Preparation of MSBF reinforced PP composites & research on their mechanical and sound insulation properties

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Abstract
Nowadays, most researchers at home and abroad are studying the planting of seashore mallow and the efficient extraction of mallow seed oil, while the remaining mallow stems are generally incinerated or discarded. In fact, there are a lot of bundle fibers in mallow stem bark (MSB), and their physical properties such as strength and modulus are close to those of jute and flax. So developing the fiber-reinforced composites of mallow stem bark fiber (MSBF) has high potential application value. Using mallow stem bark as raw material, MSBF was extracted by alkali degumming process, and MSBF reinforced polypropylene (PP) composites with fiber mass fraction of 40%, 30%, 20%, 10% and 0% were prepared by using alkali-treated MSBF as reinforcement and PP fiber as matrix. The effects of fiber mass fraction on tensile, bending and sound insulation properties of MSBF reinforced PP composites were discussed. The results show that when the fiber mass fraction was 30%, the tensile breaking strength, tensile modulus and bending modulus of the composite were the highest, and the bending strength gradually enhances with the increase of the fiber mass fraction of MSBF. When the fiber mass fraction exceeds 10%, the voltage peak attenuation rate of MSBF reinforced PP composites increases first and then decreases with the increase of sounding frequency. When the sound frequency is the same, the lower the fiber mass fraction, the worse the sound insulation performance of the composite. When the fiber mass fraction exceeds 30%, the voltage peak attenuation rate decreases.

Introduction
In today's era, people's requirements for ideal materials are getting higher and higher, which not only require them to be firm, reliable, lightweight, ecologically adaptable and biodegradable, but also require their mechanical properties to be superior to those of traditional materials. Natural fiber reinforced composites can satisfy most of the above conditions. Therefore, natural fiber reinforced resin matrix composites have become the key research direction in the field of composites [1, 2]. Natural fiber has low density, easy degradation, energy saving and environmental protection, which accords with sustainable development. However, natural fibers contain many hydroxyl groups, showing strong hydrophilic properties. Resin matrix usually has hydrophobic properties, which will lead to poor interface compatibility between them, and will destroy various properties of composites [3–6]. Therefore, natural fibers need to be modified [7–9], so as to improve the compatibility between natural plant fibers and resin matrix and prepare composites with good properties. Zuo Yingfeng et al [10] modified bamboo fiber with NaOH solution, and studied the effects of NaOH solution concentration, soaking time and temperature on the properties of polylactic acid/bamboo fiber degradable composites. The results showed that the fiber became unsmooth after alkali treatment, and it bonded better with polylactic acid matrix. When the fiber treated with the concentration (NaOH solution) of 3% and the temperature of 60 °C for 4 h, the properties of the composites were the best. Sepe et al [11] studied and prepared hemp fiber reinforced composite...
epoxy resin composites, and modified hemp fiber with silane coupling agent KH560. The mechanical testing results and scanning electron microscope (SEM) showed that hemp fiber was well bonded with epoxy resin matrix, and the tensile and bending properties of the composites were enhanced. When the concentration of silane coupling agent solution was 1%, the mechanical properties of the composites were the best. Jacob et al. [12, 13] studied the elastic composites with sisal/oil palm fiber reinforced polymer properties according to the focus and change of fiber surface. The results showed that the ductility and tear resistance of fibers decreased, but the tensile modulus increased. Idicula et al. [14] studied the dynamic and static mechanical properties of short banana/sisal hybrid fiber reinforced polyester composites at any position. The research showed that the arrangement of composites were realized by changing the relative volume of each layer of fibers. Medeiros et al. [15] focused on the mechanical properties of jute/cotton semi-phenolic composites and used them as reinforcing elements introduced by fibers. The results showed that this mixture of regular fibers was suitable for composites with light foundation applications.

Kosteletzkya virginica is a perennial herb suitable for growing in coastal areas, and it is very suitable for growing in saline soil (salt content is 0.6 ~ 1%) such as Dafeng in northern Jiangsu, China. Nowadays, most researchers at home and abroad are studying the planting of seashore mallow and the efficient extraction of mallow seed oil, while the remaining mallow stems are generally incinerated or discarded. In fact, there are a lot of bundle fibers in mallow stem bark, and the physical properties of MSBF are close to those of jute and flax [16]. As we all know, composites are gradually developing towards green, environmental protection and low price [17–19]. Many universities such as Tianjin Polytechnic University and Donghua University have been devoted to the research and development of some natural high-performance fiber reinforced composites [20–22], which have been widely used in construction, automobile, decoration and other industries. However, the research on the fiber-reinforced composites of mallow stalk bark by domestic and foreign scholars is still rare so far, so developing the fiber-reinforced composites of mallow stalk bark has high potential application value.

In this work, MSBF treated by alkali degumming process act as reinforcement while PP act as matrix. MSBF reinforced PP composites were prepared by molding method [23, 24]. The mechanical properties and sound insulation properties of composites with different mass fractions of MSBF were discussed.

**Experimental materials and equipment**
The stem bark of mallow was provided by Dafeng North Farm in Jiangsu Province; PP fiber was purchased from Yancheng Weibang Engineering Materials Co. Ltd; Alkali (NaOH) was purchased from Shanghai Nanxiang Reagent Co. Ltd.

After degumming, MSBF was opened by Y101 raw cotton impurity analyzer provided by Changzhou No.1 Textile Equipment Co. Ltd; The preforms of composites were made by using the small carding machine (A181AS27) provided by Suzhou Hua Fei Textile Technology Co. Ltd; The molding of composite plate was processed by flat vulcanizing machine provided by Qingdao Xincheng Yiming Rubber Machinery Co. Ltd; The tensile and bending properties of the composites were tested by INSTRON3369 universal electronic strength instrument provided by Shanghai Longhua Co. Ltd; The crystallinity of fiber was indirectly characterized by infrared spectrometer (Nicolet5700) provided by Thermo Electron Company of America; The microstructure of fibers was observed by Japanese electron JSM-5600 IV scanning electron microscope; The sound insulation performance of the composites was characterized by self-developed testing device.

The moisture regain of a single MSBF was tested according to the standard NY/T 243–2011; The fineness of a single MSBF was tested according to the standard GB/T18147.4-2015; The tensile properties of a single MSBF were tested in accordance with the standard NY-T 2635-2014.

**Extraction of fiber from mallow stem bark**
Mallow Stem Bark was degummed by alkali scouring. The process route of alkaline scouring was: pickling → washing → beating → alkaline scouring → pickling → washing → beating, and the alkaline scouring was repeated twice at 98 °C for 1.5h and the bath ratio was 1:30. The NaOH concentration were 30 g l−1 and 20 g l−1 respectively during the two alkali scouring [7, 8].

**Alkali treatment of MSBF**
Before alkali treatment, it was prepared into an aqueous alkali solution according to the mass percentage of 7.5%. The degummed MSBF was placed on the raw cotton impurity analyzer for single fiberization and dry for use, then soaked for 1 h at normal temperature in NaOH solution with concentration of 7.5% (the fiber should remain fluffy). After treatment, MSBF was washed with dilute acid solution and clean water to be neutral, and dry it in an oven at 80 °C to constant weight for later use [25, 26]. Then we cut the alkali–treated MSBF into short lengths of 2 ~ 3 mm for the preparation of composite.
Preparation of sound insulation performance testing device

The physical map and schematic diagram of self-made sound insulation performance testing device is shown in figure 1, which includes acoustic emission device and acoustic detection device. Acoustic emission device composed of loudspeaker and signal generator in virtual sound card instrument makes sound, and acoustic signal acquisition and processing system is composed of acoustic sensors, such as microphone, oscilloscope and computer in virtual sound card instrument. In order to ensure the preparation of experimental data, the distance between the tested plate and the microphone should be kept constant during the test [27, 28]. Sound insulation performance is characterized by peak attenuation rate of acoustic voltage ($\Delta U$). The larger the value of $\Delta U$ is, the larger the sound attenuation is, and the better the sound insulation performance of the composite material is. $\Delta U$ is calculated as follows:

$$\Delta U = \frac{\Delta U_0 - \Delta U_1}{\Delta U_0} \times 100\%$$

Where: $\Delta U_0$ is the voltage peak during blank test, $\Delta U_1$ is the voltage peak during plate test, and the units of $\Delta U_0$ and $\Delta U_1$ are volts.

Preparation and performance testing of composites

PP fiber was opened manually. In order to avoid the phenomenon of winding doffer due to static electricity during carding, which will affect the carding into the web, a certain amount of water was sprayed on the surface...
of the fiber before carding. In order to ensure the uniform performance of the prepared MSBF reinforced PP composite, when carding into the web, the PP fiber needs to be laid on the upper and lower layers, while MSBF is laid between the upper and lower PP fiber layers. According to the mass fraction of 40%, 30%, 20%, 10% and 0% for MSBF, 100g preform-MSBF/PP fiber mixed web [22] was prepared, the fiber web formed by one carding was then combed a second time to ensure the uniformity of the web. Before preparing the composite plate, the mold was coated with release agent, and the standby MSBF/PP mixed fiber web was dried in a constant temperature oven at 80 °C for 60 min. At the same time, after preheating the flat vulcanizing machine for 30 min, the mixed fiber web (the preform) was thermoformed at constant temperature and constant pressure for a certain time according to the pressure method shown in figure 2 and cooled for use. The thermoplastic plate was trimmed after stripping to obtain the composite [29–31]. According to the previous groping experiment, considering that the composite was well formed without affecting the properties of MSBF and improving the processing speed of composite products, the preparation process of compression molding composite was as follows: pressure was 10 MPa, time was 8 min, and compression temperature was 190 °C.

For convenience of comparison, 100g pure PP plate was prepared at the same time, and the crystallinity of MSBF was measured by infrared spectroscopy. Tensile properties of fiber reinforced composites and pure PP plate were tested according to ISO 527-4: 1997 testing method. The bending properties were tested according to ISO 14125:1998. The load cell size for the instron machine was a dumbbell-shaped specimen. The total length of the dumbbell-shaped specimen was 115 mm, the end width was 25 mm, the middle parallel part length was 33 mm, and the middle parallel part width was 6 mm. The mold dimension was 45 cm x 45 cm in length and
width, and the depth was 8 mm. The microstructure of tensile fracture surface of MSBF reinforced composites was analyzed by SEM. The sound insulation performance of composites and pure PP plate was tested on the self-made sound insulation performance testing device.

**Results and discussion**

**Microstructure and performance analysis of MSBF**

*Microstructure of MSBF*

Figure 3 is a scanning electron microscope of the surface of MSBF before and after alkali treatment. In figure 3(a), a large amount of pectin remained on the surface of MSBF before alkali treatment, but after alkali treatment, the gum on the surface of MSBF was not only removed, but also many grooves appeared on the fiber surface, and the surface roughness was improved (figure 3(b)). This showed that alkali can greatly separate and dissolve most non-cellulose substances, such as hemicellulose, pectin, lignin, etc, and reduce the microfiber rotation angle of fibers, thus making the surface etched [25, 32]. Obviously, after the surface of MSBF was etched, the slippage resistance between MSBF and matrix can be effectively improved, that is, the bonding effect between fiber and matrix can be enhanced.

**Basic properties of fibers**

The testing results of the conventional physical properties about single MSBF were shown in table 1. It can be seen from table 1 that the hygroscopicity of MSBF decreased after alkali treatment, which was mainly due to the formation of alkali cellulose between hydroxyl groups and alkali on the surface of MSBF, which reduced a certain number of hydrophilic groups [20]. Alkali treatment reduced the fineness of MSBF, but its tensile strength increased by 2.43% compared with MSBF before treatment, which was mainly due to the fact that pectin, lignin and other substances greatly decreased after alkali treatment, which made the free range of macromolecular chains in MSBF expand, and made the molecular chains regularly arranged along the fiber length direction as much as possible, resulting in the improvement of fiber crystallinity [24, 25]. For further confirmation, we made infrared spectra of MSBF before and after alkali treatment, as shown in figure 4. After alkali treatment, the absorption peak of group (–OH) of MSBF at 3400 cm\(^{-1}\) ~ 3200 cm\(^{-1}\) decreased. At the absorption peak of

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**Table 1. Conventional physical properties of MSBF.**

| Physical index      | Moisture regain/\% | Fineness/dtex | Tensile strength/MPa | Tensile modulus/GPa | Elongation at break/\% |
|---------------------|---------------------|---------------|-----------------------|---------------------|------------------------|
| Untreated MSBF      | 12.04 ± 0.97        | 15.77 ± 1.21  | 352.25 ± 12.17        | 11.96 ± 1.18        | 3.55 ± 0.42            |
| Alkali treated MSBF | 11.23 ± 0.91        | 14.54 ± 1.16  | 396.03 ± 14.32        | 9.43 ± 0.94         | 3.73 ± 0.51            |

Figure 4. Infrared spectrum of MSBF
1740 cm\(^{-1}\), the absorption peak of \(>\text{C}=\text{O}\) stretching vibration of hemicellulose of MSBF after alkali treatment also decreased, and the absorption peak of MSBF after alkali treatment increased obviously at 2916 cm\(^{-1}\). The unchanged bands were used to analyze the changes of conformation and crystallization. The crystallinity of MSBF before and after alkali treatment was determined by infrared spectroscopy [33], and the crystallinity was analyzed by three bands of 2916 cm\(^{-1}\), 1054 cm\(^{-1}\) and 1740 cm\(^{-1}\). It can be confirmed that the crystallinity of MSBF after alkali treatment is 0.92, which is higher than that of untreated MSBF by 0.87.

### Strength of composites

The tensile strength and bending strength of the composites were shown in table 2. Those composites with the fiber mass fraction of 40\%, 30\%, 20\%, 10\% and 0\% about MSBF were respectively designated as A40, A30, A20, A10 and A. Figure 5 shows the microstructure of tensile fracture surface of composites.

| Samples | Tensile strength/MPa | Bending strength/MPa |
|---------|----------------------|----------------------|
| A       | 17.4 ± 1.27          | 40.3 ± 3.45          |
| A10     | 22.5 ± 1.34          | 45.6 ± 4.13          |
| A20     | 32.4 ± 1.46          | 51.4 ± 4.57          |
| A30     | 37.5 ± 1.52          | 55.6 ± 4.69          |
| A40     | 36.3 ± 1.59          | 59.7 ± 4.85          |

It can be seen from table 2 that when the fiber mass fraction of MSBF increases from 0\%, the tensile strength of the composite increase at first and then decrease, and when the mass fraction is 30\%, the tensile strength reaches the maximum value of 37.5 MPa. The reasons were as follows: as shown in figure 5(a), when the content of MSBF was about 10\%, the dispersion of fiber was high, and the hydroxyl groups on the surface of MSBF were obviously reduced, so good adhesion can be formed between MSBF and matrix with small pores. However, the strength of composite depends on the strength of matrix(PP), and the reinforcing effect of MSBF in matrix was not obvious enough. When the fiber mass fraction of MSBF was about 20\%, as shown in figure 5(b), the aggregation degree of MSBF was improved, and certain compression occurred between MSBF during compression molding, which lead to an increase in the porosity between fiber and matrix, a decrease in the adhesion between fiber and matrix, and a few fibers were pulled out from matrix, but the distribution of MSBF tend to be uniform, and the total infiltration area of matrix arond MSBF increase. The efficiency of load transfer between MSBF was improved, so the total adhesive force between MSBF and matrix was increased. At this time, the pore had little effect on the strength of the composite, so the tensile strength of the composite still showed an increasing trend. If the fiber mass fraction was too high, when the fiber mass fraction of MSBF reached 30\%, as shown in figure 5(c), the density of MSBF in the matrix was higher, the fiber clustered and larger pores formed between MSBF and the matrix, so the adhesion between fiber and matrix, i.e., mechanical locking force, obviously decreased, the coating and wetting effect of matrix on MSBF became worse, and the continuity of matrix and adhesion with fiber decreased in composites.

However, the bending strength gradually increased with the increase of fiber mass fraction. Compared with pure PP, the flexural strength of composites with fiber mass fraction of 10\%, 20\%, 30\% and 40\% increased by 13\%, 27\%, 38\% and 48\%, respectively. The reason was that there was no fracture in the bending process, and the bending strength reflected the load when a certain displacement occured at the midpoint of the testing sample. Therefore, when the fiber distribution in the matrix was uniform, the more the fiber mass fraction of MSBF, the more it can bear certain bending moment.

### Modulus of composites

The tensile and flexural modulus testing results of the composites were shown in table 3. It can be seen from table 3 that the tensile modulus and flexural modulus of the composites first increase and then decrease with the increase of the fiber mass fraction of MSBF. The reason was that the elongation at break of pure PP was larger. When the mass fraction of MSBF gradually increased, on the one hand, a good bonding interface can be formed between MSBF and matrix [17]. On the other hand, the modulus of MSBF itself was relatively high, which limited the tensile and bending deformation of the composite, so the tensile and bending modulus gradually increased. However, when the fiber mass fraction of MSBF exceeded 30\%, the gap between fiber and matrix increased obviously, which weakened the effect of stress transfer between fiber and matrix, and the composite was easy to be stretched or bent by external force, so the modulus decreased.
Figure 5. Tensile fracture surface’s microstructure of composites with different fiber mass fraction of MSBF: (a) 10%–200 times; (b) 20%–200 times; (c) 30%–200 times.
Figure 6. Stress strain-curve for the tensile test of different composites.

Table 3. Modulus of MSBF reinforced PP composites

| Samples | Tensile modulus/GPa | Bending modulus/GPa |
|---------|---------------------|---------------------|
| A       | 0.82 ± 0.22         | 2.74 ± 0.64         |
| A10     | 1.17 ± 0.28         | 3.46 ± 0.78         |
| A20     | 1.33 ± 0.31         | 4.18 ± 0.79         |
| A30     | 1.54 ± 0.35         | 5.57 ± 0.83         |
| A40     | 1.47 ± 0.39         | 5.23 ± 0.86         |

Figure 7. Sound insulation performance of the composites.
Tensile elongation at break of composites
When the fiber mass fraction of MSBF were 0%, 10%, 20%, 30% and 40%, the tensile elongation at break of the MSBF reinforced composites were 4.22%, 3.13%, 2.87%, 2.64% and 2.45%, respectively. It can be seen that the tensile elongation at break of pure PP was the largest, and the tensile elongation at break of composites decreased gradually with the increase of mass fraction about MSBF. The reason was that after the addition of MSBF, the elongation at break of the composites decreased obviously because the elongation at break of MSBF was lower than that of pure PP. When the mass fraction about MSBF increased, the area covered by matrix decreased gradually, and there were obvious holes between MSBF and matrix, so the adhesive force was small. When subjected to external load, it was easy to slip between the MSBF and the matrix.

Stress strain curve for the tensile test
Stress-strain curve for the tensile test of different composites was shown in figure 6. Since the tensile stress-strain curve of the composite didn’t change much when the fiber mass fraction was low, we compared the stress-strain curves of the four composites of A, A20, A30, and A40. It could be seen from figure 5 that the tensile fracture strength of the pure PP was the smallest, but the area under the stress-strain line was the largest, which meant that it absorbed more energy and had greater toughness during the failure process. After adding MSBF, the tensile state of the composites had changed, and the tensile breaking strength had been greatly improved. When the mass fraction of MSBF in the composite changed from 20% to 30%, the breaking strength of the composites and their elongation at break both increased, but when the fiber mass fraction of MSBF in the composites exceeded 30%, the breaking strength of the composite decreased slightly, which indicated that the optimal fiber mass fraction was 30% for producing the alkali treated MSBF reinforced PP composite.

Sound insulation performance of composites
The testing results of sound insulation performance of the composites were shown in figure 6. In figure 7, the sound testing frequency changed from 1000 Hz to 8000 Hz, and the interval frequency was 1000 Hz. According to formula (1), we know that the greater the attenuation rate of the measured peak acoustic voltage, the better the sound insulation performance of the composites.

It also could be seen from figure 7 that when the sound frequency was lower than 6000 Hz, the sound insulation performance of all composites increased with the enhancement of sound frequency, which showed that they had good attenuation effect on high-frequency sound waves. In addition, when the fiber mass fraction of MSBF exceeded 10% in composites, the sound insulation effect of each composite first increased and then decreased with the increase of sound frequency, and the sound insulation effect was the best when the sound frequency was 6000 Hz. When the fiber mass fraction of MSBF was less than 10%, the lower the fiber mass fraction was, the worse the sound insulation effect was. The reason was that the roughness of the fiber surface of MSBF after alkali treatment had been obviously improved, so that when the sound was projected on the fiber surface, it can’t pass through the composite after multiple refraction and reflection, and the sound insulation effect was improved. When the fiber mass fraction of MSBF exceeded 30%, the sound insulation effect decreased instead. The reason was that when the mass fraction of MSBF was too high, even though the rough MSBF could effectively achieve sound insulation effect, the interface between MSBF and matrix increased, and the number of voids in the composite increased, resulting in some sound passing through.

Conclusions
When the fiber mass fraction of MSBF increased from 0%, the tensile strength of its reinforced PP composites increased at first and then decreased, and when the fiber mass fraction was 30%, the tensile strength of the composites reached the maximum value of 37.5 MPa. Compared with pure PP, the flexural strength of composites with fiber mass fraction of 10%, 20%, 30% and 40% increased by 13%, 27%, 38% and 48% respectively.

When the fiber mass fraction of MSBF increased from 0%, the tensile and flexural modulus of its reinforced PP composites increased at first and then decreased. When the fiber mass fraction was 30%, the tensile modulus and flexural modulus reached the maximum. The tensile elongation at break of pure PP was the largest, and the elongation at break of the composite decreased gradually with the increase of fiber mass fraction of MSBF.

When the fiber mass fraction of MSBF exceeded 10% and the sounding frequency increased, the peak attenuation of acoustic voltage of the composite increased first and then decreased, and the best blocking effect was achieved at 6000 Hz. For the same sounding frequency, the lower the fiber mass fraction of MSBF, the worse the sound insulation effect. When the fiber mass fraction of MSBF exceeded 30%, the sound insulation effect decreased.
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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

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