Effect of pyrolysis conditions on sewage sludge derived biochars for high value composites applications

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EFFECT OF PYROLYSIS CONDITIONS ON SEWAGE SLUDGE DERIVED BIOCHARS FOR HIGH VALUE COMPOSITES APPLICATIONS

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The economy of the wastewater treatment system is significantly burdened by the increasing amounts of sewage sludge and by the complexity of the treatments required for guaranteeing a safe handling and a proper end-of-life of the sludge.
Objectives

To explore the effect of temperature, heating rate and feedstock composition on the conductivity of the chars and char-polymer composites.

Pyrolysis at different heating rate and temperatures

- Char F1-HR-T
  - Chemical, physical and electrical characterization
  - Char F1-HR1-T

Slow pyrolysis at different temperatures

- Char F1-T
  - Char F1-T1

Pyrolysis at the selected temperature T1

- Char F2-T1
- Char F3-T1

Preparation and characterization of the epoxy resin composites
### Feedstock characterization

<table>
<thead>
<tr>
<th>Material</th>
<th>Acronym</th>
<th>C</th>
<th>H</th>
<th>N</th>
<th>O</th>
<th>Volatiles</th>
<th>Fixed Carbon</th>
<th>Ash</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sewage Sludge</td>
<td>S</td>
<td>39.4</td>
<td>5.6</td>
<td>6.7</td>
<td>24.2</td>
<td>65.3</td>
<td>10.6</td>
<td>24.1</td>
</tr>
<tr>
<td>Walnut Shells</td>
<td>WS</td>
<td>42.5</td>
<td>6.0</td>
<td>0.1</td>
<td>51.1</td>
<td>79.7</td>
<td>20.0</td>
<td>0.3</td>
</tr>
<tr>
<td>Lignin Rich Residue</td>
<td>LRR</td>
<td>47.3</td>
<td>5.9</td>
<td>0.7</td>
<td>38.2</td>
<td>69.6</td>
<td>22.4</td>
<td>8.0</td>
</tr>
</tbody>
</table>

### Inorganics content

**Phosphate in the urban wastewater**

- **Compounds added during the treatment process**
Pyrolysis Set-Up

Slow pyrolysis

- Heating rate (°C/min): 5
- Final temperature (°C): 500, 600, 700
- H layer (mm): 3
- Feedstock layer amount (g): 1.5 g

Fast pyrolysis

- $d_{\text{sand}}$ (mm): 0.3-0.4
- $m_{\text{sand}}$ (g): 180
- $d_p$ (mm): 1.5-2.8
- Temperature (°C): 500, 600, 700
- $U$ (@T, m/s): 0.4

(1) Gas preheating section;
(2) Gas distributor;
(3) Fluidization column;
(4) Electrical heaters;
(5) Ceramic insulator;
(6) Thermocouple;
(7) Digital mass flow meters;
(8) Steel basket.
Polymer Composite Preparation

Biochar (40 wt%) Epoxy Resin Ultraturrax 3000÷25000 rpm Hardener Mixing Vacuum Oven curing

Easy mixing process
Good dispersion (shape, dimensions)

Composite dogbone or cylindrical shape for testing
## Characterization Methods

### Feedstock

<table>
<thead>
<tr>
<th>S</th>
<th>WS</th>
<th>LRR</th>
</tr>
</thead>
</table>

### Char

<table>
<thead>
<tr>
<th>Elemental analysis</th>
<th>CEN/TS 15104</th>
</tr>
</thead>
<tbody>
<tr>
<td>Proximate analysis</td>
<td>ASTM E870</td>
</tr>
<tr>
<td>ICP/MS</td>
<td>US-EPA 3051 and 3052</td>
</tr>
<tr>
<td>N\textsubscript{2} adsorption porosimetry</td>
<td>BET surface, total pore volume</td>
</tr>
<tr>
<td>Raman Spectroscopy</td>
<td>Renshaw Ramascope MicroRaman, equipped with an Argon green laser (excitation at 514.5 nm at 50 mW)</td>
</tr>
<tr>
<td>SEM analysis</td>
<td>ZEISS SUPRATM 40 Field Emission Scanning Electron Microscope</td>
</tr>
</tbody>
</table>

### Char+resin composite

| Electrical conductivity | Home-made measurement device |

### Electrical conductivity

\[
\sigma = \frac{1}{\rho} = \frac{l}{RS}
\]

- Thickness [m]
- Conductivity [S/m]
- Surface [m\textsuperscript{2}]
- Resistance [\Omega]
Yields and Carbon Content

The high ash content of S is responsible of the highest char yields.

Differently from the typical behavior of the lignocellulosic biomasses at low pyrolysis temperature carbon content is lower than in the raw feedstock due to the high content of ash.

The decrease of H/C ratios with the temperature in S-chars and in all the chars produced at 700 °C indicates the tendency of the Carbon to form aromatic structures.
Slow pyrolysis creates a more ordered carbon structure (deeper canyon between D and G peaks) for WS and LRR chars.

The higher G peaks observed for WS and LRR chars indicate a higher level of Carbon graphitization.
The high concentration of conductive metals in the S-char provide for the lower Carbon content in determining its final electrical properties.

The increase of Carbon content and its tendency to form aromatic structures at increasing pyrolysis temperature contribute to improve chars electrical properties.
Chars with comparable electrical conductivities provide different conductive properties to the corresponding polymer composites!
Coarser particles were obtained for S-char after grinding the chars in the same conditions before the composites preparation.

In the composite coarser particles could form aggregates separated by a thick polymer layer acting as an insulating barrier.
Higher pore volumes favor the penetration of the polymer inside the char matrix.
For S-chars fast pyrolysis conditions slightly improve chars’ conductivity but have a great positive effect on the conductivity of the corresponding composites.
Conclusions

• The increasing carbon content and aromatization induced by the high pyrolysis temperature affects positively the chars electrical conductivity.

• The presence of conductive metals in the sludge derived chars provides for the low carbon content in determining the electrical conductivity.

• The electrical conductivity of the composites is strongly affected by the particle size and porosity of the chars.

• Fast pyrolysis conditions promote the improvement of the electrical conductivity of both the sludge derived chars and the corresponding composite.