Transylvanian Review

Vol XXVI, No. 29, 2018

Transylvanian Review
Centrul de Studii Transilvane| str. Mihail Kogalniceanu nr. 12-14, et.5, Cluj-Napoca
Email: transylvanianreview@gmail.com
Online Submission System: http://transylvanianreviewjournal.org/

Mercerized Milletia sp Fiber Epoxy Composites: Fiber Loading, Aspect Ratio and Mechanical Properties

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Abstract

The fiber loading capacity and poor adhesion between fibers and matrix remains an area of concern in fiber polymer composites. The fiber loading capacity and aspect ratio of mercerized milletia sp fiber on mechanical properties (tensile strength, tensile modulus, tensile toughness, elongation, flexural and impact strength) of Milletia sp fiber epoxy composites was investigated. *Milletia sp* fiber was extracted using water retting approach. The extracted fiber was mercerized at optimum treatment conditions of 5.84 wt% NaOH and 42.58mins. The epoxy composite samples were prepared using hand layup method for epoxy 103 and amine hardner in a ratio 2: 1 and commingled with untreated and treated fiber, respectively, at optimum treatment conditions. The fiber loading of 10 – 50 wt% and aspect ratio of 42.68 – 213.38 mm/mm were used. The mechanical properties of mercerized milletia sp fiber epoxy composite were determined and optimized using RSM of MATLAB statistical tool box. The tensile strength, tensile modulus, toughness, elongation, flexural strength and impact strength for epoxy resin was 44.2 MPa, 1.9716 GPa, 0.8177 MPa, 3.1%, 85.4 MPa and 360 Kj/m^2, respectively. At optimum aspect ratio and fiber loading, the mercerized milletia sp fiber increased the tensile strength, tensile modulus, toughness, elongation, flexural strength and impact strength of the epoxy resin by 46.38, 77.75, 97.99, 18.81, 125.06 and 217.78%, respectively. Thus, mercerized *Milletia sp* fiber enhanced the mechanical properties of epoxy composites.

Keywords: Milletia sp fiber, epoxy resin, mercerization, mechanical properties.

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Introduction

The widely use of plant fiber as reinforcement of polymeric substrates for many applications such as automotive, marine, military, production of adhesives, ethanol, lactic acid, active carbon and building industry (Chandramohan & Bharanichandar, 2014, Drdlová & Frank, 2016, Kelly, 2002, Maheswari, Reddy, Muzenda & Shukla, 2012) have attracted the attention of the research community. This is not only because it turns out to be an alternative solution to the ever depleting petroleum resources but it stimulated by the weight saving and cost of manufacturing energy-intensive synthetic fibers such as glass, carbon and kevlar (Kalia, Kaith, & Kaur, 2009). However, they do not undergo biodegradation easily, resulting in generation of solid waste with release of hazardous odours (Azeez, Walter, Onukwuli & Menkiti, 2016, Njoku & Obikwelu, 2008). Globally, the problem of developing new and advanced technologies to treat solid waste especially with synthetic fiber reinforced composites at the end of their service time is a serious public health challenge due to intensive cost for decomposition (Asnani, 2006). Moreover, the poor adhesion with polymer matrix and hydrophilic nature of fiber which resulted to low mechanical properties remains major issue in polymer composites after the fiber loading capacity had been exceeded (Isa *et al.*, 2014, Kalia, Sheoran, Mittal, & Kumar, 2013, Njoku & Obikwelu, 2008, Pai & Jagtap, 2015). Several fiber chemical surface techniques have been employed for improvement of strength and durability of polymer composites fiber surface (Adekunle, 2015, Azeez & Onukwuli, 2016, Kalia, Kaith, & Kaur, 2009b, Thiruchitrambalam, Alavudeen, & Venkateshwaran, 2012), but mercerization (treatment with sodium hydroxide "NaOH") technique seems to be widely employed due to availability, cost and efficacy. Researchers have reported the ultimate improvement in mechanical properties of epoxy resin with the modification of existing natural fibers such as hybrid of banana and sisal fiber at 6% NaOH for 2 hours (Chandramohan & Bharanichandar, 2014), commingled Jute and sisal fibers at 5% NaOH for 30 minutes (Gupta & Srivastava, 2016), 5% lycra and jute fiber at 5% NaOH for 8 hours (Harane & Annamalai, 2014), okra/glass fiber at 10% NaOH for 24 hours (Isa *et al.*, 2014) and Napier fiber at 10% NaOH for 2 hours (Kommula, Kanchireddy, Shukla & Marwala, 2013). This may be attributed to increase in interfacial bond strength or adhesion (Gentian, Secara & Kelapa, 2010, Lee & Cho, 2008, Thamae & Baillie, 2007). The improvement in mechanical properties may also be due to penetration of NaOH solution into the crystal lattice of the fiber and substitution of hydrogen atoms of hydroxyl groups of the fibers by sodium atoms and simultaneously removal of amorphous constituents (lignin, hemicellulose, pectin, wax and oils) of the fibers (Azeez & Onukwuli, 2016, Thiruchitrambalam *et al.*, 2012). It has been reported that the favourable mechanical properties of composites are function of treatment conditions and cellulose content of the fibers (Gentian *et al.*, 2010, Kushwaha & Kumar, 2012, Lee & Cho, 2008, Phong, Fujii, Chuong, Viet, & Okubo, 2012). However, availability of fibers is a requirement for its usefulness in polymer applications which necessitate the search for more fibers and efficacy. *Milletia sp* plant is source of milletia sp fiber, abundant in the tropical rain forest especially in the South eastern part of Nigeria and used as ropes and mats but its applications in polymer composites have not been reported. This work is aimed at studying the effect of fiber loading and aspect ratio of mercerized *Milletia sp* fiber on mechanical properties (tensile strength, modulus, elongation, flexural and impact strength) of *Milletia sp* fiber - epoxy composites.

Materials and Methods

Epoxy base resin of epoxide family 103 and amine hardener 301 (poly aniline) are commercial grade and obtained from NYCIL Nigeria Limited. NaOH used is analytical grade chemical obtained from Chemisciences Nigeria Limited. The *Milletia sp* plant, the source of *Milletia sp* fiber was obtained Amakohia in Owerri, Imo state, Nigeria.

Milletia sp Fiber Extraction and Cellulose Content

The water retting method described by Azeez *et al.* (2016) and Phong *et al.* (2012) was used. The epicarp of the bark of matured stems of *Milletia sp* plant was scrapped after disentangled from the core sticks. The 30 kg of bark of *Milletia sp* plant was immersed in 20 liters of deionized water, washed every three days for four weeks and then, sun dried for two weeks after the fiber have been extracted.

Mercerization (Treatment with NaOH) of Milletia Fiber

Milletia sp fiber was mercerized as described by Suardana, Piao, & Lim (2011) after cut to length of 10 -30mm using 5.84 wt% NaOH for 43 minutes based on preliminary batch optimization treatment of fiber, washed until a pH of 7± 0.2 was achieved and then, sun dried. The fiber diameter was measured at five different points using micrometer screw gauge and the average diameter was evaluated. The aspect ratio was determined using equation 1 as reported by Dedeepya, Raju & Kumar (2012),

$$A = \frac{l_f}{d_f} \qquad (1)$$

Where A, l_f and d_f are fiber aspect ratio, length and diameter, respectively.

Preparation and Mechanical Properties of Milletia Fiber - Epoxy Composites

Randomly oriented *Milletia sp* fiber - epoxy composites containing fiber of varying aspect ratio (fiber length) and fiber volume fractions as presented in Table 1 were prepared by hand lay-up method as described by Gupta & Srivastava. (2016) using a stainless steel sheet female mould and a marble tile male mould having with dimension of 300 x 300 x 30 mm^3. Prior to the composite preparation, the mould was polished with a releasing agent (mirror glaze) and epoxy base resin was thoroughly mixed with amine hardener 301 in the ratio of 2: 1 by weight at room temperature as recommended by the suppliers. The mould was pressed in a hydraulic press at a pressure of 20 kg/m^2 at room temperature. During curing, the excess resin was allowed to flow out as "Flash". Tensile and flexural tests were conducted on the samples using a Hounsfield Monsanto Universal Tensometer Machine at a constant rate of traverse of the moving grip of 5mm min^{-1} for randomly oriented fiber composites with a dimension of 160 x 20 x 3 mm^3 in accordance with ASTM D 638 – 99 standard and 200mm x 20 mm x 3 mm^3 in accordance with ASTM D 780-99respectively. Tensile strength, tensile modulus, toughness and elongation were determined from stress strain plot of tensile test. Flexural strength was determined by equation 2 as reported by Olaitan *et al.* (2013),

$$F_s = \frac{3PL}{2bd^2} \quad (2)$$

Where P is the load on test specimen, L is the support span, b is the width of specimen and d is the thickness of the specimen. Unnotched Izod impact test was conducted on samples with a dimension of 130 x 10 x 10 mm^3 in accordance with ASTM D 256-97 standard in which the measured height of fall of a sharp object of 1.2 kg mass to shatter the test specimen was determined. The impact strength was measured using equation 3 as reported by Atuanya, Olaitan, Akagu & Onukwuli (2013),

$$I = \frac{E_{ab}}{A} \quad (3)$$

Where I is the impact strength, E_{ab} = mgh, E_{ab} is the energy absorbed in Joules, m is the mass of sharp object, g is the acceleration due to gravity m/s^2, h is the height of the sharp object to break the test sample and A is the area of impact.

Table 2.1: Preparation of mercerized *Milletia sp* fiber - epoxy composites

Composite sample	Fiber length (mm)	Fiber aspect ratio (mm/mm)	Weight fraction (wt %)
1	10	42.6767	10
2	30	128.03	10
3	50	213.3834	10
4	10	42.6767	30
5	30	128.03	30
6	50	213.3834	30
7	10	42.6767	50
8	30	128.03	50
9	50	213.3834	50

d_f of NaOH treated fiber is 0.0234 mm

Modelling and Statistical Analysis

The response surface methodology (RSM) of MATLAB statistical tool Box was used to model and optimize the mechanical properties of *Milletia sp* fiber - epoxy composites. The statistical parameters was determined using analysis of variance (ANOVA) with inferior p - value of 0.05 considered to be significant. The composites mechanical property was modelled based on input variables (weight fraction and aspect ratio) using response equation 4:

$$Y_i = a_0 + a_1x_1 + a_2x_2 + a_3x_1x_2 + a_4x_1^2 + a_5x_2^2 \quad (4)$$

Where Y_i is the response or output variable (tensile strength, modulus, toughness, elongation, flexural strength and impact strength), x_1 and x_2 are aspect ratio and volume fraction (fiber loading), respectively, while a_0, a_1, a_2, a_3, a_4 and a_5 are coefficients of the models determined by the RSM.

Results

The mechanical responses of mercerized milletia sp fiber epoxy composites based on the fiber loading capacity and aspect ratio are presented in Table 2.

Table 2: Mechanical properties of *Milletia sp* fiber - epoxy composites

X_1 (mm/mm)	X_2 (wt %)	T_s (MPa)	T_m (GPa)	T (MPa)	E (%)	F_s (MPa)	I (KJ/m^2)
0	0	44.2	1.9716	0.8177	3.1	85.4	360
42.6767	10	42.3000	2.4974	0.6299	2.5000	81.0667	840
128.03	10	42.8000	2.2864	0.6796	2.7000	77.6667	888
213.3834	10	43.3000	2.0975	0.7416	2.9000	76.8667	900
42.6767	30	52.5000	3.3427	1.1628	3.2000	173.3333	984

128.03	30	54.0000	3.3397	1.2518	3.3000	185.5333	1020
213.3834	30	55.8000	3.2534	1.3188	3.4000	181.8667	1056
42.6767	50	58.5000	3.3943	1.4302	3.5000	157.0000	1080
128.03	50	61.8000	3.3768	1.5283	3.6000	171.3333	1128
213.3834	50	64.0000	3.3441	1.6184	3.7000	164.2000	1140

Discussion

The average diameter of treated *Milletia sp* fiber obtained is 0.0234 mm which is 22.43% reduction in average diameter of untreated fiber. Table 2 shows the mechanical properties of *Milletia sp* fiber - epoxy composites based on the aspect ratio and volume fraction loading of the fiber. It can be deduced that tensile strength, toughness and impact strength increases with increased aspect ratio and volume fraction of the fiber. The increase in tensile strength, toughness and impact strength of the mercerized *Milletia sp* fiber - epoxy composites may be attributed to good adhesion and wettability of the mercerized fiber and epoxy resin, even distribution of fiber loading, fiber aspect ratio and effectiveness of the mercerization which altered the bonding topography, interfacial adhesion and interlocking between the fiber and resin. This is in agreement with report of researchers (Azeez & Onukwuli, 2017, Cao, Sakamoto, & Goda, 2007, Thiruchitrambalam *et al*., 2012). Tensile modulus is a measure of the stiffness of a composite or a measure of the extent to which a composite resist deformation in a given strain region. Initially, the increase in tensile modulus was observed but later decreased with increased aspect ratio. It can be observed that tensile modulus increased with increase in fiber loading from 10 - 50% fiber at lower aspect ratio of 42.6767 mm/mm. The decrease in tensile modulus of the composites with increased aspect ratio at 30 – 50 % fiber loading may be attributed to unfavorable length of the fiber in the epoxy resin poor stiffness with increased fiber. Elongation is a measure of ductility of a composite. A ductile composite after its elastic limit is exceeded, elongates continuously under increasing loads suffering plastic deformation until the breaking point. It can be observed that elongation reduces with aspect ratio at lower fiber loading of 10% but later increases. The initial reduction in elongation may be attributed to uneven distribution of the fiber which caused uneven stress transfer. This is similar to the report of Atuanya *et al.* (2013). At higher fiber loading, elongation of the mercerized *Milletia sp* fiber - epoxy composites increases with increased aspect ratio. This indicated that elongation of the composites increases with increased aspect ratio. Flexural strength of the composites reduced with increased aspect ratio at lower fiber loading of 10% but increases with increased aspect ratio up to 128.03 mm/mm at 30 and 50% fiber loading and then, reduced at aspect ratio above 128.03 mm/mm. The equation (5) – (10) represent the reduced response surface models of MATLAB tool box software for tensile strength, tensile modulus, toughness, elongation, flexural strength and impact strength, respectively. The fitness of the model can be determined by the R^2 value which indicated the observed variability of the responses. The reduced response models were obtained due to truncation of insignificant model terms or variables (> 0.05) as shown in Table 3. The R^2 values of 0.9997, 0.9969, 0.9999, 0.9976, 0.9962 and 0.9980 represent 99.97, 99.69, 99.99, 99.76, 99.62 and 99.8% observed variability of the composites tensile strength, tensile modulus, toughness, elongation, flexural strength and impact strength, respectively, based on the aspect ratio and loading capacity of the fiber in epoxy resin with residue of 0.03, 0.31, 0.01, 0.24, 0.038 and 0.2%. The adjusted R^2 value revealed the correlation of the observed variability based on addition factors of quadratic of variables and interaction between the variables. It can be observed that the difference in R^2 value and adjusted R^2 value are closer and less than 0.2. This indicated that the models can be used for mercerized Milletia sp fiber - epoxy composites in any applications since the difference between R^2 and adj. R^2 less than 0.2. This is in agreement with the report of Dufour (2011).

$$T_s = 35.5326 + 0.6790x_2 + 0.0007x_1x_2 - 0.005x_2^2 \quad (5)$$

$$T_m = 1.7367 + 0.0923x_2 + 5.12e^{-5}x_1x_2 - 0.0012x_2^2 \quad (6)$$

$$T = 0.2229 + 0.0007x_1 + 0.0406x_2 + 1.12e^{-5}x_1x_2 - 0.0003x_2^2 \quad (7)$$

$$E = 1.975 + 0.0488x_2 - 0.0004x_2^2 \quad (8)$$

$$F_s = 10.7607x_2 - 0.1472x_2^2 \quad (9)$$

$$I = 719 + 0.7967x_1 + 9.6x_2 - 0.06x_2^2 \quad (10)$$

Table 3: Statistics analysis for mechanical properties of *Milletia sp* fiber - epoxy composites

Variable	T_s		T_m		T		E		F_s		I	
	Coeff	P-value	Coeff	P-value	Coeff	P-value	Coeff	P-value	Coeff	P-value	Coeff	P-value
Constant	35.5326	7.32e-6	1.7367	0.0005	0.2229	0.0004	1.9750	0.0001	-22.1254	0.1221	719.000	3.024e-5
X_1	0.0041	0.5826	-0.0021	0.1987	0.0007	0.0275	0.0024	0.0718	0.1759	0.2655	0.7967	0.0339
X_2	0.6790	0.0002	0.0923	0.0005	0.0406	1.11e-5	0.0488	0.0010	10.7607	0.0003	9.6000	0.0018
$X_1 X_2$	0.0007	0.0028	5.12e-5	0.0369	1.120e-5	0.0082	-2.93e-5	0.0577	0.0017	0.3212	-2.67e-17	1.0000
x_1^2	-1.83e-5	0.4996	-1.75e-6	0.7358	-4.05e-7	0.5426	-1.07e-11	1.0000	-0.0008	0.1872	-0.0016	0.1240
x_2^2	-0.0050	0.0015	-0.0012	0.0008	-0.0003	6.43e-5	-0.0004	0.0079	-0.1472	0.0004	-0.0600	0.0240
Model	R^2 = 0.9997, Adj.R^2 = 0.9991, P-val = 2.0425e-5		R^2 = 0.9969, Adj.R^2 =0.9917, P-val =0.00058		R^2 = 0.9999, Adj.R^2 = 0.9997, P-val =3.2831e-6		R^2 = 0.9976, Adj.R^2 =0.9936, P-val =0.0004		R^2 = 0.9962, Adj.R^2 =0.9898, P-val =0.0008		R^2 = 0.9980, Adj.R^2=0.9946, P-val = 0.0003	

Moreover, it can be deduced that the tensile strength and modulus of mercerized *Milletia sp* fiber - epoxy composites significantly increased with increase in quadratic of fiber loading and interaction with favorable fiber aspect ratio. This indicated that the increase in tensile strength and modulus of the composites, respectively, depends on interaction of fiber aspect ratio and fiber volume fraction (fiber loading), and quadratic of fiber aspect ratio. This is in agreement with the report of Moses, Simon & Maxwell (2013). For the case of toughness of the composites, the increase in aspect ratio, fiber loading and interaction of both aspect ratio and fiber loading significantly determined the increase in toughness of the composites. Elongation and flexural strength significantly increased by the linear fiber loading, while the increase in impact strength depends on increase in aspect ratio and quadratic of fiber loading. Figure 1 shows the response surface plot of tensile strength, tensile modulus, toughness, elongation, flexural strength and impact strength mercerized *Milletia sp* fiber - epoxy composites. From Figure 1(a), (c) and (d), the optimum tensile strength, toughness and elongation of 64.7 MPa, 1.619 MPa and 3.683% for mercerized *Milletia sp* fiber - epoxy composites which is 46.38, 97.99 and 18.81% more than that of epoxy resin, respectively, was obtained at aspect ratio of 212.5 mm/mm and fiber loading of 50%. The improvement in tensile properties of epoxy resin is more effective when using mercerized *Milletia sp* fiber than that of using mercerized Jute fiber lycra (Harane & Annamalai, 2014) and hybridized Okra /glass fiber treated with 5 and 10% NaOH (Isa *et al.*, 2014). The optimum tensile modulus of 3.5046 GPa at aspect ratio of 42.5 mm/mm and 40% volume fraction (fiber loading) was obtained for mercerized *Milletia sp* fiber - epoxy composites amounted to 77.75% more than the tensile modulus of epoxy resin (see Figure 1b). The optimum flexural strength of the composites of 192.2 MPa amounted to 125.06% more than flexural strength of epoxy resin at aspect ratio of 145.1mm/mm and volume fraction of 38%. The effectiveness of the mercerized *Milletia sp* fiber on flexural strength was found to be more than that of the combination of mercerized banana and palmyra fibers reinforced epoxy composites as reported by Prasanna, Shivananda, & Subbaiah (2011). The optimum impact strength of 1144 KJ/m^2 was obtained which is 217.78% more than impact strength of the epoxy resin at 213.5 mm/mm of aspect ratio and 50% volume fraction as shown in Figure 3(f). The increased in impact strength may be attributed to increased stress transfer and interfacial adhesion between the fiber and resin as reported by (Prasanna, Subbaiah & Rajulu, 2012). It can be deduced that mercerized Milletia sp fiber is better and effective compared to fiber of carbon (Drdlová & Frank, 2016), metallic (Viswanatha, Kumar, Basavarajappa, & Kiran, 2013), jute and sisal (Gupta & Srivastava, 2016), roselle, banana and palmyra (Prasanna *et al.*, 2011) for enhancement of the mechanical properties of epoxy resin in many applications such as automotive part, door panels, dashboards, mobile cases and laptop cases (Harane & Annamalai, 2014).

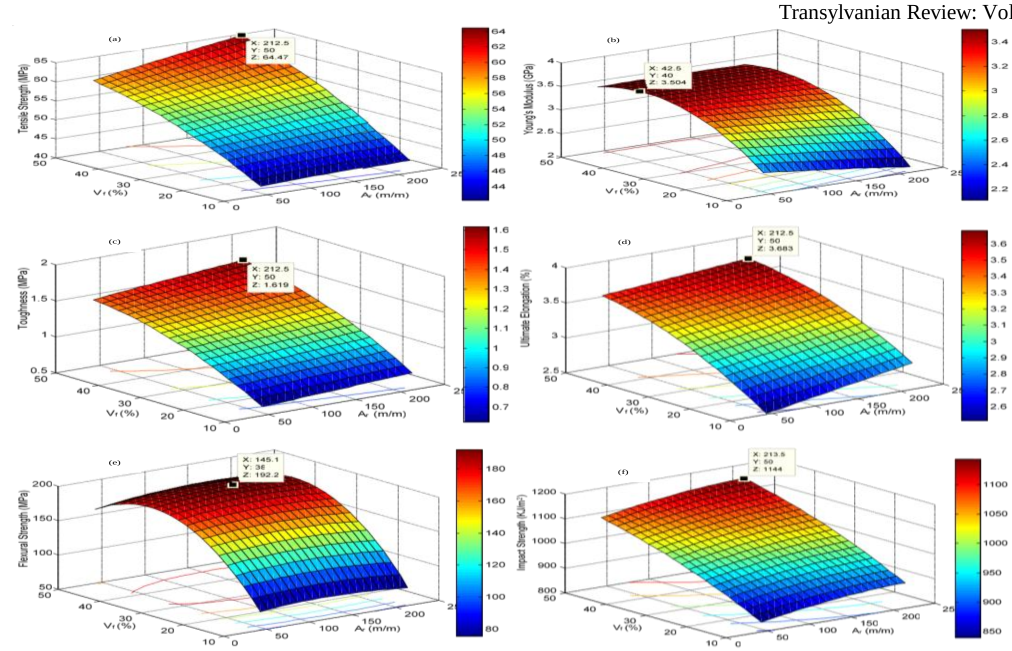

Figure 1: Response surface plot for mechanical properties of *Milletia sp* fiber - epoxy composites: (a) Tensile strength (b) Tensile modulus (c) Toughness (d) Ultimate elongation (e) Flexural strength (f) impact strength.

Conclusion

The results produced from this work have shown that mercerization significantly enhanced the mechanical properties (Tensile strength, tensile modulus, tensile toughness, tensile elongation, flexural strength and impact strength) of *Milletia sp* fiber - epoxy composites. Apart from effect of chemical treatment, the aspect ratio and fiber loading significantly improved the mechanical properties of epoxy composites through improvement in stress transfer and interfacial adhesion between mercerized *Milletia sp* fiber and epoxy resin. Hence, this make the mercerized *Milletia sp* fiber to be more effective than mercerized banana and palmyra hybridized fiber, jute fiber lycra, carbon and metallic fiber for epoxy composite applications.

Acknowledgement

The authors acknowledged the support of Federal University of Technology, Owerri in Nigeria.

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