

## Optimized Pretreatment of Kenaf (*Hibiscus cannabinus*) Phloem Insulation Cotton

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Using response surface methodology, the pretreatment conditions of kenaf fibers were optimized to improve the tensile strength of kenaf phloem insulation cotton (KPIC). The effects and interactions of three parameters—sodium hydroxide concentration ( $X_1$ ), soaking time ( $X_2$ ), and beating time ( $X_3$ )—on the tensile strength of the kenaf fibers were investigated. The Fourier transform infrared (FTIR) spectroscopy, X-ray diffraction (XRD), and thermal conductivity of the KPICs further confirmed the validity of the optimal pretreatment conditions. Sodium hydroxide concentration had the greatest effect on kenaf fibers. The maximum tensile strength of 117.6 N resulted from a sodium hydroxide concentration of 4%, soaking time of 50 h, and beating time of 12 min. As shown by FTIR and XRD, optimized pretreatment generated surface functional groups and increased the tensile strength of fibers. It has a low thermal conductivity of 0.032 W/mK. In conclusion, the pretreatment of kenaf fiber significantly improves the tensile strength of KPIC and also improves the retention rate of the chemicals used during the preparation of KPIC. As an environment friendly and renewable material, the KPIC has a great application prospect with its good thermal conductivity.

*Keywords:* Kenaf phloem insulation cotton; Pretreatment; Mechanical property; Optimization; Characterization

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### INTRODUCTION

The depletion of petroleum resources and increasing awareness of environmental pollution has stimulated industries (*i.e.* automotive, building, and nautical) to replace conventional materials with more sustainable ones. As environmentally friendly and renewable materials, natural fibers have great advantages over other materials and, thus, are the most promising for building. Thermal insulation materials have been developed from easily obtained biomass. Zhou *et al.* (2010) used cotton stalk fibers and high frequency hot pressing to develop a binderless fiberboard with no chemical additives. A thermal and sound insulation nonwoven mat has been produced from waste wool and recycled polyester fibers (Patnaik *et al.* 2015). A thermal insulation-like material from sunflower stalk, textile waste, and stubble fibers was made by Binici *et al.* (2014). Coconut husk, bagasse (Panyakaew and Fotios 2011), silkworm cocoon (Zhang *et al.* 2013), and even crop by-products (Palumbo *et al.* 2015) can be used to develop raw materials for thermal insulation.

Kenaf-reinforced composites are primarily used as low-cost materials with practical structural properties. Kenaf has been manufactured in nonwoven mats because

of its high elastic modulus (Hao *et al.* 2012), and kenaf fibers have been used to improve the mechanical properties of various polymer composites (Abu Bakar *et al.* 2011; Arsad *et al.* 2013; Kim *et al.* 2014; Paridah *et al.* 2014; Saba *et al.* 2015).

There is increasing interest in the utilization of kenaf in papermaking (Ashori *et al.* 2006), particleboard (Xu *et al.* 2004), and fiberboard manufacturing (Kawasaki *et al.* 1998). However, limited research has been conducted on the thermal insulation materials of kenaf.

Kenaf phloem insulation cotton (KPIC) is manufactured by liquid frothing. In our previous work, the raw materials were mechanical pine (*Pinus massoniana*) pulp, kraft pulp, or the fiber for medium density fiberboard (MDF) (3:7 of *Pinus massoniana* mixture of hardwoods) (Xie *et al.* 2004, 2008a, 2008b). The cited work represents a new experimental approach, using the renewable and environment-friendly kenaf as the raw material. This process efficiently lowers material density and modifies its internal structure (Xie *et al.* 2004, 2008a, 2008b). The mechanical properties of foaming materials have been improved by inorganic compounds (Xie and Liu 2012; Lin *et al.* 2013; Niu *et al.* 2014). However, pretreatments for foaming materials have not received attention in previous studies.

Pretreatments can improve the performance of natural fibers by bridging the gap in compatibility between hydrophilic fibers and hydrophobic matrices (Tang and Liang 1999). Physical treatments change the structural and surface properties of fibers and thereby influence their mechanical bonding to polymers. Chemical methods, such as alkaline treatment, are effective because they increase the interfacial bonding strength between lingocellulosic fibers and chemical fillers. Alkali treatment improves fiber-matrix interfacial adhesion and increases the amount of cellulose exposed on the fiber surface; these effects improve fiber-polymer bonding (Zheng *et al.* 2012; Fiore *et al.* 2015; Li *et al.* 2015). Physical and chemical pretreatments are rarely used together, but they were both included in this study.

The objective of this study was to improve the mechanical properties of KPIC by kenaf phloem fiber pretreatments. The pretreatment conditions, including the concentration of sodium hydroxide (NaOH), soaking time, and beating time, were optimized by a standard RSM design, which is referred to as a Box-Behnken design (BBD). Fourier transform infrared (FTIR) spectroscopy and X-ray diffraction (XRD) were used to determine the effect of pretreatments on the microstructure of KPIC. And thermal conductivity of KPIC was studied by Thermogravimetric Analysis.

## EXPERIMENTAL

### Materials

Kenaf (*Hibiscus cannabinus*), a raw material containing 78.1% cellulose, 24.3% hemicelluloses, and 15.6% lignin, was purchased from Hena Tianli Products Co., Ltd. Henan, China.

Sodium hydroxide (NaOH) and glue were purchased from Sinopharm Chemical Reagent Co., Ltd. (Shanghai, China). Sodium dodecylbenzene sulfonate surfactant (foaming agent) and alkyl ketene dimer (AKD, the water proofing additive) were purchased from Jiangsu Qingting Washing Products Co., Ltd., Yancheng, China.

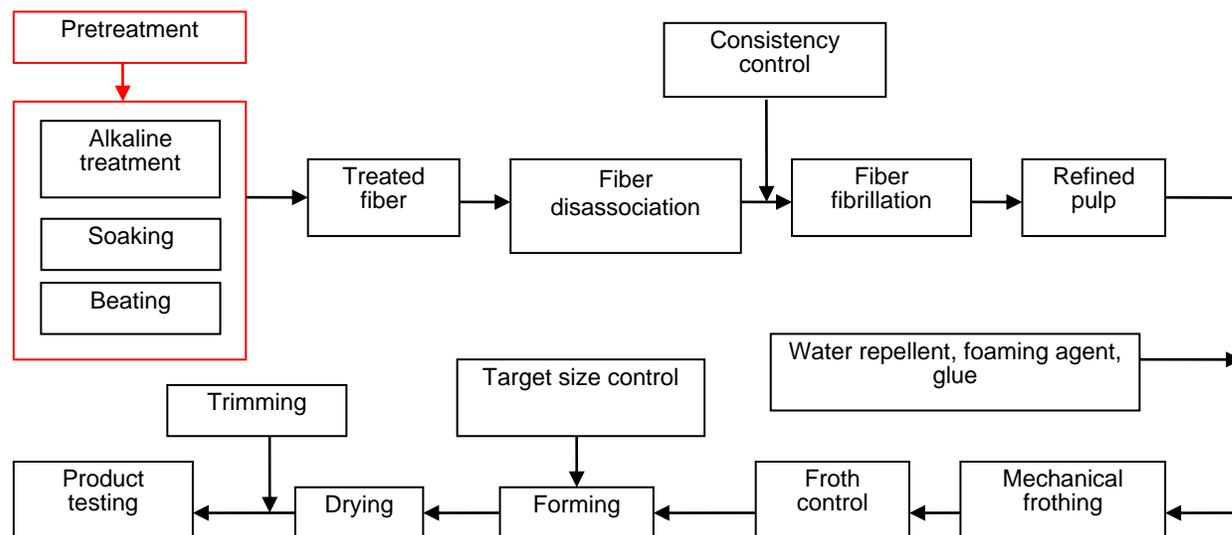
## Methods

### *Pretreatment of kenaf phloem fibers*

Phloem fibers were removed from kenaf and cleaned with purified water. First, they were chipped to an average length of 60 to 80 mm. The chips were soaked in asodium hydroxide (concentration 2, 4, or 6 %) for a variable period of time (36, 50, or 64 h) at room temperature (around 30 °C). They were ground in an extrusion machine (JS 10, Wenrui Machinery (Shandong) Co., Ltd., Anqiu, China). The pretreated fibers were the base material for KPIC.

### *Preparation of kenaf phloem insulation cotton (KPIC)*

The mixture solvent containing water, AKD (5.0 wt.%), glue (2.5 wt.%), and sodium dodecylbenzene sulfonate (3.0 wt.%) was transferred into an open foaming tank attached to a fiber dissociator (model GBJ-A, Changchun Yueming Miniature Tester Co., Ltd., Changchun, China) and mixed with the kenaf phloem pulp. KPIC was prepared as described in Xie *et al.* (2011). KPIC samples of 190 × 190 × 50 mm ( $L \times W \times H$ ) were manufactured individually using various parameters (Fig. 1) with a target bulk density of 50 to 90 g/m<sup>3</sup>.



**Fig. 1.** The preparation process of kenaf phloem insulation cotton

### *Experimental design*

The effects of three independent variables on the response variable were investigated using a Box-Behnken design (BBD) (Table 1).

**Table 1.** Code and Level of Factors Chosen for the Trials

Coded-variables	Levels		
	-1	0	+1
Sodium Hydroxide Concentration ( $X_1$ , %)	2	4	6
Soaking Time ( $X_2$ , h)	36	50	64
Beating Time ( $X_3$ , min)	8	12	16
-1 means low level and +1 means high level, and a center point was run to evaluate the linear and curvature effects of the variables			

The three independent variables were sodium hydroxide concentration ( $X_1$ ), soaking time ( $X_2$ ), and beating time ( $X_3$ ). The response variable was the tensile strength (TS) of KPICs ( $Y$ ). The specific parameters and levels are shown in Table 1. Regression analysis was performed with Design Expert 7.0 Trial (Static Made Easy, Minneapolis, MN, USA).

#### *Characterization*

The tensile strength of KPICs was tested in accordance with GB/T 6344 (2008) using a micro control electronic universal testing machine (model CMT 5504, Xinsansi Material Testing Co., Ltd., Shenzhen, China). The size of the specimens was 15 mm × 15 mm × 100 mm.

Chemical groups in the original KPIC specimen and KPIC prepared with the optimal conditions were characterized by a Fourier transform infrared spectrometer (Bruker Vertex 70, Ettlingen, Germany) in the scanning range of 4000 to 400  $\text{cm}^{-1}$ . The KBR pellet method was employed, taking 64 scans for each sample with a resolution of 2  $\text{cm}^{-1}$ .

The crystal structure of specimens was analyzed with an X-ray diffractometer (X'Pert PRO, PANalytical, Almelo, Netherlands). Samples were dried and ground into powders for XRD. The diffracted intensity of Co  $K\alpha$  radiation (40 kV and 40 mA) was measured in a  $2\theta$  range between 4.0° and 65.0°.

The thermal conductivity of thermal insulation specimens of 200 mm × 200 mm × 15 mm were analyzed by thermal conductivity analyzer (HFM 436/3/1E, NETZSCH-Gerätebau GmbH, Selb, Germany).

## RESULTS AND DISCUSSION

### Modeling and Optimization of KPICs

#### *Model fitting*

BBD was employed to develop correlations between the KPIC preparation variables and its TS ( $Y$ ). Results from all 17 tests are shown in Table 2.

The experimental data shown in Table 2 were fitted into the quadratic polynomial equations to predict the responses. The coefficients of the parameter variables ( $X_1$ ,  $X_2$ , and  $X_3$ ) for the response variable ( $Y$ ) were expressed by the following second-order polynomial equation in terms of coded values:

$$Y = 117.60 + 4.88X_1 + 0.14X_2 - 0.98X_3 - 4.32X_1X_2 + 1.04X_1X_3 - 3.50X_2X_3 - 13.81X_1^2 - 9.39X_2^2 - 13.69X_3^2 \quad (1)$$

Coefficients for a model for the responses were estimated using a multiple regression analysis technique included in the RSM. An adjusted coefficient of correlation ( $R^2_{\text{adj}}$ ) was the correlation measure for testing the goodness-of-fit of the regression equation with a higher value being more favorable. For Eq. 1, the  $R^2_{\text{adj}}$  was 0.9274. These indicate that 92.74% of the total variation in TS was attributable to the experimental variables studied and these models could be used to navigate the experimental design space.

**Table 2.** Box-Behnken Design (BBD) and the Corresponding Tensile Strength

Run No.	Coded levels			Tensile Strength (N)	
	X <sub>1</sub>	X <sub>2</sub>	X <sub>3</sub>	Experimental	Predicted
1	-1	-1	0	84.87	85.09
2	0	-1	+1	96.87	96.93
3	-1	0	-1	83.68	87.27
4	0	-1	-1	95.71	91.90
5	+1	-1	0	99.96	103.49
6	-1	0	+1	83.52	83.23
7	0	+1	+1	86.39	90.21
8	+1	0	+1	98.65	95.06
9	0	+1	-1	99.24	99.18
10	+1	0	-1	94.67	94.96
11	+1	+1	0	95.35	95.13
12	-1	+1	0	97.54	94.01
13	0	0	0	116.82	117.62
14	0	0	0	117.16	117.62
15	0	0	0	117.31	117.62
16	0	0	0	118.53	117.62
17	0	0	0	118.3	117.62

\*Note: X<sub>1</sub>, NaOH concentration (%); X<sub>2</sub>, soaking time (h); X<sub>3</sub>, beating time (min)

The significance of the fitted model for the TS was evaluated by analysis of variance (ANOVA) (Table 3). A model was deemed significant if its 'Prob> F' (*p*-value) was smaller than 0.05 and its F-value was relatively high, suggesting only a 5% chance that a 'Model F-value' could occur due to confounders. On the basis of ANOVA, statistical analysis was carried out to check whether these parameters for a response were significant in terms of a *p*-value. The *p*-value of the model was 0.0002, which was less than 0.05, the Lack of Fit value was 47.71, and the Lack of Fit *p*-value of 0.0014 was significant, suggesting that the model fitted well with all data. The result indicated that the model was considered to be statistically significant. In addition, the values of R<sup>2</sup> and adjusted R<sup>2</sup> were 0.97 and 0.93, respectively. This meant that the model achieved a high degree of fit and could provide a satisfying prediction for the experimental results (Chen *et al.* 2012).

The sodium hydroxide concentration (X<sub>1</sub>), interaction term (X<sub>1</sub>X<sub>2</sub>), and three quadratic terms (X<sub>1</sub><sup>2</sup>, X<sub>2</sub><sup>2</sup>, X<sub>3</sub><sup>2</sup>), affected the TS significantly, whereas the soaking time (X<sub>2</sub>), beating time (X<sub>3</sub>), X<sub>1</sub>X<sub>3</sub>, and X<sub>2</sub>X<sub>3</sub> did not.

According to the sum of squares of the parameter variables, the effect of the parameters on the TS were as follows: sodium hydroxide concentration (X<sub>1</sub>) > beating time (X<sub>3</sub>) > soaking time (X<sub>2</sub>).

**Table 3.** Analysis of Variance (ANOVA) for Regression Equations

Source	Sum of Squares	Degree of freedom	Mean Square	F-value	p-value
Model	2510.49	1	278.94	23.72	0.0002
X <sub>1</sub>	190.32	1	190.32	16.18	0.0050
X <sub>2</sub>	0.15	1	0.15	0.013	0.9121
X <sub>3</sub>	7.74	1	7.74	0.66	0.4438
X <sub>1</sub> X <sub>2</sub>	74.65	1	74.65	6.35	0.0398
X <sub>1</sub> X <sub>3</sub>	4.28	1	4.28	0.36	0.5651
X <sub>2</sub> X <sub>3</sub>	49.07	1	49.07	4.17	0.0804
X <sub>1</sub> <sup>2</sup>	802.81	1	802.81	68.27	< 0.0001
X <sub>2</sub> <sup>2</sup>	370.91	1	370.91	31.54	0.0008
X <sub>3</sub> <sup>2</sup>	788.63	1	788.63	67.06	< 0.0001
Residual	82.32	7	11.76		
Lack of Fit	80.08	3	26.69	47.71	0.0014
Pure Error	2.24	4	0.56		
Corrected Total	2592.80	16			

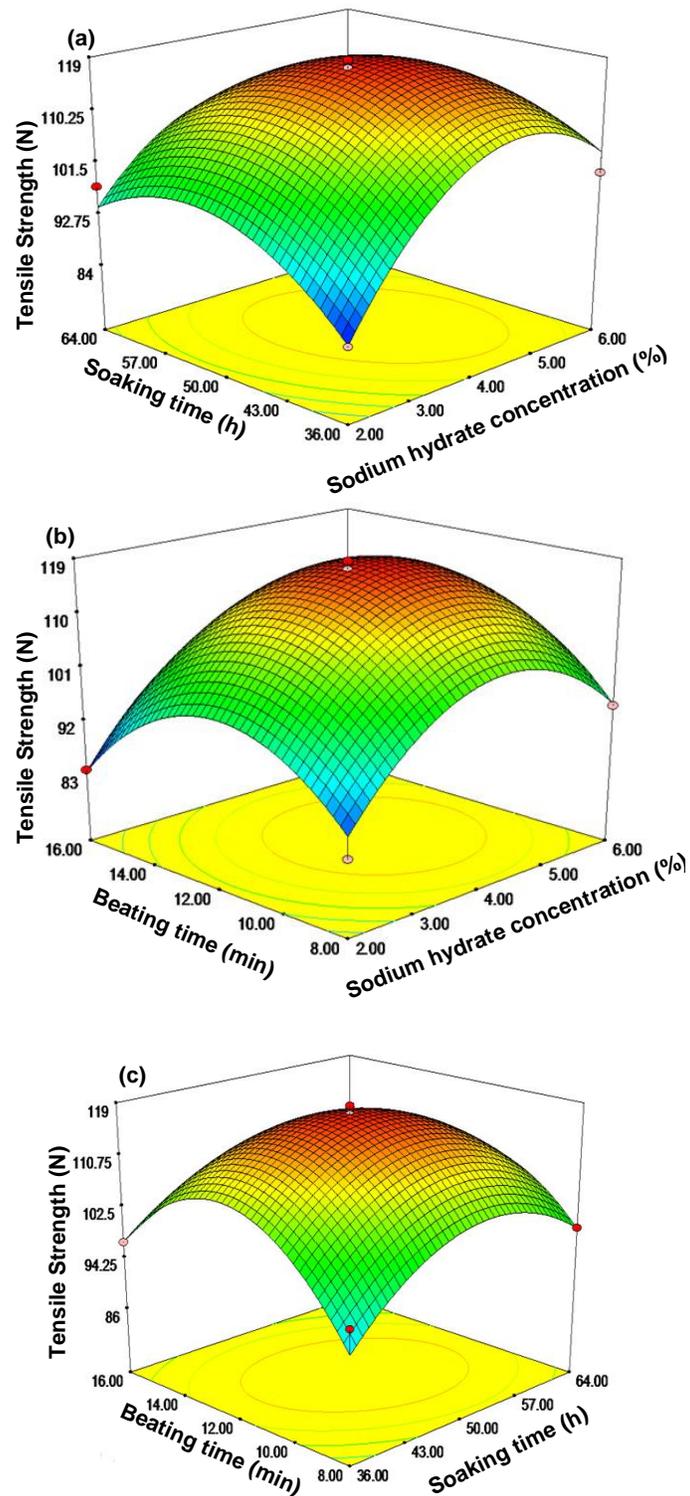
\*Note: p < 0.01 highly significant; 0.01 < p < 0.05 significant; p > 0.05 insignificant.

#### Analysis of response surface and optimization

KPIC pretreatment response surface graphs showed the relationships between the parameters and the response variable, as illustrated in a 3D representation of the response surface (Fig. 2).

Figure 2(a) shows the effects of sodium hydroxide concentration and soaking time on TS. The maximal TS occurred when the NaOH concentration was 4% and soaking time was 12 h. Both factors had positive effects on the response. Increasing the NaOH concentration, from 2% to 4%, and soaking time, from 36 h to 50 h, produced an increased TS up to its maximum value; further increases showed an adverse effect. This adverse effect might be caused by the reaction of free hydrogen bonds in cellulose after alkali treatment. The reactivity of the hydrogen bonds on the surface of cellulose is strengthened by increasing the NaOH concentration (Tang and Liang 1999). However, the fiber structure was destroyed and the fiber strength decreased at very high NaOH concentrations (Tang and Liang 1999).

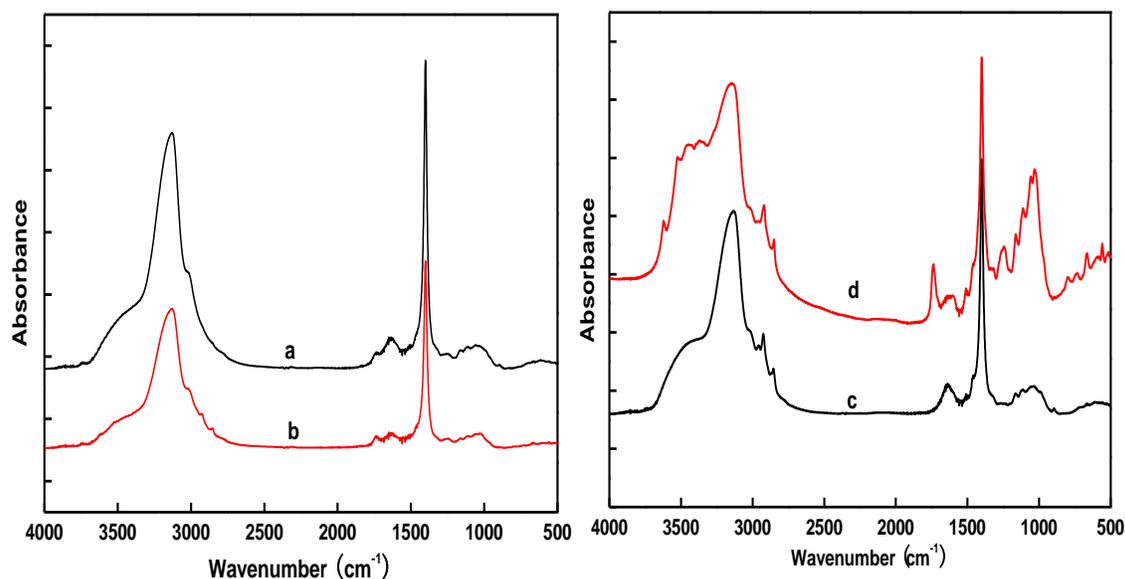
Figure 2(b) depicts the interaction of NaOH concentration and beating time relative to TS. This effect was not obvious. The beating time exhibited a weaker effect, whereas the NaOH concentration produced a significant effect because bonding forces were enhanced when the fibers were treated with the alkaline condition. However, the fibers became shorter, which might have harmed the material strength (Yang *et al.* 2005).



**Fig. 2.** Response surface plots for the maximum TS of KPIC: (a) sodium hydroxide concentration vs. soaking time; (b) sodium hydroxide concentration vs. beating time; (c) soaking time vs. beating time.

Figure 2(c) shows that the TS improved initially and then decreased with increasing soaking time. The effect of the beating time on TS followed a similar trend to that of the soaking time, mostly due to the increased bonding force of the kenaf fibers.

Taken together, these data indicated that the optimal pretreatment conditions were 4% sodium hydroxide, 50 h soaking time, and 12 min beating time.



**Fig. 3.** FTIR spectra of (a) kenaf phloem fiber (KPF), (b) KPF pretreated with the optimal conditions, (c) KPIC made with untreated KPF, and (d) KPIC made from KPF with the optimal pretreatment

### Micro-morphology of KPICs

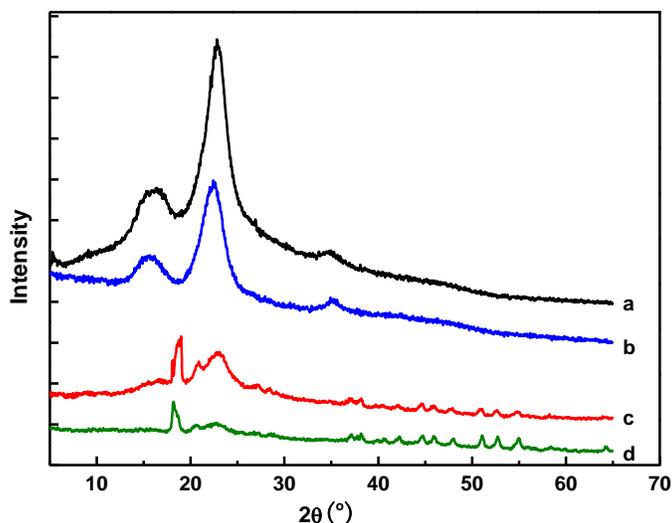
#### FTIR analysis

Changes in the structural configuration of treated and untreated specimens were analyzed by FTIR spectroscopy (Fig. 3). The change in absorbance and alteration of the bands are clearly shown. There were few differences in the FTIR spectra, indicating that the chemical structure of KPF was only degraded slightly by pretreatment (Fig. 3). The broad absorption band observed at  $3140\text{ cm}^{-1}$  for all specimens was attributed to the OH stretching from the intermolecular and intramolecular hydrogen bonds of cellulose. The peak at  $1620\text{ cm}^{-1}$  indicated the bending vibration of residual water molecules. The band at  $1400\text{ cm}^{-1}$  corresponded to asymmetric bending of  $\text{CH}_3$  that was lost during pretreatment, again verifying the structural degradation of the biomass (Spiridon *et al.* 2010). However, the three bands ( $3140$ ,  $1620$ , and  $1400\text{ cm}^{-1}$ ) were weakened compared with those of the KPF without pretreatment. These results confirmed that pretreatment affected the oxidative degradation of KPF, primarily because sodium hydroxide and hydroxyl radicals generated by the decomposition of hydrogen peroxide were responsible for delignification (Jiang *et al.* 2015). In addition, the intensity of other bands was mostly constant, suggesting that the structure of KPF was not obviously changed by pretreatment.

The peaks between  $2800$  and  $3000\text{ cm}^{-1}$  in Figs. 3(c) and (d) showed C-H stretching. The peak at  $1730\text{ cm}^{-1}$  almost disappeared in the extracted cellulose microfibrils because alkali treatment removes most hemicelluloses and lignin from kenaf phloem (Uma Maheswari *et al.* 2012). Figures 3(c) and (d) show that pretreatment

affected the reaction between inorganic fillers and fibers, which helped retain inorganic fillers in the KPIC. The additional peaks absent from Fig. 3(c) could be attributed to the inorganic fillers. An absorption value and area increased the -OH groups in Fig. 3(d), which implied the weakening of hydrogen bonds in pretreated KPIC. Thus, pretreatment changed crystalline cellulose from cellulose I to cellulose II (Yang *et al.* 2010).

The changes in the band intensities after treatment clearly indicated that the interfacial bonding strength between kenaf fibers and chemical fillers was developed.



**Fig. 4.** X-ray diffraction curves of: (a) kenaf phloem fiber (KPF), (b) KPF pretreated with the optimal conditions, (c) KPIC made with untreated KPF, and (d) KPIC made from KPF with the optimal pretreatment

#### XRD analysis

X-ray diffraction was applied to characterize the crystalline structure and diffraction patterns (Fig. 4). The transformation of crystal structure could be clearly found according to Fig. 4. The diffraction peaks at  $2\theta = 18^\circ$ ,  $22^\circ$ , and  $34^\circ$  were perfectly indexed to the (101), (002), and (040) crystal faces of cellulose I in the native fiber, respectively (Chen *et al.* 2015). After pretreatment, the peaks at  $18.9^\circ$  and  $23.1^\circ$  weakened (cp. Fig. 4(a), (b)). The diffraction intensity in KPIC samples (Fig. 4(c), (d)) decreased and slightly broadened. The intensity of peaks was limited by a substantial proportion of chemical additives (Chen *et al.* 2015).

Untreated specimens had characteristic peaks with a weak diffractive pattern at  $2\theta = 15^\circ$ ,  $16^\circ$ , and  $22^\circ$ . The peak at  $22.5^\circ$  represented the amorphous area (Zheng *et al.* 2012). This might be evidence of enhancement of thermal insulating properties of (d), since its structure was amorphous. In general, it is well known that amorphous materials possess low thermal conductivity. The treated specimens had characteristic peaks at  $18^\circ$  and  $22^\circ$ . These differences showed that the hydroxyl groups in the fiber surface were removed by the alkaline pretreatment (Wang *et al.* 2014).

During the pretreatment, cellulose was first fully exposed to alkali solution accompanied by increased reactivity of cellulose toward chemical modification and structural transformation from cellulose I to cellulose II (Tang and Liang 1999). In this case, the alkaline treatment penetrated the adjacent amorphous region of cellulose, leading to its dissolution. In sum, the pretreatment changed the kenaf cellulose structure

and destroyed hydrogen bonds, which had a positive effect on combining the chemical and base materials.

#### *Thermal conductivity*

Table 4 compares the thermal conductivity of KPIC (untreated), KPIC (optimization), and other common thermal insulation materials. The results showed that the KPIC (with optimization) had a low thermal conductivity that ranged from 0.043 and 0.032 W/mK. The thermal conductivity and density of KPIC were both improved by pretreatment. It could be seen that the thermal conductivity values of both KPICs were lower than textile waste panel, kenaf, and cotton stalk insulation boards. KPIC was similar to or slightly lower than that of similar products reported in the literature (Ultra-low-density fiberboard and low-density binder less particleboard), and it was also similar to that of rockwool. Low fiber content (low density) and low thermal conductivity made the KPIC a good thermal insulation material. Therefore, it could be concluded that KPIC was an excellent insulating material for building component.

**Table 4.** Thermal Conductivity of Various Materials

Materials	Density (kg/m <sup>3</sup> )	Thermal conductivity (W/mK)	Source
KPIC (untreated)	59–63	0.039–0.055	This work
KPIC(optimization)	61–68	0.032–0.043	This work
ULDF	56.3	0.035	Xie <i>et al.</i> (2011)
Ultra-low-density fiberboard	≤100	0.039	Kawasaki <i>et al.</i> (1998)
low-density binderless particleboard	≤100	0.043	Xu <i>et al.</i> (2004)
cotton stalk fibers thermal insulation board	150–450	0.0585–0.0815	Zhou <i>et al.</i> (2010)
Textile waste panel	203–491	0.053–0.041	Valverde <i>et al.</i> (2013)
Kenaf binder less board	150–200	0.051–0.058	Zou (2008)
Wood (pine)	450–630	0.151	Zou (2008)
Rockwool	80–200	0.025–0.035	Zou (2008)
Low-density wheat straw board	150–250	0.0481–0.0521	Zou (2008)
Fiberglass	24–120	0.034–0.047	Zou (2008)
Vermiculite	80–200	0.047–0.07	Zou (2008)

## CONCLUSIONS

1. The tensile strength of KPIC was increased by pretreatment. The response parameter of tensile strength was affected by the soaking time, the sodium hydroxide concentration, and the beating time. An optimal tensile strength of 117.4 N was obtained after pretreatment with 4% sodium hydroxide concentration, a soaking time of 50 h, and a beating time of 12 min.
2. Structural analysis from FTIR and XRD studies showed that no noticeable change on KPIC macromolecular structure occurred and the major structural changes took place in the pretreatment, during which the crystal structure was partially destroyed and the crystal phase transition from cellulose I to cellulose II appeared. These results elucidated the increased deposition of chemical fillers and the improved tensile strength of KPIC.
3. Thermal conductivity of panels manufacturing in this conditions was obtained in a range between 0.043 and 0.032 W/mK. The obtained values were similar to other commercial insulation materials. Since it was made from kenaf phloem and without any chemical binders, it can be regarded as environmentally friendly and renewable. Furthermore, the manufacturing process of KPIC was very simple. KPIC could possibly compete with conventional insulation materials and has a bright future.

## ACKNOWLEDGMENTS

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