Preparation and characterization of activated carbon fibers from liquefied wood by KOH

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Outline

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  - Research Objective

- Experimental
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  - Surface Morphology Examination
  - Surface Chemistry Examination
  - Pore Structure Characterization

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Introduction

Activated carbon fibers (ACFs)

- High surface areas and functionalized surface
- Used as sorbent-materials in air purification, water treatment, etc.

Fossil resources
Exhausting

Wood (biomass)
Renewable
Introduction

Electric double layer capacitors (EDCLs)

- Wide pore size distributions (PSDs) and more mesopores
- Surface chemistry, especially the O functionalities.

Physical activation (steam)
- High SSA
- But narrow microporous

Chemical activation (KOH)
- Wide PSDs
- More O-containing surface groups
1. To control the porosity and surface chemical properties of wood liquefied-based ACFs

2. To investigate the effect of the KOH/fiber ratio in terms of ACFs’ porous structure and surface chemistry
Experimental

Preparing Liquefied Wood, As-spun Fibers, Precursors

Wood powder + Phenol → Liquefaction → Spinning solution

Liquefied wood

Curing → As-spun fibers

Precursors

Single-step synthesis of the spinning solution

Spinning solution

Temperature

Spinneret orifice

Spinning roller
Preparing Activated Carbon Fibers

**Carbon fibers**

500°C 1h

Carbonization

KOH (KOH/C 1~3) 850°C 1h

Activation

Steam 850°C 1h

Activation

Activated carbon fibers

ACF-1

ACF-2

ACF-3

ACF-S

Precursors
Surface Morphology Examination

More rugged surfaces for the KOH-activated ACFs
The holes become larger gradually with increasing mass ratios
XRD analysis

![XRD analysis graph](image-url)
N2-adsorption-desorption isotherms

Hysteresis loop

Pore structure parameters

<table>
<thead>
<tr>
<th>Sample</th>
<th>( S_{\text{BET}} ) (m(^2)/g)</th>
<th>( S_{\text{micro}} ) (m(^2)/g)</th>
<th>( S_{\text{meso}} ) (m(^2)/g)</th>
<th>( V_{\text{tot}} ) (cm(^3)/g)</th>
<th>( V_{\text{meso}} ) (cm(^3)/g)</th>
<th>( V_{\text{meso}}/V_{\text{tot}} ) (%)</th>
<th>Yield (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>ACFs-1</td>
<td>536</td>
<td>35</td>
<td>501</td>
<td>0.504</td>
<td>0.471</td>
<td>93.5%</td>
<td>66.8</td>
</tr>
<tr>
<td>ACFs-2</td>
<td>700</td>
<td>175</td>
<td>525</td>
<td>0.611</td>
<td>0.516</td>
<td>84.5%</td>
<td>58.2</td>
</tr>
<tr>
<td>ACFs-3</td>
<td>1371</td>
<td>1024</td>
<td>347</td>
<td>0.777</td>
<td>0.352</td>
<td>45.3%</td>
<td>35.6</td>
</tr>
<tr>
<td>ACFs-S</td>
<td>1250</td>
<td>969</td>
<td>281</td>
<td>0.644</td>
<td>0.060</td>
<td>9.3%</td>
<td>29.7</td>
</tr>
</tbody>
</table>
Pore size distributions (PSDs)
FTIR analysis
### XPS analysis

#### Table 1: C, O, and K At.% for Different ACFs Samples

<table>
<thead>
<tr>
<th>Sample</th>
<th>C(at.%)</th>
<th>O(at.%)</th>
<th>K(at.%)</th>
<th>O/C(%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>ACFs-1</td>
<td>85.2</td>
<td>13.9</td>
<td>1.0</td>
<td>16.3%</td>
</tr>
<tr>
<td>ACFs-2</td>
<td>86.1</td>
<td>12.4</td>
<td>1.5</td>
<td>14.4%</td>
</tr>
<tr>
<td>ACFs-3</td>
<td>88.6</td>
<td>9.8</td>
<td>1.6</td>
<td>11.1%</td>
</tr>
<tr>
<td>ACFs-S</td>
<td>91.4</td>
<td>8.6</td>
<td>-</td>
<td>9.4%</td>
</tr>
</tbody>
</table>

#### Table 2: BE(eV) and M(%) for Different ACFs Samples

<table>
<thead>
<tr>
<th>Sample</th>
<th>Graphite</th>
<th>C-OH</th>
<th>C=O</th>
<th>-COOH</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>BE(eV)</td>
<td>M(%)</td>
<td>BE(eV)</td>
<td>M(%)</td>
</tr>
<tr>
<td>ACFs-1</td>
<td>284.7</td>
<td>66.4</td>
<td>285.6</td>
<td>15.1</td>
</tr>
<tr>
<td>ACFs-2</td>
<td>284.7</td>
<td>58.8</td>
<td>285.7</td>
<td>20.2</td>
</tr>
<tr>
<td>ACFs-3</td>
<td>284.7</td>
<td>58.6</td>
<td>285.6</td>
<td>16.6</td>
</tr>
<tr>
<td>ACFs-S</td>
<td>284.7</td>
<td>68.1</td>
<td>285.5</td>
<td>15.0</td>
</tr>
</tbody>
</table>

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[Graphical Illustration of XPS analysis]
In case of KOH/fiber ratio 3, the BET surface area of ACFs could reach values up to 1371 m²/g.

Compared with the ACFs activated by steam at the same temperature, the samples ACF-3 have more small micropores (<0.7 nm) in addition to more mesopores in the range of 2-4 nm.

The KOH-activated ACFs had more oxygenated functional groups on their surfaces than the steam-activated ACF-S.
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Thank you for your attention