SYNTHESIS AND CHARACTERIZATION
OF ACTIVATED CARBON

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Synthesis

Sludge → Sludge-based adsorbent
Water polluted with heavy metals is a serious environmental problem.

Adsorption is a preferable treatment method.

The most widely used adsorbent is activated carbon (AC).

The price of coal-based AC has risen up to 80%.

Alternative precursors to replace non-renewable and expensive commercial AC.
Why Activated Carbon

- High surface area.
- Relatively cheap compared to other adsorbents.
- Economically viable.
- Chemically stable.
- Highly durable in acidic and basic environments.
Why Adsorption

- **Best** pollution control technology.
- Filtration, coagulation and ion-exchange are sometimes **ineffective**.
- Able to remove **low concentration** pollutants.
- Easy to **scale-up**.
- Relatively **cheap**.
- No further **sludge** treatment required.
<table>
<thead>
<tr>
<th>Method of treatment</th>
<th>Disadvantage</th>
</tr>
</thead>
<tbody>
<tr>
<td>Chemical precipitation</td>
<td>Slow process, poor settling, sludge production, high operating and handling costs for chemicals used and sludge treatment prior to disposal.</td>
</tr>
<tr>
<td>Coagulation-flocculation</td>
<td>Sludge generation, high operational costs due to high chemicals consumption and sludge disposal.</td>
</tr>
<tr>
<td>Dissolved air flotation</td>
<td>High operating cost, imperfect removal performance.</td>
</tr>
<tr>
<td>Membrane filtration</td>
<td>Membrane fouling, high operating and maintenance costs, high energy consumption.</td>
</tr>
<tr>
<td>Ion-exchange</td>
<td>Low surface area, high capital cost, varying metal removal ability of different resins, difficult to scale-up.</td>
</tr>
<tr>
<td>Electrochemical treatments</td>
<td>High operational cost, need periodic maintenance, high energy consumption.</td>
</tr>
</tbody>
</table>
Pore Characteristics

Macropore: widths greater or equal to 50 nm
Mesopore: widths between 2 nm and 50 nm
Micropore: widths lesser or equal to 2 nm

## Possible Functional Groups

<table>
<thead>
<tr>
<th>Acidic oxygen groups</th>
<th>Basic oxygen groups</th>
<th>Nitrogen groups</th>
</tr>
</thead>
<tbody>
<tr>
<td><img src="image" alt="Carboxyl" /></td>
<td><img src="image" alt="Quinone" /></td>
<td><img src="image" alt="Amine" /></td>
</tr>
<tr>
<td><img src="image" alt="Lactone" /></td>
<td><img src="image" alt="Chromene" /></td>
<td><img src="image" alt="Pyridine" /></td>
</tr>
<tr>
<td><img src="image" alt="Phenol" /></td>
<td><img src="image" alt="Furan" /></td>
<td><img src="image" alt="Pyrrol" /></td>
</tr>
<tr>
<td><img src="image" alt="Carbonyl" /></td>
<td><img src="image" alt="Pyrone" /></td>
<td><img src="image" alt="Tertiary nitrogen" /></td>
</tr>
</tbody>
</table>
Proposed acidic and basic oxygen functionalities on carbon surfaces
Chemical Activation

N₂, 300 mL/min

500 °C, 1 h

H₂, H₂O, HCl, CO₂, N₂, etc.

Reagent: Precursor

Ceramic tube

Furnace

PRECURSOR

One-step chemical activation
Physical Activation

Steam activation

80 mL/min N₂, 13.5 mL/h H₂O

900 °C, 1 h

Volatiles

CO₂ activation

300 mL/min CO₂

900 °C, 1 h

Volatiles

CARBONIZED PRECURSOR
**Characterization**

- $\text{pH}_{\text{PZC}}$: pH drift method.
- Surface chemistry: Boehm titration method.
- Pore texture: BET analyzer
- Elemental compositions
- Heavy metal concentration
Boehm Titration

Basicity

strong

NaOH

Na₂CO₃

NaHCO₃

-COOH

-COO-

Ar-OH

weak

Acidity

strong

weak
Post-Activation Treatment

H₂, H₂O, CO₂, CO, He, etc.

Precursor-based Activated carbon

Quartz tube
Furnace

1000 °C, 1 h

Before out-gassing

AC surface

After out-gassing

AC surface
Suggested Mechanisms

- Pair of σ and localized π electrons
- Delocalized π electrons
- Tertiary nitrogen
- Pyridinic or pyridonic
- Pyrrolic
Suggested Mechanisms

Degree of cyclization can avoid the removal of nitrogen during activation.
• Two surface sites of different affinity.
• Mesopores offered higher affinity at lower $C_e$.
• Micropores offered lower affinity towards copper ions.
Suggested Mechanisms

Legend
(a) Outer surface of carbon
(b) Mesopore channel (2-50nm)
(c) Micropore channel (<2nm)
● Copper ions
THANK YOU