Preparation of a Rough Hydrophobic Surface on Jute Fibers *via* Silica Hydrosol Modification and Properties of Fiber-reinforced Polylactic Acid Composites

Weiming Wang,[a,b,*] Aixue Dong,[a,b] and Zuoqiang Wen [a]

Scoured jute fiber was modified with silica hydrosol and then mixed with polylactic acid to form bio-based fiber-reinforced composites. The effects of modification methods on the morphology and hydrophobicity of the fibers, as well as the tensile strength and fracture structure of the composites were studied. Also, the influence of sodium hydroxide concentration, MTMOS concentration, and numbers of dipping steps on the tensile strength of the composites were examined. The results indicated that silica hydrosol condensation could be used to prepare a smooth hydrophobic surface on modified fibers, while a rough hydrophobic surface could be prepared when alkaline treatment was carried out onto the surface of incompletely condensed silica hydrosol film. Rough hydrophilic fibers revealed poor interfacial compatibility and adhesion strength. Smooth hydrophobic surfaces improved interfacial compatibility and dispersibility of the fibers in hydrophobic matrix, whereas a rough hydrophobic structure further improved the adhesion strength through mechanical interlock. The data illustrated that morphology of the silica hydrosol modified fiber surface depended on the alkaline treatment conditions and amounts of adsorbed silica hydrosol, while the effect of MTMOS precursor concentration was negligible.

Keywords: SiO_2 hydrosol; Jute/PLA composite; Tensile strength; Interfacial structure

Contact information: a: *College of Textile and Garment, Shaoxing University, Shaoxing Zhejiang 312000, China; b: Zhejiang Key Laboratory of Clean Dyeing and Finishing Technology, Shaoxing Zhejiang 312000, China*; **Corresponding author*: *wangwm@usx.edu.cn*

INTRODUCTION

The increasing awareness of the environmental concerns and limited petroleum resources has resulted in the replacement of synthetic fibers (glass fiber, carbon fiber, aramid fiber) in polymer composites by natural fibers due to their numerous advantages, such as light weight, low cost, recyclability, biodegradability, and easy availability (Liu *et al.* 2012; Baheti *et al.* 2014; Shih *et al.* 2014). Jute fibers are promising natural fibers used for reinforcing polymeric composites due to their abundant availability and low microfibril angle (Khan *et al*. 2012, 2014). However, like other cellulosic fibers, jute fibers often suffer from poor interfacial adhesion and compatibility in contact with hydrophobic matrices due to their hydrophilic nature, leading to unsatisfactory mechanical properties for the composites (Seki *et al*. 2010; Khan *et al*. 2014). To overcome this limitation, surface modification of natural fibers becomes one of the main research directions.

Many methods have been attempted to modify the surface of natural fibers to enhance their compatibility and interfacial adhesion properties with polymer matrices. These include alkaline treatment (Mwaikambo and Ansell 2006; Lee *et al*. 2008; Sinha and Rout 2009), oxidation (Khan *et al*. 2014), silane coupling (Jandas *et al*. 2012; Zafar *et al*.

2016), plasma (Seki *et al*. 2010), and sol-gel coating (Wu *et al*. 1997a,b). Goriparthi *et al*. (2012) revealed that surface treatments such as alkali, oxidation, and silane could enhance the tensile strength of jute-reinforced PLA composites. Li *et al*. (2006) studied the interfacial adhesion of sisal fiber-reinforced composites using a single fiber pullout testing method. The silane coupling agent treated sisal debonded during testing, while in the case of potassium permanganate and diisopropyl peroxide treated fiber debonding did not occur due to increased roughness of the fiber surface. Utilizing of silica sol nanoparticles to create a rough hydrophobic surface on fiber materials has become a research hotspot in the super-hydrophobic finishing field (Xu *et al*. 2015). The basic conditions for applying sol-gel coatings include stable adhesion to the surface of fiber. Kowalczyk and Kaminska (2017) showed that the adhesion of silicone-organic coating to polyamide fiber surface could be improved by creating a rough surface. Wang *et al.* (2009) pointed out that sodium hydroxide treatment improves the tenacity and roughness of jute fiber due to removal of non-cellulosic materials, namely, pectin, wax, lignin, and hemicelluloses. For preparing rough surfaces, alkaline treatment is an easier method compared with plasma and irradiation. The surface of sodium hydroxide treated fiber has bad interfacial compatibility due to its hydrophilicity, while the rough surface of treated fiber can improve the adhesion force of silica sol coating on the fiber surface through mechanical interlock.

However, the roughness degree of sol-gel coatings is often measured in nano size, which is good enough to create a hydrophobic surface, but the interlock force between fiber and polymer matrix is too weak to improve their interfacial adhesion. Wu *et al.* (2011) studied the stability of silica sol system by acid-base two-step catalytic process, finding that condensation rate of silicon hydroxyl (Si-OH) could be significantly speeded up under alkaline conditions. Therefore, it is speculated that a larger size rough surface could be obtained by adding alkaline solution onto the surface of fiber adsorbed silica sol before the condensation process.

Silica sol nanoparticles are usually prepared from tetraethyl orthosilicate (TEOS) or silane coupling agent using two steps: hydrolysis and condensation (Hsieh *et al.* 2010; Xu *et al*. 2010a). The hydrolysis is often controlled under acidic conditions of pH 1 to 3 (Shih *et al.* 2014), which will decrease the fiber tensile strength due to the degradation of the cellulose chain. Shen *et al*. (2010) revealed that silica sol is very stable at pH 6, indicating that damage of cellulose fibers could be reduced by adjusting the pH of the sol solution to 6 at the beginning of fiber modification. However, there is no published research on utilizing silica sol nanoparticles to create large-scale rough surfaces on lignocellulosic fibers, and then using them as reinforcement in resin composites.

In this work, water-based silica (SiO_2) sol nanoparticles were prepared using methyltrimethoxysilane (MTMOS) and hydrochloric acid (HCl) as precursor and catalyst, respectively. The bacterial-retted jut fiber was scoured using sodium hydroxide (NaOH), modified with a SiO_2 hydrosol, and then it was mixed with PLA to prepare jute reinforced PLA composites using an injection model. The effects of NaOH treatment and SiO_2 hydrosol modified conditions on the morphology and hydrophobicity of jute fiber were examined by scanning electron microscopy (SEM) and static water contact angle testing. The tensile strengths of the obtained jute reinforced PLA composites were evaluated to optimize SiO_2 hydrosol modification conditions. The fractured surfaces of the composites were tested to identify the effects of SiO_2 hydrosol on the interfacial adhesion and compatibility of jute reinforced PLA composites.

EXPERIMENTAL

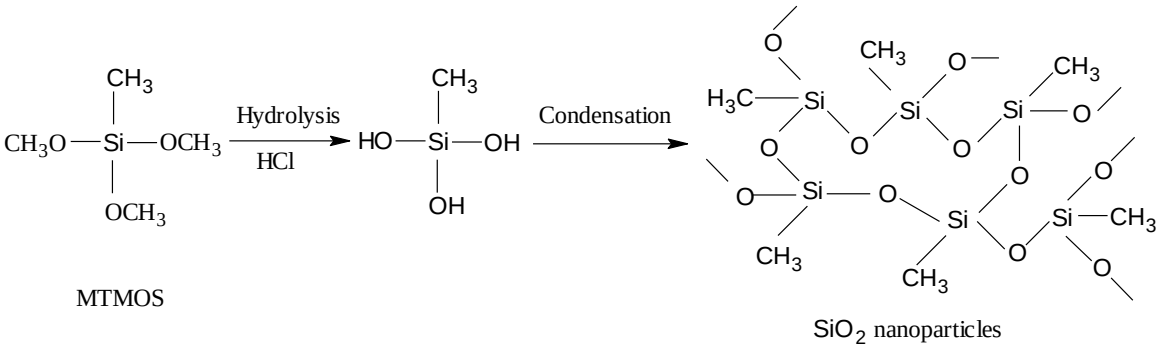
Materials

Bacteria-retted jute fibers were obtained from Hangzhou Hongbo New Materials, Hangzhou, China. The precursor methyltrimethoxysilane (MTMOS) was purchased from Nanjing Chengong Organic Silicone Material, Nanjing, China. The granular PLA 4032D was purchased from NatureWorks (USA) through local supplier JinHong Plastic Chemical, Dongguan, China. Penetrant JFC was provided by Zhejiang Transfar Group, Hangzhou, China. Sodium dodecyl benzene sulfonate (SDBS), hydrochloric acid (HCl) (36.4%, w/w), sodium hydroxide (NaOH), sodium silicate (Na_2SiO_3), and concentrated sulfuric acid (H_2SO_4) were purchased from Aladdin Industrial Corporation, Shanghai, China.

Methods

Preparation of silica hydrosol

First, HCl (0.5 g), SDBS (0.4 g), and deionized water (100 g) were added to a round-bottom flask and stirred at 30 °C until SDBS was completely dissolved. The MTMOS (1.5 to 3.0 g) was added to the mixture and stirred at 30 °C for 60 min. The resulting mixture was heated to 60 °C at a speed of 2 °C/min, followed by further stirring for 120 min to obtain SiO_2 hydrosol. The stirring speed of SiO_2 hydrosol preparation process was controlled at 150 r/min. The preparing process of SiO_2 nanoparticles hydrosol is shown in Scheme 1.



Scheme 1. Preparation of SiO_2 nanoparticles hydrosol with MTMOS

Modification of jute fiber

The scouring of retted jute fibers was carried out in a sealed glass conical flask using a vibrating laboratory dyeing machine (Xiamen Rapid, Xiamen, China) at fiber to liquor ratio of 1:15. The aqueous scouring solution consisted of NaOH (12 g/L), Na_2SiO_3 (6 g/L), and penetrating agent (JFC, 2 g/L). The bath was heated to 95 °C, and then kept at 95 °C for 120 min. After treatment, the fibers were neutralized with 2 g/L H_2SO_4, thoroughly washed with distilled water, and dried in an oven.

Prior to modification of the SiO_2 hydrosol, the pH was adjusted to 6 using buffer solution. The scoured jute was immersed in SiO_2 hydrosol at a fiber-to-liquor ratio of 1:15 then kept at 25 °C for 10 min. The fibers were pre-dried in an oven then heat-treated at 120 °C for 10 min. Next, the samples were thoroughly washed with distilled water until the fiber surface became neutral, followed by oven-drying to obtain SiO_2-jute-1.

To prepare SiO_2-jute-2, scoured jute was first immersed in SiO_2 hydrosol for 10 min at 25 °C and then dried at 80°C. Next, the fibers were dipped in NaOH solution (0.5 to 3 g/L) for 1 min at 25 °C. The fibers were pre-dried in an oven and then heat-treated at 120 °C for 10 min. Subsequently, the samples were thoroughly washed with distilled water

until the fiber surface became neutral followed by drying in an oven. The fiber to liquor ratio of all modification processes was maintained at 1:15.

Preparation of composites

Prior to compounding, the jute fibers were cut into pieces (length, 1 to 2 cm). Both jute and PLA were pre-dried at 80 °C for 2 h. Subsequently, both the fibers and PLA were melt blended in a Micro Hybrid Rheometer (Mini Lab II, ThermoFisher Scientific, Waltham, MA, USA) at a fiber to PLA ratio of 1:9. The mixing process was conducted at 190 °C and a rotor speed of 60 r/min. The melt mixed composite was cooled to room temperature, granulated, and conditioned at 80 °C for 2 h prior to specimen preparation. Dumb-bell specimens were prepared using an injection mold (HAAKE MiniJet PRO, ThermoFisher Scientific) at an injection temperature of 190 °C.

Characterization

The morphologies of the fibers and fracture surface of composites were observed by scanning electron microscopy (SNE-3000, SEC, Suwon, Korea). All samples were coated with a thin layer of gold before observation.

The breaking tensile of pure PLA and jute reinforced PLA were tested according to GB/T 1040.4 (2006) using a Universal Testing Machine (H-10K-L Tinius Olsen, Philadelphia, PA, USA).

The static water contact angles (Static WCA) of the specimens were tested on an OCA50 micro contact angle system (Dataphysics, Stuttgart, Germany) using 30 μL of water at ambient temperature.

RESULTS AND DISCUSSION

Effect of Modification on Morphology of Jute Fiber

The interfacial adhesion properties of fiber-reinforced composites mostly depend on surface morphology of the reinforcing material. As shown in Fig. 1, the scoured jute fibers exhibited surfaces with numerous grooves along the vertical section due to partial removal of non-cellulosic materials (pectin, wax, lignin, hemicellulose, *etc*.) (Wang *et al*. 2009; Xu *et al*. 2010b). Figure 1(b) shows that SiO_2-jute-1 had a smooth surface, while SiO_2-jute-2 exhibited a rough surface (Fig. 1(c)). This may be explained by adsorbed SiO_2 hydrosol, which forms uniform smooth films on fiber surfaces through condensation reaction of silicon hydroxyl (Si-OH) (Xu *et al*. 2010a, 2015; Shih, *et al*. 2015). The formed smooth SiO_2 hydrosol film could be etched by sodium hydroxide during dipping process, and the adsorbed sodium hydroxide could catalyze the condensation reactions of Si-OH to form irregular grooves during heat-treating process, especially during treatment at high temperature. Subsequently a rough hydrophobic film deposited on SiO_2-jute-2 was created.

Effect of Modification on hydrophobic performance of Jute Fiber

The main purpose of fiber modification is to prepare hydrophobic surface. Therefore, static WCA of modified jute fibers was measured as a function of hydrophobic performance, and the data are listed in Table 1. The static WCA of SiO_2-jute-2 was greater than that of SiO_2-jute-1. It was speculated that the hydrophilic Si-OH on the surface of SiO_2-jute-1 could not completely condense under the common applied conditions, while adsorbed sodium hydroxide could catalyze the condensation reaction of Si-OH to result in

complete condensation. In sum, the alkaline treatment performed on pre-dried SiO_2 hydrosol modified fibers could improve the hydrophobic performance of SiO_2 hydrosol modified jute fiber.

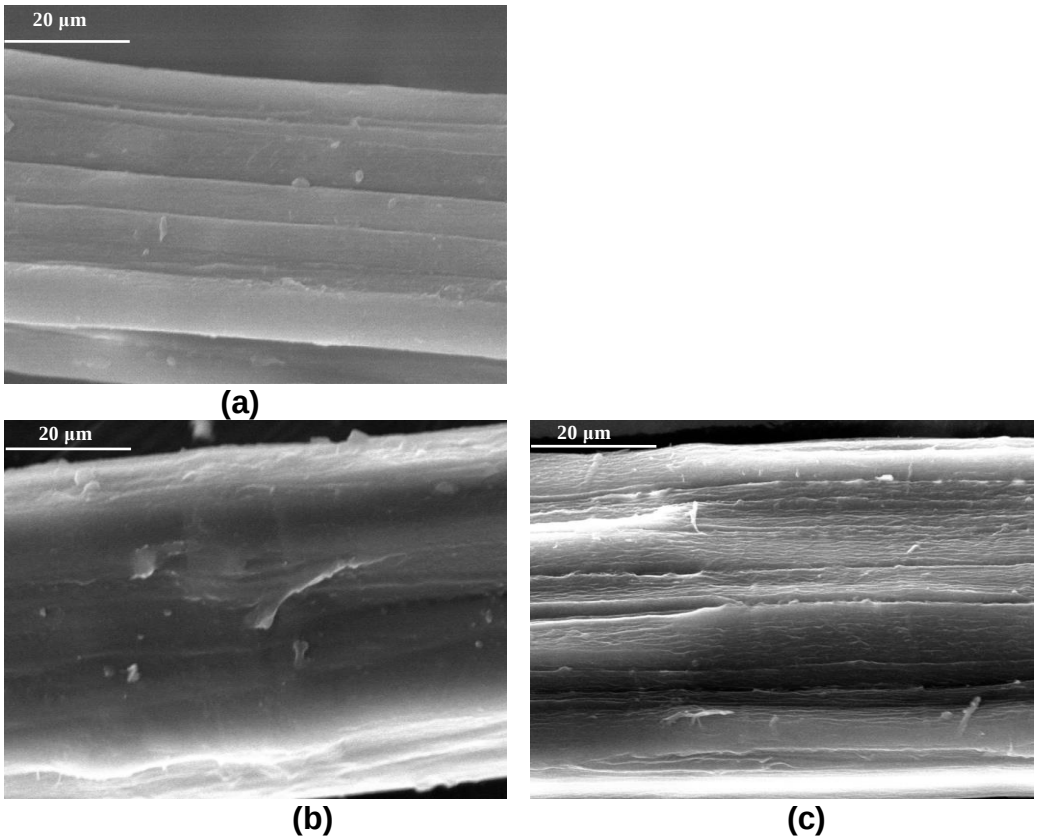


Fig. 1. SEM photographs of jute fibers: (a) scoured jute, (b) SiO_2-jute-1, and (c) SiO_2-jute-1

Table 1. Static WCA of Jute Fibers

Samples	Static WCA (°)
Scoured jute	-
SiO_2-jute-1	110
SiO_2-jute-2	121

Effect of Modification on Tensile Strength of Composites

Figure 2 depicts the tensile strength of jute fiber reinforced PLA composites modified by different methods. The tensile strength of scoured jute/PLA composite showed the lowest value of all three composites, while SiO_2-jute-2/PLA composite exhibited the maximal tensile strength. This may be explained by the weaker interfacial adhesive force and the poor compatibility of scoured jute fiber in PLA due to its good hydrophilicity, which makes the jute/PLA composite have weaker tensile strength. Furthermore, the hydrophilic scoured jute fibers are prone to aggregation in the mixture of jute and PLA, resulting in uneven tensile strength of the composite materials.

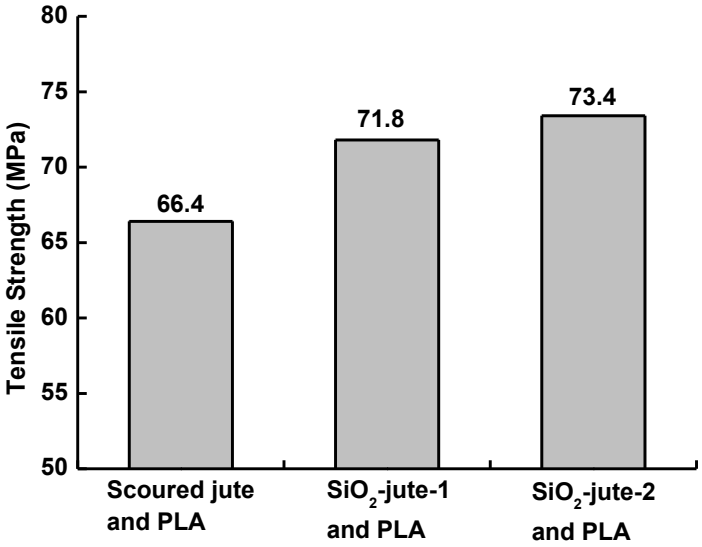


Fig. 2. Effect of modification method on the tensile strength of composites

Although the surface of SiO_2-jute-1 was hydrophobic, the higher flowing speed of melted jute and PLA mixture during the injection process could slip over the fiber to form pores in the composite. These pores form breakable points. Figure 1 and Table 1 show that SiO_2-jute-2 had a rough hydrophobic surface, which could improve the interfacial adhesion between SiO_2-jute-2 and PLA due to the good interfacial compatibility and mechanical interlocking. Therefore, both roughness and hydrophobicity play important roles in the improvement of tensile strength of fiber reinforced resin composites.

Effect of NaOH Concentration on Tensile Strength

Wu *et al*. (2009) reported that alkaline conditions could catalyze the condensation reaction of Si-OH to form Si-O-Si. Therefore, the surface structure of hydrophobic surface could be affected through adding sodium hydroxide onto the not fully condensed SiO_2 hydrosol film due to changes in condensation speed. This, in turn, affects the tensile strength of the resulting composites.

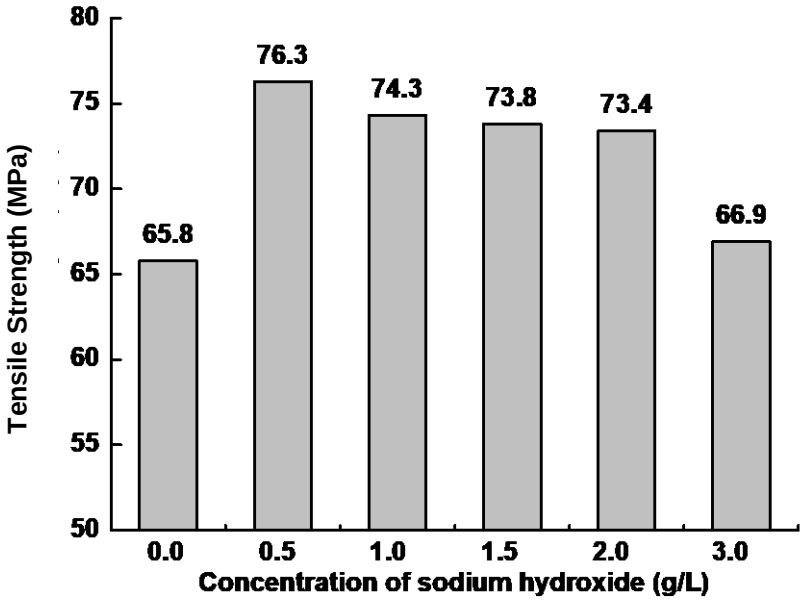


Fig. 3. Effect of NaOH concentration on the tensile strength of composites

Figure 3 shows that the tensile strength of jute/PLA composites increased as sodium hydroxide concentration rose from 0 g/L to 0.5 g/L. However, reverse changes in tensile strength appeared with further increases in sodium hydroxide concentration. This effect could be related to the rough hydrophobic surface, which could be obtained by surface etching and quick condensation of Si-OH. Also, roughness was improved by increasing sodium hydroxide concentration. However, strong etching or rapid condensation at higher sodium hydroxide concentrations could destroy the SiO_2 hydrosol film to form cracks, resulting in the hydrophilic surface of scoured jute could not be completely covered by hydrophobic SiO_2 hydrosol film. Therefore, the optimal sodium hydroxide concentration was determined as 0.5 g/L.

Influence of MTMOS Concentration on Tensile Strength

Figure 4 illustrates the influence of MTMOS precursor concentration on the tensile strength of modified jute/PLA composites. The change in tensile strength could be ignored when the concentration of MTMOS rose from 15 g/L to 30 g/L. It was speculated that 15 g/L MTMOS was good enough to endow jute fiber with relevant hydrophobicity. The tensile strength of composites mostly depended on the roughness of SiO_2 hydrosol film.

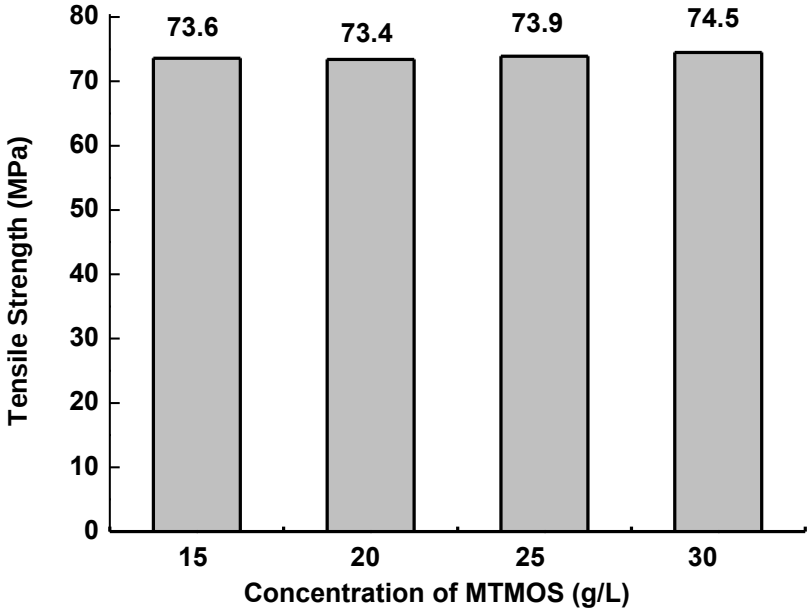


Fig. 4. Effect of MTMOS concentration on tensile strength of composites

The roughness of SiO_2 hydrosol film depended on the conditions of alkaline treatment. However, all alkaline treatments of SiO_2 hydrosol modified jute fibers were kept under the same conditions. Another probable reason to explain the negligible changes in tensile strength of composites may be fiber surface adsorbed SiO_2 hydrosol particles, which would remain unchanged due to electrostatic repulsions between particles at elevated concentrations of MTMOS. Therefore, no visible change in tensile strength of the composites was observed as TMOS concentration rose.

Effect of Number of Dipping Stepson Tensile Strength

To estimate the effect of adsorbed SiO_2 hydrosol amounts on composite tensile strength, the number of dipping steps of SiO_2 hydrosol was also changed during the preparation process. Every dipping step was comprised of immersion treatment of scoured jute fiber in SiO_2 hydrosol solution for 10 min followed by pre-drying. The results of these variations are gathered in Fig. 5.

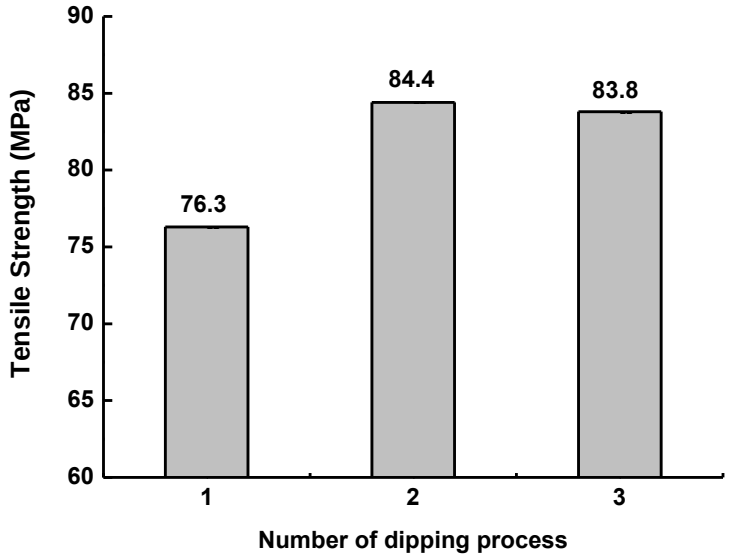


Fig. 5. Effect of number of dipping steps on tensile strength of composites

Figure 5 shows that the tensile strengths of jute-reinforced PLA composites prepared by two steps were higher than those of composites prepared using one step. There were no obvious changes in tensile strength when the number of dipping steps was increased to three. This may be explained by the formed SiO_2 hydrosol film obtained after one step, which did not fully cover the fiber surface. This resulted in a decrease in hydrophobicity and subsequently reduced interfacial adhesive force. The adsorption of SiO_2 hydrosol using a 2-step dipping could condense the deposited film to completely cover the fiber surface. Compared with 2-step dipping, one additional step (3-step dipping) would in theory increase the adsorbed SiO_2 hydrosol amounts. However, the rough film surface would not change because the alkaline treatment was kept under the same conditions. Therefore, 2-step dipping was enough to obtain good rough hydrophobic surfaces.

Influence of Modification on Composite Interfacial Structure

To clarify the mechanism of tensile breaking of jute reinforced PLA composites, fracture surfaces were analyzed by SEM, as shown in Fig. 6. A few deep grooves were detected on the fractured surface of scoured jute reinforced PLA composite (Fig. 6(a)), with some single gathered fibers. The latter were associated with the fact that scoured jute has good hydrophilicity. This rendered the fibers ineffectively dispersed in the hydrophobic PLA matrix. Good hydrophilicity could affect the interfacial adhesive force between jute fiber and PLA, resulting in interfacial slipping.

Compared with scoured jute reinforced PLA composite, both SiO_2-jute-1 (Fig. 6(b)) and SiO_2-jute-2 (Fig. 6(c)) dispersed well in PLA matrix as single fibers because of their high hydrophobicities. The fractured surface of SiO_2-jute-1 reinforced PLA (Fig. 6(b)) was rough with some visible grooves due to slipping fibers at the interfacial surface, while SiO_2-jute-2 reinforced PLA (Fig. 6(c)) was relatively smooth with a flat fractured surface. Therefore, the tensile strength of fiber reinforced PLA composites could be improved by building rough structures on hydrophobic surfaces.

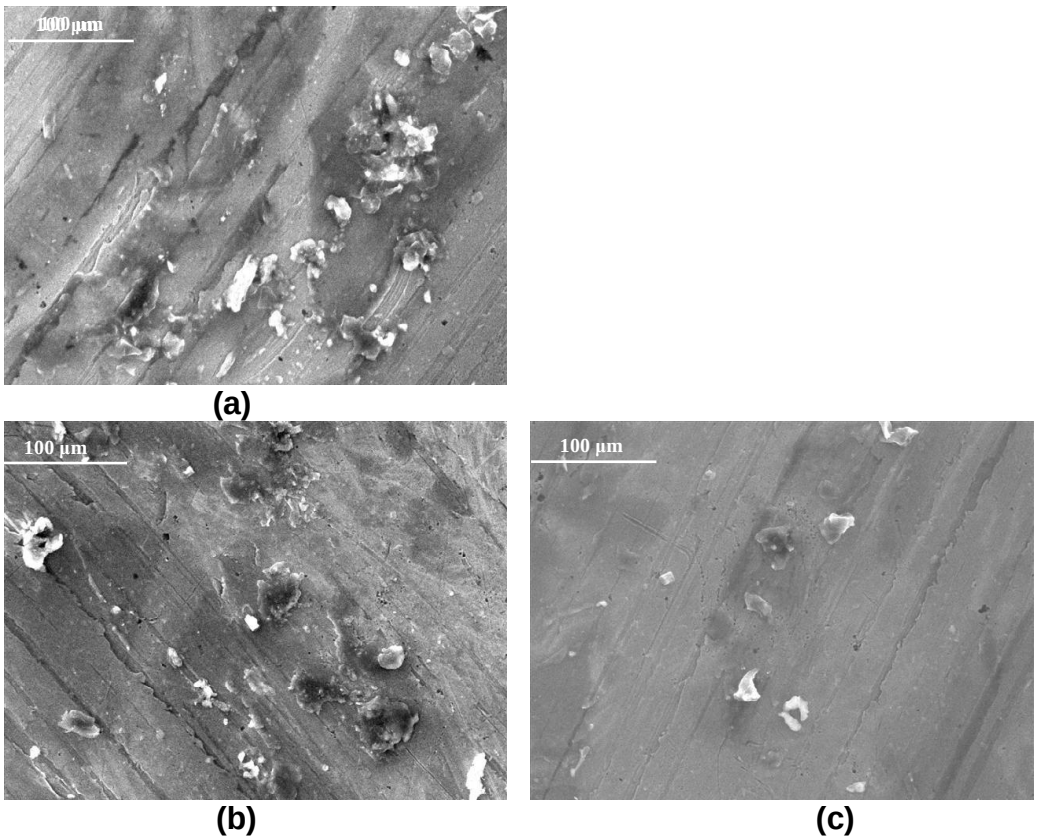


Fig. 6. Morphologies of fractured surface of composites: (a) scoured jute and PLA, (b) SiO_2–jute-1 and PLA, and (c) SiO_2–jute-2 and PLA

CONCLUSIONS

1. Scoured jute fibers exhibited a rough, hydrophilic surface.
2. SiO_2 hydrosol modified jute fibers had a smooth hydrophobic surface.
3. A rough hydrophobic surface was prepared when the SiO_2 hydrosol modified jute fibers were treated with alkaline solution.
4. The sodium hydroxide concentration and the dipping number of SiO_2 hydrosol had effects on the tensile strength of jute fiber-reinforced polylactic acid composites, while the methyltrimethoxysilane (MTMOS) precursor concentration had no effect.
5. During the alkaline treatment on SiO_2 hydrosol modified jute fibers, the conditions were adjusted according to the adsorbed amounts of SiO_2 hydrosol.
6. The fractured surface of jute/PLA composite indicated that dispersibility of jute fiber in a poly-(lactic acids) (PLA) matrix depended on hydrophobicity of jute fiber surface, while the interfacial adhesive force between jute and PLA depended on the roughness of the hydrophobic surface.

ACKNOWLEDGEMENTS

This work was financially supported by National Science & Technology Innovation Plan Project for College Student, China (Granted No. 201710349010), and the Science Technology Department of Zhejiang Provincial, China (Grant No. LGG18E030005).

REFERENCE CITED

Baheti, V., Mishra. R., Militky, J., and Behera, B. K. (2014). "Influence of noncellulosic contents on nano scale refinement of waste jute fibers for reinforcement in polylactic acid films," *Fiber Polym.* 15(7), 1500-1506. DOI: 10.1007/s12221-014 -1500-5

GB/T 1040.4 (2006). "Plastics—Determination of tensile properties—Part 4: Test conditions for isotropic and orthotropic fiber-reinforced plastic composites," Standardization Administration of China, Beijing, China.

Goriparthi, B. K., Suman, K. N. S., and Rao, N. M. (2012). "Effect of surface treatments on mechanical and abrasive wear performance of polylacide/jute composites," *Compos. Part A-Appl. Sci. Manuf.* 43(10), 1800-1808. DOI: 10.1016/j.compostesa.2012.05.007

Hsieh, C. T., Wu, F. L., and Chen, W. Y. (2010). "Surperhydrophobicity and superoleophobicity from hierarchical silica sphere stacking layers," *Mate. Chem. Phys.* 121(1-2), 14-21. DOI: 10.1016/j.matchemphys.2009.12.031

Jandas, P. J., Mohanty, S., and Nayak, S. K. (2012). "Renewable resource-based biocomposites of various surface treated banana fiber and polylactic acid: Characterization and biodegradability," *J. Polym. Environ.* 20, 583-595. DOI: 10.1007/s10924-012-0415-8

Khan, J. A., Khan, M. A., and Islam, R. (2014). "Mechanical, thermal and degradation characteristics of jute fabric-reinforced polypropylene composites. Effect of potassium dichromate as oxidizing agent," *Fiber Polym.* 15(11), 2386-2394. DOI: 10.1007/s12221-014-2386-y

Khan, J. A., Khan, M. A., and Islam, R. (2012). "Effect of mercerization on mechanical, thermal and degradation characteristics of jute fabric-reinforced polypropylene composites," *Fiber Polym.* 13(10), 1300-1309. DOI: 10.1007/s12221-012-1300-8

Kowalczyk, S., and Kaminska, I. (2017). "Effect of corona discharge on the stability of the adhesion of thin silicone-organic coating to polyamide fiber surface made by the sol–gel method," *J. Coat. Technol. Res.* 14(5), 1115-1125. DOI: 10.1007/s11998-016-9899-9

Lee, H. S., Cho, D., and Han, S. O. (2008). "Effect of natural fiber surface treatments on the interfacial and mechanical properties of henequen polypropylene biocomposites," *Macromol. Res.* 16(5), 411-417. DOI: 10.1007/bf03218538

Li, Y., and May, Y. W. (2006). "Interfacial characteristics of sisal fiber and polymeric matrices," *J. Adhes.* 82(5), 527-554. DOI: 10.1080/00218460600713840

Liu, D. G., Song, J. W., Anderson, D. P., Chang, P. R., and Hua, Y. (2012). "Bamboo fiber and its reinforced composites: Structure and properties," *Cellulose* 19, 1449-1480. DOI: 10.1007/s10570-012-9741-1

Mwaikambo, L. Y., and Ansell, M. P. (2006). "Mechanical properties of alkali treated plant fibres and their potential as reinforcement materials. II. Sisal fibres," *J. Mater.*

Sci. 41, 2497-2508. DOI: 10.1007/s10853-006-5075-4

Seki, Y., Sarikanat, M., Sever, K., Erckin, S., and Gulec, H. A. (2010). "Effect of the low and radio frequency oxygen plasma treatment of jute fiber on mechanical properties of jute fiber polyester composite," *Fiber Polym.* 11(8), 1159-1164. DOI: 10.1007/s12221-010-1159-5

Shen, F., Cui, Y. H., Jin, X., and Lu, T. (2010). "Preparation and stability of silica sol catalyzed by hydrochloric acid and ammonia," *Chemical Research* 21(1), 15-18 (In Chinese). DOI: 10.14002/j.hxya.2010.01.005

Shih, Y. F., Huang, R. H., and Yu, Y. H. (2014). "Preparation and characterization of sol–gel-modified pineapple leaf fiber polylactic acid composites," *J. Sol-Gel Sci. Technol.* 70, 491-499. DOI: 10.1007/s10971-014-3311-y

Sinha, E., and Rout, S. K. (2009). "Influence of fibre-surface treatment on structural, thermal and mechanical properties of jute fibre and its composite," *Bull. Mater. Sci.* 22(1), 65-76. DOI: 10.1007/s12034-009-0010-3

Wang, W. M., Cai, Z. S., Yu, J. Y., and Xia, Z. P. (2009). "Changes in composition, structure, and properties of jute fibers after chemical treatments," *Fiber Polym* .10(6), 776-780. DOI: 10.1007/s12221-009-0776-3

Wu, J., Jones, F. R., and James, P. F. (1997a). "Continuous fiber reinforced mullite matrix composites by sol–gel processing Part II. Properties and fracture behavior," *J. Mater. Sci.* 32, 3629-3635. DOI: 10.1023/A:1018630429960

Wu, J., Jones, F. R., and James, P. F. (1997b). "Continuous fiber reinforced mullite matrix composites by sol–gel processing. Part I. Fabrication and microstructures," *J. Mater. Sci.* 32, 3361-3368. DOI: 10.1023/A:1018604229535

Wu, Q. J., Wu, Q. R., Yang, Y., Cao, Q., Ren, Y. P., Shui, C. S., and Luo, Y. Z. (2011). "Study on preparation and stability of silica sol system by acid-base two-step catalytic process," *Journal of Wuhan University of Technology* 33(7), 14-19 (In Chinese). DOI: 10.3963/j.issn.1671-4431.2011.07.004

Xu, B., Cai, Z. S., Wang, W. M., and Ge, F. Y. (2010a). "Preparation of superhydrophobic cotton fabrics based on SiO_2 nanoparticles and ZnO nanorod arrays with subsequent hydrophobic modification," *Surf. Coat. Technol.* 24, 1556-1561. DOI: 10.1016/j.surfcoat.2009.09.086

Xu, X. F., Chen, P., Chen, C. X., Liu, H. M., and Yang, Y. R. (2010b). "Study on the fast degumming process of jute by microwave-accelerated alkalization treatment," *Shanghai Textile Science & Technology* 38(12), 26-29. DOI:10.16549/j.cnki.issn.1001-2044.2010.12.009

Xu, L. H., Wang, L. M., Shen, Y., Ding, Y., and Cai, Z. S. (2015). "Preparation of hexadecyltrimethoxysilane-modified silica nanocomposite hydrosol and surperhydrophobic cotton coating," *Fiber Polym.* 16(5), 1082-1091. DOI: 10.1007/s12221-015-1082-x

Zafar, M. T., Maiti, S. N, and Ghosh A. K. (2016). "Effect of surface treatment of jute fibers on the interfacial adhesion in poly(lactic acid) jute fiber biocomposites," *Fiber Polym.* 17(2), 266-274. DOI: 10.1007/s12221-016-5781-8

Article submitted: August 1, 2019; Peer review completed: November 3, 2019; Revised version received and accepted: November 11, 2019; Published: November 14, 2019.
DOI: 10.15376/biores.15.1.290-301