Preparation and Properties of Composite Materials Mulberry Nano-Cellulose Whiskers

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Abstract. To prepare environmentally friendly PVA composite material, using easy crystallinity strong Hydrophilic and high strength Mulberry bark nano-cellulose whiskers with special optical properties, Rheological properties and mechanical behavior to make a NCW/PVA composite material with PVA by casting method. The Experiment indicates nano-cellulose whiskers have obvious reinforcing effect to the physical and mechanical properties of PVA. With the 6cm air layer, the Sound absorption coefficient increased clearly in medium low frequency sounds. Noise reduction coefficient (NRC) can be raised by 10%. Keeping Air cavity structure between the material and wall, the thermal insulation performance of the material with 3% NCW will be increased more.

Introduction

With the severe shortage of resources and people's increasing emphasis on environmental protection, it is significant to use the cheap and abundant green renewable cellulose resources, recombining and modifying nano-cellulose with new technology in microscopic field to develop a variety of new materials with excellent properties[1]. Nano-cellulose can be divided into nano-crystalline cellulose (whiskers), nano-cellulose composites and nano-cellulose fibers based on the shapes. The length of NCW is 10~1000nm and the cross section is only 5~20nm. The ratio of length to diameter is 1~100[2-5]. NCW is easy crystallinity, strong hydrophilic and high strength, which determined its widely usage because of its rheological properties and mechanical behavior. What’s more, NCW can be used in preparing environment friendly nano composite material according to its biocompatibility and biodegradability, that’s the reason for researching NCW is becoming more and more popular.

Preparing mulberry cellulose whiskers is to prepare an environmentally friendly nanocomposites by combining PVA and the white odorless powder particles from natural cellulose treated by hydrolyzing and other methods. PVA is a non-toxic polymer, with many hydroxyl on its molecular chain, coming from the hydrolysis of polyvinyl acetate. PVA has a character of good water solubility, film formation, adhesive strength and emulsifying and resistance of oil and solvent. Therefore, PVA is widely used as adhesion agent, finishing & sizing agent for paper making, textile pulp, emulsifier & protective colloid in emulsion polymerization, temporary adhesive in Ceramic Industry. Considering the water solubility of PVA and the excellent performance of Mulberry nano-cellulose whiskers, the article researches the performance and preparing of the composite material from mulberry nano-cellulose whiskers and PVA.

Preparation of Mulberry Nano-cellulose Whisker/PVA Composite Material

Experimental Materials

PVA; Mulberry nano-cellulose whiskers: self-made, as shown in Fig.1.
Preparing Composite Material

Take some PVA, put some amount of water in constant temperature water bath and heat to 90 °C, churning to dissolve PVA in water. According to PVA used, put the amount of mulberry nano-cellulose whiskers separately from 1% to 5%. Keep on churning for 30 min. Make it a film on a glass plate by salivation after defoaming. Dry the film with infrared lamp after the moisture dry naturally to prepare composite membrane of mulberry NCW & PVA.

Study on Physical and Mechanical Properties of Composite Materials

Test of Physical and Mechanical Properties

Adopting the experiment used for tensile property of plastic in GB/T 1040-92 to test the tensile strength, tensile modulus and elongation at break of the Composite material (NCW/PVA). Cutter: Type II. Stretch rate: 100 mm/min.

Results and Analysis

Fig.2 is the physical and mechanical properties influence caused by different amount of Mulberry nanometer cellulose whiskers for the Composite membrane of mulberry NCW & PVA.

![Figure 2](image)

Figure 2. Physical and mechanical properties of composite caused by different amount of mulberry nano-cellulose whiskers.

Fig.2 shows the influence of Physical and mechanical properties of NCW/PVA composite material caused by different amount of NCW from 0% to 5%. From Picture 1, the result is little NCW putting in PVA can cause great changes of material’s physical and mechanical properties. Tensile strength and tensile modulus rise first, then drop after the highest point. While Elongation at break keeps on dropping with the continuous adding of NCW.

It is found that the Overall performance reach the best point when NCW amount is 3%. Tensile Strength increased 66.02% and Tensile modulus raised 147.33 %. This means MCW have an obvious Reinforcing effect to PVA. But the disadvantage is brittleness of PVA increased indicated by the decrease of Elongation at break.
Performance Testing and Characterization of Composites

Research the Sound Absorption Performance of Composite Material

Adopt standing wave tube test device to test the sound insulation performance of composites based on the 3 % NCW.

Sound absorption coefficient is on the basis of American Standard: ASTMC384-98.96mm diameter of round disk was used for the sample composites. Two ways applied in the test, the one is putting the sample pasted on the wall of the standing wave tube, the other one is putting the sample in the wave tube but preserving a 6cm Air layer between the sample and the tube wall. The Sound absorption coefficient is obtained under 6 frequency and the acoustic frequency Corresponded are 125Hz, 250Hz, 500Hz, 1000Hz, 2000Hz and 4000Hz. Tab.1 shows the designing and the result.

<table>
<thead>
<tr>
<th>Sheet</th>
<th>Model</th>
<th>Air Layer</th>
<th>Test Frequency /HZ</th>
<th>NRC</th>
</tr>
</thead>
<tbody>
<tr>
<td>1#</td>
<td>/</td>
<td>/</td>
<td>0.56 0.12 0.36 0.56 0.87 0.90</td>
<td>0.482</td>
</tr>
<tr>
<td>1#</td>
<td>6cm</td>
<td></td>
<td>0.08 0.32 0.46 0.91 0.78 0.90</td>
<td>0.621</td>
</tr>
<tr>
<td>2#</td>
<td>/</td>
<td>/</td>
<td>0.04 0.11 0.23 0.41 0.72 0.82</td>
<td>0.373</td>
</tr>
<tr>
<td>2#</td>
<td>6cm</td>
<td></td>
<td>0.15 0.52 0.89 0.63 0.57 0.89</td>
<td>0.657</td>
</tr>
<tr>
<td>3#</td>
<td>/</td>
<td>/</td>
<td>0.09 0.14 0.32 0.73 0.91 0.93</td>
<td>0.529</td>
</tr>
<tr>
<td>3#</td>
<td>6cm</td>
<td></td>
<td>0.22 0.76 0.60 0.57 0.88 0.83</td>
<td>0.709</td>
</tr>
<tr>
<td>4#</td>
<td>/</td>
<td>/</td>
<td>0.03 0.07 0.17 0.34 0.50 0.89</td>
<td>0.276</td>
</tr>
<tr>
<td>4#</td>
<td>6cm</td>
<td></td>
<td>0.07 0.16 0.23 0.43 0.67 0.93</td>
<td>0.379</td>
</tr>
<tr>
<td>5#</td>
<td>/</td>
<td>/</td>
<td>0.05 0.13 0.20 0.43 0.85 0.91</td>
<td>0.409</td>
</tr>
<tr>
<td>5#</td>
<td>6cm</td>
<td></td>
<td>0.11 0.23 0.41 0.51 0.78 0.94</td>
<td>0.486</td>
</tr>
<tr>
<td>6#</td>
<td>/</td>
<td>/</td>
<td>0.03 0.12 0.21 0.34 0.65 0.88</td>
<td>0.335</td>
</tr>
<tr>
<td>6#</td>
<td>6cm</td>
<td></td>
<td>0.06 0.23 0.43 0.89 0.50 0.91</td>
<td>0.515</td>
</tr>
</tbody>
</table>

From Tab.1, the sound absorption properties of the NCW/PVA composites are better in the range of high frequency while not so good with low frequency. If the noise reduction coefficient (NRC) of the material is 0.276~0.529. It is not a good sound-absorbing material. On this basis, the structure of air layer was used in the evaluation of Sound absorption coefficient. From the experiment, it is found that with the 6cm air layer, the sound absorption coefficient of the sample increased obviously in low frequency sound and NRC raised to 0.379~0.709 which makes the result improved more than 10% totally.

Research the Thermal Insulation Performance of Composites

The heat pulse in non-steady heat source method was adopted to evaluate the thermal physical performance of the composites based on 3% NCW. Heat pulse method is applicable to the measurement of temperature coefficient, Specific heat capacity and thermal conductivity and many other thermal physical parameters for homogeneous solid materials, Heterogeneous material and porous material.
Formula 1 is the calculating formula for thermal conductivity coefficient based on the thermal conductivity. Tab. 2 is the test result.

\[
\lambda = \frac{I^2 \times R \times \sqrt{a} \times (\sqrt{t_2} - \sqrt{t_1})}{A \theta(0, t_2) \sqrt{\pi}}
\]

PS:  
- \(\lambda\) — Thermal Conductivity of the sample, \((\text{W/m} \cdot \text{K})\);  
- \(\theta(0, t_2)\) — Surplus Temperature on the Heat source surface during cooling process \((\degree \text{C})\);  
- \(t_2\) — The theoretical corresponding time of the surface during cooling process(s);  
- \(t_1\) — The theoretical corresponding time of closing the heat source (s);  
- A — Area of the heater \((\text{m}^2)\);  
- I — Current passing the heater \((\text{A})\);  
- R — The resistance of the heater \((\Omega)\);  
- a — Temperature coefficient \((\text{m}^2/\text{s})\);

<table>
<thead>
<tr>
<th>Sample No</th>
<th>Thickness /cm</th>
<th>Direction of Heat Flow</th>
<th>Coefficient (10^{-4} \text{m}^2/\text{s})</th>
<th>Coefficient (/\text{W/m} \cdot \text{K})</th>
</tr>
</thead>
<tbody>
<tr>
<td>1#</td>
<td>0.2</td>
<td>vertical</td>
<td>0.000654</td>
<td>0.254</td>
</tr>
<tr>
<td>2#</td>
<td>0.2</td>
<td>vertical</td>
<td>0.000686</td>
<td>0.279</td>
</tr>
<tr>
<td>3#</td>
<td>0.2</td>
<td>vertical</td>
<td>0.000723</td>
<td>0.307</td>
</tr>
<tr>
<td>4#</td>
<td>0.2</td>
<td>vertical</td>
<td>0.000748</td>
<td>0.325</td>
</tr>
<tr>
<td>5#</td>
<td>0.2</td>
<td>vertical</td>
<td>0.000898</td>
<td>0.358</td>
</tr>
<tr>
<td>6#</td>
<td>0.2</td>
<td>vertical</td>
<td>0.000734</td>
<td>0.398</td>
</tr>
</tbody>
</table>

It can be found from Tab. Π, in the experiment, test sample is vertical to the direction of the heater. The thermal conductive coefficient values of composite material are between 0.000654～0.000898 \((10^{-4}\text{m}^2/\text{s})\). Based on the analysis of thermal conductivity, when the thermal conductivity coefficient is 0.254～0.398 \(/\text{W/m} \cdot \text{K}\), composite material occurs a smaller thermal performance, so it can be used as indoor insulation material. Refer to the design of sound absorbing structure, if air layer reserved between the material and wall, thermal insulation performance of the material with 3% NCW will be increased more.

**Conclusions**

Composite material was made by mulberry nano-cellulose whiskers/PVA, churning PVA under 90°C thermostatic bath till the dissolution, then adding mulberry nano-cellulose whiskers separately at an amount of 1%-5% comparing to the amount of PVA, and keeping on churning 30min, lastly, making it a film on a glass plate by salivation after defoaming and drying the film with infrared lamp after the moisture becomes dry naturally. The conclusion can be got from researching the performance of Composites Material as follow:

1. Putting a certain amount of NCW in PVA, the material’s physical and mechanical properties changed significantly. Tensile strength and tensile modulus of the material rise to the top and then dropped. But elongation at break keep on decreasing with the increasing of NCW. The composite material’s tensile strength raised 66.02% and the tensile modulus increased by 147.33% when the amount of 3% NCW. The point gets the best overall performance which indicates NCW has a significant reinforcement to PVA.
2. NCW/PVA material has a better sound absorption in high frequency than that of the low
frequency when being tested by pasting the wall. Air layer was adopted in the evaluation of sound absorption coefficient. The test shows when 6cm air layer is used, the material’s sound absorption coefficient increased obviously which made the NRC raised to 0.38~0.71 and made the result improved more than 10% totally.

(3) Analyzing the thermal conductivity of the material, when thermal conductive coefficient values are $0.000654 \sim 0.000898 \times 10^{-4} \text{m}^2/\text{s}$, the material can be used as indoor insulation material. Referring to the design of sound absorbing structure, if an air layer reserved between the material and car wall, the thermal insulation performance of the material with 3% NCW will be increased more.

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