Xylan Reinforcement on Poplar Cellulose Nanowhiskers films

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Poplar cellulose nanowhiskers preparation

1. Poplar ground to powder
2. Hydrolyzed with 64 wt% sulfuric acid and neutralized with 10 fold water
3. Settled cellulosics are under centrifuging to remove excessive acid
4. Solids sample was dialyzed for 3 days against DI water
5. The suspension was then sonicated and centrifuged
6. Cloudy supernatant, containing nanowhiskers was collected and concentrated

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Xylan reinforced CNW film preparation

Materials: Poplar cellulose nano-whiskers; Birchwood Xylan; Sorbitol; GPC measurement of Birch Xylan: Mw: 1.51 \times 10^4 \text{ g/mol}; Mn: 1.15 \times 10^4 \text{ g/mol};

(1) pH adjustment to deposit xylan on poplar cellulose nanowhiskers, thereby mix them with sorbitol and prepare films via solution casting technology.

(2) Density test, Mechanical Tensile strength---Strain test, Optical microscope and AFM analysis, Water vapor transmission rate (WVTR), film composition analysis to test the absorption amount of xylan, components via FTIR, film transparency via UV.

Xylan dissolved in NaOH (pH 11.30) at room temp. for 30 min with stirring

Cellulose whiskers adding in with adjusting pH to 9, and heat \Delta to 70^\circ\text{C} for 30 min, then cool to RM

Dialysis undergoes for 3 days with DI water at room temp.

Xylan deposited on CNW, solution is stirred at room temp. for 30 min and put into dialysis tubing

Adding sorbitol in and mix them thoroughly together at room temp. for 30min

\( \text{H}_2\text{SO}_4 \) adding in with adjusting pH to 5.0

Film is prepared by solution casting tech

Property testing

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### Formulation of various films

<table>
<thead>
<tr>
<th>No.</th>
<th>Sample</th>
<th>Mass weight (gram)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>CNW+S (control)</td>
<td>CNW 0.4; Sorbitol 0.2</td>
</tr>
<tr>
<td>2</td>
<td>X2%/CNW+S*</td>
<td>CNW 0.4; Sorbitol 0.2; Xylan 0.008</td>
</tr>
<tr>
<td>3</td>
<td>X4%/CNW+S</td>
<td>CNW 0.4; Sorbitol 0.2; Xylan 0.016</td>
</tr>
<tr>
<td>4</td>
<td>X6%/CNW+S</td>
<td>CNW 0.4; Sorbitol 0.2; Xylan 0.024</td>
</tr>
<tr>
<td>5</td>
<td>X8%/CNW+S</td>
<td>CNW 0.4; Sorbitol 0.2; Xylan 0.032</td>
</tr>
<tr>
<td>6</td>
<td>X10%/CNW+S</td>
<td>CNW 0.4; Sorbitol 0.2; Xylan 0.04</td>
</tr>
<tr>
<td>7</td>
<td>X20%/CNW+S</td>
<td>CNW 0.4; Sorbitol 0.2; Xylan 0.08</td>
</tr>
<tr>
<td>8</td>
<td>X30%/CNW+S</td>
<td>CNW 0.4; Sorbitol 0.2; Xylan 0.12</td>
</tr>
</tbody>
</table>

*: CNW means poplar cellulose whiskers; S means sorbitol; Xylan content percentage based on CNW mass weight

### Specific density of xylan reinforced CNW films

<table>
<thead>
<tr>
<th>Film samples</th>
<th>Density (g/cm$^3$)</th>
<th>Film samples</th>
<th>Density (g/cm$^3$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>CNW+S</td>
<td>1.1333</td>
<td>Control X2</td>
<td>1.0872</td>
</tr>
<tr>
<td>Control X2</td>
<td>1.0872</td>
<td>pH X2</td>
<td>1.2292</td>
</tr>
<tr>
<td>Control X4</td>
<td>1.2519</td>
<td>pH X4</td>
<td>1.2667</td>
</tr>
<tr>
<td>Control X6</td>
<td>1.27339</td>
<td>pH X6</td>
<td>1.1846</td>
</tr>
<tr>
<td>Control X8</td>
<td>1.1818</td>
<td>pH X8</td>
<td>1.3194</td>
</tr>
<tr>
<td>Control X10</td>
<td>1.1273</td>
<td>pH X10</td>
<td>1.275</td>
</tr>
<tr>
<td>Control X20</td>
<td>1.1333</td>
<td>pH X20</td>
<td>1.1534</td>
</tr>
<tr>
<td>Control X30</td>
<td>1.1389</td>
<td>pH X30</td>
<td>1.1590</td>
</tr>
</tbody>
</table>
Products— Xylan reinforced CNW films

Tensile strength analysis of films

Elongation at break (%)
Products— Xylan reinforced CNW films

Specific water vapor transmission rate of films

Deposited amounts of xylan on the cellulose nanowhiskers film
Products— Xylan reinforced CNW films

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Transparency measurements via UV/vis

FT-IR of xylan reinforced cellulose nanowhisker films

AFM images of cellulose nanowhiskers
Products— Xylan reinforced CNW films

3D Optical microscope images of films CNW+S

3D Optical microscope images of films control X8

3D Optical microscope images of films pH adjusted X8

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Results

- Addition of 8 % xylan improved the film tensile strength to the largest extent, increasing the tensile strength by 88.9 %, and density by 16.4%, but decreased the elongation by 15.6%.

- In addition, various amounts of xylan addition increased the film water vapor transmission rate to different extents. pH adjusted sample X8 exhibited better surface uniformity than controlled sample. Moreover, there is a large reduction in the amount of light being transmitted through the pH adjusted X8 films compared to controlled sample.
Acknowledgements

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