The Influence of Alkaline Treatment on Tensile Properties of Short Bamboo Fiber Reinforced Epoxy Resin Biocomposites

W Nhuapeng1,2, T. Chintonguyad3 and W Thamjaree1,2*

1Department of Physics and Materials Science, Faculty of Science, Chiang Mai University, Chiang Mai 50200, Thailand
2Materials Science Research Center, Faculty of Science, Chiang Mai University
3The Graduate School, Chiang Mai University, 239 Huaykaew Road, Suthep, Meuang Chiang Mai 50200, Thailand
*wandee.th@cmu.ac.th

Abstract. In this work, the influence of alkaline treatment on tensile properties of short bamboo fiber reinforced epoxy resin biocomposites was investigated. Sodium hydroxide (NaOH) was used as a treatment agent. To improve the ability of adhesion, bamboo fibers were treated in NaOH solution in different concentrations and soaking times. The treated fibers were then fabricated to composites via casting technique. Mechanical properties and microstructure of biocomposites were determined using tensile testing and scanning electron microscope (SEM), respectively. From the results, it could be seen that the alkaline treatment can improve the fiber-matrix adhesion which have effect to better mechanical properties of biocomposites.

1. Introduction

Nowadays, natural fibers, plant fibers becomes popular materials that worldwide using in daily life and many composites industries, for example, automotive, building components and furniture, respectively. The plant fibers are used as reinforced phase instead of the commercial synthetic fibers such as glass fiber, because they are ecofriendly to the living things and environment. It is also show acceptable specific strength, low density, good thermal properties and low cost. Therefore, it is promoted to use instead of the synthetic glass fiber. There are many species of plants which offer various properties. Bamboo (Thrysostachys Siamensis Gamble of in Thai Pai Luak) is one of these plant that exhibits interesting properties [1-4]. Bamboo stems morphology consist of elementary fiber together with fiber bundle and culm wall. The high quantity of elementary fiber can promote the high mechanical properties such as strength and flexibility. Many parts of bamboo can uses in many utilities such as bamboo stem are utilized as the materials for musical instruments, furniture and building components. Besides, it is also easy to cultivate in Thailand, rapid growing, renewability, low cost and ecofriendly. Therefore, bamboo is selected to use as reinforced phase in composite materials instead of commercials synthetic fibers. The mechanical properties of bamboo fibers are included in Table 1.
Table 1. Mechanical properties of bamboo fiber.

| Types of fiber | Density (g/cm$^3$) | Tensile strength (MPa) | Young modulus (GPa) | Elongation at break (%) | References |
|----------------|---------------------|------------------------|---------------------|-------------------------|------------|
| Bamboo         | 1.16                | 610                    | 46                  | 1.5-1.8                 | [5]        |
| Jute           | 1.3                 | 393-773                | 26.5                | 2.7-3.2                 | [6]        |
| Flax           | 1.5                 | 343-1035               | 27.6                | 2.0-2.5                 | [6]        |
| Cotton         | 1.5-1.6             | 287-597                | 5.5-12.6            | 7.0-8.0                 | [6]        |
| Sisal          | 1.5                 | 511-635                | 9.4-22.0            | 1.6-4.0                 | [7]        |
| Hemp           | 1.48                | 350-800                | 30-60               |                         | [7]        |
| Coir           | 1.2                 | 300-500                | 10-30               | 20-40                   | [7]        |

However, it is found that the component of plant fiber such as lignin, pectin and hemicellulose are the obstacle of adhesion between reinforcing and matrix phase in composites. Researchers tried to improve the internal adhesion by modifying the surface of fiber using chemical treatment [8-10]. Alkaline treatment is one of chemical method which use for cleaning and treating the fiber surface that can enhance the interfacial adhesion between a natural fiber and a polymeric matrix. Nevertheless, the treatment parameters for an optimal composite quality are still not clearly specified. Several authors mention different values of time, alkaline concentration and duration to immerse the fiber. Many works reviewed about the effect of alkaline treatment on properties of natural fibers [11-12]. Hence, in this research, the effect of alkaline treatment on tensile properties of biocomposites between natural short bamboo fiber and epoxy resin was focused. Tensile properties and microstructure of samples were investigated. Since mechanical behaviour of composites directly depend on the length of fiber and adhesion between fiber and matrix. But using the long fiber as reinforcement phase can made the gap and bad adhesion between natural fiber and matrix which can affect to interfacial shear strength of composites. Therefore, to avoid this problem, the short fiber (about 10 mm) which has the length nearly to critical length of natural fiber (about 10-20 mm) was used instead to long fiber in this work.

2. Experimental Procedure

For fiber reinforced phase preparation, firstly, the bamboo plants (Thyrsostachys Siamensis Gamble) were collected from Noonsonbon Village, Roi-Et Province, Thailand. Secondly, bamboo fiber was then carefully extracted from its bundles by pen knife technique with the diameter of fiber was in the range of 250-300 µm. To obtain the optimum condition of treatment, the fibers were treated with alkaline treatment solution, sodium hydroxide (NaOH 97%, Acilabsan Ar1171-P1KG) with different concentration of 0.25 M and 0.5 M for various immersed time of 60 min, 4 hr and 8 hr, respectively. Next, fibers were rinsed with deionized water until pH of the rinsed water reached to 7. Thereafter, treated fibers were exposed and let to dry at room temperature for 5 days. The bamboo fiber was then cut with the length of 10 mm and mixed with epoxy resin for 20 wt% to fabricate biocomposites via casting technique. The sample was cast into the dog-bone shape (Figure 1) with the thickness of 1 mm.
Finally, the composite samples were left to settle at the room temperature for one day and then removed from the mould. Physical and mechanical properties such as density and tensile properties were examined. Density and tensile strength of the samples were measured using Archimedes and the Hounsfield mechanical testing machine using a cross-head speed of 50 mm/min and 250 mm extensometer, respectively. Furthermore, microstructure of biocomposites samples were also investigated by scanning electron microscope (SEM) technique.

3. Results and Discussion

Physical properties of bamboo fiber in which before and after treatment were shown in Figure 2. It was observed that color of fiber was changed after NaOH alkaline treatment. Color of bamboo fiber turn to dark brown compared with that of untreated fiber whereas density of samples were not significantly changed. It was found that density of bamboo fiber was about 1.03 g/cm$^3$ and density of neat epoxy resin was about 1.31 g/cm$^3$.

The untreated and treated bamboo fiber with different conditions were prepared to biocomposites via casting technique with the ratio of 20 wt%. Thereafter, tensile properties of biocomposites were then examined. Tensile strength result was shown in Figure 3. From the results, it was observed that the tensile strength of biocomposites fabricated from treated bamboo fiber was higher than those of untreated fiber sample and neat epoxy resin sample. It was also observed that concentration and soaking time of the treatment had affected to tensile strength of biocomposites. The optimum condition was obtained from alkaline treatment with concentration of 0.25 M and soaking for 1 hr. Tensile strength of
treated bamboo fiber biocomposites was increased about 53% and 32% when compared with those of neat epoxy resin and untreated fiber biocomposites, respectively.

The enhancement of tensile strength in alkaline treated (0.25 M of NaOH for 1 hr) could explained that biocomposites was attributed to the improvement of wettability between natural fiber and synthetic polymer matrix which correspond to previous work [12]. Therefore, it could be said that the alkaline treatment could remove the lignin, pectin, impurities and waxy substances from fiber surface. Moreover, the rougher surface also found after alkalization which enhanced the mechanical interlocking between reinforced fiber and polymer matrix that had affected to better interfacial bonding. Besides, the size of fiber becomes smaller after treatment thus, the extent of stress concentration derived from the fiber would decrease, which leads to the increasing of the strength [13]. However, the results was also found that tensile strength decrease with increasing of alkaline concentration. This may be due to the over treatment that could damage of fiber morphology in higher alkaline concentration which agree with Mwaikambo and Ausell [14].

![Graph showing tensile strength of untreated and treated short bamboo fiber reinforced epoxy resin biocomposites with soaking time.](image)

**Figure 3** Average tensile strength of untreated and treated short bamboo fiber reinforced epoxy resin biocomposites with soaking time.

The elongation of break (ε) of biocomposites results was shown in Figure 4. The results showed that the elongation of break of treated fiber biocomposites was not affected significantly by the alkaline concentrations and soaking times even the diameter and aspect ratio was changed. It was found that the percent elongation at break of the biocomposites was lower than that of the neat epoxy resin. This could be affected of low fracture strain of fibers [15].
Figure 4 Average elongation percentage at break of untreated and treated short bamboo fiber reinforced epoxy resin biocomposites with soaking time.

Furthermore, SEM micrographs of untreated and treated bamboo fiber were shown in Figure 5. It could be seen that untreated bamboo fiber (Figure 5(a)) contains impurities, wax, fatty substances, hemicelluloses, and lignin [16]. Since the SEM micrograph (Figure 5(a)) exhibited morphology of untreated bamboo fiber which exposed the bamboo wall layer. The bamboo wall layer composed with multilayer of vascular bundle which each layer has cellulose microfibrils oriented at various angles to the fibre axis surrounded by matrix of hemicellulose, lignin and wax that correspond to Sayyad M et al. [17]. Therefore, the heterogeneous surface with smooth and rough surface could be observed. However, after the treatment (Figure 5(b), it was found that the undesirable substance such as pectin, lignin, hemicellulose and fatty-deposits was removed from bamboo fiber surface. The rougher surface was obtained that the resulted in good adhesion between natural fiber and epoxy resin polymer matrix which could reduce the fiber pullout defects. This results is similar to Bismarck et.al who reported that alkaline treatment resulted in a higher amount of cellulose exposed on the fiber surface, thereby increasing the number of possible reaction sites [18].

Figure 5 SEM surface studies of (a) untreated and (b) NaOH-treated short bamboo fibers.
Moreover, the SEM micrographs of fracture surface of untreated and treated bamboo fiber reinforced epoxy resin biocomposites after tensile loading were shown in Figure 6. It could be seen that poor adhesion between bamboo fiber and resin matrix (Figure 6 (a)) was observed whereas the extend fiber and small gap appeared after tensile loading was found in treated fiber biocomposites samples (Figure 6 (b) and (c)). However, fiber was not pulled out of the matrix which could be said that the strong interfacial bonding of fiber and matrix could obtain after the alkaline treatment. Therefore, it could be concluded that the alkaline treatment had affected to the tensile properties of biocomposites. Since it could enhance the interfacial and adhesion between hydrophilic natural fiber and hydrophobic epoxy resin polymer.

![SEM micrographs of (a) untreated and treated (b) 0.25 M NaOH and (c) 0.5 M NaOH bamboo fiber reinforced epoxy resin biocomposites after tensile loading.](image)

4. Conclusion

The influence of fiber treatment using alkaline technique on tensile properties of short bamboo fiber reinforced epoxy resin biocomposites was investigated. It was found that biocomposites prepared from treated bamboo fiber showed significantly improved in tensile strength but had not affected to the elongation at break of the biocomposites. The optimum tensile strength obtained from biocomposites sample which bamboo fiber was treated with 0.25 M of NaOH soaking for 1 hr. The hydrophilic property of natural short bamboo fiber had been reduced whereas the the interfacial bonding between fiber and matrix was increased.

Acknowledgement

This research project is supported by TSRI. The authors would also like to thank The Composites and Reinforced Nanocomposites Unit, Department of Physics and Materials Science, Faculty of Science and the Graduate School of Chiang Mai University for all supporting.
References
[1] Bachtiar D, Sapuan S M and Hamdan M M 2008 Materials and Design 29 1285
[2] Van de Weyenberg I, Chi Truong T, Vangrimde B and Versoest I 2006 Composites: Part A 37 1368
[3] Abdul Khalil H P S, Bhat U H, Jawaid M, Zaidon A, Hermawan D and Hadi Y S 2012 Materials and Design 42 353
[4] Ridzqo I F, Susanto D, Panjaitan T H and Putra N 2020 IOP Conf. Series: Mater. Sci. Eng. 713 012010
[5] Amada S and Untao S 2001 Composites: Part B 32 451
[6] Gassan J and Bledzki A K 1999 Comp. Sci. Tech. 59 1303
[7] Raja T, Anand P, Karthik M and Sundaraj M 2017 Inter. J. Mech. Eng. Tech. (IJMET) 8[7] 915
[8] Van de Weyenberg I, Ivens J, De Coster A, Kino B, Baetens E and Verpoest I 2003 Comp. Sci. Tech. 63 1241
[9] Takagi H and Ichihara Y 2004 JSME International Journal 47 551
[10] Rahman M M and Khan M A 2007 Comp. Sci. Tech. 67 2369
[11] Wong K J, Tousif B F and Low K O 2010 Proc. IMechE 224
[12] Rout J, Misra M, Tripathy S S, Nayak S K and Mohanty A K 2001 Comp. Sci. Tech. 61 1303
[13] Liu W, Mohanty A K, Askeland P, Drzal L T and Misra M 2004 J. Mater. Sci. 39 1051
[14] Mwaikambo L and Ansell M 2002 J. Appl. Polym. Sci. 84 2222
[15] Chumsamrong P, Sutapun W, Kiaw-on S and Tonuloon W 2005 31st Cong. Sci. Tech. http://sutir.sut.ac.th:8080/jspui/handle/123456789/2204
[16] Wong K J, Zahid S, Low K O, Lim C C 2010 Materials and Design 31 4147
[17] Sayyad M, J Paul K and Sumit B, 2017 Royal Society Open Science, 4[1] https://doi.org/10.1098/rsos.160412
[18] Bismarck A, Mohanty A K, Aranberri-Askargorta I, Czapla S, Misra M, Hinrichsenb G, Springera J 2001Green Chemistry 3 100