Analysis of Sugar Beet Pulp by X-ray Photoelectron Spectroscopy

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Analysis of Sugar Beet Pulp by X-ray Photoelectron Spectroscopy

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Determining the chemical structure and composition of biomass fuels using x-ray photoelectron spectroscopy (XPS) can provide fundamental knowledge of their structures that is useful in understanding and predicting their combustion behavior. Sugar beet pulp is an example of an agricultural residue (byproduct of food and feed production) of potential interest for biomass combustion. The XPS spectra of sugar beet pulp provide both its elemental composition and indications of its bonding. Traditional fuel analyses of this fuel are also provided. These include: ultimate analysis — the elemental composition of the overall fuel (C, H, N, S, and O); chlorine analysis — reported here as part of the ultimate analysis but formally a separate procedure; proximate analysis — the proximate composition of the fuel (moisture, fixed carbon, volatiles, and ash); heating value — the specific heat of combustion. These data are summarized with the XPS spectra. © 2005 American Vacuum Society. [DOI: 10.1116/11.20040803]

Keywords: biomass; sugar beet pulp; XPS; fuel

PACS: 82.80.Pv, 01.30.Kj, 84.60.Rb, 82.33.Vx, 82.60.Cx

SPECIMEN DESCRIPTION

Host Material: sugar beet pulp
Host Material Characteristics: homogeneous; amorphous; unknown electrical characteristics; biological material; powder
Chemical Name: cellulose
Host Composition: see entry for History & Significance
Form: powder

History & Significance: Sugar beet pulp is an example of an agricultural residue (byproduct of food and feed production) of potential interest for biomass combustion. The XPS spectra of sugar beet pulp provide both its elemental composition and indications of its bonding. Traditional fuel analyses of this fuel are also provided. These include: ultimate analysis — the elemental composition of the overall fuel (C, H, N, S, and O); chlorine analysis — reported here as part of the ultimate analysis but formally a separate procedure; proximate analysis — the proximate composition of the fuel (moisture, fixed carbon, volatiles, and ash); heating value — the specific heat of combustion. These data are summarized with the XPS spectra. The chemical composition of sugar beet pulp is summarized in Table 1.

As Received Condition: powder
Analyzed Region: same as host material

Ex Situ Preparation/Mounting: Sawdust powders were used as received. The powders were pressed onto a piece of nonconductive double-sticky tape mounted on a piece of silicon, which was then mounted on the sample stage with a piece of the same tape.

In Situ Preparation: none
Pre-Analysis Beam Exposure: No damage was observed in the sample even after several hours of exposure to x-ray radiation.

In Situ Pre-Analysis Beam Exposure: sawdust powders were used as received. The powders were pressed onto a piece of nonconductive double-sticky tape mounted on a piece of silicon, which was then mounted on the sample stage with a piece of the same tape.

After 4 h of exposure to x rays, the intensity of the N 1s scan did not change.

Charge Control: A flood gun was applied. The flood gun voltage was 4 V, and its current was less than 50 mA. A metal screen was used to mask the sample. The charge control was determined by observing zirconia Zr 3p3/2 peak positions under different flood gun settings. XPS spectra showed a Zr 3d5/2 at 182.3 eV. The metal screen used was nickel, 1 mm distance, 70 lines/in. and 90% transmission.

Temp. During Analysis: 298 K
Pressure During Analysis: <2.0 × 10⁻⁶ Pa

INSTRUMENT DESCRIPTION

Manufacturer and Model: Surface Science Instruments SSX-100
Analyzer Type: spherical sector
Detector: resistive anode position detector
Number of Detector Elements: 128

Table 1: Fuel analysis of bio-fuel sunflower shells (ash free basis except for ash and LHV, which are on an as-received basis).

<table>
<thead>
<tr>
<th>% by weight</th>
<th>Moisture</th>
<th>C</th>
<th>H</th>
<th>O</th>
<th>N</th>
<th>S</th>
<th>Ash</th>
<th>Sum</th>
<th>LHV*, MJ/kg</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>9.84</td>
<td>46.40</td>
<td>5.85</td>
<td>34.33</td>
<td>0.88</td>
<td>0.18</td>
<td>2.52</td>
<td>100.0</td>
<td>18.816</td>
</tr>
</tbody>
</table>

*Lower heating value
INSTRUMENT PARAMETERS COMMON TO ALL SPECTRA

- **Spectrometer**
  Analyzer Mode: constant pass energy
  Throughput \( T = E^N \): \( N = 0 \)
  Excitation Source Window: 12 \( \mu \)m aluminum foil
  Excitation Source: Al \( K_a \) monochromatic
  Source Energy: 1486.6 eV
  Source Strength: 200 W
  Signal Mode: multichannel direct

- **Geometry**
  Incident Angle: 55°
  Source to Analyzer Angle: 70.8°
  Emission Angle: 55°

Specimen Azimuthal Angle: 0°
Acceptance Angle from Analyzer Axis: 0°

DATA ANALYSIS METHOD

- Peak Shape and Background Method: Shirley background function

Quantitation Method: Sensitivity factors were obtained from ESCA 2000 NT software supplied by Service Physics. The peak areas are the areas above a linear background.

ACKNOWLEDGMENTS

The authors acknowledge U.S. DOE Biomass Power Program for financial support, and Elsam engineering and Eltra, both Danish companies, which provided complementary analyses and some financial support for this investigation.
### SPECTRAL FEATURES TABLE

<table>
<thead>
<tr>
<th>Spectrum ID #</th>
<th>Element/Transition</th>
<th>Peak Energy (eV)</th>
<th>Peak Width FWHM (eV)</th>
<th>Peak Area (counts)</th>
<th>Sensitivity Factor</th>
<th>Concentration (at. %)</th>
<th>Peak Assignment</th>
</tr>
</thead>
<tbody>
<tr>
<td>00897-02</td>
<td>O 1s</td>
<td>527.8</td>
<td>3.0</td>
<td>138000</td>
<td>2.5</td>
<td>23.6</td>
<td>⋮</td>
</tr>
<tr>
<td>00897-03</td>
<td>C 1s</td>
<td>281.0</td>
<td>3.9</td>
<td>175000</td>
<td>1</td>
<td>74.6</td>
<td>⋮</td>
</tr>
<tr>
<td>00897-04</td>
<td>N 1s</td>
<td>395.6</td>
<td>3.9</td>
<td>71800</td>
<td>1.68</td>
<td>1.8</td>
<td>⋮</td>
</tr>
<tr>
<td>00897-05</td>
<td>O 1s</td>
<td>527.8</td>
<td>2.4</td>
<td>50700</td>
<td>2.5</td>
<td>⋮</td>
<td>⋮</td>
</tr>
<tr>
<td>00897-06</td>
<td>C 1s</td>
<td>280.1</td>
<td>2.7</td>
<td>60200</td>
<td>1</td>
<td>⋮</td>
<td>⋮</td>
</tr>
<tr>
<td>00897-07</td>
<td>N 1s</td>
<td>395.1</td>
<td>1.6</td>
<td>92200</td>
<td>1.68</td>
<td>⋮</td>
<td>⋮</td>
</tr>
</tbody>
</table>

### GUIDE TO FIGURES

<table>
<thead>
<tr>
<th>Spectrum (Accession) #</th>
<th>Spectral Region</th>
<th>Voltage Shift*</th>
<th>Multiplier</th>
<th>Baseline</th>
<th>Comment #</th>
</tr>
</thead>
<tbody>
<tr>
<td>897-1</td>
<td>Survey</td>
<td>0</td>
<td>1</td>
<td>0</td>
<td>1</td>
</tr>
<tr>
<td>897-2</td>
<td>O 1s</td>
<td>0</td>
<td>1</td>
<td>0</td>
<td>1</td>
</tr>
<tr>
<td>897-3</td>
<td>C 1s</td>
<td>0</td>
<td>1</td>
<td>0</td>
<td>1</td>
</tr>
<tr>
<td>897-4</td>
<td>N 1s</td>
<td>0</td>
<td>1</td>
<td>0</td>
<td>1</td>
</tr>
<tr>
<td>897-5</td>
<td>O 1s</td>
<td>0</td>
<td>1</td>
<td>0</td>
<td>2</td>
</tr>
<tr>
<td>897-6</td>
<td>C 1s</td>
<td>0</td>
<td>1</td>
<td>0</td>
<td>2</td>
</tr>
<tr>
<td>897-7</td>
<td>N 1s</td>
<td>0</td>
<td>1</td>
<td>0</td>
<td>2</td>
</tr>
</tbody>
</table>

* Voltage shift of the archived (as-measured) spectrum relative to the printed figure. The figure reflects the recommended energy scale correction due to a calibration correction, sample charging, flood gun, or other phenomenon.
1. 800 μm x-ray beam diameter, 150 eV pass energy
2. 300 μm x-ray beam diameter, 50 eV pass energy
<table>
<thead>
<tr>
<th><strong>Accession #</strong></th>
<th>00897-01</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Host Material</strong></td>
<td>sugar beet pulp</td>
</tr>
<tr>
<td><strong>Technique</strong></td>
<td>XPS</td>
</tr>
<tr>
<td><strong>Spectral Region</strong></td>
<td>survey</td>
</tr>
<tr>
<td><strong>Instrument</strong></td>
<td>Surface Science Instruments SSX-100</td>
</tr>
<tr>
<td><strong>Excitation Source</strong></td>
<td>$\text{Al } K_{\alpha}$ monochromatic</td>
</tr>
<tr>
<td><strong>Source Energy</strong></td>
<td>1486.6 eV</td>
</tr>
<tr>
<td><strong>Source Strength</strong></td>
<td>200 W</td>
</tr>
<tr>
<td><strong>Source Size</strong></td>
<td>0.8 mm $\times$ 0.8 mm</td>
</tr>
<tr>
<td><strong>Analyzer Type</strong></td>
<td>spherical sector</td>
</tr>
<tr>
<td><strong>Incident Angle</strong></td>
<td>55°</td>
</tr>
<tr>
<td><strong>Emission Angle</strong></td>
<td>55°</td>
</tr>
<tr>
<td><strong>Analyzer Pass Energy</strong></td>
<td>150 eV</td>
</tr>
<tr>
<td><strong>Analyzer Resolution</strong></td>
<td>1.5 eV</td>
</tr>
<tr>
<td><strong>Total Signal Accumulation Time</strong></td>
<td>2200 s</td>
</tr>
<tr>
<td><strong>Total Elapsed Time</strong></td>
<td>2400 s</td>
</tr>
<tr>
<td><strong>Number of Scans</strong></td>
<td>10</td>
</tr>
<tr>
<td><strong>Source Beam Size at Specimen Surface</strong></td>
<td>0.8 mm $\times$ 1.392 mm</td>
</tr>
<tr>
<td><strong>Effective Detector Width</strong></td>
<td>19 eV</td>
</tr>
<tr>
<td><strong>Analyzer Width</strong></td>
<td>1500 $\mu$m $\times$ 12000 $\mu$m at 84 eV</td>
</tr>
</tbody>
</table>
**Accession #:** 00897-03
**Host Material:** sugar beet pulp
**Technique:** XPS
**Spectral Region:** O 1s

Instrument: Surface Science Instruments SSX-100
Excitation Source: Al K$_\alpha$ monochromatic
Source Energy: 1486.6 eV
Source Strength: 200 W
Source Size: 0.8 mm × 0.8 mm
Incident Angle: 55°
Analyzer Type: spherical sector
Analyzer Pass Energy: 150 eV
Analyzer Resolution: 1.5 eV
Emission Angle: 55°
Total Signal Accumulation Time: 306.5 s
Total Elapsed Time: 475.5 s
Number of Scans: 5
Source Beam Size at Specimen Surface: 0.8 mm × 1.392 mm
Effective Detector Width: 19 eV
Analyzer Width: 1500 μm × 12000 μm at 84 eV

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**Accession #:** 00897-02
**Host Material:** sugar beet pulp
**Technique:** XPS
**Spectral Region:** C 1s

Instrument: Surface Science Instruments SSX-100
Excitation Source: Al K$_\alpha$ monochromatic
Source Energy: 1486.6 eV
Source Strength: 200 W
Source Size: 0.8 mm × 0.8 mm
Incident Angle: 55°
Analyzer Type: spherical sector
Analyzer Pass Energy: 150 eV
Analyzer Resolution: 1.5 eV
Emission Angle: 55°
Total Signal Accumulation Time: 306.5 s
Total Elapsed Time: 475.5 s
Number of Scans: 5
Source Beam Size at Specimen Surface: 0.8 mm × 1.392 mm
Effective Detector Width: 19 eV
Analyzer Width: 1500 μm × 12000 μm at 84 eV
Accession #: 00897-04
Host Material: sugar beet pulp
Technique: XPS
Spectral Region: N 1s
Instrument: Surface Science Instruments SSX-100
Excitation Source: Al K$_\alpha$ monochromatic
Source Energy: 1486.6 eV
Source Strength: 200 W
Source Size: 0.8 mm $\times$ 0.8 mm
Incident Angle: 55°
Analyzer Type: spherical sector
Analyzer Pass Energy: 150 eV
Analyzer Resolution: 1.5 eV
Emission Angle: 55°
Total Signal Accumulation Time: 3065 s
Total Elapsed Time: 3234 s
Number of Scans: 50
Source Beam Size at Specimen Surface: 0.8 mm $\times$ 1.392 mm
Effective Detector Width: 19 eV
Analyzer Width: 1500 $\mu$m $\times$ 12000 $\mu$m at 84 eV

---

Accession #: 00897-05
Host Material: sugar beet pulp
Technique: XPS
Spectral Region: O 1s
Instrument: Surface Science Instruments SSX-100
Excitation Source: Al K$_\alpha$ monochromatic
Source Energy: 1486.6 eV
Source Strength: 200 W
Source Size: 0.3 mm $\times$ 0.3 mm
Incident Angle: 55°
Analyzer Type: spherical sector
Analyzer Pass Energy: 50 eV
Analyzer Resolution: 0.5 eV
Emission Angle: 55°
Total Signal Accumulation Time: 613 s
Total Elapsed Time: 782 s
Number of Scans: 10
Source Beam Size at Specimen Surface: 0.3 mm $\times$ 0.523 mm
Effective Detector Width: 6.6 eV
Analyzer Width: 750 $\mu$m $\times$ 6000 $\mu$m at 84 eV
Host Material: sugar beet pulp
Technique: XPS
Spectral Region:

Instrument: Surface Science Instruments SSX-100
Excitation Source: Al K$_\alpha$ monochromatic
Source Energy: 1486.6 eV
Source Strength: 200 W
Source Size: 0.3 mm $\times$ 0.3 mm
Incident Angle: 55°
Analyzer Type: spherical sector
Analyzer Pass Energy: 50 eV
Analyzer Resolution: 0.5 eV
Emission Angle: 55°
Total Signal Accumulation Time: 613 s
Total Elapsed Time: 782 s
Number of Scans: 10
Source Beam Size at Specimen Surface: 0.3 mm $\times$ 0.523 mm
Effective Detector Width: 6.6 eV
Analyzer Width: 750 $\mu$m $\times$ 6000 $\mu$m at 84 eV

Instrument: Surface Science Instruments SSX-100
Excitation Source: Al K$_\alpha$ monochromatic
Source Energy: 1486.6 eV
Source Strength: 200 W
Source Size: 0.3 mm $\times$ 0.3 mm
Incident Angle: 55°
Analyzer Type: spherical sector
Analyzer Pass Energy: 50 eV
Analyzer Resolution: 0.5 eV
Emission Angle: 55°
Total Signal Accumulation Time: 24520 s
Total Elapsed Time: 24689 s
Number of Scans: 400
Source Beam Size at Specimen Surface: 0.3 mm $\times$ 0.523 mm
Effective Detector Width: 6.6 eV
Analyzer Width: 750 $\mu$m $\times$ 6000 $\mu$m at 84 eV