Nanoindentation characterization of microwave-pyrolysis biochar

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Nanoindentation Characterization of Microwave-Pyrolysis Biochar

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Biochar II: Production, Characterization and Applications
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Research Motivation & Goals

Strong potential for biochar in biocomposites manufacturing
- Can aid in compressive and flexural strength
- Can increase flammability resistance

- Need a better understanding of biochar mechanical properties

- Goals
  - Produce biochar via microwave pyrolysis
  - Characterize biochar through common techniques and nanoindentation
  - Evaluate the Young’s modulus and hardness of char
Research Methodology – Feedstocks

- Softwood Chips
  - Mixture of spruce/fir
  - Non-uniform sizes
  - Moisture content ~12%

- Hemp
  - Dried Bale
  - Hand shredded, Non-uniform sizes
  - Moisture content ~13%

- Both feedstocks were locally sourced
Research Methodology – Microwave Pyrolysis Reactor

- Experimental parameters
  - 1 kg, feedstock
  - 100 grams, carbon microwave absorber
  - 1 hr run time
  - 2700-Watt microwave power level
Scaled-up microwave reactor, UNB

- 3000 Watt max
- Large insulated single batch reactor
- Real time temperature data
- Volatiles exit through condenser
- Max. 5 kg sample

MW power supply (1), Magnetron head (2), Sub-tuner (3), Waveguide (4), SS 309 reactor (5), N₂ gas generator (6), Flow meter (7), Main power switch (8), Condenser (9), Bio-oil collector (10), Computer and data logging system (11), Temperature data acquisition system (12)

Research Methodology – Biochar Characterization

Biochar Characterization

1) Ultimate and Proximate analysis
2) Physiosorption analysis
3) SEM imaging
4) Nanoindentation
Research Methodology – Biochar Characterization

Nanoindentation

1) Cold-mounting samples
2) Polishing with decreasing grit sizes (300-1200 microns)
3) Testing with iMicro Nanoindentor (10 indentations per sample)

nanomechanicsinc.com
Results – Microwave Pyrolysis Temperature

<table>
<thead>
<tr>
<th>Feedstock</th>
<th>Residence Temperature (°C)</th>
<th>Heating Rate (°C/min)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Softwood</td>
<td>659.8 ± 59.9</td>
<td>38.7 ± 13.3</td>
</tr>
<tr>
<td>Hemp</td>
<td>604.2 ± 3.3</td>
<td>49.2 ± 10.8</td>
</tr>
</tbody>
</table>
Results – Biochar

Softwood

Hemp
Results – Biochar Proximate/Ultimate

<table>
<thead>
<tr>
<th>Specimen</th>
<th>Proximate Analysis</th>
<th>Ultimate Analysis</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>M.C. %</td>
<td>V.M. %</td>
</tr>
<tr>
<td>Raw Hemp</td>
<td>10</td>
<td>76</td>
</tr>
<tr>
<td>Raw Softwood</td>
<td>10</td>
<td>78</td>
</tr>
<tr>
<td>Hemp Char</td>
<td>3.0</td>
<td>26.8</td>
</tr>
<tr>
<td>Softwood Char</td>
<td>4.0</td>
<td>26.0</td>
</tr>
</tbody>
</table>

- Large increase in Fixed Carbon % and Carbon elemental % after pyrolysis due to release of volatiles
- Hemp biochar has larger carbon weight percentages than softwood (more full release of volatiles)
## Results – Biochar Porosity

<table>
<thead>
<tr>
<th>Sample</th>
<th>BET Surface Area (m²/g)</th>
<th>Average Pore Diameter (Å)</th>
<th>Micropore Area (m²/g)</th>
<th>Average Pore Volume (cm³/g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Raw Hemp</td>
<td>2.97</td>
<td>78.06</td>
<td>N/A</td>
<td>0.0058</td>
</tr>
<tr>
<td>Raw Softwood</td>
<td>0.76</td>
<td>134.86</td>
<td>N/A</td>
<td>0.0026</td>
</tr>
<tr>
<td>Hemp Char</td>
<td>12.18</td>
<td>52.75</td>
<td>2.58</td>
<td>0.0161</td>
</tr>
<tr>
<td>Softwood Char</td>
<td>9.96</td>
<td>46.44</td>
<td>1.63</td>
<td>0.0116</td>
</tr>
</tbody>
</table>

- Increase in BET Surface Area and Micropore Area due to development of new micropores during pyrolysis
- Hemp Biochar has better porosity properties due to faster heating rate allowing a faster release of volatiles
Results – Biochar SEM softwood (A) and hemp (B)

Images taken at 1000x magnification. Power level 27kW

Results – Biochar Nanoindentation

Hemp biochar has a higher Young’s modulus (resistance to deformation)
Results – Biochar Nanoindentation

Both biochar show partially elastic load-depth curves

Conclusion

- Hemp biochar showed a higher average Young’s Modulus than the softwood char.

- Young’s modulus and hardness of biochar are dependent on the amount of carbon percentage and development of healthy micropore lattice.

- Both samples would be suitable for biocomposite manufacturing due to their high Young’s Modulus values.
Acknowledgements
THANK YOU!

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