The Effect of Hot Water Treatment on the Properties of Lotus Leaves, Reed Leaves, and Basho Leaves Combined with Gelatin Composites

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This study investigated the feasibility of preparing biodegradable composites, such as food packing materials, from plant leaves as a substitute plastic. Lotus leaves, reed leaves, and basho leaves were treated with hot water and combined with gelatin to form composite samples. The effects on the morphology, thermal properties of leaves, and the mechanical properties and moisture absorption of the composites were studied. The Fourier-transform infrared spectroscopy (FTIR) analysis showed that the amorphous components such as lignin, wax, and pectin were removed after the hot water treatment. The treatment had the most beneficial effect on the reed leaf. The reed/gelatin composites had the best mechanical properties, of which the tensile strength and the flexural strength were 14.0% and 77.1%, respectively, higher than that of the lotus/gelatin composites and 121.5% and 192.5%, respectively, higher than that of the basho/gelatin composites. The morphology of the cross-section of the composites showed that there were numerous holes and gaps in the basho/gelatin composites which induced a high moisture absorption performance.

Keywords: Leaf; Gelatin; Compression molding technique; Hot water treatment; Composites

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INTRODUCTION

Currently, more than 90 countries and regions have issued relevant policies or regulations to control or prohibit disposable plastic products (Krishnaiah *et al*. 2017; Shanmugasundaram *et al*. 2018). In 2020, the Chinese government issued a series of policies to restrict the use of disposable plastic products. By the end of 2020, the Chinese government will prohibit the production and sale of disposable foam plastic tableware and nondegradable disposable plastic straws in the catering industry across the country. Currently, disposable plastic is still used in food packaging and tableware. This disposable plastic cannot be recycled, which can cause harm to the natural environment. As people become more involved in environmental protection efforts, there has been a surge in the development of biodegradable materials that can replace plastics to reduce environmental pollution (Sanjay *et al*. 2015; Mittal *et al*. 2016; Sepe *et al*. 2018).

There are many advantages to utilizing natural materials for the research and development of biomass composite food packing materials. Biomass composites can be degradable and economically viable substitutes for disposable plastics in some scenarios (Hanifi *et al*. 2014; Vivek and Kanthavel 2019). There has been some research on the use of biomass as food packaging materials. For instance, Ortega-Toro *et al.* (2015) prepared food packaging material composites made of corn starch and polycaprolactone (PCL). The composites had no toxic compounds and had good food compatibility, so they could be used in food packaging. Jancikova *et al.* (2019) prepared a biodegradable composite membrane by using the hydrolysate of gelatin, red algal glue, and dry leaves. The dry leaves were found to improve the oxidation resistance. The composite membrane had the best performance when the content of the dry leaves was 20%. Such a composite can be used to package food. In a study on the effect of gelatin on the properties of fiber-based packaging composites, the gelatin improved the plasticity, ductility, and mechanical properties of packaging materials (Khakalo *et al.* 2014).

Gelatin is extracted from animals' skin, bones, and other tissues. Although gelatin is a completely biodegradable biomaterial, it has poor mechanical properties and its aqueous solution will deteriorate after boiling for a long time (Oustadi *et al.* 2020; Yang *et al*. 2020). Plant leaves are an ideal raw material for biomass composites, as they are bio-degradable and widely available. However, leaves are commonly treated as domestic waste or made into feed, and there is little research regarding the application of plant leaves as a biomass composite material (Jiang *et al.* 2018).

Based on the lack of research on plant leaves and the wanton use of plastics, the main objective of this work was to investigate the application of plant leaves to replace plastics in food packing materials. Lotus leaves, reed leaves, and basho leaves were treated with hot water as preparation to produce gelatin composites *via* the compression molding technique. The mechanical and moisture absorption properties of these samples was measured. Additionally, in order to investigate the influence of the hot water treatment on the mechanical properties of the composite materials, the leaves were characterized through Fourier-transform infrared spectroscopy (FTIR), thermogravimetric analysis (TGA), and scanning electron microscopy (SEM). The cross section of the composites were observed through SEM.

EXPERIMENTAL

Materials

The lotus leaves and the reed leaves were obtained from a farm in Jining in the Shandong Province of China, while the basho leaves were obtained from Zhanjiang in the Guangdong Province of China. The leaves were hand-picked. The collected leaves were washed and cleaned with running water and dried at room temperature. Edible grade gelatin was obtained from the Henan Boyang Gelatin Company (Henan, China).

Preparation of Composites

The leaves were soaked in 95 ℃ water for 30 min, left out to dry naturally, and cut into 100 mm × 100 mm samples. The cut leaves were immersed in 100 mL of 30 wt.% gelatin aqueous solution, dried naturally at room temperature, and stacked in layers in a mold and compressed by a XLB-D400×400×2-Z flat vulcanizer (Shanghai Qicai Hydraulic Machinery Co., Shanghai, China). The materials were pressed into a 2 mm thick sheet at 80 ℃ under 100 MPa of pressure for 30 min. The chemical composition of the raw leaves and the treated leaves was tested according to the standards of NREL, and the percentage of cellulose, hemicellulose, lignin, and ash are shown in Table 1.

Table 1. Chemical Composition of the Leaves

Parameter (%)	Lotus leaf		Reed leaf		Basho leaf	
	Untreated	Treated	Untreated	Treated	Untreated	Treated
Cellulose	12.33	15.30	27.75	32.29	23.78	25.95
Hemicellulose	9.02	7.27	20.06	15.13	17.79	14.43
Lignin	50.79	48.19	35.30	30.35	32.87	31.36
Ash	12.17	0.33	11.20	3.59	4.03	0.68

FTIR Analysis

The characteristic functional groups of the leaves were analyzed by a Nicolet iS-10 spectrometer (Thermo Fischer Scientific, Waltham, MA) at room temperature. The FTIR transmittance spectra were recorded after an average of 16 scans from 4000 cm^{-1} to 500 cm^{-1} with a resolution of 4 cm^{-1}.

TGA

The thermal stability of leaves was studied by using an STA 449 F3 Jupiter simultaneous thermal analyzer (NETZSCH, Selb, Germany). Eight mg of leaves were taken in each sample, and the tests were carried out in argon atmosphere from 35 °C to 800 °C at a rate of 20 °C/min and a flow rate of 20 mL/min.

Morphological Studies

The surface morphology and the cross-section images of the untreated and treated composites were investigated using a Hitachi S-4800 scanning electron microscope (Tokyo, Japan).

Mechanical Properties

The tensile strength and the modulus and flexural strength properties were determined according to the GB/T standards 1040.1-2006 and 9341-2008, respectively. A universal testing machine (CMT6104, Metz Industrial System (China) Co., Ltd., Shanghai, China) was used to measure the mechanical properties of the composites. The test rate was set at 2 mm/min, and each specimen was tested three times to obtain the average value.

Moisture Absorption

The moisture absorption was carried out in a HZ-2004G constant temperature and humidity box (Dongguan Hengzhun (Lixian) Instrument Scientific Co., Ltd., Dongguan, China) according to the GB/T standard 20312-2006. The dried samples were placed in the box and the temperature was set to 23 °C ± 0.5 °C and the relative humidity was 85%. The samples were removed and weighed at time intervals of 6, 24, 48, 72, 96, 120, 144, and 168 h. The moisture absorption balance was achieved when the mass of the sample did not change between drying intervals. The moisture absorption rate of the composites was calculated according to Eq. 1,

$$c = \frac{W_1 - W_0}{W_0} \times 100\% \tag{1}$$

where W_1 is the mass of the specimen at a certain time and W_0 is the initial mass of the specimen.

RESULTS AND DISCUSSION

FTIR Analysis

The FTIR spectra of the treated and untreated leaves can be seen in Fig. 1. The broad peak at approximately 3400 cm^{-1} corresponds to the hydrogen bonded O-H stretching. The peak near 1919 cm^{-1} is related to the C-H stretching vibration in the lignin. The peak at 1605 cm^{-1} is associated with the C-C in plane symmetrical stretching vibration of aromatic rings present in lignin. The peaks at 1385 cm^{-1} and 1349 cm^{-1} are attributed to the C-H asymmetric bending vibration of the $-C(CH_3)_3$ group in lignin. The peak at 1074 cm^{-1} represents the C-O stretching of the lignin (Rosa *et al.* 2010; Al Maadeed *et al*. 2013; Orue *et al.* 2015). The intensity of these peaks decreased after the hot water treatment, which indicates that the lignin content of the leaves diminished, and this was consistent with the results of the tests of the chemical composition. The peak at 1740 cm^{-1} is related to the C=O stretching in the carbonyl group of wax and pectin (Kabir *et al.* 2013; Belaadi *et al.* 2014). The reduction in this peak intensity showed that the hot water could decrease the content of the wax and pectin.

Figure 2 displays the FTIR spectra comparison of the treated leaves. The peak intensity of the reed leaf was the highest at 3400 cm^{-1}, which indicates that the -OH content in the reed leaf was the largest among the three leaves. The peak intensity of the reed leaf at 2919 cm^{-1}, 1605 cm^{-1}, 1385 cm^{-1}, and 1349 cm^{-1} was lower than that of the lotus leaf and the basho leaf. This shows that the treatment effect was more effective on the reed leaf than the other two plant leaves which was consistent with the results of the leaf composition test. The peak of the reed leaf and the lotus leaf disappeared at 1740 cm^{-1}, but it was still observed in the basho leaf. Therefore, the hot water treatment removed the pectin and wax from the plant leaves but the treatment effect on the basho leaf was not optimal. The existence of the wax layer may have a negative effect on the combination of the leaves and gelatin. In addition, the hot water treatment can hydrolyze wax to fatty acids and fatty alcohols (Jiang *et al.* 2015), which results in a stronger peak intensity of the reed leaf and lotus leaf at 3400 cm^{-1}.

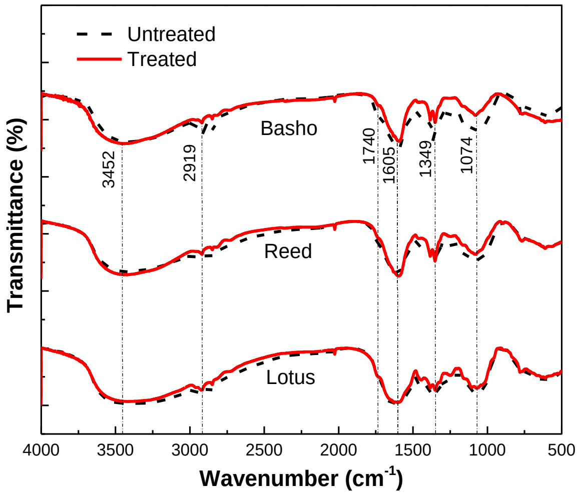

Fig. 1. The FTIR spectra of the treated and untreated leaves

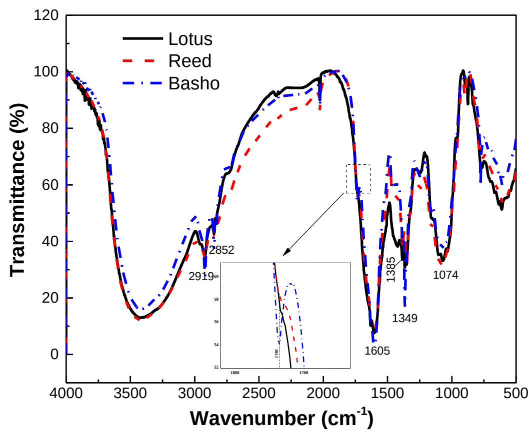

Fig. 2. The FTIR spectra comparison of the treated leaves

TG Analysis

Figure 3 shows the thermogravimetric curves of treated leaves. The degradation of the leaves was comprised of three stages. The first stage occurred at 40 °C to 120 °C, corresponding to the vaporization of the moisture content in the leaves. This occurs in all the natural fibers due to the high moisture content (Shanmugasundaram *et al*. 2018). The second stage was at 200 °C to 300 °C, which was the main degradation stage of the leaves caused by the decomposition of the non-cellulosic materials such as hemicellulose and lignin (Izani *et al.* 2013; Indran and Raj 2015). The final stage was related to the thermal decomposition of the cellulose in the fibers (Mittal and Sinha 2017).

In the first degradation stage, the mass loss of the lotus leaf, the reed leaf, and the basho leaf were 8%, 5%, and 3%, respectively. This indicated that the basho leaf had the lowest moisture content of all the leaves, which was consistent with the results of the FTIR analysis. Compared to the lotus leaf and the reed leaf, the basho leaf may have a higher hydrophobicity. In the second stage, the mass loss of the lotus leaf, the reed leaf, and the basho leaf were 37%, 45%, and 30%, respectively. In this stage, the basho leaf had the smallest amount of mass loss, which shows there was less hemicellulose, lignin, and other non-cellulose components in the basho leaf. In the third stage, the mass loss of the lotus leaf, the reed leaf, and the basho leaf were 22%, 15%, and 36%, respectively.

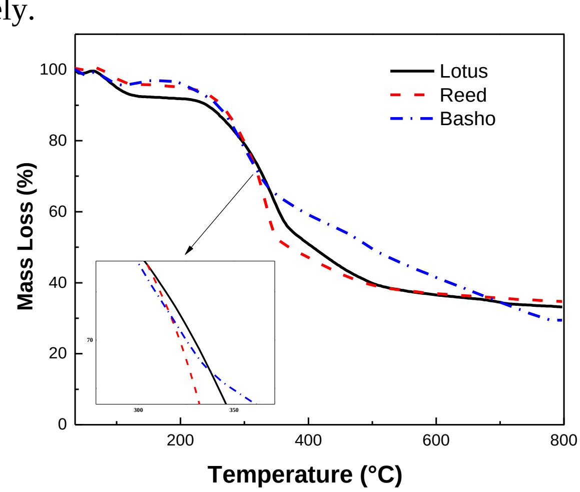

Fig. 3. The TGA curves of the treated leaves

Morphology Analysis

Figure 4 illustrates the surface morphology of the untreated and treated leaves. The surface of the untreated lotus leaf was uniform, and there were many plump bright particles distributed throughout it. These particles were the papillary structures composed of waxy layer fluff. After the hot water treatment, the bulge structure under the papillae of lotus leaf surface shrunk, the papillae changed irregularly, the sunken part between the structures was destroyed, and the wax density was reduced. This showed that the hot water treatment had the ability to change the wax layer structure of the lotus leaf. There were similar papillary structures and cilia on the surface of the reed leaf. The papillary structures of the untreated reed leaf were plump and evenly arranged. The wax layer was wrapped on the surface of the reed leaf. The cilia on the surface of the reed leaf were destroyed by the hot water, and the number of papillary structures diminished. The density of the papillary structure decreased, there was an irregular distribution, and the wax layer on the surface of the reed leaf disappeared. After the hot water treatment, there were many scaly structures on the basho leaf. The surface of the basho leaf was damaged and peeled, many holes were exposed, and the structure became loose.

After the hot water treatment, the surface of the plant leaves was rough and holes were present. This was because the lignin, wax, and other impurities in the plant leaf fibers were removed through the treatment (Rosa *et al*. 2010). The rough surface of the fiber can be conducive to the combination of the plant leaves and the gelatin, thus improving the performance of the composites.

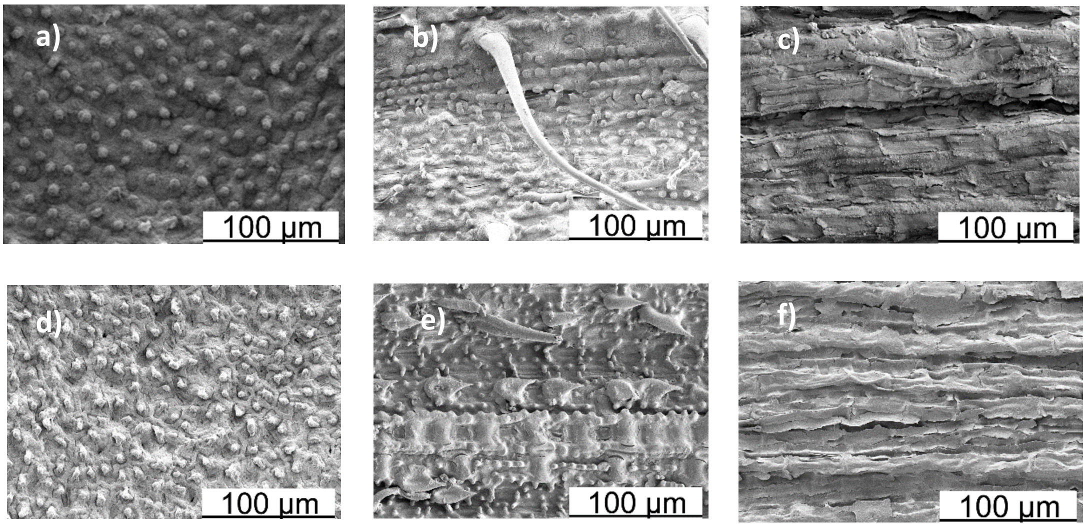

Fig. 4. The surface morphology of the untreated a) lotus, b) reed, and c) basho leaves and the treated d) lotus, e) reed, and f) basho leaves

Figure 5 shows the cross-section morphology of the lotus, reed, and basho gelatin composites. The composites were stacked in layers, and the gaps between the leaves were filled with gelatin. Gelatin plays an important role in the adhesion of the composites, as it can enhance their properties. Therefore, the addition of the gelatin can have a large effect on the composites. In the reed/gelatin composite, the gaps within the leaves were sufficiently filled with gelatin. The space between the layers was small, and there was a tight combination between the gelatin and the leaves. In the lotus/gelatin composite and the basho/gelatin composite, the gap between the layers was large. The gelatin did not fill sufficiently, and the combination between the gelatin and the leaves was not tight. Therefore, the performance of the reed/gelatin composite was better than the other two composites.

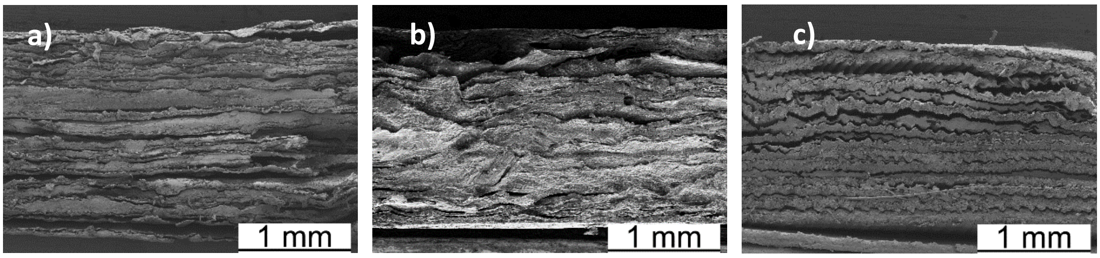

Fig. 5. The cross-section morphology of the a) lotus, b) reed, and c) basho gelatin composites

Mechanical Properties

Figures 6a and 6b show the tensile and flexural strength moduli of the composites. Among the treated composites, the reed/gelatin composite had the largest tensile strength and modulus of 26.1 MPa and 509 MPa, followed by the basho/gelatin composite, which was 22.9 MPa and 396 MPa. The lotus/gelatin composite had the weakest tensile strength and modulus of 11.8 MPa and 166 MPa, respectively. The tensile strength and modulus of the reed/gelatin composite was 14.0% and 28.4% higher than the basho/gelatin composite, and 121.5% and 206.2% higher than the lotus/gelatin composite. The reed/gelatin composite had the largest flexural strength and modulus of 41.1 MPa and 869.1 MPa, respectively, followed by the basho/gelatin composite, which was 23.2 MPa and 718.2 MPa. The lotus/gelatin composite had the weakest flexural strength and modules of 14.1 MPa and 619 MPa, respectively. The tensile strength and modulus of the reed/gelatin composite was 77.1% and 21.0% higher than the basho/gelatin composite, and 192.5% and 40.5% higher than the lotus/gelatin composite. This is due to the close combination between the reed leaf/gelatin composite and the orientation of the leaf fibers in the outer layer of the composites (Dehghani *et al*. 2013).

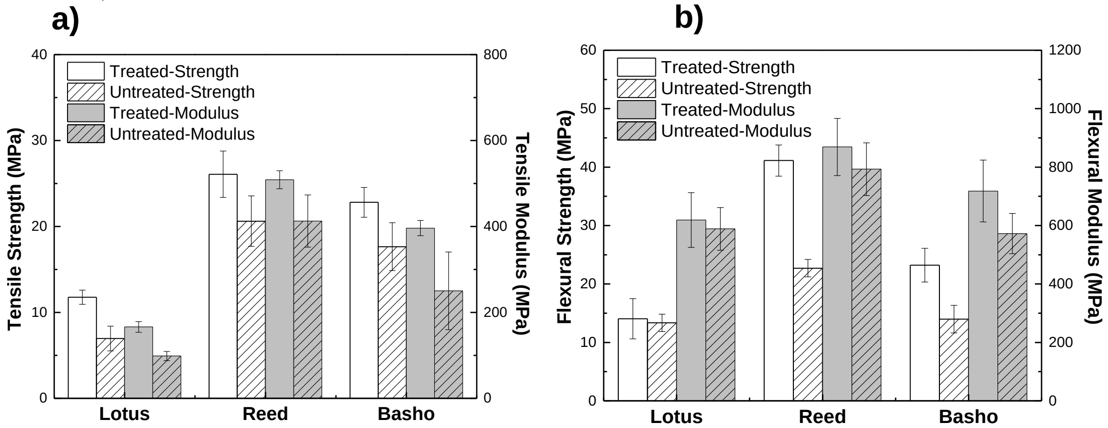

Fig. 6. The a) tensile strength and modulus and the b) flexural strength and modules of the leaf/gelatin composites

The mechanical properties of the composites improved after the leaves were treated with hot water, due to the removal of the non-cellulose components from the fiber (Boopathi *et al.* 2012; Shanmugasundaram and Rajendran 2016). It can be seen from the SEM images of the leaves that the hot water treatment formed caves and holes on the surface of the leaf, which increased the roughness of the leaf surface and improved the mechanical interlock performance of the composite. In addition, the hot water treatment exposed more cellulose on the fiber surface of the leaf, which increased the possible reaction position of the leaf (Mittal *et al*. 2017; Shanmugasundaram *et al*. 2018).

The TGA and SEM analysis of the leaves showed that the combination of the basho leaves and gelatin was poor, which may have been caused by the presence of wax. Therefore, the mechanical properties of the reed/gelatin composite were better than the other two composites.

Moisture Absorption

Figure 7 displays the moisture absorption curves of the composites. All three composites had similar moisture absorption rate curves. The moisture absorption rate grew rapidly in 6 h, then it began to slow down gradually. After 48 h, the moisture absorption rate basically remained unchanged, indicating that the composites reached the moisture absorption equilibrium. The equilibrium moisture absorption rates of the lotus/gelatin, reed/gelatin, and basho gelatin composites were 18.6%, 19.2%, and 23.8%, respectively. The equilibrium moisture absorption rate of the lotus/gelatin and the reed/gelatin composites were very similar. However, the equilibrium moisture absorption rate of the basho/gelatin was higher than that of the other two composites, which showed that the moisture absorption performance of the basho/gelatin composite was higher than that of the other two composites.

It can be seen from the SEM images of the composites cross section that there were gaps between the composite layers and caves and holes in the leaves, which caused large quantities of water vapor to gather here. The caves and holes play an important role in the moisture absorption test of the composites, which causes the composites to show a high moisture absorption. However, there were more caves and holes in the basho/gelatin composite than the other two composites, so the moisture absorption performance of the basho/gelatin composite was higher than the other two composites.

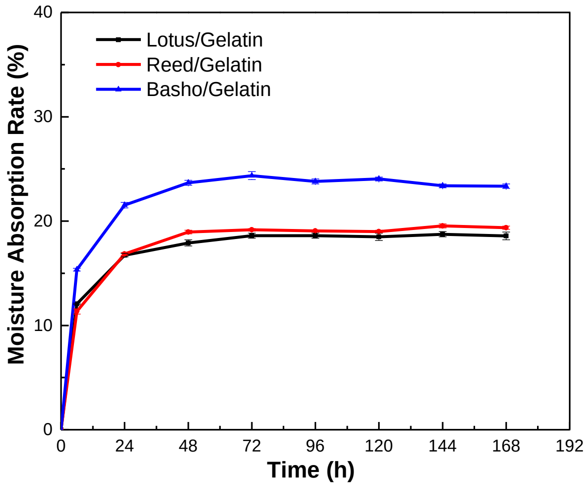

Fig. 7. The moisture absorption curve of the composites

CONCLUSIONS

1. The hot water treatment can remove the non-cellulose components in plant leaves and make the leaf surface rough to present a better adhesion with gelatin to impart better mechanical properties on the composites. The treatment had the best effect on the reed leaf, and the reed/gelatin composite had a better mechanical performance.
2. The composites had similar moisture absorption characteristics, and they all reached equilibrium after 48 h. The poor adhesion between the basho/gelatin layers caused numerous caves and holes, which gathered plenty of moisture in the basho/gelatin composite which induced a high moisture absorption rate.
3. Overall, the reed leaf/gelatin composite has the best performance.

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