MECHANICAL PROPERTIES IMPROVEMENT OF
NATURAL FIBER GREEN COMPOSITES
BY CYCLIC LOAD APPLICATION

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Abstract
The purpose of this study is to improve the strength and stiffness of natural fiber green composites through mechanical treatment, and to equalize the mechanical properties of the composites with those of glass fiber composites. Cyclic tensile stress at 50% or 70% level of their initial strengths was applied five or twenty times for single ramie fibers, twenty or a hundred times for ramie fiber green composites. Tensile strength of the as-supplied fibers was improved approximately 50% higher than their initial value, if the cyclic stress is their 70% level and the number of cycles is twenty, while this mercerized fibers was improved in strength approximately 20% higher than the original value. On the other hand, the mechanical treatment was not so largely affected in strength for ramie fiber green composites, approximately 10% higher than that of the untreated green composites, while Young’s moduli of cyclic-loaded green composites were significantly improved, 56% to 67% higher than that of untreated green composites.

1. Introduction
Nowadays, there is an increase in the number of ecologically aware studies and their practical applications, such as the development and use of alternative environment-friendly materials. The use of natural fiber-reinforced composites may be a suitable method for replacing glass fiber-reinforced plastics (GFRP). Although exhibiting several merits in cost, handling, energy consumption and so on, natural fibers are not comparable for glass fibers in terms of mechanical properties.

The purpose of this study is thus to improve mechanical properties of plant-based natural fibers through cyclic load application. This treatment was also applied for natural fiber-reinforced biopolymer matrix composites. The results obtained in this study show a dramatic improvement in strength and stiffness of ramie fibers and their composites.

2. Experimental

2.1 Materials
A plied yarn of ramie fibers (supplied from TOSCO Co., No.16, five twists) was used as a reinforcing material of the green composites. As a matrix, cornstarch-based biodegradable resin (Randy CP-300, supplied from Miyoshi Oil & Fat Co.) was used, which is decomposed easily and naturally in soil. This resin is made from a blend of polycaprolactone and cornstarch, and hydrophilic nature. The resin was supplied in water emulsion with micro-order particles. Mechanical properties of ramie fibers and the biodegradable resin are shown in Tables 1 and 2, respectively.

In this study, furthermore, high concentration alkali treatment, called mercerization [1], was performed for as-supplied ramie fibers aiming to improve the toughness of green composites. The

<table>
<thead>
<tr>
<th>Density (Mg/m³)</th>
<th>Cellulose (wt%)</th>
<th>Lignin (wt%)</th>
<th>Hemichellulose (wt%)</th>
<th>Pectin (wt%)</th>
<th>Wax (wt%)</th>
<th>Microfibrillar angle (°)</th>
<th>Moisture content (wt%)</th>
<th>Tensile strength (MPa)</th>
<th>Fracture Strain (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.50</td>
<td>68.6-76.2</td>
<td>0.6-0.7</td>
<td>13.1-16.7</td>
<td>1.9</td>
<td>0.3</td>
<td>7.5</td>
<td>8.0</td>
<td>0.031</td>
<td>610</td>
</tr>
</tbody>
</table>
treatment was carried out in 15wt% concentrated sodium hydroxide (NaOH) solutions for 2hs at room temperature. After alkali treatment, the fibers were washed for a few minutes in a 1wt% acid acetic solution. Finally, the fibers were washed in water and dried at room temperature for 24hs. As-supplied ramie fibers have not been treated in our laboratory, i.e. untreated, so hereinafter denoted as ‘UT’, and alkali-treated ramie fibers are denoted as ‘AT’.

2.2 Cyclic load application of single fibers

To study the effect of cyclic load application on the tensile strength of ramie fibers, single fiber specimens were prepared by bonding a monofilament of UT or AT fiber to a paperboard with a rectangular hole of 10 mm length, as shown in Fig.1. The gage length was therefore 10 mm. Fiber width on each specimen was measured before cyclic load application using an optical microscope, and then the diameter of each fiber was calculated assuming that the cross-section of each fiber has circle morphology. The fiber specimen was attached to a testing machine with a low capacity of load-cell (9.8N), and left and right hand parts of the paperboard along the fiber axis were cut out. Crosshead speed of the testing machine was 0.8 mm/min for both of cyclic loading and tensile testing. For several fiber specimens, the distance between the both ends of the paperboard was measured through a laser system to obtain the fiber elongation. The cyclic load application was carried out such that a single fiber is loaded in tension up to 50% or 70% level of the fiber strength and then unloaded. This cycle was repeated five or twenty times and thereafter, conventional tensile test was performed with the same crosshead speed. The cyclic loaded specimens are hereinafter denoted as ‘Fiber - stress level - cyclic number’. For example, if the single fiber is alkali-treated, and this specimen is cyclic-loaded at 70% stress level twenty times, then this is denoted as ‘AT-70-20’.

2.3 Fabrication of ramie fiber green composites and testing condition

The composites were fabricated by use of compression molding method. Several ramie yarns were placed in parallel and the emulsion resin was pasted on the yarns. One set of the yarns is called pre-form. After drying water in the pre-form at room temperature, a pair of pre-forms was inserted in the mold and pressed with 6.5 MPa at 150°C for 30min. The heating process was then stopped but the pressure was maintained until the temperature fell to near room temperature.

To carry out a cyclic loading and tensile tests, aluminum plates with 0.8mm in thickness were attached onto the both ends of the fabricated composites. The configuration and dimension of the tensile specimen is shown in Fig.2. A strain gage was fixed at the center of each specimen for measuring uniaxial strain. Cyclic loading was applied for the tensile specimen in order to improve mechanical properties of the composites. The number of cycle was twenty or one hundred, and the stress level was 70% of the composite strength using a fatigue testing machine (Hydraulic Servo-Pulser, Shmadzu Co.). Frequency at cyclic loading of the testing machine was 1Hz with sine signal. After the cyclic load application, the specimen was attached to an Instron-type testing machine (Autograph, Shimadzu Co.), and tensile-tested statically. Crosshead speed of the testing machine was 1 mm/min.

3. Results and discussion

3.1 Tensile properties improvement of single ramie fibers by cyclic load application

<table>
<thead>
<tr>
<th>Density (Mg/m³)</th>
<th>Melting Point (°C)</th>
<th>Tensile Strength (MPa)</th>
<th>Fracture Strain (%)</th>
<th>Young's Modulus (GPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.16</td>
<td>58</td>
<td>10.6</td>
<td>6.5</td>
<td>0.531</td>
</tr>
</tbody>
</table>

Table 2. Properties of cornstarch-based biodegradable resin.

Fig. 1. Tensile specimen of a single fiber

Fig.2. Tensile specimen of a ramie yarn green composite

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Tables 3 and 4 show the effect of cyclic load application on tensile properties of UT and AT ramie fibers, respectively. Table 3 shows the tensile strength increases with increases in the number of cycles and stress level. Especially, tensile strength of UT-70-20 fibers increased about 50% more than that of UT fibers, while fracture strain of UT-70-20 fibers decreases to 2.76%. The cyclic load application is also effective for AT fibers, as shown in Table 4. But the effect did not occur successfully in the conditions of 70% stress level and 20 times, which was effective for UT fibers. As a future subject, an optimal number of cycles to bring higher strength of AT fibers should be studied. While AT fibers is largely improved in fracture strain in comparison with UT fibers, this strain decreases with increase in the number of cycles and stress level. Furthermore, this application was effective for improvement of stiffness in the fibers. Figure 3 shows typical stress-strain diagrams of UT, UT-70-20, AT and AT-70-20 fibers. By cyclic load application of 70% stress level and 20 times, it is seen that both of untreated and alkali-treated fibers became stiffer, while these cyclic loaded fibers decrease in fracture strain. Young’s moduli of UT, UT-70-20, AT and AT-70-20 fibers were calculated from the diagrams as 23.1, 30.7, 16.5 and 22.2GPa, respectively. We should take care that distance between both ends of the specimen paperboard includes deformation of bonding agent between the paper and fiber surface as well as fiber’s deformation, and therefore the above values of Young’s moduli may be underestimated. However, the Young’s moduli after cyclic load application increase undoubtedly. Even in AT fibers, its Young’s modulus almost recovers up to the level of UT fibers, as seen in Fig.3, and deforms largely with non-linearity up to around 5.0% strain. In this case the tensile strength did not so largely decrease. In other words, as-supplied ramie fibers can be largely toughened through alkali-treatment and cyclic load application. The reason of such improvement of the tensile properties is guessed that the orientation of microfibrils in the fiber was changed to near fiber axis’s orientation. And also the crystallinity index in the microfibrils may increase by cyclic loading. Thus, we consider that these mechanisms are closely related with improvements of the tensile strength and stiffness of ramie single fibers.

3.2 Tensile properties improvement of ramie fiber green composites by cyclic load application

Table 5 shows the effect of cyclic load application on tensile properties of green composites reinforced with UT and AT ramie fibers. In this case also this application increases tensile strength of the both composites, but the degree of increase is not so large in comparison with single fibers. Increasing rate of tensile strength is only 8.0% in the conditions of 70% stress level and 20 times. In the condition of 100 times, furthermore, the effect was hardly seen as in Table 5.

Figures 4 and 5 show typical stress-strain diagrams of cyclic-loaded composites reinforced with UT and AT fibers, respectively. Stiffness of the both composites increases obviously. That is to say,
Young’s modulus of these composites measured by strain gage indicates 1.32 to 1.67 times higher than that of composites without cyclic loading. Young’s modulus of general GFRP, e.g. 50vol% E-glass fiber reinforced unsaturated-polyester is about 38GPa [2]. This means, cyclic-loaded green composites obtained here are almost comparable in Young’s modulus to general GFRP. Thus, it is concluded that cyclic load application for green composites is not so effective for improvement of tensile strength, but effective for improvement for stiffness. Young’s modulus of the composites increased through 100 times load application, but not so different from the level of 20 times load application. In other words, this cyclic load application almost saturated the change in Young’s modulus of the composites around 20 times load application. It is considered that such improvement of the tensile properties is brought from changes in the orientation of microfibrils in the fiber and crystallinity index in the microfibrils, as mentioned earlier. It is also considered that increase in Young’s modulus is related with decrease in twisted angle of yarns in the composites during cyclic loading. On the other hand, tensile strength of the composites decreased through 100 times load application in comparison with 20 times. It is considered that this is due to occurrence of fatigue damage inside the composites.

4. Conclusion

In order to improve mechanical properties of natural fiber reinforced green composites through mechanical treatment, ramie fibers and green composites were cyclic-loaded. Cyclic tensile stress at 50% or 70% level of their original strengths was applied five or twenty times for single ramie fibers, and twenty or a hundred times for ramie fiber green composites. Tensile strength of the as-supplied fibers was improved approximately 50% higher than their original value, if we choose 70% stress level and twenty number of cycles, while alkali-treated
fibers were improved in strength approximately 20% higher than the original value. On the other hand, the cyclic load application was not so largely affected for tensile strength of green composites, approximately 10% higher than the original value, while Young’s moduli of cyclic-loaded green composites were significantly improved, 56% to 67% higher.

References
