClO₂ solution. Once the treatment was complete, the fiber was filtered and washed thoroughly with distilled water and oven-dried at 60 °C until a constant weight was obtained. The cellulose content was determined by treating the holocellulose with 6 wt. % potassium hydroxide solution at 25 °C for 24 h. The mixture was filtered, and the solid residue obtained was washed thoroughly with distilled water and oven-dried at 60 °C until a constant weight was obtained. Afterwards, the difference between the holocellulose and cellulose contents represented the hemicellulose content of the fibers.

The lignin content was determined by submerging the borax-treated and untreated EFBF, OPMF, and KCF, respectively, in 72 wt. % H₂SO₄ at 30 °C for 1 h. Then, the solution was diluted to 3% H₂SO₄ and refluxed for 2 h. The mixture was filtered and the

insoluble solid residue obtained was washed thoroughly with distilled water and oven-dried at 60 °C until a constant weight was obtained.

Determination of the moisture and ash contents

The moisture content was determined by placing weighed amounts of EFBF, OPMF, and KCF (both untreated and treated) in an oven at 105 °C for 4 h, respectively. Thereafter, the weights of the oven-dried samples were recorded. The moisture content was calculated by obtaining the difference between the weight of the fibers before and after oven-drying.

To determine the ash content, weighed amounts of EFBF, OPMF, and KCF (both untreated and treated) were heated in a furnace at 600 °C for 2 h, respectively. Afterward, the fibers were cooled in a desiccator and re-weighed. The ash content was calculated as the difference between the weight of the fibers before and after furnace heating.

Fabrication of hybrid fibers-PLA biocomposites

To prepare the hybrid fiber-PLA biocomposites, oven-dried untreated kenaf core fiber (KCF), as a secondary fiber, was randomly mixed manually with untreated empty fruit bunch fiber (EFBF) and oil palm mesocarp fiber (OPMF), at a hybrid-fiber weight ratio of 5 wt.% (KCF), 55 wt.% (EFBF, or OPMF), respectively, together with 40 wt.% (PLA). Similarly, oven-dried borax-treated kenaf core fiber (BXKCF), as a secondary fiber, was randomly mixed manually with borax-treated empty fruit bunch fiber (BXEFBF) and oil palm mesocarp fiber (BXOPMF), at a hybrid-fiber weight ratio of 5 wt.% (BXKCF), 55 wt.% (BXEFBF, or BXOPMF), respectively, together with 40 wt.% (PLA). The manually mixed hybrid fibers (Table 1) were melt-blended with PLA using a Brabender Internal Mixer (Germany) at 170 °C, with a 50 rpm rotor speed for 15 min, in accordance with the method reported by Then *et al.* (2015). Initially, PLA pellets were loaded into the mixing chamber to melt for 2 min, and then a weighed amount of hybrid fibers were subsequently added into the molten PLA and the mixed for 13 additional minutes. The compounded hybrid fiber-PLA biocomposites were further compression molded into sheets with the dimensions 1 mm × 150 mm × 150 mm and 3 mm × 150 mm × 150 mm (thickness × length × width). The compression molding was performed using a hydraulic hot press under the following conditions: 170 °C, 150 kgf^m-², and 10 min. Lastly, cooling was performed at 30 °C for 5 min.

Table 1. Treated and Untreated Hybrid Fiber Formulations

Designation	Hybrid fiber formulations			Borax concentration (wt. %)
	EFBF (wt. %)	OPMF (wt. %)	KCF (wt. %)	
EFBF-KCF	55	-	5	0
OPMF-KCF	-	55	5	0
BXEFBF-BXKCF	55	-	5	5
BXOPMF-BXKCF	-	55	5	5

EFBF-KCF = untreated hybrid empty fruit bunch fiber and kenaf core fiber

OPMF-KCF = untreated hybrid oil palm mesocarp fiber and kenaf core fiber

BXEFBF-BXKCF = borax treated hybrid empty fruit bunch fiber and kenaf core fiber

BXOPMF- BXKCF = borax treated hybrid oil palm mesocarp fiber and kenaf core fiber

Mechanical properties analysis

The tensile properties of treated and untreated hybrid fiber PLA biocomposites were tested using a universal testing machine (Model 3365, Instron Corp., Norwood, MA), equipped with a 5-kN load cell, 5 mm/min crosshead speed, and 25 °C processing temperature. Five dog-bone shaped specimens were tested as specified by the ASTM D638-5 (2000) testing standard. Average values and standard deviation were reported for the tensile strength (TS), tensile modulus (TM), and elongation at break (EB) tests.

The flexural properties (three-point testing) of the biocomposites were performed using a universal testing machine (Model 3365, Instron Corp., Norwood, MA), equipped with a 5-kN load cell, 1.3 mm/min crosshead speed, and 48 mm span length. The test was performed at 25 °C on five specimens with the dimensions 127.0 mm × 12.7 mm × 3.0 mm, in accordance with the ASTM D790 (2000) testing standard. Average values and standard deviation were reported for the flexural strength (FS) and flexural modulus (FM) tests.

The impact strength (IS) of the composites was examined following the un-notched IZOD impact test, as specified by ASTM D256 (2000) testing standard. The impact tester (Izod, Computerized, International Equipments, India) was equipped with a 7.5-J pendulum. The test was performed at 25 °C on five specimens with the dimensions, 63.5 mm × 12.7 mm × 3.0 mm, and the average values and standard deviations were reported.

Density measurement

The density of the biocomposites was measured in accordance with the BS EN 323 (1993) testing standard, as reported by the European Committee for Standardization (Khalil *et al.* 2010b). The mass of the test samples having dimension *i.e.* 1 mm × 150 mm × 150, were obtained using an analytical balance. Afterwards, the volume of the test samples was determined using their dimensions, *i.e.*, multiplying their length, width, and thickness, respectively. The density was subsequently calculated using equation 1. Five tests were conducted and average values and standard deviations were reported.

$$\text{Density} = \text{Mass/Volume} \quad (1)$$

Dimensional stability measurement

To test for water uptake and thickness swelling of the biocomposites, test samples with the dimensions, 10.0 mm × 10.0 mm × 1.0 mm, were cut according to the ASTM D570 (2005) and EN 317 (1993) testing standards, respectively. The initial weight (W_1) and thickness (T_1) of the oven-dried test samples were measured and recorded. Test samples were then immersed in distilled water for 24 h at 25 °C. Thereafter, the samples were towel dried and measured for their second weight (W_2) and thickness (T_2). Five specimens for each sample were tested to determine the mean and standard deviation. The water uptake and thickness swelling were calculated using the following equations:

$$\text{Water Uptake (\%)} = \frac{W_2 - W_1}{W_1} \times 100 \quad (2)$$

$$\text{Thickness Swelling (\%)} = \frac{T_2 - T_1}{T_1} \times 100 \quad (3)$$

Scanning electron microscopy (SEM)

The surface morphology of the treated and untreated fibers, as well as the fractured surfaces of the various hybrid-fiber PLA biocomposites, was analyzed using scanning electron microscopy (SEM). The instrument used to conduct the test was a LEO 1455 VP scanning electron microscope (Zeiss, Jena, Germany) operated at a 10 kV accelerating voltage. The metal holder of the instrument was used to hold the oven-dried samples in place. Thereafter, a gold coating was applied on the samples for 3 min using a Bio-Rad™ coating system (Hercules, CA) to enhance the conductivity before the commencement of the analysis.

Fourier transform infrared (FTIR) spectroscopy

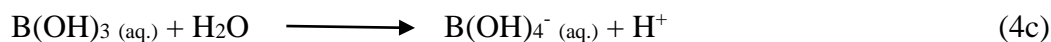
The chemical properties, *i.e.*, the functional groups, the bonding type, and the chemical components of both the treated (BXEFBF, BXOPMF, and BXKCF) and untreated fibers (EFBF, OPMF, and KCF), were analyzed using a Perkin Elmer Spectrum 100 series spectrophotometer (Waltham, MA). The instrument was equipped with an attenuated total reflectance (ATR) capacity. The wave number ranged from 400 to 4000 cm^{-1} and was employed to record the FTIR spectra.

RESULTS AND DISCUSSION

The effect of the borax decahydrate treatment of KCF, EFBF, and OPMF, at a 5 wt.% modifier concentration, on the dimensional stability (water uptake and thickness swelling), mechanical properties (tensile and flexural), impact strength, and density of their hybrid fiber PLA biocomposites, was investigated.

Characterizations of Fibers*Modification of fibers*

The hypothetical mechanism for the modification of the fibers (EFBF, OPMF, and KCF) is depicted in Eqs. 4a through 4d. First, the sodium tetraborate decahydrate (borax) solution was dissolved in water to form borate ions. Then, the borate ions reacted with the hydroxyl groups on the fiber's surface.

*Determination of chemical components of treated and untreated fibers*

The contents of cellulose, hemicellulose, lignin, moisture, and ash for untreated and treated EFBF, OPMF, and KCF are presented in Table 2. It was observed that the cellulose content of untreated EFBF, OPMF, and KCF (initially 39.50%, 33.75%, and 19.82%, respectively) increased after the borax treatment (62.18%, 61.00%, and 32.16%, respectively). This represented a total increase of 57.42%, 80.74%, and 62.26% for the EFBF, OPMF, and KCF, respectively. The EFBF and OPMF exhibited the highest

cellulose content, while the KCF was the lowest. The cellulose content of borax-treated EFBF(BXEFBF) and borax-treated OPMF (BXOPMF) agreed with the contents reported in the previous literature (Sreekala *et al.* 1997; Law *et al.* 2007). Similarly, the cellulose content of borax-treated KCF(BXKCF) was in agreement with Aisyah *et al.* (2013).

On the other hand, the hemicellulose content of untreated EFBF, OPMF, and KCF (initially, 34.87%, 30.40%, and 37.10%) tended to decline after the borax treatment, (17.30%, 15.42%, 26.65%, respectively). This finding was in agreement with Yue *et al.* (2015). The removal of hemicellulose with the borax treatment was validated by FTIR analysis. The lignin concentration also partially decreased after the fibers were treated with 5 wt. % aqueous borax. The minimal removal of lignin after treatment indicates that the aqueous borax can slightly delignify the fibers at 25 °C. Complete delignification of natural fiber is often achieved at higher temperatures, using different modifiers (Hubbell and Ragauskas 2010).

Table 2. Chemical Components of Treated and Untreated Fibers

Sample	Cellulose (%)	Hemicellulose (%)	Lignin (%)	Moisture (%)	Ash (%)
EFBF	39.50 ± 0.31	34.87 ± 0.42	14.32 ± 0.38	8.19 ± 0.28	3.12 ± 0.69
OPMF	33.75 ± 0.33	30.40 ± 0.35	22.93 ± 0.53	8.14 ± 0.08	4.78 ± 0.17
KCF	19.82 ± 0.17	37.10 ± 0.60	33.15 ± 0.50	8.86 ± 0.47	1.07 ± 0.53
BXEFBF	62.18 ± 1.70	17.30 ± 0.15	10.13 ± 0.81	9.38 ± 0.29	1.01 ± 0.23
BXOPMF	61.00 ± 0.63	15.42 ± 0.45	12.33 ± 0.39	10.67 ± 1.09	0.58 ± 0.24
BXKCF	32.16 ± 1.02	26.65 ± 0.96	30.79 ± 0.18	9.96 ± 0.16	0.44 ± 0.05

'±' refers to standard deviation.

EFBF = untreated empty fruit bunch fiber, OPMF = untreated oil palm mesocarp fiber

KCF = untreated kenaf core fiber, BXEFBF = borax treated empty fruit bunch fiber

BXOPMF = borax treated oil palm mesocarp fiber, BXKCF = borax treated kenaf core fiber

The partial removal of lignin after the borax treatment tended to increase the moisture content of the fibers. The low ash content in the borax-treated fibers compared to the untreated fibers was indicative of the significant removal of hemicellulose and partial decrease in lignin content, which undergo thermal decomposition to yield ash. Cellulose rich EFBF and OPMF are more susceptible to thermal degradation, thereby yielding high ash content relative to the lignin-rich KCF, which is more resistant to thermal decomposition. Then *et al.* (2015) stated that although cellulose and hemicellulose decompose at 550 °C, their decomposition begins within the range of 160 to 400 °C.

Fourier transform infrared spectra analysis of untreated and treated fibers

The FTIR spectra of both untreated and treated EFBF, OPMF, and KCF are given in Fig. 1. Fourier transform infrared spectroscopy was employed to study the different functional groups, chemical composition, and the bonding present in both untreated and borax-treated fibers.

The cellulose and hemicellulose O-H stretching appeared as broad bands at 3335, 3344, and 3342 cm⁻¹ for KCF, OPMF, and EFBF, respectively. These peaks slightly shifted to lower intensities, *i.e.*, 3327, 3327, and 3326 cm⁻¹ for BXKCF, BXOPMF, and BXEFBF, respectively, thereby implying the removal of hemicellulose because of the borax treatment. The peaks at 2899, 2918, and 2903 cm⁻¹ for KCF, OPMF, and EFBF decreased slightly to lower intensities of 2889, 2890, and 2891 cm⁻¹, for BXKCF, BXOPMF, and

BXEFBF, respectively. These peaks are related to the C-H stretching of cellulose and hemicellulose. The shifting to lower absorption bands after the borax treatment can be ascribed to the hemicellulosic group elimination from the fiber's surface. This concurred with previous observations (Sgriccia *et al.* 2008).

The absorption bands appeared at 1725, 1718, and 1721 cm^{-1} in the spectra of KCF, OPMF, and EFBF, respectively, but disappeared in the spectra of BXKCF, BXOPMF, and BXEFBF. These bands were attributed to the C=O stretching of carbonyl groups of hemicellulose or lignin. The disappearance of these peaks in the borax-treated fibers explained the considerable removal of hemicellulose and the partial removal of lignin from the fibers (Then *et al.* 2015; Yue *et al.* 2015). The aromatic C=C stretching in lignin in the spectra for KCF, OPMF, and EFBF appeared at the absorption bands 1614, 1619, and 1610 cm^{-1} , respectively. These peaks were reduced in the spectra of BXKCF, BXOPMF, and disappeared in the spectrum of BXEFBF. The peak intensity diminution can be ascribed to the partial removal of lignin in KCF and OPMF because of the borax treatment, while the disappearance could be attributed to the more significant elimination of the lignin in EFBF relative to that of KCF and OPMF. The KCF and OPMF recorded the highest lignin contents, which was greater than EFBF (Table 2). Thus, the extent of its removal using the borax treatment could perhaps be more pronounced in the EFBF.

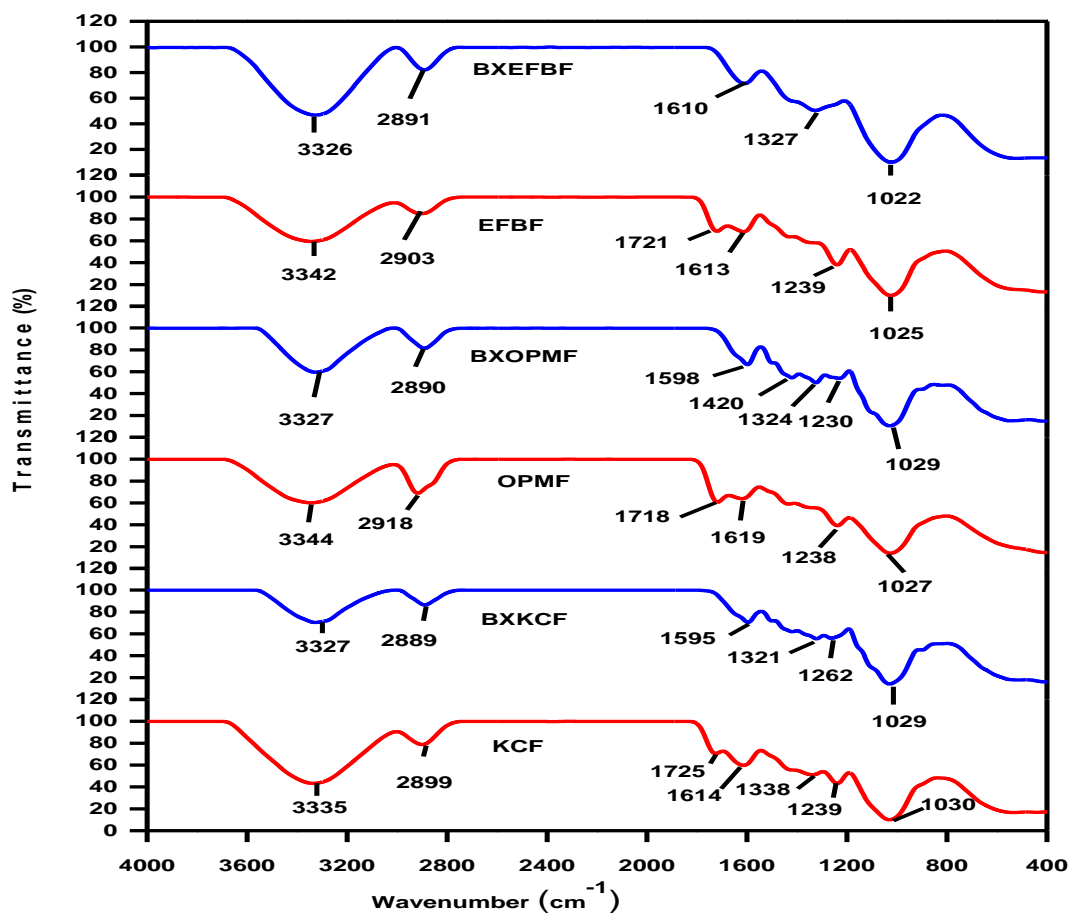


Fig. 1. Fourier transform infrared spectroscopy of treated and untreated fibers

A band at 1338 cm^{-1} was noted only in the spectra of KCF. This band can be ascribed to the C=C bond stretching of benzene present in lignin. Its appearance, only in the KCF spectrum, explained the high lignin content in this fiber, compared to that of OPMF and EFBF. Similar peaks appeared at the absorption bands of 1321 , 1324 , and 1327 cm^{-1} for the BXKCF, BXOPMF, and BXEFBF, respectively. The alcoholic, phenolic, and ether C-O stretching peaks for waxy residue and lignin in KCF, OPMF, and EFBF appeared at 1239 , 1238 , and 1239 cm^{-1} , respectively. Related peaks were observed at 1262 and 1230 cm^{-1} for BXKCF and BXOPMF, and disappeared in the spectra of BXEFBF. The disappearance can be attributed to the extent of waxy residue removal, as well as the low lignin content of EFBF. The peaks at 1030 , 1027 , and 1025 cm^{-1} can be assigned to the C-H and C-O stretching of aliphatic and aromatic primary alcohols present in cellulose, hemicellulose, and lignin in KCF, OPMF, and EFBF, respectively. Comparable peaks were observed at 1029 , 1029 , and 1022 cm^{-1} in the spectra of BXKCF, BXOPMF, and BXEFBF, respectively. This agreed with the observations reported by Maizatul *et al.* (2013) and Nasir *et al.* (2013).

Scanning electron microscopy analysis of treated and untreated fibers

The morphologies of the EFBF, OPMF, KCF, and their corresponding borax-treated BXEFBF, BXOPMF, and BXKCF, are presented in Fig. 2. The EFBF (Fig. 2a) presented residual impurities and a smoothly intact surface, as previously observed by Then *et al.* (2013). These impurities were removed after the borax treatment, and the surface became rough and textured (Fig. 2d). Few pores were noted on the OPMF surface (Fig. 2b), which multiplied after the borax treatment (Fig. 2e). These pores provided good bonding points with the PLA matrix, thereby enhancing the mechanical interlocking capabilities between the fiber and the PLA matrix (Then *et al.* 2014a).

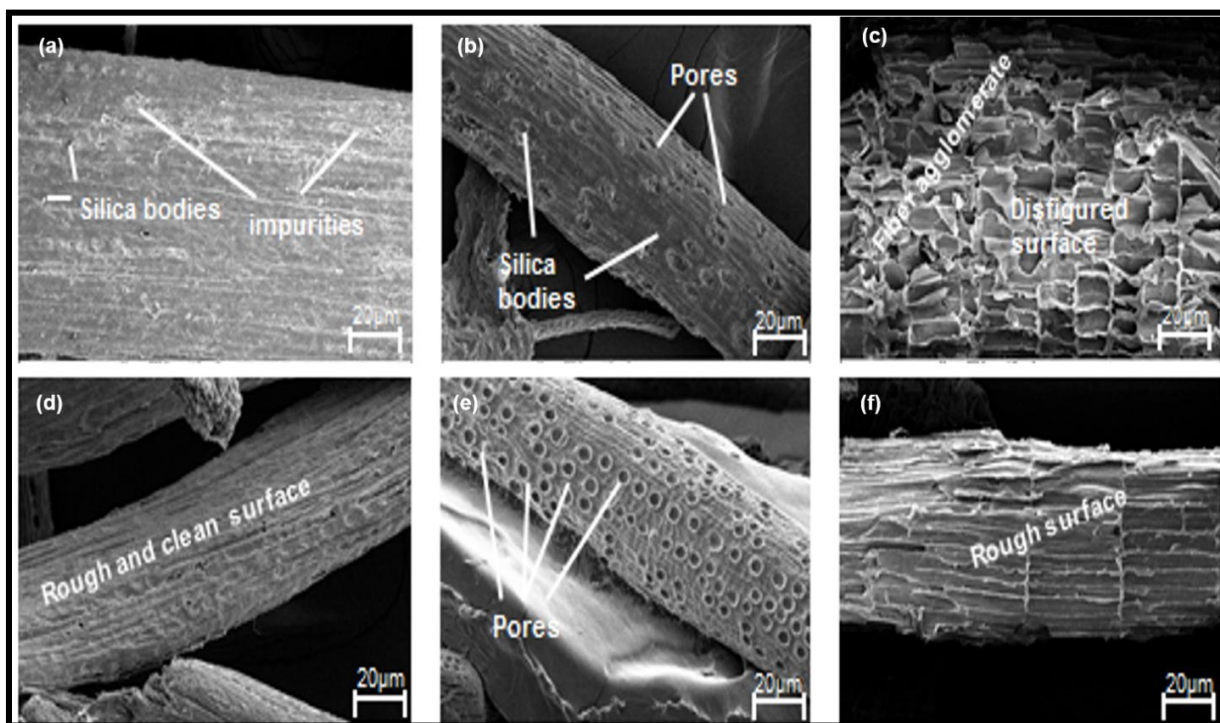


Fig. 2. Scanning electron micrographs of (a) EFBF, (b) OPMF, (c) KCF, (d) BXEFBF, (e) BXOPMF, and (f) BXKCF

The KCF surface presented disfigured morphology along with fiber agglomerates (Fig. 2c). These features were previously described to be the weak points that decreased the KCF's strength (Khalil *et al.* 2010c; Fiore *et al.* 2014). After the borax treatment, the KCF yielded improved morphology, having both clean and rough surfaces (Fig. 2f). Generally, the morphologies of the fibers were improved by the borax treatment, resulting from the removal of hemicellulose and waxy residue.

Characterization of Untreated and Borax-Treated Hybrid Fiber PLA Biocomposites

Mechanical properties

In this work, PLA was employed as the polymer matrix, owing to its attractive mechanical and environmental properties. Poly(lactic acid) was previously reported to possess high strength, modulus, and biocompatibility (Chieng *et al.* 2014). The tensile and flexural properties of the borax-treated and untreated hybrid fiber-reinforced PLA biocomposites are presented in Table 3.

Table 3. Tensile and Flexural Properties of Borax-Treated and Untreated Hybrid Fiber-PLA Composites

Sample	TS (MPa)	EB (%)	TM (MPa)	FS (MPa)	FM (GPa)
EFBF-KCF	35.59 ± 1.32	8.59 ± 0.84	469.39 ± 24.11	25.08 ± 1.80	4.19 ± 0.20
OPMF-KCF	28.96 ± 1.44	8.70 ± 0.75	335.86 ± 14.79	26.87 ± 1.43	2.57 ± 0.16
BXEFBF-KCF	37.44 ± 2.56	12.23 ± 0.45	305.92 ± 20.64	40.26 ± 1.19	5.23 ± 0.15
BXOPMF-KCF	33.66 ± 1.32	9.23 ± 0.53	344.28 ± 19.61	44.39 ± 2.07	5.02 ± 0.23

'±' refers to standard deviation.

It can be seen that the tensile strength (TS) of the hybrid fiber composites (HFCs) increased slightly after the borax treatment. The TS of EFBF-KCF and OPMF-KCF increased by approximately 5% and 16% after the borax treatment, *i.e.* BXEFBF-BXKCF and BXOPMF-BXKCF, respectively. This indicates enhanced internal bond strength between the fibers and the PLA matrix as a result of the removal of hydrophilic components from the fiber's surface (hemicelluloses). This observation concurred with the previous findings from Aziz and Ansell (2004). The elongation-at-break also improved after the borax treatment. Perhaps the strengthening of the internal bonds between the fibers and PLA facilitated the prevention of micro-crack generation and propagation, enhancing the elongation. The increase in the elongation of natural fiber-reinforced biocomposites, as a result of the borax treatment, was also observed by Cavdar *et al.* (2015). The aqueous borax pre-treatment of the fibers considerably influenced the tensile modulus (TM) of the HFCs. The TM of the BXEFBF-BXKCF-PLA was lower than that of the EFBF-KCF-PLA; meanwhile, the opposite trend was observed for the BXOPMF-BXKCF-PLA, which exhibited a higher TM than that of the OPMF-KCF-PLA. This disparity in TM among the treated and untreated HFCs can be ascribed to the degree of fiber compatibility and different pattern of fiber orientation that occurred during the melt-blending and compression molding (Ibrahim *et al.* 2010).

The flexural strength (FS) of both the EFBF-KCF-PLA and OPMF-KCF-PLA increased approximately 61% and 65%, respectively, after the borax treatment. It can be seen in Table 3 that the FS followed a similar trend as the TS. Similar observations have

been previously reported (Asumani *et al.* 2012). The flexural modulus (FM) also showed enhancement after the borax treatment, indicating that the stiffness of the treated hybrid fibers was higher than the untreated fibers, as previously observed by Razak *et al.* (2014) and Then *et al.* (2015). This increment in FM may be due to the improved interfacial adhesion between the fibers and the PLA matrix.

Generally, slight increment in both the tensile and flexural properties was observed for BXEFBF-BXKCF- PLA and BXOPMF-BXKCF- PLA compared to those of their corresponding untreated ones (EFBF-KCF- PLA and OPMF-KCF- PLA) and may be attributed to the removal of fiber surface impurities, thus facilitating a better bonding ability to the PLA matrix (Then *et al.* 2014a). Additionally, the hybridization of the lignin-rich KCF with the cellulose-rich EFBF and OPMF also influenced the mechanical performance of the borax-treated HFCs. The rigidity and stiffness of lignin has been described to aid its reinforcing abilities (Kubo and Kadla 2005; Nasir *et al.* 2013; Ghaffar and Fan 2014; Thakur *et al.* 2014; Then *et al.* 2015).

The impact strength (IS), presented in Fig. 3, of the BXEFBF-BXKCF and BXOPMF-BXKCF reinforced PLA composites exhibited higher values than those of the EFBF-KCF and OPMF-KCF reinforced PLA composites, having approximately 36% and 16% increases, respectively. The increase in IS can be attributed to strengthened internal bonding because of the removal of impurities from the fiber's surface, as well as the relatively rich lignin content in KCF and rich cellulose content in EFBF and OPMF, respectively. Improved IS was often obtained as a result of lignin, especially when the lignin is modified (Thakur *et al.* 2014). Clearly, the results of treated EFBF-KCF- PLA recorded better impact strengths than those of the treated OPMF-KCF- PLA hybrid; perhaps because the EFBF is tougher than the soft and flexible OPMF.

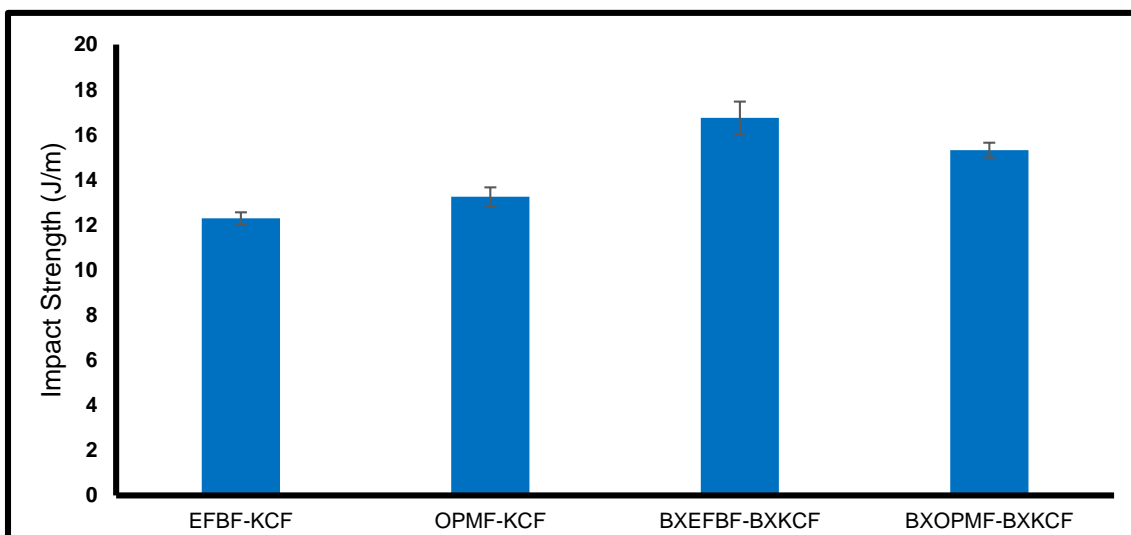


Fig. 3. Impact strength of borax-treated and untreated HFCs

Dimensional stability

The dimensional stability, *i.e.*, the water uptake and thickness swelling, of both the borax-treated and untreated HFCs, are presented in Fig. 4. The water uptake measured the moisture absorption of the natural fibers.

The high hygroscopicity of the natural fibers limits their suitability for biocomposites application. Thus, the hygroscopic property of the natural fiber should be reduced in order to make it suitable for biocomposite application (Then *et al.* 2014b).

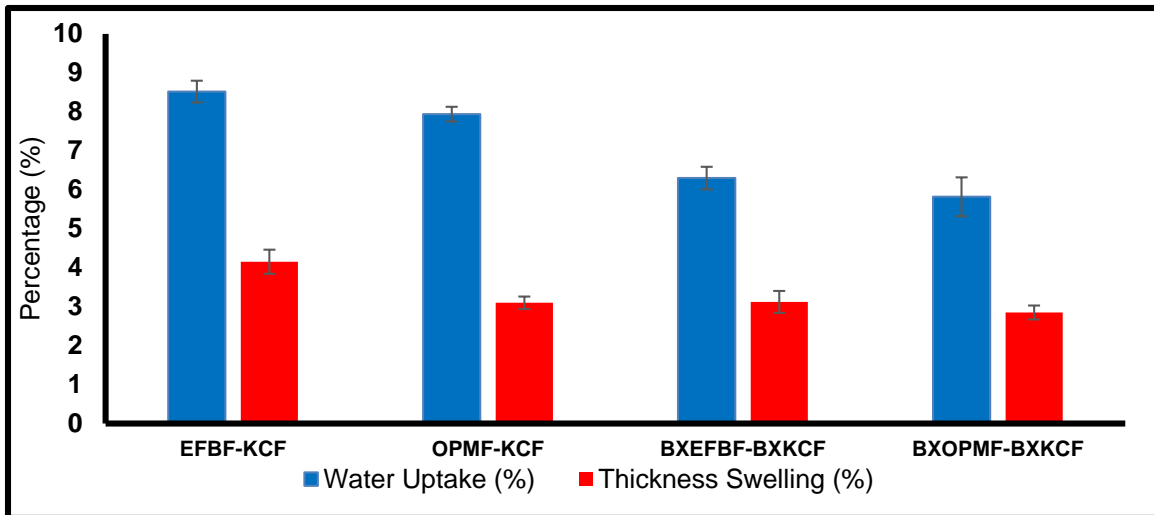


Fig. 4. Dimensional stability of borax-treated and untreated HFCs

It can be seen from Fig. 4 that the water uptake of the untreated HFCs drastically decreased after the borax treatment of the fibers. This can be a result of the removal of hemicellulose from the fibers, thereby decreasing the amount of hydrophilic O-H groups from the fibers' surface (Khalil *et al.* 2010a). This observation was validated by the FTIR analysis (Fig. 1) and chemical components results (Table 2).

Similarly, the thickness swelling of the untreated HFCs also decreased after the borax treatment by approximately 35% and 36% for BXEFBF-KCF-PLA and BXOPMF-KCF-PLA, respectively. This was expected because the thickness swelling is directly proportional to the water uptake of the biocomposites. Thus, the borax treatment imparts favorable bondability between the fibers and the PLA matrix, as well as enhancing the hydrophobicity of the borax-treated fibers for better dimensional stability of the HFCs (Ayrilmis 2012).

Densities of untreated and borax-treated HFCs

The densities (Fig. 5) of the borax-treated hybrid fiber-reinforced PLA biocomposites drastically declined compared to those of their corresponding untreated biocomposites. This was attributed to the removal of water absorbing O-H groups (hemicellulose) on the surface of the fiber by the borax treatment.

The untreated fibers contained the highest amounts of hemicelluloses, which absorbed moisture from the atmosphere and added density of the composites (Khazaeian *et al.* 2015). All of the HFCs exhibited higher densities than conventional medium density fiberboard (MDF).

The density of MDF ranges from 496 to 801 kgm⁻³ (Rivela *et al.* 2007). Therefore, natural fiber thermoplastic composites, having densities within this range, can be employed as an alternative for MDF. Also, those with densities greater than 900 kgm⁻³ are more suitable as alternatives for high density fiberboards (HDF).

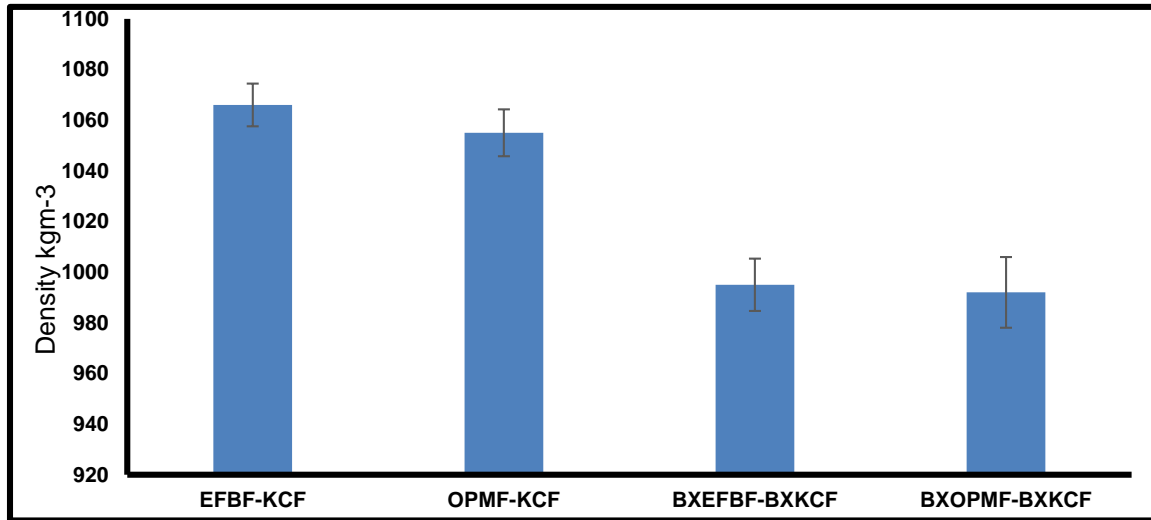


Fig. 5. Densities of borax-treated and untreated HFCs

However, the significant difference between the borax-treated and untreated hybrid fiber-PLA composites regarding their mechanical properties, dimensional stability, and density was statistically analyzed using the one-way analysis of variance (ANOVA) and the results are presented in Table 4. The variance of the mechanical properties (*i.e.* TS, EB, TM, FS, FM, and IS), dimensional stability (*i.e.* water uptake and thickness swelling), and density of the samples was decomposed into two components by ANOVA analysis *viz.* sum of square between group (SSBG) and sum of square within group (SSWG). The F-ratio was calculated as a ratio of the SSBG estimate to the SSWG estimate.

Table 4. ANOVA Test for Mechanical, Dimensional Stability and Density Properties of Borax-treated and Untreated Hybrid Fiber-PLA Composites

Source	TS	EB	TM	FS	FM	IS	WU	TW	DT
TSS	634.8835	81.4249	234145.7781	3093.3531	32.7478	66.4964	31.7667	8.1632	30104
SSWG	435.6236	37.2852	155846.8980	1932.5919	12.6036	5.5526	7.0537	3.1821	7334
SSBG	199.2599	44.1398	78298.8801	1160.761	20.1442	60.9438	24.7129	4.9811	22770
DFBG	3	3	3	3	3	3	3	3	3
DFWG	16	16	16	16	16	16	16	16	16
F-ratio	2.4395	6.3138	2.6795	3.2033	8.5242	58.5372	18.6856	8.3487	16.5585
P-value	0.1021	0.0049	0.081	0.052	0.0013	0.0000	0.0000	0.0014	0.0000

ANOVA: analysis of variance; TS: tensile strength; EB: elongation at break; TM: tensile modulus; FS: flexural strength; FM: flexural modulus; IS: impact strength; WU: water uptake; TW: thickness swelling; DT: density; TSS: total sum of square; SSWG: sum of square within group; SSBG: sum of square between group; DFBG: degree of freedom between group; DFWG: degree of freedom within group; F: F test for ANOVA, P-value: probability value; Number of sample = 4; Number of observations = 20.

It can clearly be seen from Table 4 that the P-value of F-test for both water uptake and thickness was less than 0.05, which indicates that there was a statistically significant difference between the mean water uptake and thickness swelling from untreated hybrid fiber-PLA composites to their corresponding borax-treated samples at the 95.0% confidence level. A similar observation was made for density and impact strength,

respectively. However, among the tensile and flexural properties, only EB and FM showed a significant difference whereas, regarding TS, TM, and FS there was no significant difference between the mean of these properties from untreated hybrid fiber-PLA composites to their corresponding borax-treated samples at the 95.0% confidence level.

This anomaly in the statistical significance of the mechanical properties of the hybrid fiber-PLA composites could perhaps be associated with the brittleness of PLA matrix. The PLA used in the present research was unmodified and may possibly generate an interfacial tension by restricting dispersion of the hybrid fiber in the PLA matrix, and consequently, reduce the stress transfer efficiency between the fibers and the PLA (Dos Santos and Tavares 2014). Accordingly, only a small increase in mechanical properties was observed for the borax-treated hybrid fiber-PLA composites, providing almost a comparable significant difference with the untreated hybrid fiber-PLA composites. A major increment in mechanical properties of borax-treated hybrid fiber-PLA composites may be achieved by modification of the PLA *via* reactive compatibilization with the application of a suitable coupling agent, as previously posited by (Fowlks and Narayan 2010).

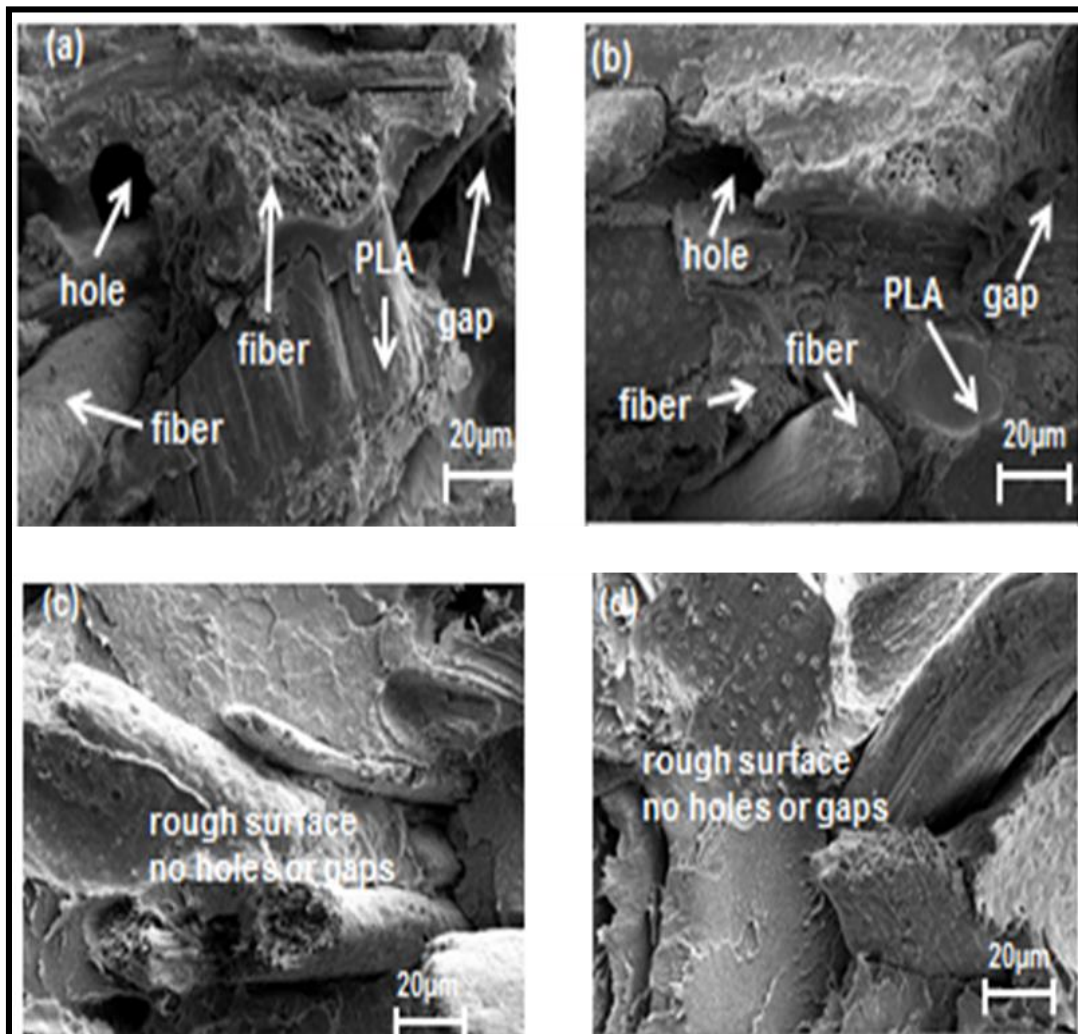


Fig. 6. Scanning electron microscopy imaging of fractured surfaces of (a) OPMF-KCF-PLA, (b) EFBF-KCF-PLA, (c) BXOPMF-BXKCF-PLA, and (d) BXEFBF-BXKCF-PLA

Morphology of the fractured surfaces of the HFCs

Scanning electron microscopy was employed to probe the morphology of the fractured surfaces of both borax-treated and untreated hybrid fiber-reinforced PLA biocomposites. The results are depicted in Fig. 6.

The untreated fibers contained a high level of impurities and hydrophilic groups, such as hemicelluloses, which made the hybrid fibers less bondable, as well as weaker in strength than those of the borax-treated HFCs. Thus, the biocomposites fabricated from the untreated HFCs exhibited fractured surfaces, revealing holes and gaps (Fig. 6a through 6b). Biocomposites that possess these features (gaps and holes) imply weak mechanical performance and dimensional stability (Ibrahim *et al.* 2010). Conversely, these weak features (gaps, holes, and fiber debris) are relatively unrevealed in the morphologies of the fractured surfaces of the borax-treated HFCs (Fig. 6c through 6d). Therefore, this implies that the borax-treated HFCs had better mechanical behavior and dimensional stability. Previous studies have noted that the chemical treatment of natural fibers improves the morphology of biocomposites (Thiruchitrambalam *et al.* 2009; Asumani *et al.* 2012).

CONCLUSIONS

1. The aqueous borax modification of oil palm empty fruit bunch fiber (EFBF), oil palm mesocarp fiber (OPMF), and kenaf core fiber (KCF) effectively achieved fibers with cleaner and rougher surfaces. The modification led to the partial removal of lignin along with residual waxy impurities and considerable elimination of hemicellulose, consequently increasing the cellulose content of the treated fibers.
2. The hybrid biocomposites of the borax-modified fibers-reinforced polylactic acid (PLA), *i.e.* BXEFBF-BXKCF-PLA and BXOPMF-BXKCF-PLA, had considerable improvements in their dimensional stability, and minimal increment in mechanical properties, compared with the corresponding untreated biocomposites. Optimal results were observed with the BXEFBF-BXKCF-PLA biocomposites.
3. Analysis of variance (ANOVA) revealed that, only the effects on elongation at break (EB), flexural modulus (FM), and impact strength (IS) exhibited significance, whereas tensile strength (TS), tensile modulus (TM), and flexural strength (FS) showed no significance. This anomaly in significance of mechanical properties may be due to brittleness of PLA. The ANOVA showed a statistically significant difference in dimensional stability and density of all hybrid fiber-PLA composites.
4. This study demonstrated that the aqueous borax treatment of natural fibers with a water-washing procedure can be an attractive option to borax impregnation, borax filler as well as mercerization methods, in fabricating hybrid natural fiber thermoplastic biocomposites, with a promising mechanical performance and enhanced density and dimensional stability.

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