Effect of Heat Treatment on Properties of Chinese White Poplar

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Part I

Introduction

Heat treatment
What’s changed?
Research Aim

- Improving dimensional stability
- Enhancing decay resistance
- Darken color
- Minimizing loss ratios of MOR or MOE
- Establishing regression models
- Extending application fields

CRIWI, CAF
Materials and Method
Materials
Facilities
<table>
<thead>
<tr>
<th>Temp. (℃)</th>
<th>(T)</th>
<th>(A_1)</th>
<th>(A_2)</th>
<th>(A_3)</th>
<th>(A_4)</th>
<th>(A_5)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>(B_1) = 1</td>
<td>(B_2) = 2</td>
<td>(B_3) = 3</td>
<td>(B_4) = 4</td>
<td>(B_5) = 5</td>
<td></td>
</tr>
<tr>
<td>(A_1 = 170)</td>
<td>(A_1B_1)</td>
<td>(A_1B_2)</td>
<td>(A_1B_3)</td>
<td>(A_1B_4)</td>
<td>(A_1B_5)</td>
<td></td>
</tr>
<tr>
<td>(A_2 = 185)</td>
<td>(A_2B_1)</td>
<td>(A_2B_2)</td>
<td>(A_2B_3)</td>
<td>(A_2B_4)</td>
<td>(A_2B_5)</td>
<td></td>
</tr>
<tr>
<td>(A_3 = 200)</td>
<td>(A_3B_1)</td>
<td>(A_3B_2)</td>
<td>(A_3B_3)</td>
<td>(A_3B_4)</td>
<td>(A_3B_5)</td>
<td></td>
</tr>
<tr>
<td>(A_4 = 215)</td>
<td>(A_4B_1)</td>
<td>(A_4B_2)</td>
<td>(A_4B_3)</td>
<td>(A_4B_4)</td>
<td>(A_4B_5)</td>
<td></td>
</tr>
<tr>
<td>(A_5 = 230)</td>
<td>(A_5B_1)</td>
<td>(A_5B_2)</td>
<td>(A_5B_3)</td>
<td>(A_5B_4)</td>
<td>(A_5B_5)</td>
<td></td>
</tr>
</tbody>
</table>

CRIWI, CAF
Part III

Results and discussion

3.1 Physical properties

3.2 Chemistry analysis

3.3 Color analysis

3.4 Mechanical properties
Weight loss ratio

Range of loss ratios: 0.37% ~ 12.24%
Effect of temp. > time
Air-dry shrinkage ASE

Range of ASE: 0.84% ~ 67.21%
Effect of temp. > time
Absolute dry shrinkage ASE

Range of ASE: 1.01% ~ 55.43%
Effect of temp. > time

CRIWI, CAF
Air-dry swelling ASE

Range of ASE: 3.98% ～58.52%
Effect of temp. > time
Saturated swelling ASE

Range of ASE: 2.68% ~ 56.43%

Effect of temp. > time
### Regression models

<table>
<thead>
<tr>
<th>Loss Ratio</th>
<th>Regression Model</th>
<th>R²</th>
<th>Adj. R²</th>
<th>Pr &gt; F</th>
</tr>
</thead>
<tbody>
<tr>
<td>Absolute dry density</td>
<td>$\hat{y} = 0.139x_1 + 1.240x_2 - 25.866$</td>
<td>0.964</td>
<td>0.960</td>
<td>&lt; .0001</td>
</tr>
<tr>
<td>Weight loss ratio</td>
<td>$\hat{y} = 0.130x_1 + 1.144x_2 - 24.526$</td>
<td>0.942</td>
<td>0.937</td>
<td>&lt; .0001</td>
</tr>
<tr>
<td>Air dry shrinkage ratio</td>
<td>$\hat{y} = 0.999x_1 + 4.837x_2 - 184.299$</td>
<td>0.940</td>
<td>0.934</td>
<td>&lt; .0001</td>
</tr>
<tr>
<td>Absolute dry shrinkage ratio</td>
<td>$\hat{y} = 0.838x_1 + 3.924x_2 - 153.255$</td>
<td>0.960</td>
<td>0.956</td>
<td>&lt; .0001</td>
</tr>
<tr>
<td>Air dry swelling ratio</td>
<td>$\hat{y} = 0.596x_1 + 4.578x_2 - 94.132$</td>
<td>0.880</td>
<td>0.869</td>
<td>&lt; .0001</td>
</tr>
<tr>
<td>Water absorbed swelling ratio</td>
<td>$\hat{y} = 0.771x_1 + 3.419x_2 - 134.318$</td>
<td>0.973</td>
<td>0.971</td>
<td>&lt; .0001</td>
</tr>
</tbody>
</table>

$\hat{y}$ means loss ratio, $x_1$ means temperature, $x_2$ means time

*CRIWI, CAF*
Discussion

1. Degradation of cellulose and hemicellulloses
2. Disappearance of inorganic and volatile materials
3. Change in hydrogen bond between surfaces of cellulose
Decay resistance

I : strong decay resistance, 0 ~ 10%
II : decay resistance, 11 ~ 24%
III: little decay resistance, 25 ~ 44%
IV: no decay resistance, >45%

CRIWI, CAF
Weight loss ratios

Range of loss ratios: 55.75% ~ 2.05%
Effect of temp. > time

CRIWI, CAF
### Data of weight loss ratio

<table>
<thead>
<tr>
<th></th>
<th>Control</th>
<th>170°C</th>
<th>185°C</th>
<th>200°C</th>
<th>215°C</th>
<th>230°C</th>
</tr>
</thead>
<tbody>
<tr>
<td>1h</td>
<td>55.746</td>
<td>IV</td>
<td>IV</td>
<td>III</td>
<td>III</td>
<td>II</td>
</tr>
<tr>
<td></td>
<td>44.690</td>
<td></td>
<td>41.726</td>
<td>38.374</td>
<td>26.595</td>
<td>18.648</td>
</tr>
<tr>
<td>2h</td>
<td>55.746</td>
<td>IV</td>
<td>III</td>
<td>III</td>
<td>III</td>
<td>II</td>
</tr>
<tr>
<td></td>
<td>43.423</td>
<td></td>
<td>39.880</td>
<td>34.280</td>
<td>22.504</td>
<td>8.086</td>
</tr>
<tr>
<td>3h</td>
<td>55.746</td>
<td>IV</td>
<td>III</td>
<td>III</td>
<td>III</td>
<td>II</td>
</tr>
<tr>
<td></td>
<td>42.123</td>
<td></td>
<td>36.117</td>
<td>27.829</td>
<td>18.672</td>
<td>7.317</td>
</tr>
<tr>
<td>4h</td>
<td>55.746</td>
<td>IV</td>
<td>III</td>
<td>III</td>
<td>III</td>
<td>II</td>
</tr>
<tr>
<td></td>
<td>39.014</td>
<td></td>
<td>34.450</td>
<td>25.294</td>
<td>16.432</td>
<td>3.157</td>
</tr>
<tr>
<td>5h</td>
<td>55.746</td>
<td>IV</td>
<td>III</td>
<td>III</td>
<td>III</td>
<td>II</td>
</tr>
<tr>
<td></td>
<td>37.612</td>
<td></td>
<td>31.904</td>
<td>21.474</td>
<td>12.073</td>
<td>2.052</td>
</tr>
</tbody>
</table>

*CRIWI, CAF*
Discussion

Pyrogenation reaction

Little nutritional materials were not enough to fungi

Decreased of cellulose and lignin in wood
Part III

Results and discussion

3.1 Physical properties

3.2 Chemistry analysis

3.3 Color analysis

3.4 Mechanical properties
Range of loss ratios: 2.52% ~ 23.72%
Effect of temp. > time

CRIWI, CAF
α-cellulose content

Range of loss ratios: 0.94% ~ 41.44%
Effect of temp. > time

CRIWI, CAF
**Lignin content**

Range of increase ratio: 9.06% ~ 123.64%

Effect of temp. > time

*CRIWI, CAF*
## Hemicellulose content

<table>
<thead>
<tr>
<th></th>
<th>1h</th>
<th>2h</th>
<th>3h</th>
<th>4h</th>
<th>5h</th>
</tr>
</thead>
<tbody>
<tr>
<td>control</td>
<td>32.12</td>
<td>32.12</td>
<td>32.12</td>
<td>32.12</td>
<td>32.12</td>
</tr>
<tr>
<td>170°C</td>
<td>30.58</td>
<td>30.81</td>
<td>29.70</td>
<td>29.44</td>
<td>29.07</td>
</tr>
<tr>
<td>185°C</td>
<td>29.75</td>
<td>28.63</td>
<td>26.32</td>
<td>24.64</td>
<td>27.69</td>
</tr>
<tr>
<td>200°C</td>
<td>25.15</td>
<td>22.20</td>
<td>25.60</td>
<td>27.47</td>
<td>29.76</td>
</tr>
<tr>
<td>215°C</td>
<td>24.44</td>
<td>24.33</td>
<td>24.77</td>
<td>28.87</td>
<td>31.91</td>
</tr>
<tr>
<td>230°C</td>
<td>26.05</td>
<td>28.14</td>
<td>32.22</td>
<td>32.33</td>
<td>32.76</td>
</tr>
</tbody>
</table>
Why?
Should be decrease!
But increase? ? ?

Question and key

CRIWI, CAF
Reaction mechanisms

(Source: VTT, Thermowood® handbook)
FTIR Spectra

Wavenumbers (cm$^{-1}$)

Absorbance

1736.58, 1657.11, 1604.44, 1510.69, 1459.27, 1424.21, 1372.95, 1319.12, 1268.52, 1227.02, 1159.27, 1109.68, 1077.77

CRIWI, CAF
So …

**New materials** were produced in the thermal degradation process. **However** these new materials did not extracted thoroughly from three main chemical components.
Part Ⅲ

Results and discussion

3.1 Physical properties
3.2 Chemistry analysis
3.3 Color analysis
3.4 Mechanical properties
**Color**

CIE (1976) Color space

- $L^*$: white to dark
- $a^*$: red to green
- $b^*$: yellow to blue
- $\Delta C^*$: saturation diff.
- $\Delta E^*$: color diff.
- $\Delta H^*$: hue diff.

**CRIWI, CAF**
Range of saturation diff. : 6.87 ~ -5.14
Effect of temp. > time
Range of color diff. : 13.98 ~ 62
Effect of temp. > time

△E*

CRIWI, CAF
Range of hue diff. : 3.63 ~ 10.46
Effect of temp. > time
## Regress models

<table>
<thead>
<tr>
<th>change value</th>
<th>regression model</th>
<th>R²</th>
<th>Adj. R²</th>
<th>Pr &gt; F</th>
</tr>
</thead>
<tbody>
<tr>
<td>△C*</td>
<td>$\hat{y} = 21.972 - 0.064x_1 - 2.110x_2$</td>
<td>0.819</td>
<td>0.802</td>
<td>&lt; .0001</td>
</tr>
<tr>
<td>△E*</td>
<td>$\hat{y} = 0.226x_1 + 10.310x_2 - 34.749$</td>
<td>0.960</td>
<td>0.957</td>
<td>&lt; .0001</td>
</tr>
<tr>
<td>△H*</td>
<td>$\hat{y} = 0.035x_1 + 1.092x_2 - 3.128$</td>
<td>0.942</td>
<td>0.937</td>
<td>&lt; .0001</td>
</tr>
</tbody>
</table>

$\hat{y}$ means change values, $x_1$ means temperature, $x_2$ means time.
colors

More pictures ...
Discussion

Chromophoric groups:

\[ R - \text{CH} = \text{CH} - R \]

\[ \text{Auxochrome:} \]

- \(-\text{OH}\)
- \(-\text{COOH}\)
- \(-\text{OR}\)
- \(-\text{NH}_2\)

\[ \text{CRIWI, CAF} \]
Part III

Results and discussion

3.1 Physical properties
3.2 Chemistry analysis
3.3 Color analysis

3.4 Mechanical properties
Range of loss ratios: $-15.82\% \sim 22.09\%$

Effect of temp & time

CRIWI, CAF
**Range of loss ratios:** $-11.28\% \sim 54.2\%$

**Effect of temp. > time**
Range of increase ratios: 2.73% ~ 15.8%
Effect of temp & time

CRIWI, CAF
Regression models

<table>
<thead>
<tr>
<th>loss ratio $\hat{y}$ ( % )</th>
<th>regression model</th>
<th>$R^2$</th>
<th>Adj. $R^2$</th>
<th>Pr &gt; F</th>
</tr>
</thead>
<tbody>
<tr>
<td>bending strength $\hat{y} = 0.961x_1 + 4.218x_2 - 183.8316$</td>
<td>0.953</td>
<td>0.948</td>
<td>&lt; .0001</td>
<td></td>
</tr>
<tr>
<td>MOE $\hat{y} = 0.089x_1 + 1.544x_2 - 32.172$</td>
<td>0.777</td>
<td>0.756</td>
<td>&lt; .0001</td>
<td></td>
</tr>
</tbody>
</table>

$\hat{y}$ means loss ratio, $x_1$ means temperature, $x_2$ means time
Discussion

Before 200 °C & 2hrs,
New –OH were generated between fibers of wood; thermal degradation seems to be ignored.

After 200 °C & 2hrs,
Thermal degradation became harder and harder.
Fibre chains became shorter and shorter so that mechanical strength was decreased step by step.

But, MOE was increased during the whole process.
Part IV

Conclusion
Conclusion

- Heat treatment can remarkably improve the dimensional stability and decay resistance of wood.
- Thermal degradation of chemical component is the main reason for loss of mechanical strength of wood.
- The temperature should be controlled at below 200 °C so that there is no loss or a minimum loss in the mechanical strength of wood.
Thank You!