MERCERIZATION PARAMETERS EFFECT ON NATURAL FIBER REINFORCED POLYMER MATRIX COMPOSITE: A REVIEW

Yussni, M.H.[1], Nazrul, M.R.[1], Azriszul,M.A.[1], Mujahid, A.A.Z. [2] , and Saparudin, A.

[1]Faculty of Mechanical and Manufacturing Engineering,
Universiti Tun Hussein Onn Malaysia,
86400 Parit Raja, Batu Pahat, Johor, Malaysia

[2]Faculty of Engineering, National Defense University of Malaysia,
57000 Kuala Lumpur, Malaysia

Author's email: yusni@uthm.edu.my

ABSTRACT: Environmental awareness and depletion of the petroleum resources are among vital factors that motivate a number of researchers to explore the potential of reusing natural fiber as an alternative composite material in industries such as packaging, automotive and building constructions. Natural fibers are available in abundance, low cost, lightweight polymer composite and most importance its biodegradability features, which often called "eco-friendly" materials. However, their applications are still limited due to several factors like moisture absorption, poor wettability and large scattering in mechanical properties. Among the main challenges on natural fibers reinforced matrices composite is their inclination to entangle and form fibers agglomerates during processing due to fiber-fiber interaction. This tends to prevent better dispersion of the fibers into the matrix, resulting in poor interfacial adhesion between the hydrophobic matrix and the hydrophilic reinforced natural fiber. Therefore, to overcome this challenge, fiber treatment process is one common alternative that can be use to modify the fiber surface topology by chemically, physically or mechanically technique. Nevertheless, this paper attempt to focus on the effect of mercerization treatment on mechanical properties enhancement of natural fiber reinforced composite or so-called bio composite. It specifically discussed on mercerization conditions, and natural fiber reinforced composite mechanical properties enhancement.

KEYWORDS: Mercerization treatment, mechanical properties, natural fiber and bio composite.

1.0 INTRODUCTION

The use of natural fibers as reinforced material in composite manufacturing was incredibly increased due to environmental awareness, depletion of non-renewable petroleum resource and growing demand on sustainable product development (Bledzki & Gassan, 1999; Dweib, Hu, O'Donnell, Shenton, & Wool, 2004; Graupner, Herrmann, & Müssig, 2009). A tremendous number of publications over the past two decades have agreed that natural fibers give competent potential to be used as an alternate for glass or other's man made synthetic fiber reinforcement materials in composites (Eichhorn, *et al.*, 2010; Eichhorn, *et al.*, 2001; Kalia, *et al.*, 2011; Ku, Wang, Pattarachaiyakoop, & Trada, 2011; La Mantia & Morreale, 2011; Summerscales, Dissanayake, Virk, & Hall, 2010). The applications is diversified into engineering end uses mainly for non structural applications such as in interior lining for automotive component (Davoodi, *et al.,* 2010; Holbery & Houston, 2006), packaging materials (Chaudhary, Borkar, & Mantha, 2010), insulation (Kymäläinen & Sjöberg, 2008; Zhou, Zheng, Li, & Lu, 2010), acoustic absorption panel (Hosseini Fouladi, Nor, Ayub, & Leman, 2010; Koenig, Muller, & Thoben, 2008) and building materials (Elsaid, Dawood, Seracino, & Bobko, 2011; Rodríguez, *et al.,* 2011). These eco friendly materials have several interesting properties, which make it an attractive and comparable to traditional synthetic reinforced material. Some of natural fiber advantages are abundantly available, inexpensive, low density, high specific stiffness and strength, lightweight, desirable fiber aspect ratio, minimal health hazards, non-abrasive, outstanding insulation properties, enhanced energy recovery, renewability and biodegradability (John & Anandjiwala, 2008; Joshi, Drzal, Mohanty, & Arora, 2004; Mwaikambo, 2006; Satyanarayana, Arizaga, & Wypych, 2009). However, although natural fiber reinforced composite seem to give a promising benefit as compare to synthetic fiber, there are still several critical issues need to be addressed before manufacturing industry gains full confidence to enable wide-scale acceptance of this material in a global market. The shape, size and strength of the natural plant fibers may vary widely depending on cultivation environment, geographical origin, plant maturity, retting technique, and composite manufacturing process (Hughes, 2011; Shinji, 2008).

The numerous highlight problem in dealing with natural fiber was it hydrophilic nature, which leads to an adhesion problem with hydrophobic nature of the polymer matrix (Sgriccia, Hawley, & Misra, 2008). Hydrophilic character of natural fibers is incompatible with hydrophobic polymer matrix and has a tendency to form aggregates. Furthermore, it exhibit poor resistant to moisture, which lead to high water absorption, subsequently resulting in poor mechanical properties and dimensional stability of the natural fiber reinforced composites John, Francis, Varughese, & Thomas, 2008; Sreekumar, *et al.*, 2009). Therefore, chemical modification either on natural fiber, polymer matrix or both materials is an alternative solution to overcome these challenges (De Rosa, *et al.*, 2011; Kalia, Kaith, & Kaur, 2009; Li, Tabil, & Panigrahi, 2007; Vilay, Mariatti, Mat Taib, & Todo, 2008; Xie, Hill, Xiao, Militz, & Mai, 2010). The chemical modification is attempted to improve natural fiber hydrophobic nature, interfacial bonding between matrix and fiber, surface roughness and wettability, and also decrease moisture absorption, leading to the enhancement of mechanical properties of the natural fiber reinforced composites (George, Sreekala, & Thomas, 2001; Ku, *et al.*, 2011).

Mercerization is a common fiber treatment that extensively used by the number of researcher (Akil, *et al.*, 2011; Bachtiar, Sapuan, & Hamdan, 2008; Cho, Kim, Song, & Hong, 2011; Islam, Pickering, & Foreman, 2010). However, for various types of fiber, distinct mercerization treatment conditions were used, and very limited paper took a partial review on these condition interaction effects on fiber and it composite properties enhancement. Thus, the aim of this paper is to briefly review the mercerization parameter's effect on natural fiber and it composite mechanical properties enhancement. The mercerization parameters that will be stress in this paper are sodium hydroxide (NaOH) concentration, temperature and soaking duration. This paper also attempts to discuss the interaction of mercerization parameters interaction toward mechanical properties enhancement of natural fiber reinforce composite. The next section of this paper will briefly describe the mercerization treatment. Section three discussed the common mercerization treatment conditions. The following section is about properties enhancement via mercerization treatment. The conclusion of this work and the recommendation for future work will be highlighted at conclusion section.

2.0 MERCERIZATION TREATMENT

Mercerization is an alkali treatment process. It is widely used in textile industry (Wang, Postle, & Kessler, 2003). The standard definition of mercerization as proposed by ASTM D1965 is: the process of subjecting a vegetable fiber to an interaction with a fairly concentrated aqueous solution of strong base, to produce great swelling with resultant changes in the fine structure, dimension, morphology and mechanical properties (Bledzki & Gassan, 1999). Therefore, mercerization is a chemical modification process that changed the chemical constituent behavior in natural fiber. The effect of alkali on cellulose fiber is a swelling reaction, during which the natural crystalline structure of the cellulose relaxes. The schematic illustration of the swelling process in cellulose is shown in Figure 1 (Leonard Y. Mwaikambo & Ansell, 2002). Native cellulose (i.e. cellulose as it occurs in nature) shows a monoclinic crystalline lattice of cellulose-I, which can be changed into different polymorphous forms through chemical or thermal treatments. The important forms of alkali-cellulose and cellulose-II are shown in Figure 2 (John & Anandjiwala, 2008).

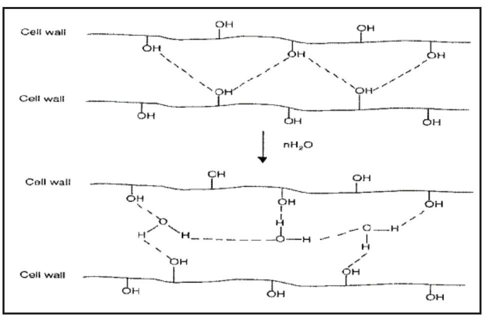

Figure 1: Schematic illustration of the swelling.

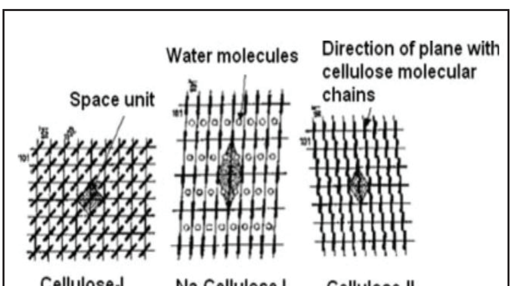

Figure 2: Lattice structure of cellulose I and cellulose II (John, M. & Anandjiwala, 2008) process in cellulose (Leonard, Y. M., & Ansell, 2002).

The type of alkali and its concentration will influence the degree of swelling, and hence the degree of lattice transformation into cellulose-II. It has been reported that Na^+ has got a favorable diameter, able to widen the smallest pores in between the lattice planes and penetrate into them. Consequently, sodium hydroxide (NaOH) treatment results in a higher amount of swelling. This leads to the formation of new Na–cellulose-I lattice, a lattice with relatively large distances between the cellulose molecules, and these spaces are filled with H_2O molecules. In this structure, the OH-groups of the cellulose are converted into O – Na-groups, expanding the dimensions of molecules. Subsequent rinsing with water will remove the linked Na-ions and convert the cellulose to a new crystalline structure, i.e. cellulose-II, which is thermodynamically more stable than cellulose-I. NaOH can cause a complete lattice transformation from cellulose-I to cellulose-II. Addition of aqueous sodium hydroxide (NaOH) to natural fiber promotes the ionization of the hydroxyl group to the alkoxide. The following reaction takes place as a result of alkali treatment (Li, *et al.*, 2007; Leonard Y. Mwaikambo & Ansell, 2002; Sreenivasan, Iyer, & Iyer, 1996):

$$\text{Fiber} - \text{OH} + \text{NaOH} \rightarrow \text{Fiber} - \text{O} - \text{Na} + H_2O \quad (1)$$

As reported in much literature, natural fiber chemical constituent is consisted of cellulose and other non cellulose constituent like hemicellulose, lignin, pectin and impurities such as wax, ash and natural oil (Abdul Khalil, Yusra, Bhat, & Jawaid, 2010; Khalil, Alwani, & Omar, 2006). This non cellulose material could be removed by appropriate alkali treatments, which affect the tensile characteristic of the fiber (Gassan & Bledzki, 1999; Sreenivasan, *et al.*, 1996). Mercerization was found to change fiber surface topography, and the fiber diameter was reported to be decreased with increased concentration of sodium hydroxide concentration(L.Y Mwaikambo & Ansell, 2006). Mercerization treatment also results in surface modifications leading to increase wettability of coir fiber polyester resin as reported by Prasad, *et al.* (Prasad, Pavithran, & Rohatgi, 1983). It is reported in that alkaline treatment has two effects on the henequen fiber: (1) it increases surface roughness, resulting in better mechanical interlocking; and (2) it increases the amount of cellulose exposed on the fiber surface, thus increasing the number of possible reaction sites (Valadez-Gonzalez, Cervantes-Uc, Olayo, & Herrera-Franco, 1999). Consequently, alkaline treatment has a lasting effect on the mechanical behavior of natural fibers, especially on their strength and stiffness.

3.0 TREATMENT CONDITIONS

An excellent discussion of mercerization effects and it observed behavior was reported by Symington, *et al.* (Symington, Banks, West, & Pethrick, 2009). They highlight two important mercerization treatments, which are NaOH concentration and processing time could increase 30% in interfacial strength in composite with a proper pre treatment process. The general concentration ranges of NaOH seem to be 1-25% and processing time of 1-60 minute. However, in real application, due to chemical constituent variation among similar types or different types of natural fiber, optimal mercerization conditions like NaOH concentration, time and temperature will vary for each fiber. Norazawa et. al study the effect of alkali treatment on kenaf –Ecoflex composites mechanical properties. The composite was prepared to use different fiber loading, and the fiber was treated with various concentrations of NaOH solution. Their found that 40% fiber loading improved the tensile strength properties and whole stem kenaf fiber treated with 4% NaOH was found to enhance composite tensile and flexural properties compared with untreated fiber (Nor Azowa Ibrahim, Kamarul Arifin Hadithon, & Abdan, 2010). Umar, *et al.* reported that 4% NaOH could clean the impurities and make a Betelnut fiber surface a bit rougher (Nirmal, Singh, Hashim, Lau, & Jamil, 2011). When NaOH concentration increased to 6%, their found that Betelnut fiber became totally free from any impurities.

Table 1 expressed a literature summary stress on mercerization treatment parameters focused on NaOH concentration, processing temperature and soaking duration. From Table 1, it shows number of research was conducted to determine the optimum NaOH solution concentration during the mercerization process at specific soaking duration and/or soaking temperature. However, there are limited work reported regarding attempts to determine the main effect and the interaction between these three factors on it effect to natural fiber and its final composite mechanical properties performance.

Table 1: Gathered common mercerization treatment parameters from literature

Natural Fiber	Matrix	NaOH Treatment Parameters			Effect/Comments	Ref.
		Concentration %	Temp. (^{0}C)	Duration		
Pineapple, sisal	Polyester	5 & 10 %	30	1 h	At 10% NaOH, excess delignification occurred. Thus, fiber become weaker	(Mishra *et al.*, 2003)
Sisal	Polyester	0.25,0.5,1.0, 2.0, 5.0 & 10% w/w	Room temperature	1 h	NaOH treatment decrease fiber density. 10% treatment results a rougher surface than untreated	(Sydenstricker, Mochnaz, & Amico, 2003)
Hemp & kenaf	Polyester	6 %	19 ± 2	48 h	Cell wall densification identification from small positive change in fiber density observation	(Aziz & Ansell, 2004)
Curaua	Biodegradable resin	5, 10 & 15wt %	Room temperature	1 h & 2 h	Decrease of fiber diameter, fiber weight, fiber density and tensile strength with increasing NaOH content	(A Gomes, Goda, & Ohgi, 2004)
Henequen	HDPE	2% w/v	25	1 h	Surface modification increase the area of contact and further expose the cellulose microfibril. Thus, improve fiber wetting and impregnation	(Herrera-Franco & Valadez-González, 2005)
Kenaf	-	3, 6 & 9%	Room temperature and 95^{0}C for 6% NaOH	3 h	3% NaOH was ineffective to remove impurities on fiber surface, 9% NaOH show cleanest fiber surface	(Edeerozey, Akil, Azhar, & Ariffin, 2007)

Pineapple	-	2% w/v	95	2 h	Decrease in tensile strength and Young modulus were probably due to decrease in the degree of crystallinity and crystallite orientation	(Munawar, Umemura, Tanaka, & Kawai, 2008)
Kenaf, Flax & Hemp	Epoxy	5%	Room temperature	1 h	NaOH treated kenaf and hemp composite absorb more water then silane only or alkali and silane treated samples	(Sgriccia *et al.*, 2008)
Sisal & Oil palm	Natural rubber	0.5, 1, 2 & 4%	Ambient temperature	1 h	Alkali treated composite exhibited better tensile properties than silane treated composite	(M. J. John *et al.*, 2008)
Coir	Polypropylene	2, 4, 6, 8 & 10%	Room temperature	4 week	Denser NaOH solution provided more Na^+ and OH^- ions to react with the substances on the fiber, causing greater amount of lignin, pectin, fatty acid and the cellulose to leach out, this would be detrimental to the fiber strength.	(Gu, 2009)
Ramie	PLA	5% w/v	Room temperature	3 h	Alkali treated composite exhibited better tensile properties than silane treated composite	(Yu, Ren, Li, Yuan, & Li, 2010)
Hemp	PLA	5%	Ambient temperature	30 min	Show higher tensile strength (75.5MPa) and Young modulus (8.2GPa)	(Sawpan, Pickering, & Fernyhough, 2011)

4.0 PROPERTIES ENHANCEMENT

When dealing with natural fiber reinforce composite, there always have two stages of performance evaluation. First is the natural fiber characteristic evaluation (Aslan, Chinga-Carrasco, Sørensen, & Madsen, 2011; Defoirdt, *et al.,* 2010) and secondly is the fabricated composite evaluation (Asasutjarit, Charoenvai, Hirunlabh, & Khedari, 2009). Most of the literature were focused on both stages which considering some modification on the fiber (Bettini, *et al.,* 2010) or polymer matrix(Rassmann, Paskaramoorthy, & Reid, 2011) and finally evaluate the final composite performance which generally fabricated with variation of fiber matrix mixing ratio. There are tremendous research conducted to evaluate performance of natural fiber and it reinforced composite (Alawar, Hamed, & Al-Kaabi, 2009; M. John & Anandjiwala, 2008; Rodríguez, *et al.,* 2011; Sgriccia, *et al.,* 2008; Shinji, 2008; Shinoj, Visvanathan, & Panigrahi, 2010). This performance evaluation is mostly depending on composite characterization determination and presented in terms of physical, mechanical and thermal properties. These characteristics are important to determine material ability, especially under extreme and critical conditions, which are directly connected with engineering performance. In this short review, the focused was zoomed in mercerization parameters effect on natural fiber physical properties and its reinforced composite mechanical properties. The mercerization effect on common mechanical properties of several natural fibers and it composite commonly was highlight in Table 2.

Table 2: Mercerization treatment effect on polymer composite mechanical properties

Natural Fiber	Matrix	Fiber Properties		Polymer Composite Properties			Effect / Comments	Ref.
		Shear Strength (MPa)	Tensile Strength (MPa)	Tensile Strength (MPa)	Flexural Strength (MPa)	Impact Strength (KJ/m^2)		
Sisal	Polyester	6.9 at 2% NaOH	375.4 at 2% NaOH	-	-	-	Sisal surface smoother at NaOH less than 2%, rougher when more than 5%	(Sydenstricker *et al.*, 2003)
Hemp & kenaf	Polyester	-	-	-	Treated long fiber > Treated short fiber > untreated fiber	Untreated hemp polyester composite show greater work of fracture value	Flexural strength depends on fiber alignment and resin rich areas location	(Aziz & Ansell, 2004)
Curaua	Biodegradable resin	-	Decreased for treated fiber compared to untreated fiber	Treated =137 Untreated = 124 (slightly higher) (A Gomes *et al.*, 2004) Untreated PF = 275, PS = 327 10% NaOH PF = 276 PS = 334 (Alexandre Gomes, Matsuo, Goda, & Ohgi, 2007)	-	-	2 factors contribute to composite tensile strength: 1. Interfacial bonding improvement, 2. Decrease in the coefficient of variation of alkali treated fiber strength	(A Gomes *et al.*, 2004; Alexandre Gomes *et al.*, 2007)
Kenaf	-	-	6% NaOH at 95^{0}C is 243.7	-	-	-	9% NaOH was too strong and might damage the fibers, thus resulting lower tensile strength	(Edeerozey *et al.*, 2007)

Hemp	Polyprop -ylene & MAPP	-	347 at 10 wt%, Young modulus = 20.3 GPa	Increase with the increase of MAPP content	-	-	Alkali treatment separate the fiber bundles into elementary fiber by degrading the cementing material	(Beckermann & Pickering, 2008)
Ramie	PLA	-	-	66.8±1.7	170	24	Alkali treatment seem to be an effective surface treatment agent compare to silane	(Yu *et al.*, 2010)
Jute	-	Increase 50% (370± 134MPa)compare to untreated fiber	-	-	-	-	Fiber diameter decrease due to the removal of hemicellulose, pectin and lignin	(Saha *et al.*, 2010)

5.0 CONCLUSIONS

Mercerization or alkali treatment is one of the common techniques that widely used for modification of fiber surface. In addition, this treatment also considered as a simple and cheapest treatment method. Mercerization treatment changes the surface topography of the fibers and their crystallographic structure. The removal of surface impurities on plant fibers is advantageous in fiber matrix adhesion, as it facilitates both mechanical interlocking and the bonding reaction due to the exposure of the hydroxyl groups in the polymer matrix. However, due to the large variety in natural fiber chemical composition, the treatment condition also will be different according to the selected fiber. As a conclusion of this study, although tremendous research results were published regarding mercerization treatment, there are still scant works conducted in dealing with interaction and optimizing the mercerization treatment condition like alkali concentration, soaking temperature and soaking duration.

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