OPTIMIZATION OF CHEMICAL TREATMENTS OF *COMBRETUM DOLICHOPETALUM* FIBER FOR SUSTAINABLE APPLICATIONS

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ABSTRACT

Optimizing the chemical treatment conditions (concentration and time) of C. dolichopetalum fiber at room temperature was aimed to be investigated. C. dolichopetalum fibers were separately treated with NaOH and acetic anhydride after extraction with tensile strength as a response at various concentrations (c) and time (t). Optimization was done using response surface methodology (RSM) with three level factorial design (3-LFD) on tensile strength of fiber. The optimal process conditions obtained for sodium hydroxide and acetic anhydride treated dolichopetalum fibers were 14.19 % at 28.2 minutes and 14.66 % at 86 minutes with increased tensile strength by 690.75 and 1003.49 % of untreated fibers *respectively. The results showed that the developed models are suitable for prediction of tensile strength of treated C. dolichopetalum fibers with sodium hydroxide and acetic anhydride.*

Keywords: C. dolichopetalum fiber, response surface methodology, optimization, and tensile strength.

INTRODUCTION

Increase biomass on environmental and the adverse effect of non-renewable materials on nature as well as their fast depleting state have stimulated renewed interest in the use of natural fibers. The inherent properties of natural fibers offers quality advantages in engineering applications such as building materials, structural parts for automotive application where heavy weight is not tolerated and medicine (Sinha and Rout, 2007). Some of the advantages of natural fibers over synthetic fibers include availability, low density, light weight, low cost, high toughness, non-corrosive nature, good thermal properties, reduced tool wear, less dermal and respiratory irritation, renewability and less abrasion to processing equipment (Raju et al, 2012). Natural cellulose-based fibers are hydrophilic in nature covered with waxy substances and pectin among others which obstruct the hydroxyl groups from bonding with most binding materials, making them less attractive for reinforcement in polymer composites (El-shekiel et al, 2013). The hydroxyl behavior of fibers causes massive moisture absorption in humid atmosphere resulted in swelling and creation of voids at the fiber-matrix interface with a consequential decline in mechanical properties coupled with dimensional variation of composites (Sanjeevmurthy and Srinivas, 2012).

However, chemical modifications are considered to optimize the interface of fibers by introducing new moieties that can effectively interlock with the matrix (Li et al, 2007). Mercerization, acetylation, silane treatment to mention a few have been employed by several researchers to improve mechanical properties of lignocellulosic fibers (Ramadevi and Sampathkumar, 2012; Cordeiro et al, 2012; Hussain et al, 2011; Reddy et al, 2014; Tanobe et al, 2005; Tlijania et al, 2014; Herrera – Franco and Valadez – Gonzalez, 2005). The treatment conditions have also been modeled with remarkable success and reported in literature (Wang

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et al, 2008; Siva et al, 2013; Ejikeme et al, 2014). Contrarily, failure of chemical treatments to improve the properties of fibers for better efficacy in sustainable applications has been reported (Arsene et al, 2005). Yet, there is need to optimize the chemical treatment process conditions to improve strength of fiber for sustainable applications due to variation in composition and nature, geometric and anisotropic properties in composites.

C. dolichopetalum commonly known as *sun birds wine* have been used for the treatment of urinary tract infection (Gedson et al, 2012), but disposal of its fibers become an nuisance to the environment. From available literature, there is hardly any research work on the optimization of treatment parameters (i.e., concentration and time) of *C. dolichopetalum* fibers. This research work seeks to optimize the mechanical properties (i.e., tensile strength) for the mercerization and acetylation of *C. dolichopetalum* fibers using – LFD of response surface methodology (RSM).

MATERIALS AND METHODS

Materials

C. dolichopetalum plant was obtained from Bayaoje in Surulere Local Government Area of Oyo state, Nigeria. The sodium hydroxide and acetic anhydride used for fiber modification was supplied by Rovert scientific limited, Benin city in Edo state, Nigeria.

Fiber extraction

Method described by Sumaila et al (2013) and Ejikeme et al (2014) was employed for extraction of *C. dolichopetalum* fiber from the plant stem using water retting extraction process in which 30kg of plant stem was retted in deionized water for 21 days, washed every 3days until fiber is produced, selected manually to ensure the absence of defect along the uniform length of 120mm, then sun dried for 7 days and later dried at a temperature of 60^0C for 2 hours. The average tensile strength of *C. dolichopetalum* fiber was measured and recorded as 10.15593MPa.

Alkali treatment

C. dolichopetalum fiber was soaked in various range of concentration and time of NaOH solution at room temperature as presented in the Table 2. The fibers were washed with deionized water until neutral pH was ensured and then, dried in an oven at 60^0C for 2 hours.

Table 1. Fiber treatment conditions of NaOH for coded and uncoded variables

Variable	*Ranges and level*		
	-1	0	1
c (%)	3	9	15
t (minutes)	0	150	300

C is the concentration of NaOH (%) and t is the treatment time (mins)

Acetic anhydride treatment

C. dolichopetalum fiber was soaked in various range of concentration and time of acetic anhydride solution at room temperature as presented in the Table 2. The fibers were washed with deionized water until neutral pH was ensured and then, dried in an oven at 60^0C for 2 hours.

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Table 2: Fiber treatment conditions of acetic anhydride for coded and uncoded variables

Variable	*Ranges and level*		
	-1	0	1
c (%)	3	9	15
t (mins)	0	75	150

C is the concentration of NaOH (%) and t is the treatment time (mins)

Tensile strength test of fiber

C. dolichopetalum fiber strand with apparent defects were discarded and those with greater uniform length of 150mm were adopted for the test (Hu, et al, 2010; Osorio et al, 2012). Tensile strength test was carried out using a Universal Testing Machine Instron 3369 in accordance with ASTM Test Method D638-03 in which fiber strand of 100mm was cut from 150mm with a constant transverse rate of the moving grip of 40 mm /min.

Scanning electron microscope (SEM)

The SEM micrographs of *C. dolichopetalum* fibers and cross section of the treated, NaOH and acetic anhydride treatments were taken. To avoid electron - charging effects, fiber samples were spotter coated with gold.

RESULTS AND DISCUSSION

The response model express tensile strength of mercerized and acetylated fibers as a function of treatment conditions using second order polynomial equation (1) of 3 - LFD.

$$T_i = \beta_0 + \beta_a C + \beta_a t + \beta_b C^2 + \beta_b t^2 + \beta_c Ct + \varepsilon \quad (1)$$

Where T is the tensile property of *C. dolichopetalum* fiber as dependent variables; C and t are the concentration of chemical used and treatment time respectively; ε is the random error; β_0 is intercept term, and β_a, β_b and β_c are the coefficients of linear, quadratic and interaction term respectively. The experimental results were modeled and empirical models were developed as shown in equations (1) and (2) for the tensile strength of mercerized and acetylated *C. dolichopetalum* fiber respectively:

$$T_{SN} = 93.4165 - 4.4856c - 3.8806t + 0.2596c^2 + 0.0476t^2 + 0.0311ct \quad (2)$$

$$T_{SA} = 117.8491 - 7.4110c - 1.7742t + 05021c^2 + 9.4369t^2 + 7.255x10^{-4}ct \quad (3)$$

Table 3 presented the ANOVA of the quadratic regression model of *C. dolichopetalum* fiber treated with NaOH and acetic anhydride treatments. The p-value was used as an instrument to verify the significance of the coefficients and model terms. The significant model terms are *intercept*, *c*, *t*, c^2 and t^2 for the tensile strength of *C. dolichopetalum* fiber treated with NaOH while in the case of *C. dolichopetalum* fiber treated with acetic anhydride of *intercept*, *t*, C^2 *and* t^2 are significant model terms due to $p < 0.05$.

The models for both treatments shows that the experimental data were fit with predicted data due to high magnitude of $R^2 > 0.9$. Though, there is no agreement between the *adj.* R^2 and *pred.* R^2 since their magnitude are not close as expected. However, adequate precision for the response of both treatment conditions shows that the model terms as presented by equations (2) and (3) gave magnitude greater than 4 which indicated that models are adequate and may

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be used in navigating the design applications. The response models were illustrated in Figure 1(a) and 1(b).

Table 3: ANOVA of response surface quadratic model of tensile strength of NaOH and acetic anhydride treated C. dolichopetalum fiber

Source	*Sum of Squares*	*DF*	*Mean Square*	*F Value*	*P> F*	R^2	*Adj.* R^2	*Pred.* R^2	*Adeq Precision*
				C. dolichopetalum fiber treated with NaOH					
Model	9696.2532	5	1939.2506	95.259318	< 0.0001	0.9855	0.9752	0.8931	23.6767
c	201.38785	1	201.38785	9.8925168	0.016260				
t	5579.3469	1	5579.3469	274.06709	< 0.0001				
c^2	1382.2513	1	1382.2513	67.898553	< 0.0001				
t^2	2446.1725	1	2446.1725	120.16019	< 0.0001				
ct	87.094623	1	87.094623	4.2782373	0.077400				
Residual	142.50317	7	20.357595						
Lack of Fit	142.50317	3	47.501055						
Pure Error	0	4	0						
Cor Total	9838.7563	12							
				C. dolichopetalum fiber treated with acetic anhydride					
Model	17316.955	5	3463.3911	16.454632	0.00095338	0.92159	0.86558	0.20531	10.889
c	610.0699	1	610.0699	2.8984528	0.13245				
t	4185.779	1	4185.779	19.88671	0.0029380				
c^2	4738.2781	1	4738.2781	22.511642	0.0020965				
t^2	7782.4021	1	7782.4021	36.974329	0.00050055				
ct	0.4263437	1	0.4263437	0.0020256	0.96536				
Residual	1473.3686	7	210.48123						
Lack of Fit	1473.3686	3	491.12286						
Pure Error	0	4	0						
Cor Total	18790.324	12							

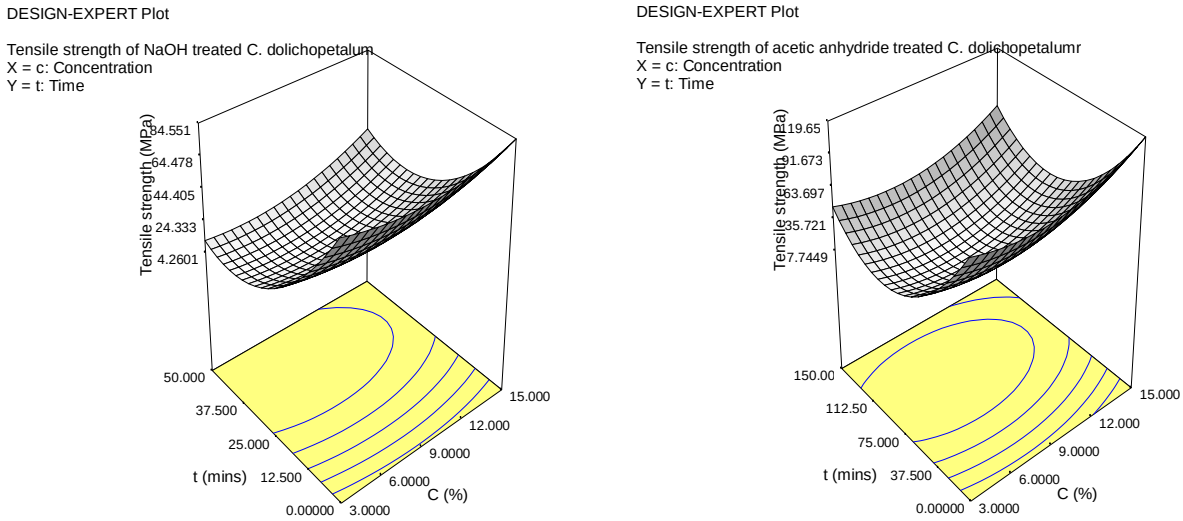

Figure 1: 3 – D plot of response surface of C. dolichopetalum fiber treated with (a) NaOH and (b) acetic anhydride

The results of both treatments were validated as presented in the Table 4 which shows that evaluated error obtained were low in magnitude which indicated effective modification of *C. dolichopetalum* fiber at optimum conditions. More so, the tensile of the untreated *C. dolichopetalum* fiber obtained is 10.15593MPa. The tensile strength of NaOH and acetic anhydride treatment of *C. dolichopetalum* increased by 690.75 and 1003.49% of untreated fibers respectively. This is similar to the report of Kalia et al (2009).

Table 4: Validation of experiments at optimum conditions

	Concentration (%)	*Time (minutes)*	*Tensile strength (MPa)*	*Error (%)*
	C. dolichopetalum with NaOH treatment			
Experimental value	14.188	28.2	80.308	1.96
Predicted value	14.188	28.2	81.878	
	C. dolichopetalum with acetic anhydride treatment			
Experimental value	14.663	86	112.07	
Predicted value	14.663	86	114.62	2.28

SEM Micrographs

Figure 2 shows the morphology of *C. dolichopetalum* fibers of untreated fiber, and NaOH and acetic anhydride treatments with magnification of x880. An irregular surface having variable roughness with porosity of the fibers and their fibrillar structure is revealed from the fiber topography of untreated C. dolichopetalum as shown in Figure 3a. The effect of alkali treatment in modifying surface of fiber was observed by scanning electron microscopy (SEM) as shown in Figure 3(b) in which the NaOH treated fiber has become coarser, cleaner and thinner compared with Figure 3(a) due to increased amorphous cellulose and removal of impurities which is in agreement with report of Ahad et al (2009). However, it was observed that acetylated fiber shrinks with reduced size and clean more than untreated.

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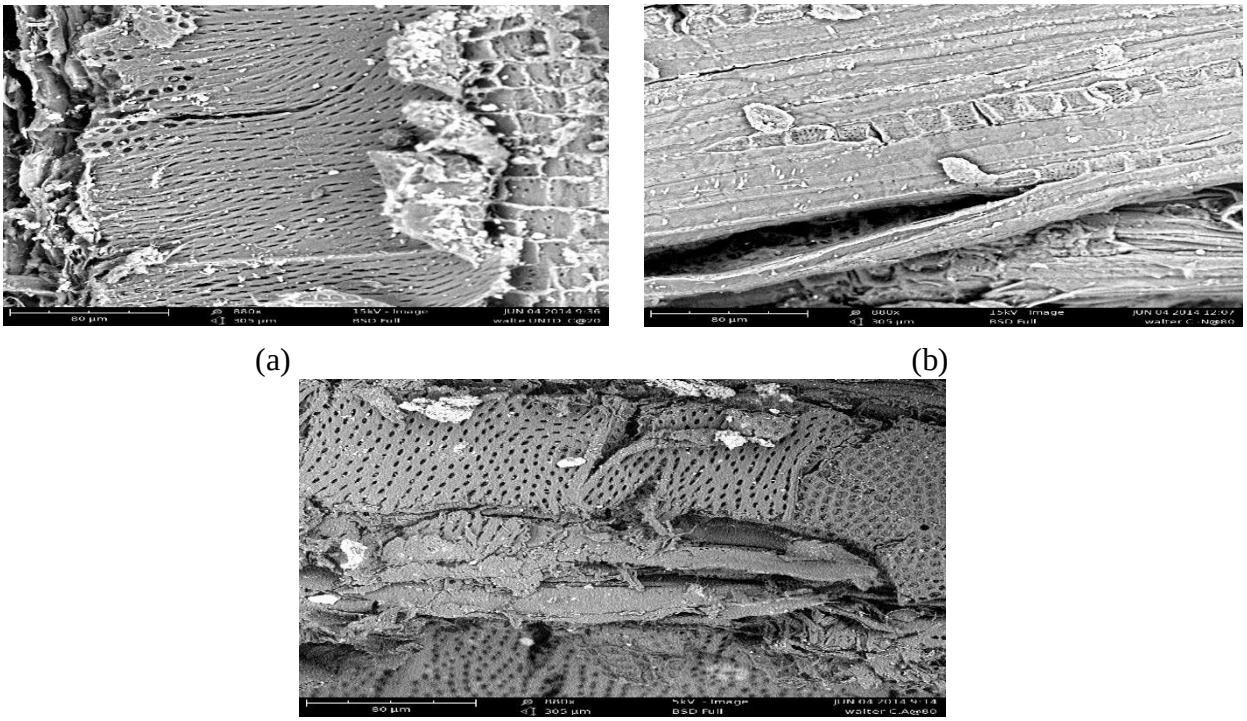

(a) (b)

Figure 3: SEM micrographs of C. dolichopetalum fiber (a) untreated (b) treated with NaOH (c) treated with acetic anhydride

CONCLUSIONS

The surface modification of *C. dolichopetalum* fiber by chemical treatment was optimized. The influence of sodium hydroxide and acetic anhydride concentration and treatment time of the fiber was analyzed using analysis of variance (ANOVA) of RSM with 3 - LFD for development of quadratic models. It was obtained that both sodium hydroxide and acetic anhydride significantly improves the tensile strength of the *C. dolichopetalum* fiber at optimal conditions due to high magnitude of correlation coefficient and adequate precision which indicated that both C. dolichopetalum fibers may be used in design and sustainable applications as load carrying materials. The optimization shows that the suitable process conditions to achieve high strength of *C. dolichopetalum* fiber with surface modification of 14.19 % at 28 minutes and 14.66 % at 86 minutes for respectively.

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REFERENCES

[1]. Kalia, S., Kaith, B. S., & Kaura, I. (2009). Pretreatments of Natural Fibers and their Application as Reinforcing Material in Polymer Composites – A Review. *Polymer Engineering and Science*, *49*: 1253-1272.

[2]. Sinha, E., & Rout, S. K. (2007). Influence of fiber-surface treatment on structural, thermal and mechanical properties of jute fiber and its composites. *Bull. Mater. Sci. 32* (1): 65 – 76.

[3]. Raju, G. U., Gaitonde, V. N., & Kumarappa, S. (2012). Experimental study on optimization of thermal properties of groundnut shell particle reinforced polymer composites, *Int. J. Emerg. Sci., 2*(3): 433-454.

[4]. El-shekiel, Y. A., Sampaun, S. M., Azaman, M. D., & Jawaid, M. (2013). Optimization of blending parameters and fiber size of Kenaf-bast-fiber-reinforced the thermoplastic polyurethane composites by Taguchi method. *Advances in Natural Science and Engineering*, *10*: 1-5.

[5]. Sanjeevmurthy, G. C. & Srinivas, G. R. (2012). Sisa/coconut coir natural fibers-epoxy composites: water absorption and mechanical properties. *International Journal of Engineering and Innovative Technology (IJEIT)*, *2* (3): 166-170.

[6]. Li, X., Tabil, L. G. & Panigrahi, S. (2007). Chemical treatments of natural fiber use in natural fiber-reinforced composites: A review. *J Polym Environ*, *15*: 25-33.

[7]. Ramadevi, P. & Sampathkumar, D. (2012). Effect of alkali treatment on water absorption of single cellulosic abaca fiber. *Bioresources*, *7*(3): 3515-3524.

[8]. Cordeiro, N., Ornelas, M., Ashori, A., Shesma, S. & Norouzi, H. (2012). Investigation on the surface properties of chemically modified natural fibers using inverse gas chromatography. *Journal of Chemical Technology*, *87* (17): 2367-2375.

[9]. Hussain, S. A., Pandurangadu, V. & Palanikumr, K. (2011). Mechanical properties of green coconut fiber reinforced HDPE polymer composite. *International Journal of Engineering Science and Technology (IJEST)*, *3* (11): 7942-7952.

[10]. Reddy T. B. (2014). Mechanical performance of green coconut fiber/HDPE composites by using flexural strength. *International Journal of Mechanical Engineering (IJME)*, *3* (1): 53-66.

[11]. Tanobe, O.A.V., Sydenstricker, T.H.D., Munaro, M. & Amico, S.C. (2005). A comprehensive characterization of chemically treated Brazilian sponge-gourds (*Luffa cylindrical*). *Polymer Testing*, *24*: 474-482.

[12]. Tlijania M., Gouadriab A., benyounesc R., Durastantid Jf. and Mazioude A. (2014). Study and optimization of palm wood mechanical properties by alkalization of the natural fiber. *Global Journal of Science Frontier Research: C Biological Science*, *14* (1): 29 35.

[13]. Harrera – Franco, P. J., & Valadez – Gonzalez, A (2005). A Study of the Mechanical Properties of Short Natural Fiber Reinforced Composites, *Composites Part B: Engineering*, *36* (8): 597 – 608.

[14]. Wang, W., Cai, Z. & Yu, J. (2008). Study on the chemical modification process of jute fiber. *Journal of Engineered Fibers and Fabrics*, *3* (2): 1-11.

ISSN: 2223-9944, e ISSN: 2223-9553

[15]. Siva, I., Joppes, J.T.W., Pitchipoo, P., Amico, S.C., Nagarajan, E.R., & Azhagesan, N. (2013). Gray optimization of process parameters of surface modification of coconut sheath reinforced polymer composites. *J Polymer Eng., 33* (7): 665-672.

[16]. Aly, M., Hashaw, M. S. J., Olabi, A. G., Benyounis, K. Y., Messeiry, M., Hussain, A. I., & Abadir, E. F. (2012). Optimization of alkaline treatment conditions of flax fiber using Box-Behnken method. *Journal of Natural Fibers, 9*: 256-276.

[17]. Ejikeme, P. C. N., Onukwuli, O. D. & Ejikeme, M. E. (2014). Optimization of chemical treatment conditions for *Adenia labata* fiber using CCD. *International Journal of Engineering and Innovative Technology (IJEIT), 4* (6): 23-32.

[18]. Arsene, M. A., Bilba, K., Soboyeyo, A. B. O., & Soboyejo, W. O. (2005). Influence of chemical and thermal treatments on tensile strength of fibers from sugar cane bagasse and banana tree trunk. Inter-American Conference on non-conventional materials and technologies in ecological and sustainable construction (IAC-NOCMAT) - Rio de Janerio, November 11-15th, 2005.

[19]. Gedson, R. M. L., et al. (2012). Bioactivities of the genus combretum (*Combretaceae*): a review. *Journal of Open Access Molecules, 17* (10): 9142-9206.

[20]. Sumaila, M., Amber, I., & Bawa, M. (2013). Effect of fiber length on the physical and mechanical properties of random oriented, nonwoven short banana (musa balbisiana) fiber/epoxy composite. *Asian Journal of Natural and Applied Sciences, 2* (1): 39 – 49.

[21]. Hu, W., Ton-That, M. T., Perrin-Sarazin, F., & Denault, J. (2010). An improved method of single fiber tensile test of natural fiber. *Journal of Polymer Engineering and Sciences, 50*: 819 – 825.

[22]. Osorio, M. C. J., Baracaldo, R. R, & Florez, O. J. J. (2012). The influence of alkali treatment on banana fiber's mechanical properties. *Ingenieria e investigacion, 32* (1): 83 – 87.

[23]. Azeez, T. O., et al. (2013). Optimization of bioremediation of cheese whey with the activity *Klebsiella* pneumonia using response surface methodology. *International Journal for Science and Engineering Technologies with Latest Trends, 11* (1): 10-21.

[24]. Nwabanne, J. T., & Ekwum F. C. (2013). Experimental design methodology applied to bleaching of palm oil using local clay. *International Journal of Applied Science and Technology*, 3(4): 69-77, 2013.

[25]. Ahad, N. A., Parimin, N., Mahmed, N., Ibrahim, S. S., Nizzam, K., & Ho, Y. M. (2009). Effect of chemical treatment on the surface of natural fiber. *Journal of Nuclear and Related Technologies, 6* (1): 155-158.

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