Analysis of Shea Nut Shells 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. Shea nut shells are an example of an agricultural residue (byproduct of food and feed production) of potential interest for biomass combustion. The XPS spectra of shea nut shells 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; ash chemistry analysis — an elemental analysis of the ash content, expressed as oxides (which does not imply that they occur as oxides in the fuel). These data are summarized with the XPS spectra.
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Keywords: *biomass; shea nut shells; XPS; fuel*

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

Accession # 00898
Technique: XPS
Host Material: shea nut shells
Instrument: Surface Science Instruments SSX-100
Major Elements in Spectrum: C, O
Minor Elements in Spectrum: N
Printed Spectra: 7
Spectra in Electronic Record: 7
Spectral Category: technical

SPECIMEN DESCRIPTION

Host Material: shea nut shells

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: Shea nut shells are an example of an agricultural residue (byproduct of food and feed production) of potential interest for biomass combustion. The XPS spectra of shea nut shells 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; ash chemistry analysis — an elemental analysis of the ash content, expressed as oxides (which does not imply that they occur as oxides in the fuel). These data are summarized with the XPS spectra. The chemical composition of Shea nut shells is summarized in Tables 1 and 2.

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,

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

	% by weight
Moisture	9.84
C	46.40
H	5.85
O	34.33
N	0.88
S	0.18
Ash	2.52
Sum	100.0
LHV*, MJ/kg	18.816

*Lower heating value

Table 2: Ash composition of sunflower shells (percent of ash basis).

	Mass %
SiO_2	1.1
Al_2O_3	0.5
Fe_2O_3	0.9
CaO	16
MgO	13.1
Na_2O	<0.2
K_2O	45.1
SO_3	11.7
P_2O_5	10.1
Cl	1.2
Other	0.3
Sum	100

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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. After 4 h of exposure to x rays, the intensity of the N 1*s* 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 $3p_{3/2}$ peak positions under different flood gun settings. XPS spectra showed a Zr $3d_{5/2}$ 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: $<2.0\times10^{-6}$ 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

INSTRUMENT PARAMETERS COMMON TO ALL SPECTRA

■ *Spectrometer*

Analyzer Mode: constant pass energy

Throughput ($T=E^N$): $N=0$

Excitation Source Window: 12 μm aluminum foil

Excitation Source: Al K_α 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

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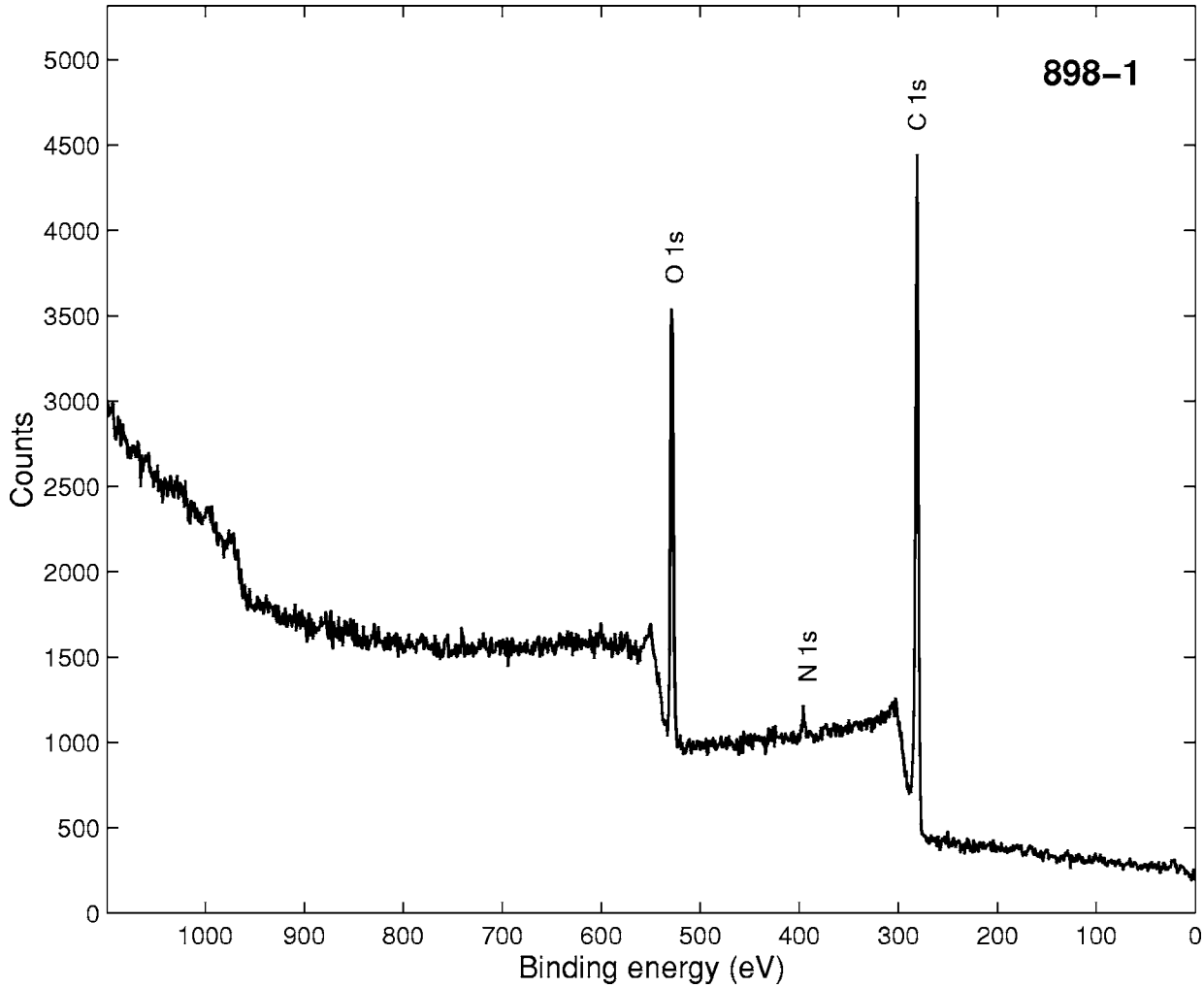
SPECTRAL FEATURES TABLE

Spectrum ID #	Element/ Transition	Peak Energy (eV)	Peak Width FWHM (eV)	Peak Area (counts)	Sensitivity Factor	Concen-tration (at. %)	Peak Assignment
00898-02	O 1*s*	528.6	3.3	67700	2.5	20	···
00898-03	C 1*s*	281.4	3.8	106000	1	78.2	···
00898-04	N 1*s*	396.2	4.8	39700	1.68	1.7	···
00898-05	O 1*s*	529.0	3.1	26400	2.5	···	···
00898-06	C 1*s*	281.9	3.2	37000	1	···	···
00898-07	N 1*s*	396.7	3.5	50000	1.68	···	···

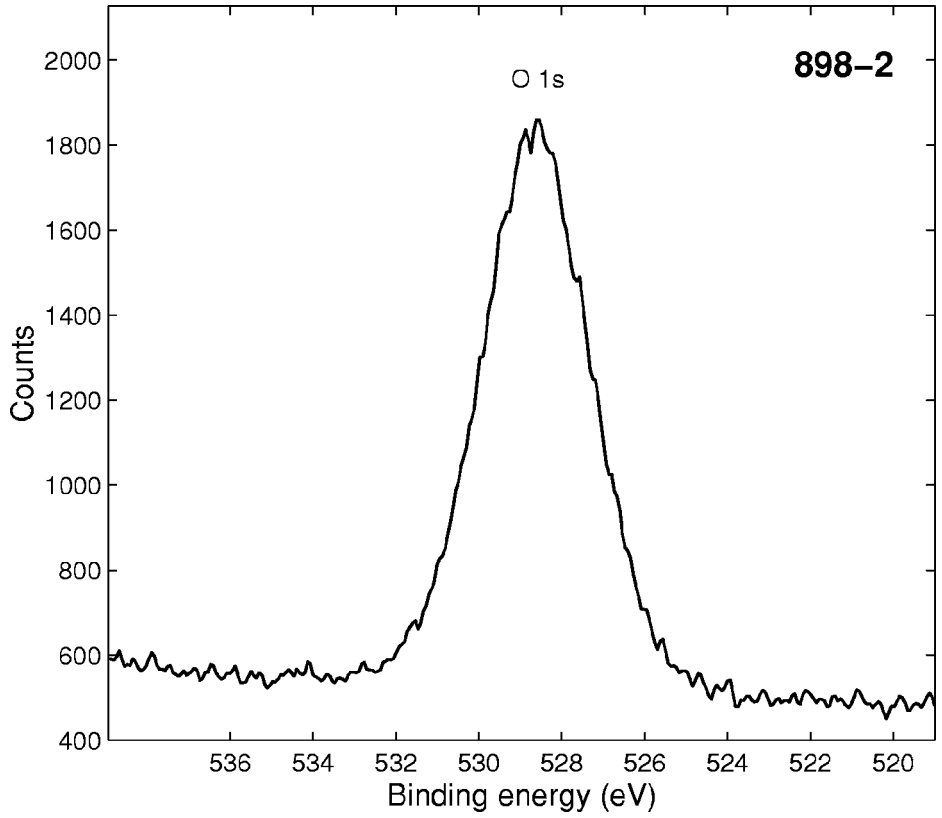
GUIDE TO FIGURES

Spectrum (Accession) #	Spectral Region	Voltage Shift*	Multiplier	Baseline	Comment #
898-1	Survey	0	1	0	1
898-2	O 1*s*	0	1	0	1
898-3	C 1*s*	0	1	0	1
898-4	N 1*s*	0	1	0	1
898-5	O 1*s*	0	1	0	2
898-6	C 1*s*	0	1	0	2
898-7	N 1*s*	0	1	0	2

* 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

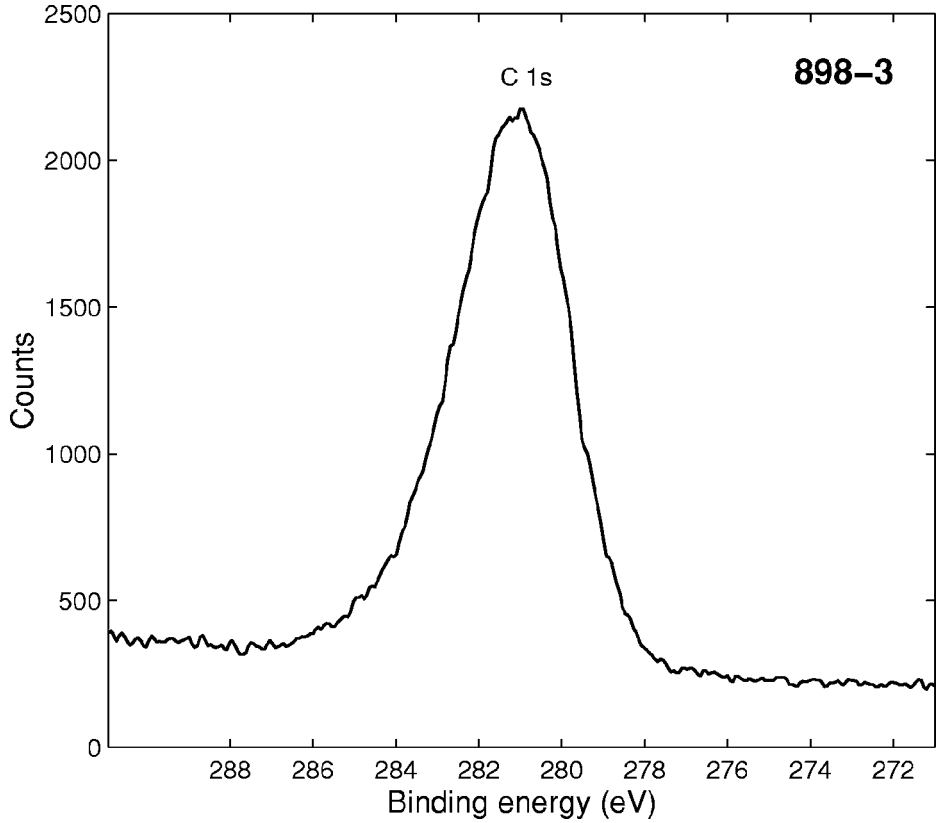


Accession #	00898-01
Host Material	shea nut shells
Technique	XPS
Spectral Region	survey
Instrument	Surface Science Instruments SSX-100
Excitation Source	Al K_α monochromatic
Source Energy	1486.6 eV
Source Strength	200 W
Source Size	0.8 mm × 0.8 mm
Analyzer Type	spherical sector
Incident Angle	55°
Emission Angle	55°
Analyzer Pass Energy	150 eV
Analyzer Resolution	1.5 eV
Total Signal Accumulation Time	2200 s
Total Elapsed Time	2400 s
Number of Scans	10
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



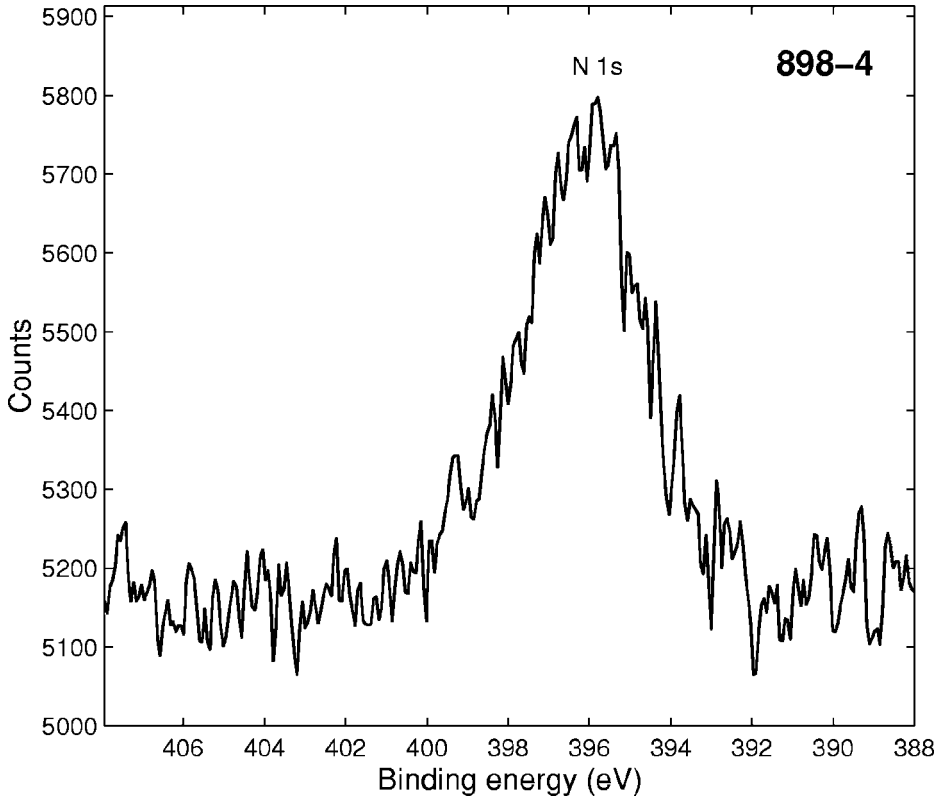
- **Accession #:** 00898-02
- **Host Material:** shea nut shells
- **Technique:** XPS
- **Spectral Region:** O 1*s*

Instrument: Surface Science Instruments SSX-100
Excitation Source: Al K_α 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



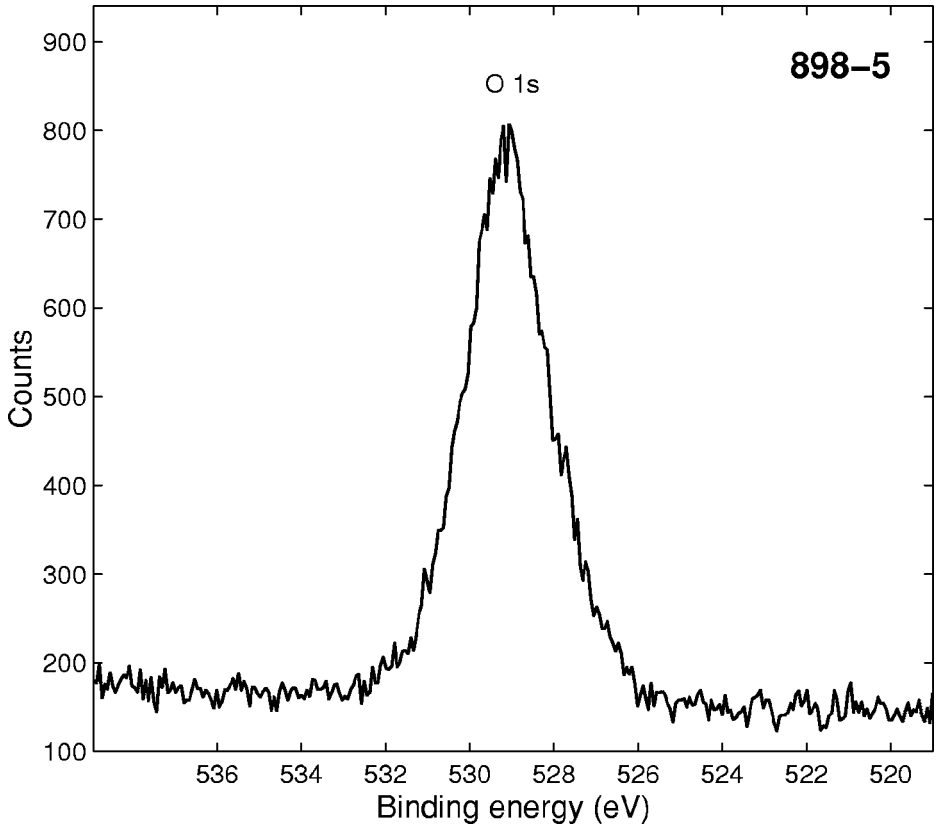
- **Accession #:** 00898-03
- **Host Material:** shea nut shells
- **Technique:** XPS
- **Spectral Region:** C 1*s*

Instrument: Surface Science Instruments SSX-100
Excitation Source: Al K_α 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



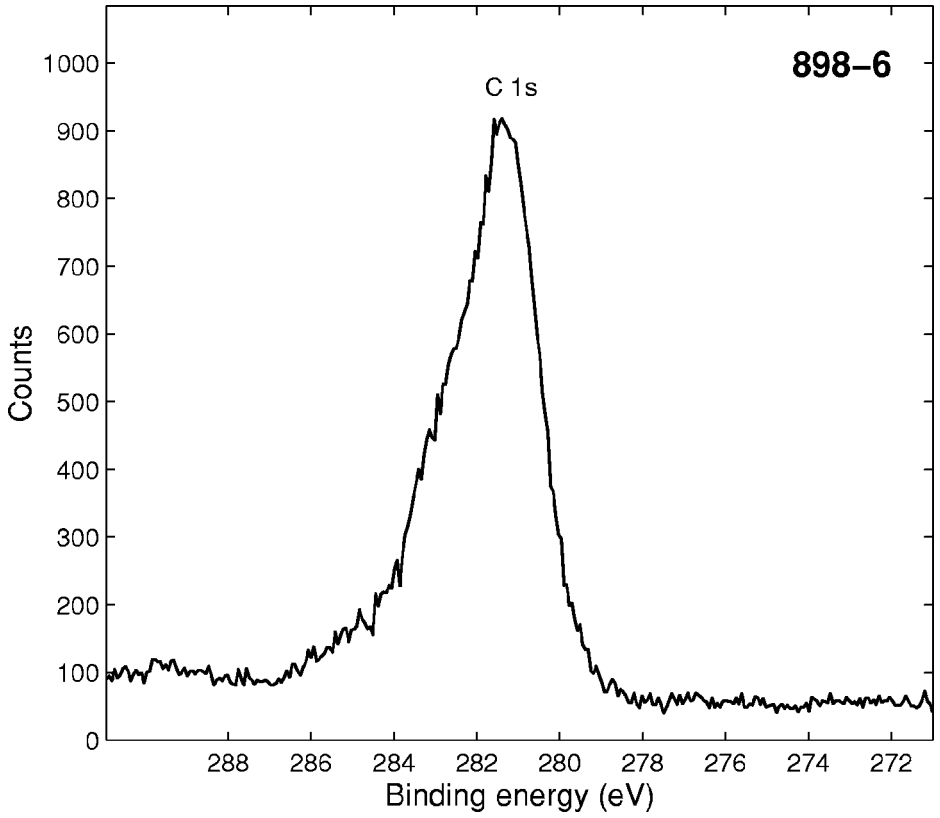
- **Accession #:** 00898-04
- **Host Material:** shea nut shells
- **Technique:** XPS
- **Spectral Region:** N 1*s*

Instrument: Surface Science Instruments SSX-100
Excitation Source: Al K_α 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: 3065 s
Total Elapsed Time: 3234 s
Number of Scans: 50
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



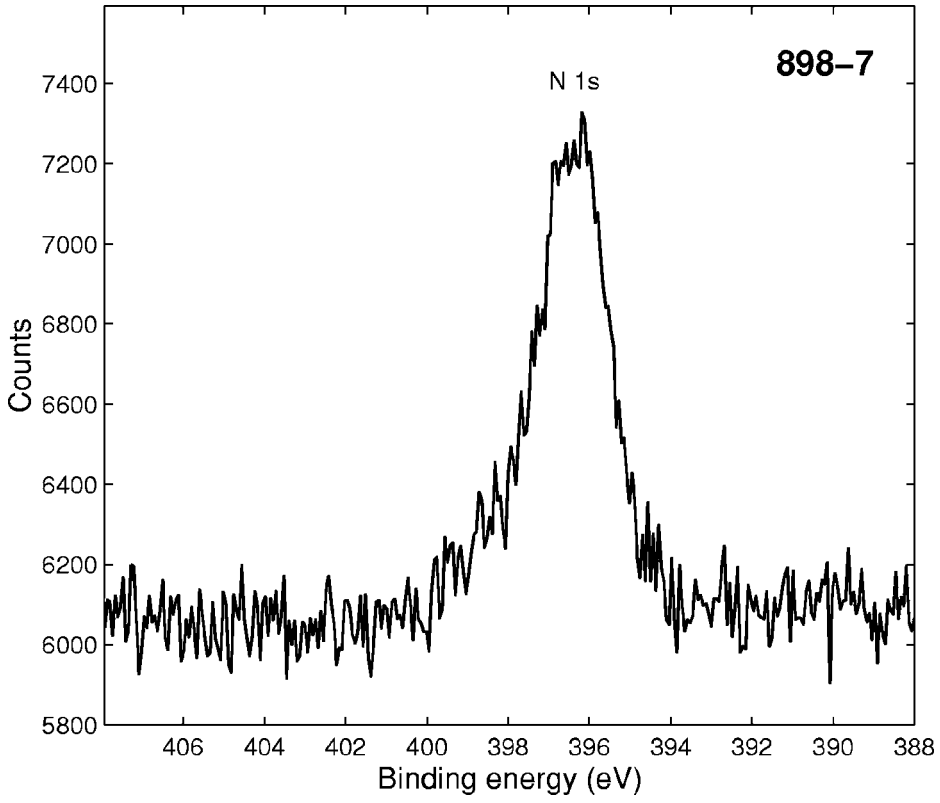
- **Accession #:** 00898-05
- **Host Material:** shea nut shells
- **Technique:** XPS
- **Spectral Region:** O 1*s*

Instrument: Surface Science Instruments SSX-100
Excitation Source: Al K_α monochromatic
Source Energy: 1486.6 eV
Source Strength: 200 W
Source Size: 0.3 mm × 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 × 0.523 mm
Effective Detector Width: 6.6 eV
Analyzer Width: 750 μm × 6000 μm at 84 eV



- **Accession #:** 00898-06
- **Host Material:** shea nut shells
- **Technique:** XPS
- **Spectral Region:** C 1*s*

Instrument: Surface Science Instruments SSX-100
Excitation Source: Al K_α monochromatic
Source Energy: 1486.6 eV
Source Strength: 200 W
Source Size: 0.3 mm × 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 × 0.523 mm
Effective Detector Width: 6.6 eV
Analyzer Width: 750 μm × 6000 μm at 84 eV



- **Accession #:** 00898-07
- **Host Material:** shea nut shells
- **Technique:** XPS
- **Spectral Region:** N 1*s*

Instrument: Surface Science Instruments SSX-100
Excitation Source: Al K_α monochromatic
Source Energy: 1486.6 eV
Source Strength: 200 W
Source Size: 0.3 mm × 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 × 0.523 mm
Effective Detector Width: 6.6 eV
Analyzer Width: 750 μm × 6000 μm at 84 eV