Effect of silane KH550 on interface of basalt fibers (BFs)/poly (lactic acid) (PLA) composites

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ABSTRACT – REZUMAT

Effect of silane KH550 on interface of basalt fibers (BFs)/poly (lactic acid) (PLA) composites

The basalt fibers/poly (lactic acid) composites, as a kind of green environment friendly materials, were prepared by vacuum perfusion method. The effect of silane coupling agent KH550 on the interface of BFs/PLA composites was analyzed. We observed the microstructure of the surface of BFs treated by silane KH550 and the interface of the composites, explored the chemical reaction among silane KH550, BFs and PLA. The silane KH550 succeeded in linking BFs and PLA and enhanced the interfacial bonding strength between BFs and PLA matrix. The crystalline properties of KH550-treated BFs/PLA composites were better than untreated BFs/PLA composites. The mechanical test suggested the silane KH550 had good effect on strengthening BFs/PLA composites.

Keywords: *basalt fibers, poly (lactic acid), silane KH550, microstructure, interfaces*

Efectul silanului KH550 asupra interfeței compozitelor din fibre de bazalt (BF)/acid (poli) lactic (PLA)

Compozitele din fibre de bazalt/acid (poli) lactic, ca material ecologic, au fost realizate prin metoda infuziei în vacuum. S-a analizat efectul agentului de cuplare silan KH550 asupra interfeței compozitelor BF/PLA. Au fost analizate microstructura suprafeței BF-urilor tratate cu silan KH550 și interfața compozitelor și a fost studiată reacția chimică dintre silan KH550, BF și PLA. Silanul KH550 a reușit să lege BF și PLA și a îmbunătățit rezistența legăturii interfeței dintre matricea BF și PLA. Proprietățile de cristalinitate ale compozitelor BF/ PLA tratate cu KH550 au fost mai bune decât cele ale compozitelor BF/PLA netratate. În urma testătii fizico-mecanice, s-a observat că silanul KH550 a condus la îmbunătăţirea rezistenței compozitelor BF/PLA.

Cuvinte-cheie*: fibre de bazalt, acid (poli) lactic, silan KH550, microstructură, interfeţe*

INTRODUCTION

Basalt fibers (BFs), a kind of silicate fiber, are made of natural volcanic exhalation-basalt. The broken basalt is added into a furnace and melted at 1450°C–1500°C, and then leaked from a platinum-rhodium alloy wire drawing plate to form continuous filament [1–5]. BFs are eco-friendly fibers, which attract lots of attention from researchers and constructors because of their strong tensile strength, high elastic modulus, high abrasion strength, good temperature-resistance, excellent heat and sound insulation, good chemical stability and so on [6–9]. Therefore, BFs are called as a green industrial material in 21st century.

In order to enhance the polymer matrix, plastics and cements, BFs are often used as reinforcements [10–14]. The composites, which is made of biomaterial-PLA as matrix and BFs as reinforcement, can meet the general requirements of light weight, high strength [15–17]. Furthermore, it possesses a large variety of excellent comprehensive properties, such as degradable, non-toxic, harmless, environmentally friendly and so on. Thus, the BFs/PLA composites are widely used in the fields of medical devices, automobile shell, leisure sports goods, wind power fan leaf and others [18–20].

However, the compatibility between BFs and PLA matrix is very poor, which leads to poor interface performance and decreased mechanical properties for the BFs/PLA composites [21–24]. Therefore, the interface between BFs and PLA is need to be modified.

Kurniawan et al. evaluated the effects of atmospheric pressure glow discharge plasma polymerization on BF to the properties of BFs/PLA composite [25]. Ying et al. reported that treatment of BFs with silane coupling agent to improve mechanical properties of composites [26].

Previous studies focused on the effect of interfacial treatment on the properties of composites, without further study on the specific process and mechanism of silane coupling agent acting on the interface [27–29]. In this paper, in order to enhance the adhesion and durability of fibers-matrix interfaces of BFs/PLA composites, the silane coupling agent of KH550 was applied to treat the surface of BFs, then the BFs and PLA were chemically linked by KH550. While the influence of silane KH550 on tensile fracture ability of composite is studied, the interface structure and interfacial bonding principle are analyzed. It provides more ideas for the latter to solve the interfacial problem of composites.

EXPERIMENTAL WORK

Material and methods

The samples of 200 mm × 200 mm basalt fabric (fiber-unidirectional cloth, plain texture), were soaked in 5% alkali solution (100 mL) for 30 minutes, and then were washed for at least 3 times and dried in a vacuum oven at 80°C. After that, the treated basalt fabric was immersed in 100 mL alcoholysis solution of KH550 with different concentrations for 1 h at room temperature. 50 g dried PLA powder (levorotatory, 51000 viscosity-average molecular weight), were added in 100 mL dichloromethane, and then they were magnetically stirred until completely dissolved together. PLA resin (viscosity, 258 mPa·s) was poured into five layers of unidirectional basalt fabrics by vacuum perfusion method. And the vacuum remained at –0.01 mPa. Then the BFs/PLA composites were dried in a vacuum oven at 80°C for 2 h.

The surface morphology of BFs and composites were inspected by scanning electron microscopy (JEM2100F). FTIR spectra of BFs, PLA and composites were recorded with a FT-IR (TENSOR27). Every spectrum was recorded from 400 to 4000 cm^{-1} using 4 cm^{-1} of resolution. The crystalline structure of composites and PLA were analyzed by an X-ray diffractometer (JSM-6700F) with a scanning range of 10–60°. The tensile tests of composites were carried out on a universal testing machine with a spline size of 250×25×2 mm and a tensile rate of 2 mm/min, and conducted in accordance to the standards GB/T 1447-2005.

RESULTS AND DISCUSSION

SEM analysis

The surface morphologies of untreated BFs, treated BFs, untreated-BFs/PLA composites and treated-BFs/PLA composites were depicted in figure 1, *a–d*. Figure 1, *a* shows that the surface of untreated BFs is not damaged, and very smooth. In contrast, the surface of treated BFs (figure 1, *b*) produces new substances that make BFs rougher and forma dense protective film on the surface of BFs. Moreover, the adhesion between fibers causes BFs to be more closely entangled. Additionally, the surface microstructure of untreated-BFs/PLA composites are showed in figure 1, *c*. Fibers are connected by a matrix, but the interface between fibers and matrix is very clear. Once the composites are damaged by force, some cracks will appear at the weak interface junction firstly. There are not any obvious interface-separations between BFs and PLA matrix, as shown in figure 1, *d*. The PLA resin completely infiltrates the BFs bundle, and be integrated with BFs tightly. These indicate that the KH550 can well link BFs with PLA matrix, and then improve the compatibility between BFs and PLA matrix.

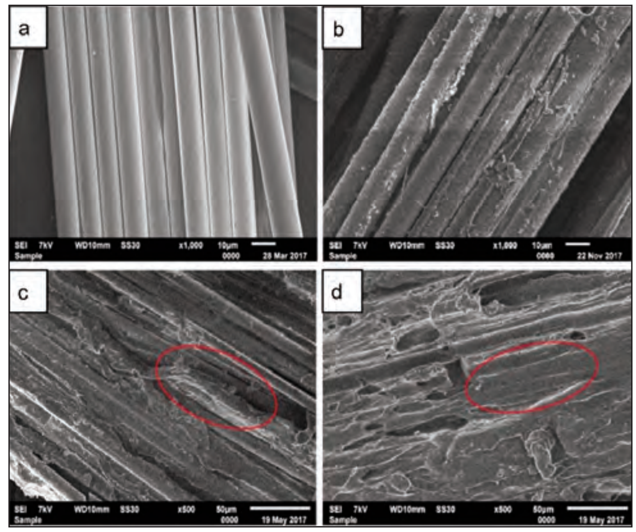

Fig. 1. SEM images of BFs and composites: *a* – untreated BFs; *b* – treated BFs; *c* – untreated-BFs/PLA composites; *d* – treated-BFs/PLA composites

FTIR analysis

In figure 2, the strong absorption peak of untreated BFs at 849 cm^{-1} is Si–OH stretching vibration, and the absorption peak at nearly 1633 cm^{-1} is the residual –OH stretching vibration. The spectra of treated-BFs shows that a characteristic peak at 1029 cm^{-1} due to Si–O–C stretching vibration of organosilicon compound or Si–O–Si asymmetric stretching vibration, and that some weak peaks at 1300–1100 cm^{-1} are assigned to the C–N stretching vibration of primary amine. These new chemical functional groups are formed on the surface of treated-BFs which suggest that the KH550 exists on the surface of BFs. The characteristic absorption peaks of PLA are the vibration peaks of C=O bond at 1754 cm^{-1}, the bending peak of $–CH_3$ group at 1380 cm^{-1} and the vibration

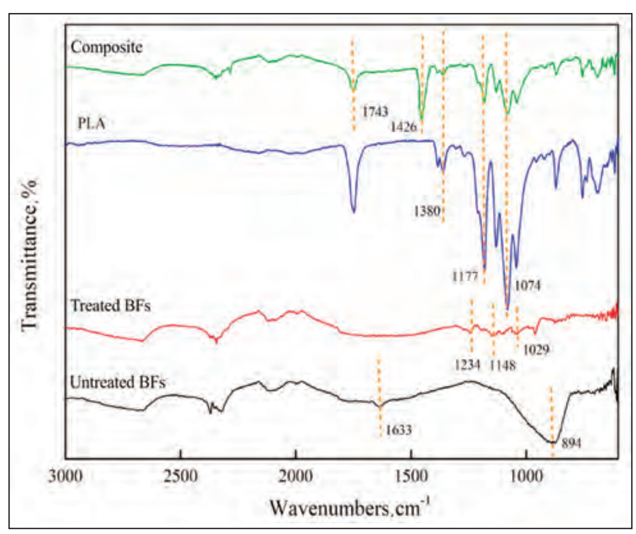

Fig. 2. FTIR spectra of untreated BFs, treated BFs, PLA and composite

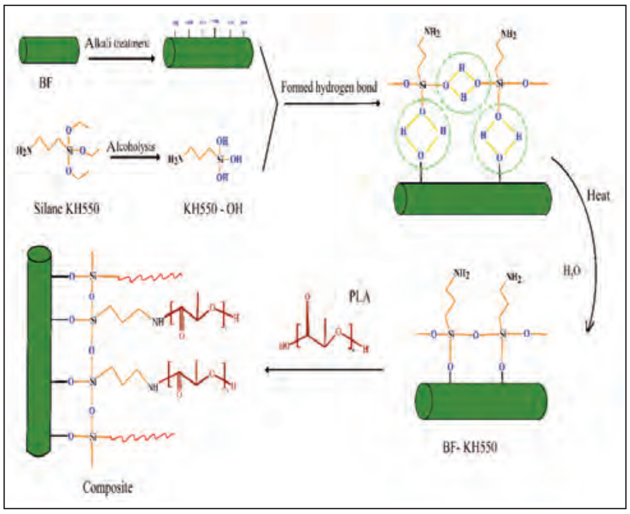

Fig. 3. The chemical reaction between PLA, BFsand silane KH550

peaks of C–O bond at 1117 cm^{-1}, and 1074 cm^{-1}. From the spectra of BFs/PLA composites, it can be clearly seen that the absorption peaks of BFs/PLA composites contain the characteristic absorption peaks of PLA and that of treated BFs. Moreover, the absorption peak of C–N stretching vibration of amide is appeared at 1426 cm^{-1}.

Thus, according to the changes of chemical bonds and functional groups, the chemical reactions among PLA, BFs and silane KH550 can be deduced, as shown in figure 3.

XRD analysis

Furthermore, the crystal structure of untreated-BFs/PLA composites, treated-BFs/PLA composites and PLA were examined by XRD patterns (figure 4). Based on the XRD results, the PLA displays two peaks at diffraction angles of 16.86° and 18.89°, which are related to relatively stable lattice planes of 110 and 203 respectively. The composites have some similar diffraction peaks, but the position of diffraction peaks moves to the right, which may be affected by grain size, lattice distortion or internal

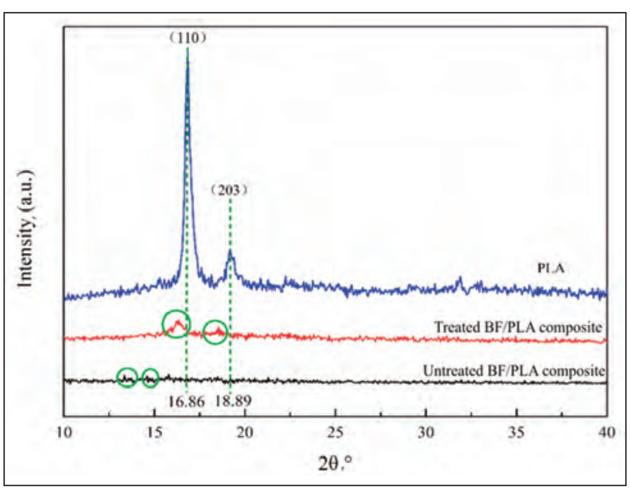

Fig. 4. XRD patterns of untreated BFs/PLA composite, treated BFs/PLA composite and PLA

stress. Moreover, the intensity of diffraction peaks becomes weaker, revealing that the added-BFs reduce the crystal grains and crystallinity of PLA matrix. However, the characteristic peak of untreated-BFs/PLA composites is extremely small and even disappeared, which demonstrates that this composite becomes a disordered amorphous state. The results that the crystallinity of treated-BFs/PLA composite is better than that of untreated-BFs/PLA composite, and the KH550, used to treat BFs, can improve the crystallinity of composites.

In addition, if the BFs are treated by different concentrations of KH550, the tensile strength of composites will be improved, as shown in figure 5.

Meachnical properties

In addition, if the BFs are treated by different concentrations of KH550, the tensile strength of composites will be improved, as shown in figure 5. The interface treatment effect was the best when the concentration of KH550 was 3%wt. The fracture failure of BFs/PLA composite mainly occurs in the interface area. The KH550 can link BFs with PLA matrix tightly, and formed a silane network layer which perform a function of micromechanical meshing, thus the interfacial bonding strength is improved. Furthermore, the KH550 between BFs and PLA matrix can promote the transfer of stress, so that the PLA matrix can bear more loading. At the same time, the KH550 can reinforce the PLA matrix directly, and enhance the bonding strength between the inner interlayer and adjacent composite layer, after that, the mechanical properties of composites are increased.

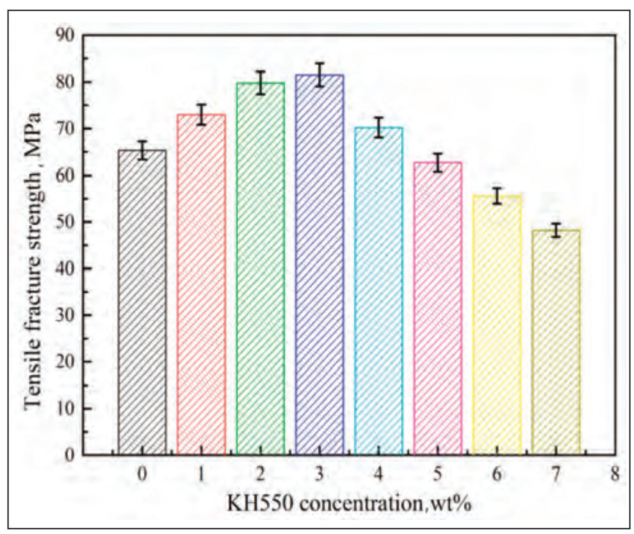

Fig. 5. Effect of KH550 concentration on tensile fracture strength of BFs/PLA composite

CONCLUSIONS

The composites, which were made of PLA as matrix and KH550-treated BFs as reinforcement, were prepared by the vacuum perfusion method. The mechanical properties of composites had been significantly improved. The results of SEM analysis indicated that the KH550 built a "bridge" between BFs and PLA matrix successfully, which combined BFs and PLA matrix tightly. The FTIR revealed that one end of KH550 was connected with the hydroxyl groups of BFs, and the other end of KH550 was connected with the carboxyl groups of PLA. The XRD analysis indicated that the crystalline properties of KH550-treated BFs/PLA composites were better than untreated-BFs/PLA composites.

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