The Characterization of Mechanical Properties of Jute/PLA Composites with Modified Nano Sio_2 by Coupling Agent

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Research Article

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Abstract

Although fibers-reinforced PLA composites show strong application prospects, their low mechanical properties limit their applications to some extent. In this paper, nano-SiO_2 particles as well as modified nano SiO_2 by coupling agents which can efficiently improve the strength and toughness of composite materials are introduced into the PLA matrix. The bending, stretching and thermal properties of designed jute/PLA nonwoven composites were studied. The study shows that the nano-SiO_2 particles are beneficial for the interface performance between the PLA matrix and jute leading to improvement in the mechanical property and thermal stability. Moreover, thermomechanical properties indicate that the addition of SiO_2 can improve the jute/PLA interfacial adhesion and increase the glass transition temperature of the material. Finally, toughening mechanism of nano-SiO_2 particles in the jute/PLA composite was analyzed.

Introduction

In recent years, thermoplastic composites are increasingly applied in high-performance engineering field due to their low density, good recyclability, and high production efficiency. Thermoplastic composites development speed has gradually surpassed thermoset material(Faruk et al. 2012). However, environmental compatibility of these material is still a huge challenge. In this case, biodegradable polymers as matrix of composite material supplies a feasible solution. Polylactic acid (PLA), a polymer material, is produced from renewable agricultural raw materials. It has tremendous advantages such as high strength, high hardness, transparency and industrial production, and is one of the most promising biodegradable polymers(Qu et al. 2015). Therefore, researchers have promoted tremendous researches on fibers reinforced PLA composites(Bax and Mussig 2008; Pan et al. 2007; Nishino et al. 2003) among which adopting natural plant fibers instead of glass fibers or synthetic fibers as filling materials to produce fibers-reinforced thermoplastic composites has attracted increasing attention(Mohammed et al. 2019). However,challenges on materials and processing relating to natural fibers reinforced composite still need to be tackled for long-term stability and high performance(Balla et al. 2019; He et al. 2019; Hossen et al. 2020). The natural fibers in these composite materials are economically and environmentally beneficial because they are abundant, recyclable, and biodegradable(Dong et al. 2014). However, Jute fibers reinforced PLA composites are poor in the mechanical properties, which limits their wider industrial applications(Pereira et al. 2019; Costa et al. 2012). Therefore, some strategies have been proposed. Kakroodi et al.(Kakroodi et al. 2012) studied the effect of coupling agent (SEBS) on the interface property between flax fibers and polyethylene matrix. Dong(Dong et al. 2020) used horseradish peroxidase-mediated functional hydrophobization of jute fabrics to enhance mechanical properties of jute/thermoplastic composites. The tensile and dynamic mechanical properties of the grafted jute/PP or jute/PLA composites were improved, providing a high-performance composite that is more environmentally friendly and has better interface adhesion.

Huda et al.(Huda et al. 2007) modified kenaf fibers combining alkalization and silane treatments. It was found that the interface compatibility between the hydrophilic natural fibers and the hydrophobic PLA

matrix was improved, and the silane-treated fibers-reinforced composites after fibers were immersed in sodium hydroxide solution (5% w/v) for 2 h at room temperature showed better bending strength and energy storage performance. Fang et al.(Fang et al. 2020) studied the effect of the hot-pressing temperature and compression molding time on mechanical properties of degradable composite materials made with needle-punched jute non-woven fabric and PLA film. Due to sufficient impregnation, the laminated composites prepared at 190 ℃ for 3 h showed excellent comprehensive mechanical properties in terms of tensile properties and bending properties. Kumar et al.(Kumar et al. 2010) added montmorillonite clay into the PLA matrix to study its effect on linen fabric reinforced composites. The results show that the Young's modulus of the hybrid composite is significantly increased, the tensile strength is slightly decreased, and the elongation at break is seriously decreased. This could be due to the weakening of the modifier/matrix interface bond and the higher stiffness of montmorillonite clay compared with PLA resin.

Based on these, the dispersed rigid organic particles have good adhesion with the matrix interface to provide better stress transfer condition. Therefore, in present research, nano-SiO_2 particles were adopted and added to the PLA matrix as a stiffening modifier. Moreover, considering the dispersibility, the nano-SiO_2 was modified with different silane coupling agents to prepare PLA-based composite films. The effect of nano-SiO_2 modified by silane coupling agents on the mechanical properties of jute/PLA laminated composites prepared by hot-pressing technology was studied.

Materials And Methods

Materials

PLA (4032D; 1.24 g/cm^3) was supplied by Nature Works, USA; Jute fibers (1.45 g/cm^3) was purchased from Shandong Jiangke Linyi Co., Ltd; Nano silica (SiO_2, 99.5%, 50 ± 5 nm) was provided by Suzhou Great Medical Technology Co., Ltd; Silane coupling agent (KH550, KH560, KH570) chemicals were provided by Jiangsu Argon Krypton Xenon Material Technology Co., Ltd.

Preparation of modified SiO_2

The absolute ethyl alcohol and deionized water were first mixed with a mass ratio of 9:1, and 5 g of SiO_2 was added into 100 mL of mixture. The SiO_2 solution was then sonicated in an ultrasonic cleaner for 1 h. The coupling agent, e.g. KH550, with a volume of 5% of the mixture was prepared and adjusted to the pH of 4 with oxalic acid. The above mixed solutions were magnetically stirred for 3 h at 60 ℃ for reaction. Finally, it was centrifuged and then washed with absolute ethanol solution for 3 times. The obtained precipitate was dried in an oven at 60 ℃ for 4 h to obtain modified SiO_2 powders, named as KH550-SiO_2, KH560-SiO_2 and KH570-SiO_2 respectively. The scheme of preparation process of modified SiO_2 is illustrated in Fig. 1.

Preparation of composite SiO_2-PLA films

3 g of PLA raw material was dissolved it in dichloromethane, stirred magnetically for 1.5 h at room temperature; different mass fractions of modified SiO_2 (0.5 wt%, 1 wt%, 2 wt%, 4 wt% and 8 wt%) were mixed into dichloromethane and ultrasonicated for 40 min. These two solutions were mixed and magnetically stirred for 2 h, followed by ultrasonication for 1 h at 35 ℃. Then, the solution was poured into a glass plate and placed in a fume hood at room temperature to make the methylene chloride solvent completely volatilize. Finally, the composite SiO_2-PLA film was peeled from the mold and placed in a vacuum oven at a temperature of 45 ℃ for 3 h, as shown in Fig. 2.

Preparation of jute non-woven

The preparation process of jute non-woven is shown in Fig. 3. First, the fluffy jute fibers was combed, opened and mixed through a coarse-garge carding machine to make the fibers looser and more uniform. Then the loose jute fibers were sent to the blender for further fine opening. The fine opening and carding process make the jute fibers finally become a fluffy and uniform fiber web. The fiber web obtained by cross-netting has higher quality. In order to obtain a certain strength of non-woven fabrics, the four-layer jute net were finally reinforced with a needle punch. The final thickness of the jute nonwoven is 0.51 ± 0.04 mm, and the surface density is 200 ± 2 g/m^2.

Preparation of composite material

To remove all moisture, SiO_2-PLA films and jute nonwoven were first dried in a vacuum oven at 80 ℃ for 6 h before hot pressing. The laminated composite material was processed through a compression mold using a film stacking method, in which two jute pads were arranged between three PLA films in parallel, as demonstrated in Fig. 4. The entire assembly was then placed in a steel mold controlled under different time and temperature. The laminated material is compacted for 0.5 h under the conditions of a temperature of 180 ℃ and a pressure of 1 MPa, and then is naturally cooled in the equipment for 8 h. After 8 h, the sample was taken out of the laminated mold with a size of 150 mm×150 mm. The samples are numbered according to the type of coupling agent and concentration. For example, 550 – 0.5, in which 550 represents the silane coupling agent of KH550, the sample untreated with the coupling agent is directly represented by SiO_2, and 0.5 represents 0.5 wt% of the modified SiO_2 mass fraction in SiO_2-PLA film.

Testing of samples

In order to study that effect of different silicon dioxide on the mechanical properties of jute/PLA composite, the SiO_2 modified by silane coupling agent was characterized by infrared transmission spectroscopy (FTIR) and particle size analyzer (DLS). At the same time, the mechanics of composite materials were characterized by scanning electron microscope, dynamic mechanical thermal analysis (DMTA) and related mechanical testing methods.

Results And Discussions

The composition and physical property of modified SiO_2

Figure 5 demonstrates the FTIR spectra of SiO_2, KH550-SiO_2, KH560-SiO_2 and KH570-SiO_2. It can be seen from Fig. 5 that the infrared spectrum of the SiO_2 before and after modification has a large absorption peak near 1100 cm^{-1}, which is the antisymmetric absorption peak of Si-O-Si bond, and near 800 cm^{-1} is the symmetrical contraction vibration of the Si-O-Si bond. The peaks at 460 cm^{-1} and 1055 cm^{-1} respectively correspond to the Si-O-Si and Si-O bending vibrations of SiO_2 nanoparticles. The infrared spectrum of KH550-SiO_2 is relatively flat compared to others in the range of 3500-3100cm^{-1}, indicating that -NH_2 in KH550 has been successfully grafted and modified on the surface of nano-SiO_2. The -CH_2- in KH550-SiO_2 has a shoulder peak at 2940 cm^{-1} and 1460 cm^{-1}, which further shows that KH550 has successfully modified nano-SiO_2. Compared with FTIR spectrum of SiO_2, in the spectrum modified by KH560-SiO_2, the stretching vibration absorption peak of methyl appears near 2987 cm^{-1}, indicating that KH560 silane coupling agent has been successfully grafted to the second Si-O surface(He et al. 2019). In the infrared spectrum of KH570-SiO_2, there is a C = O stretching vibration absorption peak on the carbonyl group at 1718 cm^{-1}, which indicates that KH570 has been successfully grafted onto nano-SiO_2.

The particle size of original and modified SiO_2 is shown in Table 1. It can be clearly seen that the unmodified particles are larger than modified particles, and KH560 has the best modification effect on SiO_2 particles. Unmodified nano SiO_2 has a relatively high polymer dispersity index (PDI), a wide molecular weight distribution, and serious agglomeration. The particle size and PDI of the modified nano-SiO_2 powder are reduced, and the molecular weight distribution is uniform, indicating that the silane coupling agent modified nano-SiO_2 can effectively prevent its agglomeration, so as to achieve the purpose of modification. On the one hand, the hydroxyl groups on the particle surfaces are replaced by organic functional groups, reducing the number of active silanol groups, thereby reducing the tendency of nanoparticles to agglomerate. On the other hand, the grafted long carbon chain increases the distance between particles and the steric hindrance of the hydroxysilyl polycondensation reaction on the particle surface, which increases the difficulty of effective collisions and more conducive to the dispersion of nanoparticles.

Table 1
The spectification of prepared nano SiO_2

Sample	Z-Average (nm)	Peak Size (nm)	Peak Intensity (%)	PDI
SiO_2	300	220.8	65.2	0.343
KH550-SiO_2	107.7	143.3	69.3	0.287
KH560-SiO_2	67.6	92.7	100	0.210
KH570-SiO_2	91	136.6	100	0.229

Bending property

Figure 6 (a) is a histogram which shows the bending strength and modulus of pure jute/PLA fibers composites involving SiO_2 and modified SiO_2 nanoparticles with different mass fractions. It can be seen that the bending modulus of jute/PLA fibers composites is relatively low. With incorporation of SiO_2, the bending strength and modulus of the composites is more or less improved. It is also found from Fig. 6 (a) that as the mass fraction of different types of added SiO_2 increases, the change trend of material bending strength and modulus is basically same.

The similar phenomena can be also observed in the bending strain and fracture toughness of jute/PLA composites. The toughness was calculated by integrating the tensile stress–strain curves. As shown in Fig. 6 (b), the increase of SiO_2 improves the bending strain and fracture toughness of jute/PLA composites.

In short, the bending performance of the sample increases with the increase of the SiO_2 mass fraction, peaks at about 1wt%, and then decreases. That is, under the same mass fraction, SiO_2 treated with different silane coupling agents has the same effect on the jute/PLA composite. The SiO_2 sample modified with KH560 has the best bending strength and modulus. It is mainly due to the fact that realize the interface combination through the coupling agent, forming a better entirety, and then strengthening the effect of stress transmission(Yang et al. 2009; Sanivada et al. 2020; Fang et al. 2020; Li et al. 2013). Sample 560-1 has best bending performance resulting from the excellent dispersibility and uniform particle size as illustrated in Table 1.

The fracture morphology

In order to illustrate the toughening mechanism, the fracture morphology of the reinforced PLA-based composite after the tensile test is shown in Fig. 7. The tensile fracture surface of the control group, illustrated in Fig. 7 (a), shows large amounts of fiber extraction leading to a lower mechanical resistance to some extent. This is consistent with comparatively large elongation ratio of the composite material

which also indicates a poor interfacial cohesiveness. Figure 7 (b) is the SiO_2 particles modified by KH560 with the mass fraction of 0.5 wt% were distributed in PLA matrix. Nano-SiO_2 particles induce local plastic deformation of the PLA matrix, showing a rougher fracture surface. As the stress concentration point, SiO_2 particles produce a large number of small cracks. As the mass fraction of SiO_2 increases, there are more contact points between SiO_2 and jute, which reduces the adhesion between jute and PLA, increases fracture cracks, and has a small amount of fibers extraction, as shown in the Fig. 7 (c). There are big cracks and holes in the fracture of 560-4 sample. The holes consume a lot of energy, as shown in Fig. 7 (d). Due to the poor dispersibility of SiO_2, the modified SiO_2 re-aggregated at 8% by mass fraction, and the mechanical properties dropped rapidly, as shown in Fig. 7 (e).

Stretching property

The tensile property of the control group and the nano-SiO_2 jute/PLA composites is shown in Fig. 8. It is concluded from Fig. 8 that the cooperation of nano-SiO_2 can effectively improve the tensile tolerance of composite materials. With the increase of nano-SiO_2 content, the change trend of the tensile properties of the composite material before and after modification is basically the same. And when the SiO_2 mass fraction is same, the sample modified by KH560 has the best tensile strength. This can be attributed to the small particle size and uniform shape of SiO_2 modified by KH560, which is consistent with the DLS data.

When preparing jute/PLA laminates, SiO_2 can be more evenly dispersed in the matrix, thereby enhancing the effective load transfer between jute and PLA, improving the tensile properties of the composite material. The silane coupling agent can serve as a bridge to improve the compatibility of the interface between SiO_2 and jute/PLA, forming a better whole, and then strengthening the stress transmission. When the mass fraction of SiO_2 is 0.5%, the tensile strength is higher.. This is because the increase of a small amount of SiO_2 produces a large number of small cracks at the fracture interface and consumes energy. It takes more energy to propagate a large number of small cracks than a few large cracks. When the mass fraction of SiO_2 is 8%, the re-agglomeration of particles increases leading to the decrease of tensile performance. The strain decreased slightly, which may be due to the increase of SiO_2, the excessively high content of nanoparticles and the serious agglomeration.

Dynamic mechanical thermal analysis (DMTA)

Figure 9 respectively show the dynamic storage modulus (E'), loss modulus (E") and loss coefficient tanδ(E"/E')of jute/PLA composites with different contents of SiO_2 modified from room temperature to 180 ℃. As shown in Fig. 9 (a), the stored modulus values and corresponding temperatures of different samples at the beginning of chain segment movement. In the temperature range studied, the storage modulus of the jute/PLA composite modified by SiO_2 is higher than that of the untreated jute/PLA composite. In addition, different variables showed the same change pattern. With the increase of SiO_2 content, the storage modulus of the modified jute/PLA composite material increases first and then

decreases. The increase in storage modulus and corresponding temperature of chain segment starting to move indicates that the interface adhesion between jute and PLA has been improved, resulting in greater stress transfer between them(Porras and Maranon 2012). That is, the increase of the SiO_2 content can improve the interfacial adhesion, but because of the poor dispersibility of SiO_2, when the content is greater than 4%, the effect of the coupling agent on SiO_2 modification is not obvious, and the jute/PLA interface adhesion begins to decrease.

The next study parameter is the loss modulus, which represents the energy dissipated by the jute/PLA composite under stress(Laly et al. 2003; Doan et al. 2007). It is observed in Fig. 9 (b) that the loss modulus value of the SiO_2-modified jute/PLA composite is much higher than that of the untreated jute/PLA composite. It is known that the maximum value of loss modulus corresponds to the glass transition temperature (Tg) of the composite material. The Tg of the untreated jute/PLA composite is around 57.3 ℃. In contrast, the Tg of the SiO_2-modified jute/PLA composite material moved to the high temperature zone, reaching a maximum of about 74.2°C. This is because the addition of SiO_2 improves the bonding strength between PLA and jute, and reduces the fluidity of PLA macromolecules in the composite material.

Finally, loss factor, tanδ, which refers to the ratio of the loss modulus of the composite material to the storage modulus of the composite material, and represents the damping energy of the material. The decrease of the tanδ value indicates Fibers inhibits the fluidity of matrix. Due to the improvement of the hydrophobicity of the SiO_2-modified Jute/PLA, the interfacial adhesion is enhanced, and the fluidity of the polymer chains at the jute/PLA composite interface is reduced. Among the unmodified SiO_2 and SiO_2 jute/PLA composites modified by KH550, KH560 and KH570, the unmodified jute/PLA composites have the highest tanδ value, and the KH560 modified SiO_2 jute/PLA composites has the highest tanδ value, indicating the dispersibility of SiO_2 modified by KH560 is best. Among all SiO_2 composite material modified by KH560, jute/PLA with a SiO_2 content of 4% has the best interface bonding. As shown in Fig. 9 (c).

Comprehensive analysis of sample 560-4 has the best thermomechanical properties. Compared with the control group, the storage modulus, glass transition temperature and loss modulus have increased; tanδ has decreased. As shown in Fig. 9 (d), the comparison of DMTA between control group and sample 560-4. The glass transition temperature of sample 560-4 is 29.5% higher than that of the control group, indicating that the experimental optimization has improved the bonding strength of the interface between PLA and jute. The storage modulus values of different samples decrease with increasing temperature. The tanδ value of the jute/PLA composite material increased with the increase of temperature when the mass fraction of SiO_2 treated by KH560 was 4%, until it reached the maximum at 69.2℃, and then the opposite trend was observed in the rubber area.

Conclusions

In order to enhance the mechanical properties of the jute/PLA composite, nano-SiO_2 particles modified by different coupling agents were introduced into the PLA matrix. The effect of the type of coupling agent and the mass fraction of SiO_2 on the bending, stretching and thermomechanical properties of the composite was studied. The study shows that the addition of SiO_2 improves the interface performance between PLA and jute and improves the mechanical properties of the composite material. Meanwhile, the smaller the particle size of SiO_2 and the more uniform the particles, the better the mechanical properties of the composite material. The analysis of thermomechanical properties shows that the addition of SiO_2 can improve the thermomechanical properties and glass transition temperature of composite materials. In addition, as shown in the tensile and bending tests, the strength, stiffness and toughness of SiO_2 composite material with mass fraction of 4% modified by the KH560 coupling agent are significantly improved, and its elongation at break is slightly affected. Its excellent mechanical properties are the result of the better interface compatibility of jute/PLA caused by the SiO_2-CN bond. In addition, the fracture morphology of the composite material shows that the matrix microcracks and cavitation caused by SiO_2 are the main toughening mechanisms.

Declarations

Conflict of interest:

The authors declare that they have no conflict of interest.

Human or animal rights

This article does not contain any studies with human participants or animals performed by any of the authors.

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Figures

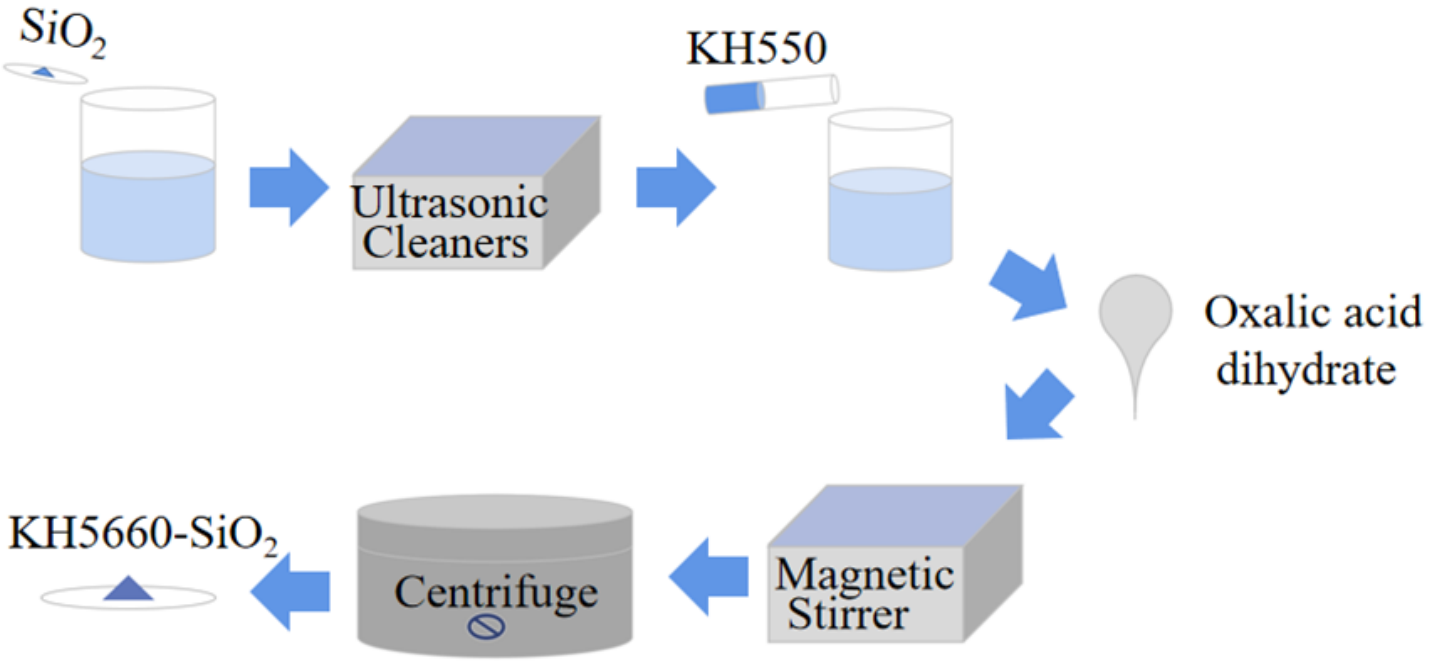

Figure 1

The scheme of preparation process of modified SiO2

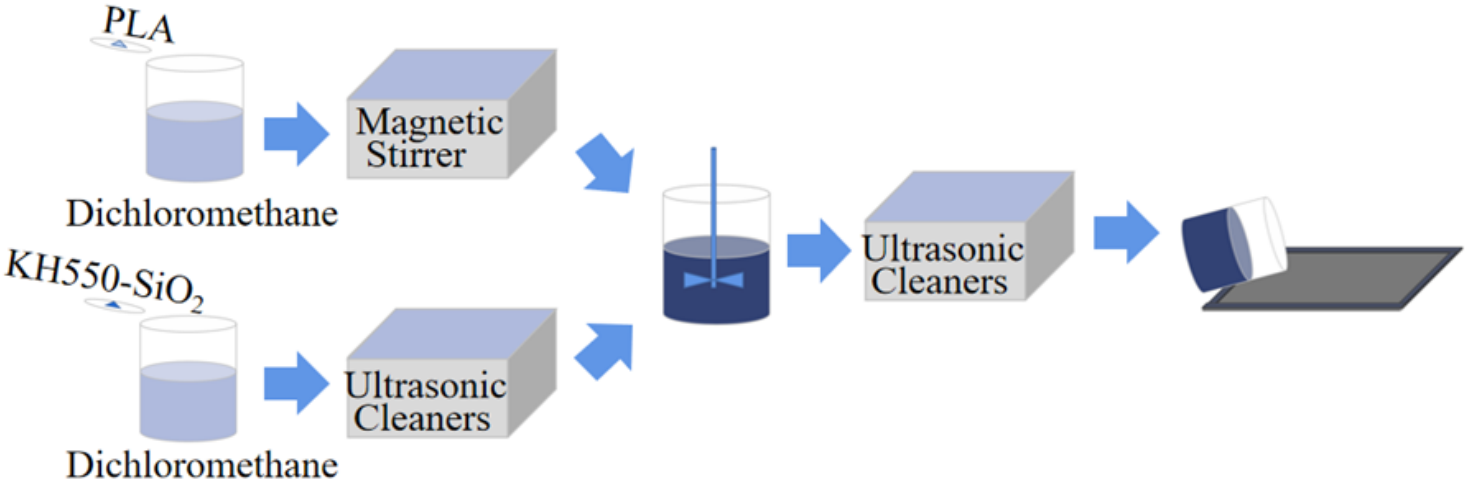

Figure 2

The scheme of preparation process of composite SiO2-PLA film

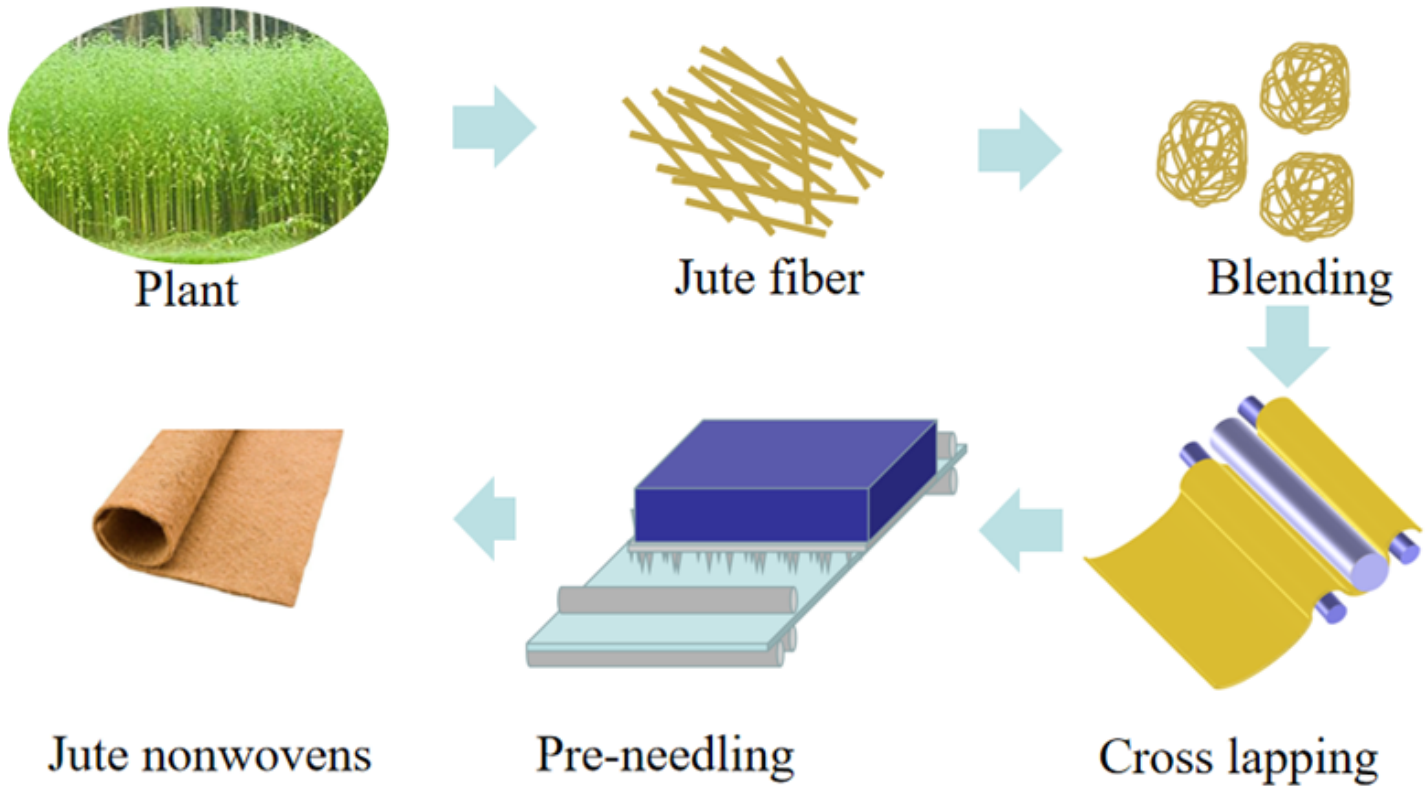

Figure 3

The preparation process of jute non-woven fabric

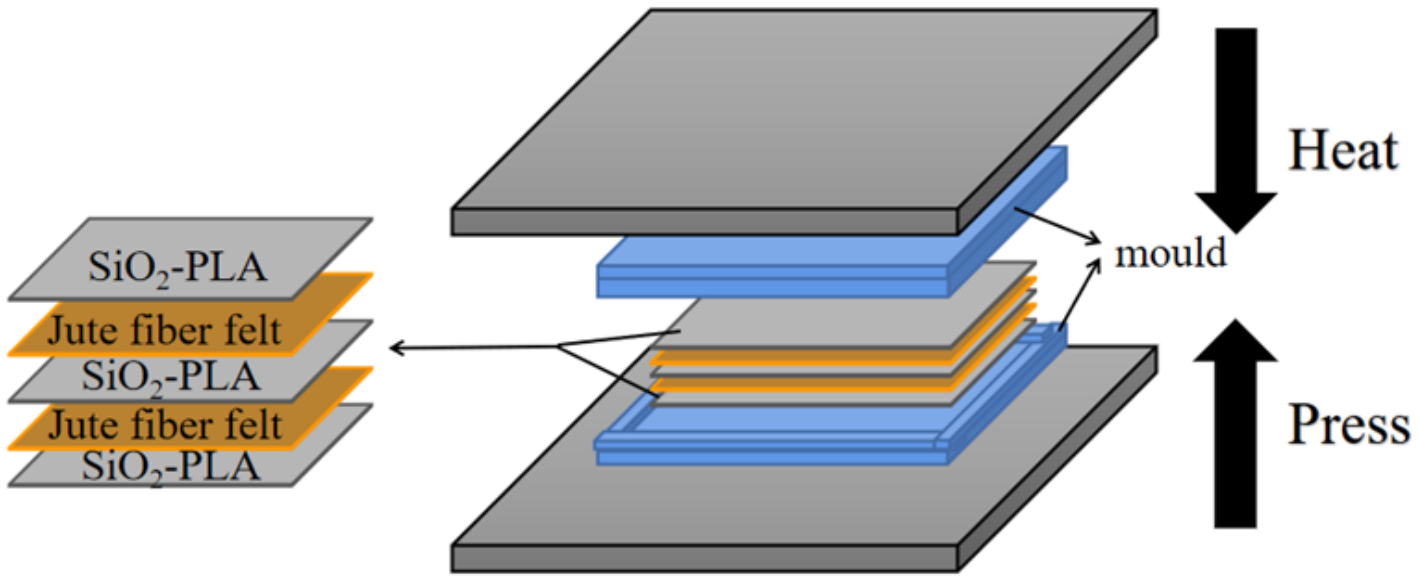

Figure 4

The preparation process of composite material

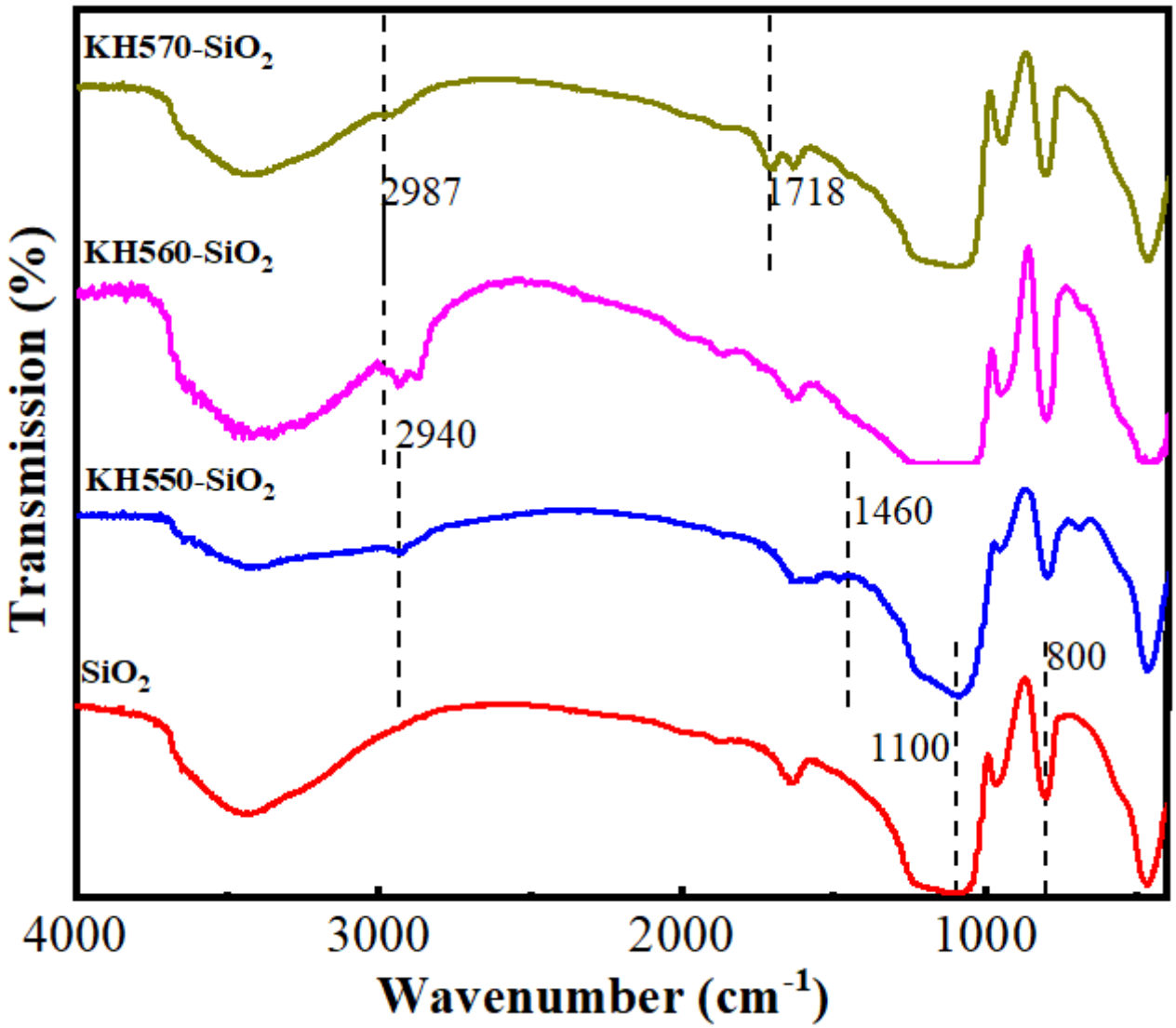

Figure 5

FTIR spectra of SiO2, KH550-SiO2, KH560-SiO2 and KH570-SiO2 nanoparticle

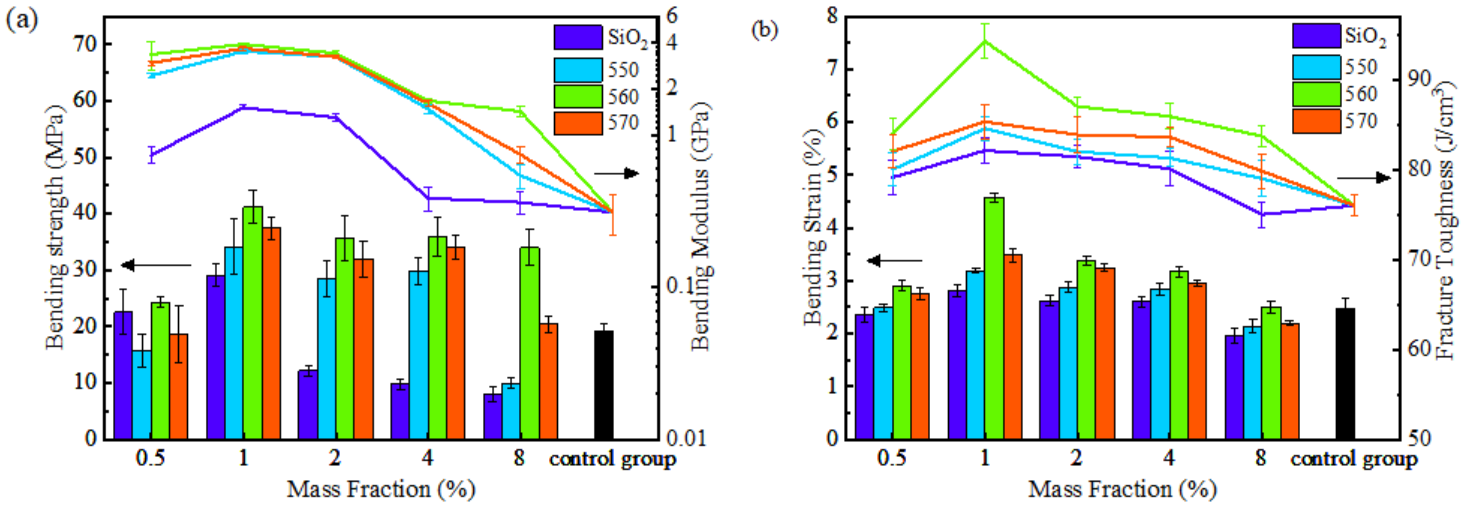

Figure 6

The effect of nano-SiO2 on tensile properties of jute/PLA composite bending performance. (a) Bending strength and modulus; (b) Bending strain and fracture toughness

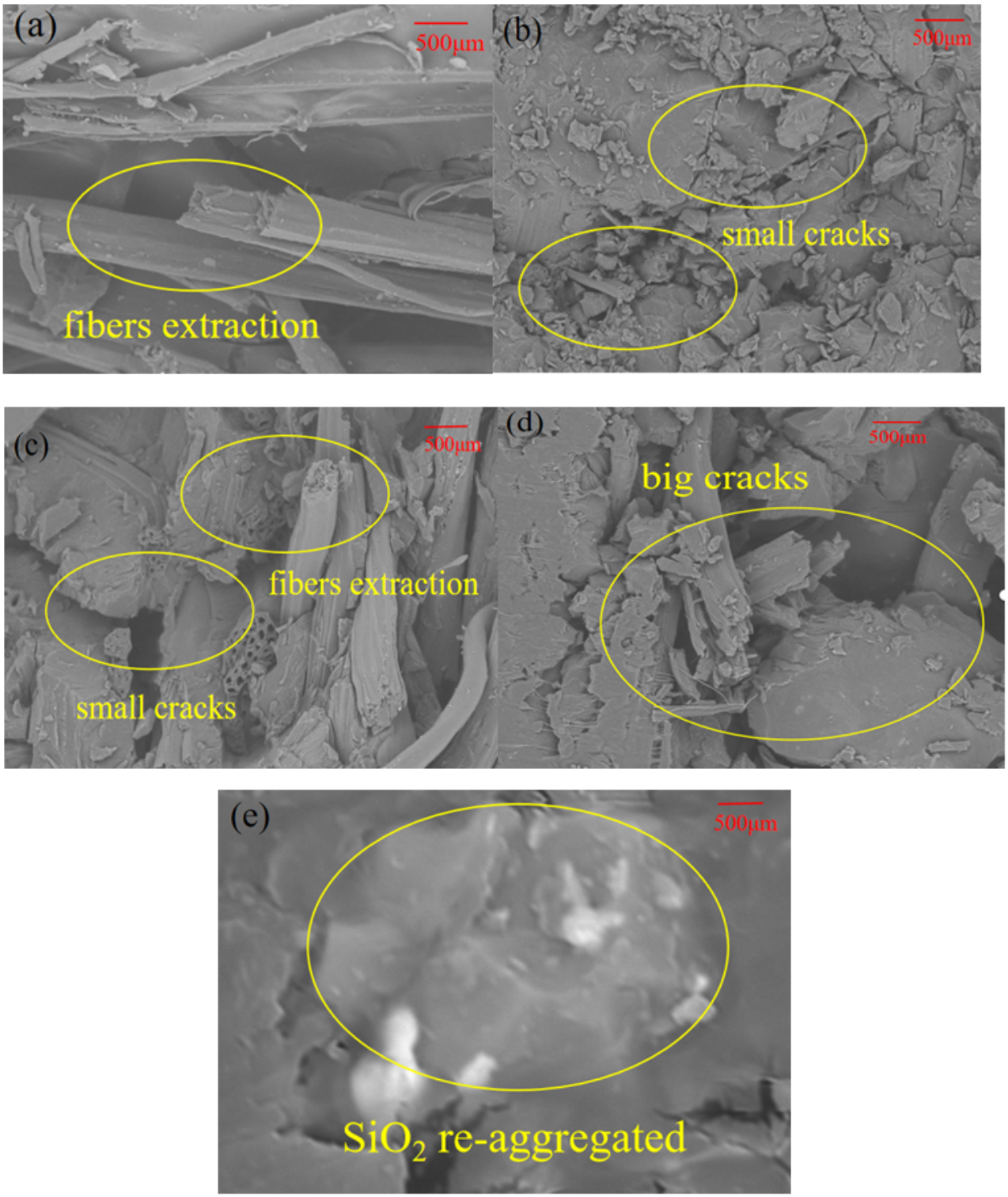

Figure 7

Fracture surface morphologies of jute/PLA composite. (a) Fracture surface morphologies of control group. (b) Fracture surface morphology of sample 560-0.5. (c) Fracture surface morphology of sample 560-2. (d) Fracture surface morphology of sample 560-4. (e) Fracture surface morphology of sample 560-8

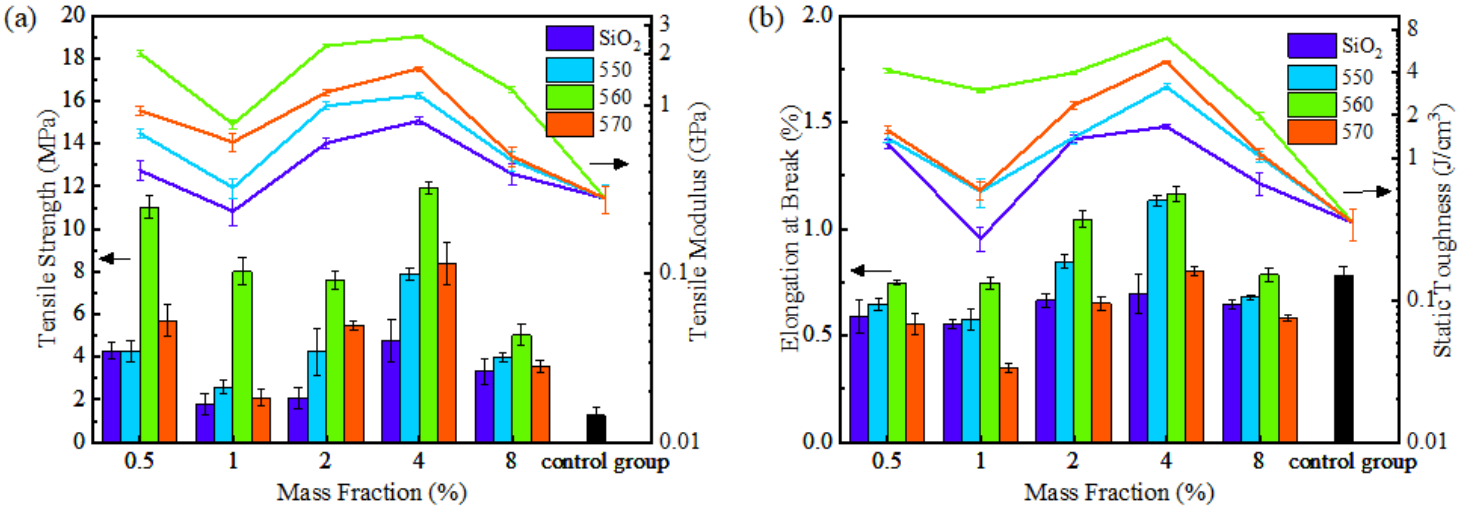

Figure 8

The effect of nano-SiO2 on tensile properties of jute/PLA fibers composites. (a) Tensile strength and modulus. (b) Elongation at break and toughness

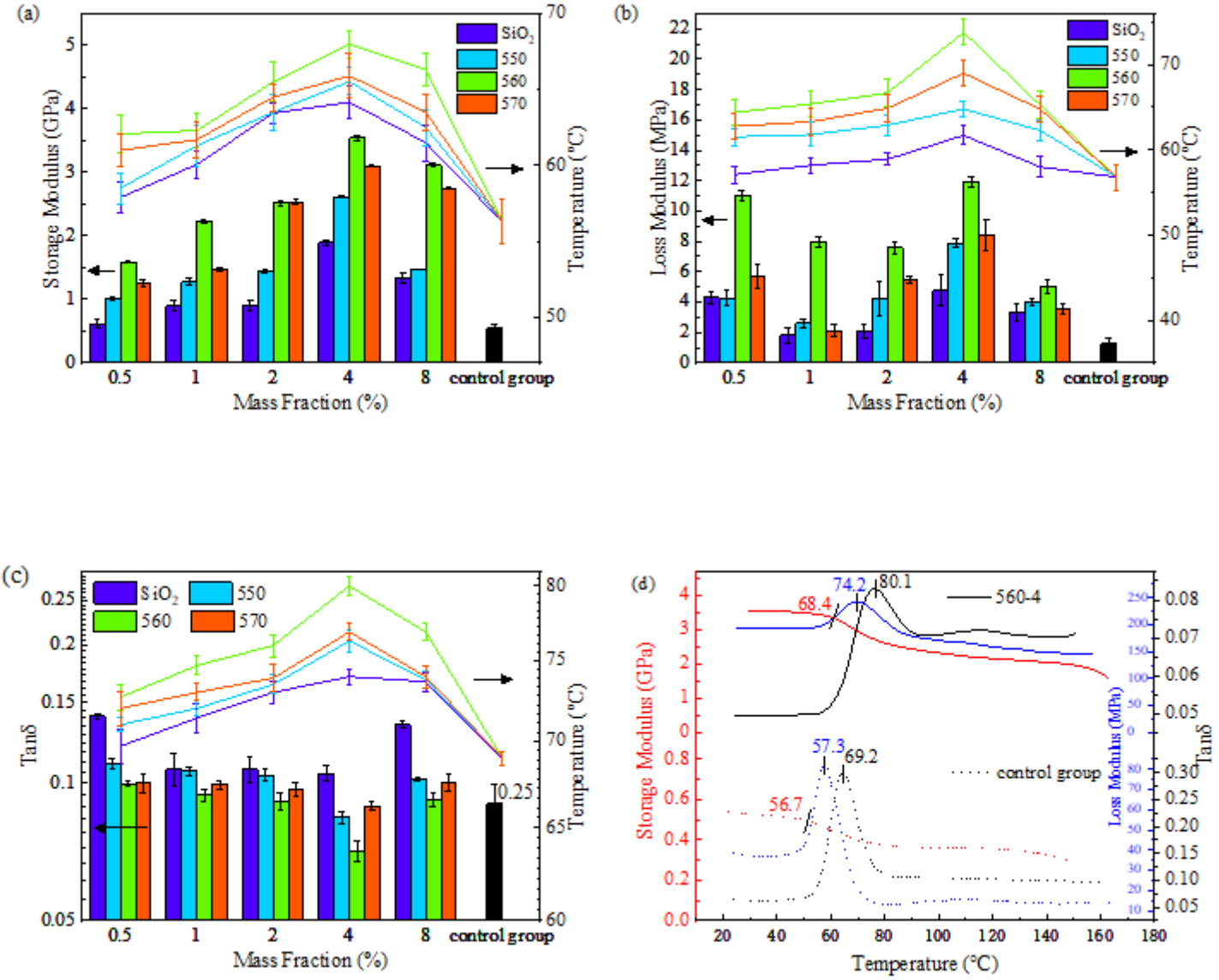

Figure 9

The DMTA of jute/PLA composite. (a) Storage modulus and corresponding temperature of chain segment starting to move. (b) The maximum loss modulus and corresponding temperature. (c) The maximum tanδ and corresponding temperature. (d) The comparison of DMTA between control group and sample 560-4