Application Note #000388

KeyWords
XPS, Food, Natural Products, Biological Material, Measurements, Surface Analysis, Charge Compensation

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XPS surface analysis of a coffee bean with EnviroESCA

This application note presents how EnviroESCA can be used to analyze the surface of untreated food samples, e.g., a roasted coffee bean. Charge compensation of this insulating natural product is accomplished by Environmental Charge Compensation enabling X-ray photoelectron spectroscopy on such biological material with ease.

Motivation

The analysis of food and natural products under environmental conditions is of great importance due to their daily use and direct interaction with humans during consumption. From an industrial perspective a detailed analysis of the food's surface during production is of enormous interest, especially when the food comes in contact with potentially harmful substances, e.g., packaging material, oils, or silicones.

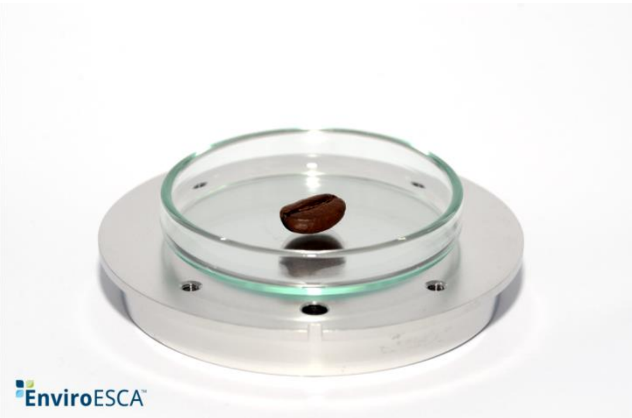

Fig. 1 Roasted coffee bean in a petri dish on an EnviroESCA sample plate

Method

EnviroESCA utilizes X-ray Photoelectron Spectroscopy (XPS) as its main analytical technique. Hereby an electron beam is generated inside the X-ray source and focused onto an X-ray anode made of aluminum. The deceleration of the electrons on the anode leads to the production of X-rays. This X-ray beam is monochromated and focused onto the sample.

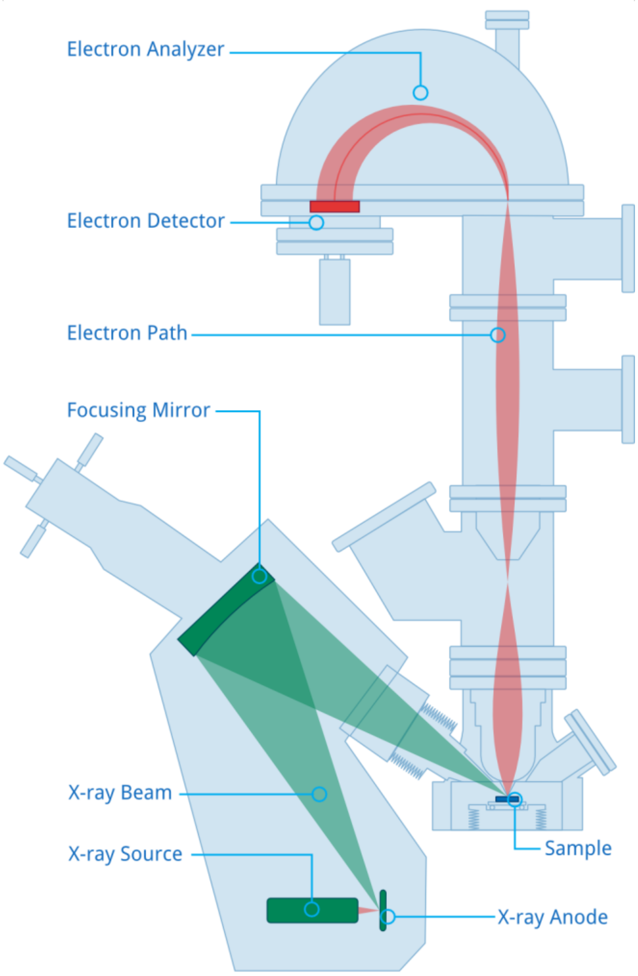

Fig. 2 XPS with EnviroESCA

X-ray photons impinging the sample excite electrons in the material which are subsequently emitted with specific kinetic energy determined by their binding energy and the photon energy of the x-rays. Thereby only

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electrons from atoms up to a depth of approx. 10nm are able to leave the surface. These electrons propagate through the lens system of the Electron Analyzer into the hemisphere which acts as a spherical capacitor forcing the electrons onto circular paths with radii depending on their kinetic energy. The electron paths end at an electron sensitive detector where the electrons are amplified and measured as an intensity in counts / second. Sweeping the voltage of the spherical capacitor while measuring the number of electrons per second on the detector results in a photoelectron spectrum. From these spectra a quantitative analysis of the atomic composition of the sample surface can be done.

Experimental Section

Roasted coffee beans exhibit caramelization and condensation products, polysaccharides, fats (oils), and proteins as main ingredients. The presence of oils, flavor and other volatile compounds makes it difficult to investigate such natural product in classical XPS systems working in (ultra-)high vacuum conditions. The volatiles will outgas during pump down cycles and will desorb continuously from the surface of the specimen.

EnviroESCA can work in pressures up to several dozens of mbar and therefore does not necessarily require vacuum conditions which overcome the problem of outgassing of almost all samples.

In classical XPS systems non-conducting (bio) organic materials tend to quickly charge up under X-ray illumination which makes charge compensation inevitable. In classical XPS low energy electron and ion sources are being used in addition to the X-ray source to compensate the surface charge of the surface.

In EnviroESCA an intrinsic charge compensation method which we call Environmental Charge Compensation makes additional electron or ion sources unnecessary.

The gas atmosphere that is surrounding the sample delivers all the free charges, when illuminated with the soft X-rays, that is needed to compensate for surface charging (see figure 3 for an illustration).

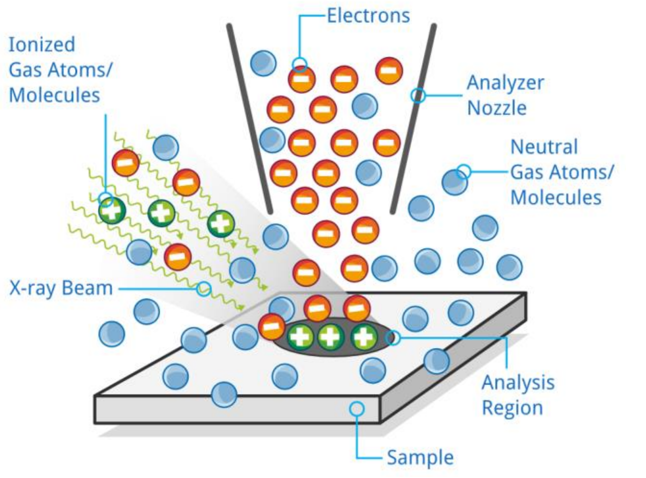

Fig. 3 Environmental Charge Compensation

In this study the surface of a roasted coffee was investigated with the EnviroESCA directly out of the package without any further pre-treatments.

Because of oils, volatile (flavor) compounds, and charge up of the surface in vacuum a working pressure of 1 mbar ambient air was chosen for this study.

Results

In the following we are presenting unmodified raw data taken with EnviroESCA. The data was not smoothened or shifted on the energy scale unless otherwise mentioned.

Fig. 4 Camera view onto the analysis area

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A single roasted coffee bean was selected, put in a petri dish, and placed on the sample plate. No additional masking or electrical contacting was performed.

First of all a survey scan was acquired in less than three minutes after starting the pump down of the Sample Environment to 1 mbar. Figure 5 displays the result of that scan which was measured with a total acquisition time of 9 minutes (5 Scans, 1 eV step width).

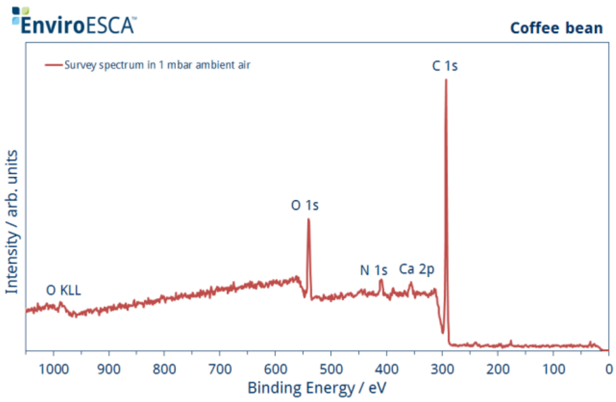

Fig. 5 XPS survey spectrum of a roasted coffee bean

Taking a closer look gives a peak position of 540.2 eV of the O 1s. Which means the spectrum is shifted about 7 eV to higher binding energies when comparing the measured O 1s position to the literature value of polysaccharides [1].

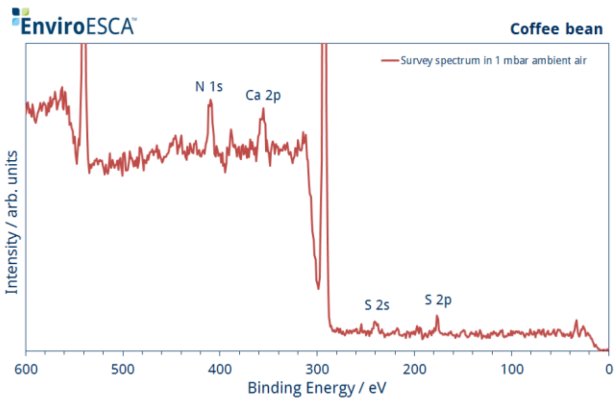

Fig. 6 Enlarged XPS survey spectrum in the energy range 0-600 eV

Zooming into the spectrum clearly shows the presence of Sulphur (2s and 2p) that originates most probably from typical coffee flavor compounds, e.g. 2-Furfurylthiol (see figure 6).

Most of these flavor compounds are formed during the roasting of coffee beans due to very complex chemical reactions, the so-called *Maillard* reaction, between reducing sugars and amino acids (from proteins).

Figure 7 displays a detail spectrum of the Carbon 1s region with the C 1s peak maximum located around 293 eV. According to a binding energy shift of about 7 eV this would correlate typically with carbon atoms bound to oxygen by a single bond (*C*-O, ~ 286.5 eV) originating, e.g., from polysaccharides.

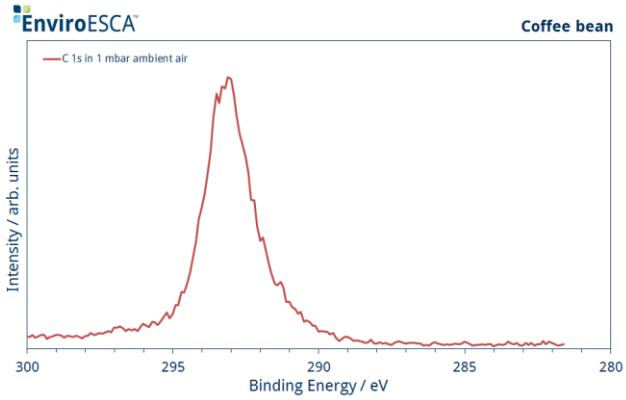

Fig. 7 High resolution spectrum of the C 1s region (recorded with a step width of 0.1 eV)

Conclusion

EnviroESCA has proven to be a powerful tool to investigate with XPS the surface of biomaterials and especially food samples. Because of the intrinsic charge compensation of the gas environment high resolution and high quality spectra are recordable. Outgassing of volatiles from the sample is no problem for EnviroESCA. And the Environmental Charge Compensation is working properly using ambient air at a pressure of 1 mbar.

[1] Beamson, G.; Briggs, D. High Resolution XPS of Organic Polymers; Wiley: Chichester, UK, 1992

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Technical Specifications

EnviroESCA	
Electron Spectrometer	▪ Hemispherical electron analyzer with 150 mm mean radius ▪ Differentially pumped lens system ▪ Delayline detector with up to 400 channels
X-ray Source	▪ Al K_α micro-focused monochromator ▪ Rowland circle diameter of 600 mm ▪ Spot sizes of 200 µm-1 mm optimized to analysis area
Charge Neutralization	▪ System immanent charge compensation by X-ray photoionization
Ion Source (optional)	▪ Scannable small spot ion source (200 eV-5 keV) or gas cluster ion source
Pumping System	▪ Turbomolecular pumps ▪ Oil-free backing pumps
Pressure Range	▪ Defined by analyzer aperture (up to 100 mbar with an aperture of 300 µm; other aperture sizes on request)
Gas Dosing	▪ Two separated mass flow controlled gas dosers at analysis position
Cameras	▪ 3 digital microscopes for sample navigation and documentation
Automation and Software	▪ Fully automated vacuum and gas dosing system ▪ Advanced software package

SampleEnvironment (standard, others on request)	
Samples Stage	▪ High precision 3-axis stage
Sample Size	▪ Up to 120 mm in diameter and 40 mm in height ▪ 50 mm inner diameter addressable
Gas Dosing	▪ Mass flow controlled process and purge gas
Cleaning	▪ Downstream RF plasma cleaner
Camera	▪ Digital microscope for sample observation and documentation

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