Application Note #000390

KeyWords
XPS, Food, Sesame Seed, Natural Products, Measurements, Surface Analysis, Charge Compensation

XPS surface analysis of sesame seeds with EnviroESCA

Results of the surface analysis of untreated food samples, e.g., sesame seeds, measured with EnviroESCA are presented. Neutralization of this insulating natural product is accomplished by Environmental Charge Compensation enabling X-ray photoelectron spectroscopy on 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 surfaces during production is of enormous interest, especially when the goods comes in contact with potentially harmful substances, e.g., packaging material, oils, or silicones.

Fig. 1 Untreated, organic sesame seeds in a watch glass 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.

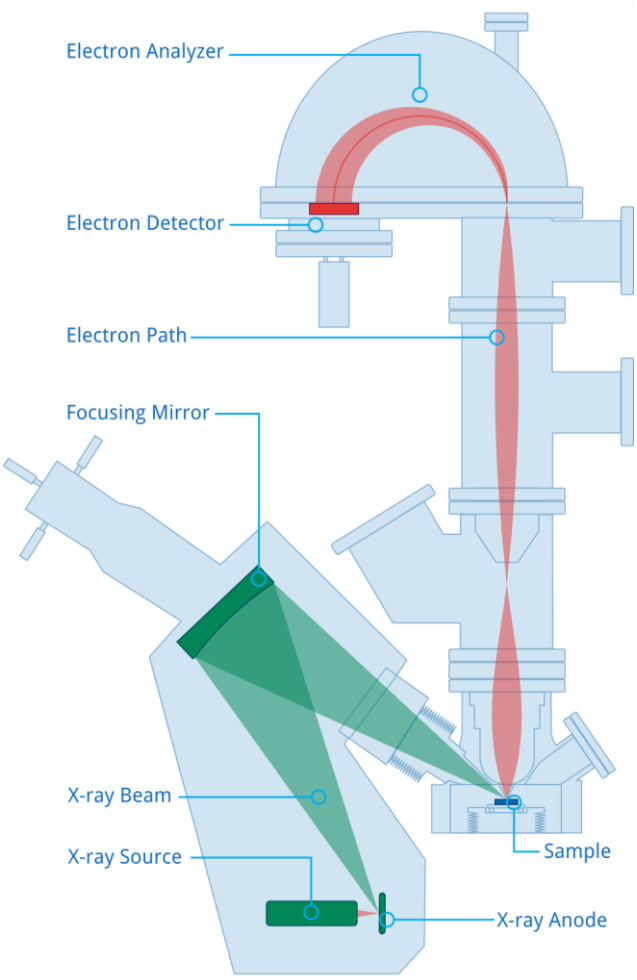

Fig. 2 XPS with EnviroESCA

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 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

Sesame seeds contain oil, sugars, polyphenols, minerals, and proteins as main ingredients. The presence of oil (fatty acids) 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. This enables the use samples in bulk quantities poured in petri dishes or watch glasses.

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 (cf. fig. 3 for an illustration).

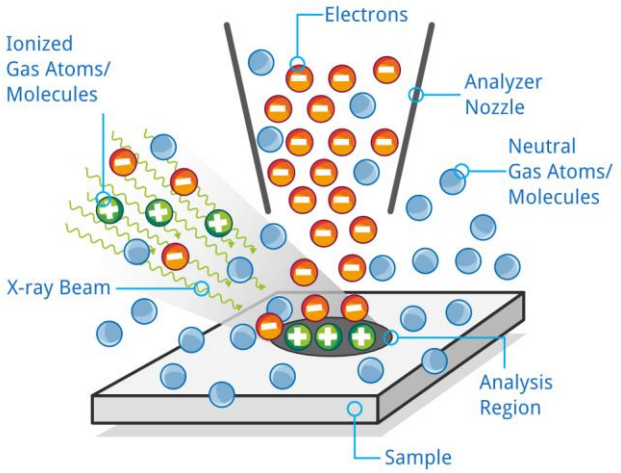

Fig. 3 Environmental Charge Compensation

In this study the surface of three different organic sesame seeds were investigated with the EnviroESCA. The sesame seeds from a local organic grocery store were used directly without any further pre-treatments.

Because of the volatile compounds and potential surface charging in vacuum a working pressure of 1 mbar (ambient air/nitrogen mix) 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 of sesame sample 1

Several sesame seeds were placed in a watch glass on the sample plate, see Fig. 4. From that stock three different seed samples were selected and analyzed without any additional treatment.

First of all survey scans were acquired on each sample in less than three minutes after starting the pump down of the Sample Environment to 1 mbar. Figure 5 displays the survey scan taken on seed sample 1. The main elements found are carbon and oxygen from the sesame seed and nitrogen from residual venting gas.

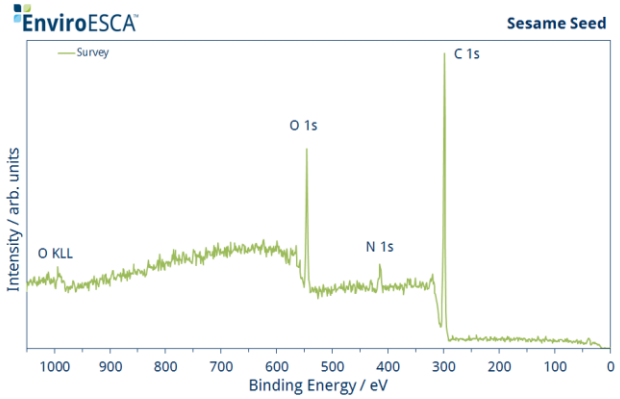

Fig. 5 XPS survey spectrum of sesame sample 1

Taking a closer look reveals a O 1s peak position of 546 eV which means the spectrum is shifted by about 13 eV to higher binding energies when comparing the measured value to literature value of oxygen in natural occurring sugars (polysaccharides), e.g., cellulose.[1]

Compared to sesame seed sample 1 additionally calcium was identified in samples 2 and 3 (cf. Fig. 6 and7).

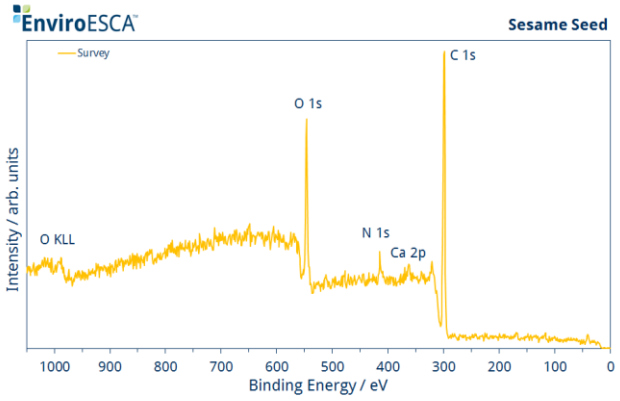

Fig. 6 XPS survey spectrum of sesame sample 2

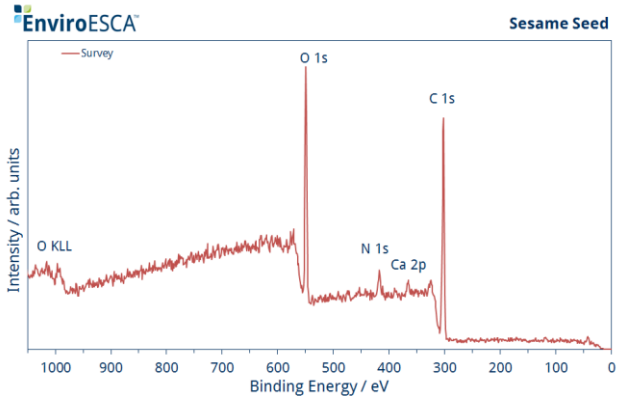

Fig. 7 XPS survey spectrum of sesame sample 3

Figure 8 shows a spectrum of the C 1s core-level region of sample 1. The energy scale was corrected by assigning the low binding energy C 1s peak component to a value of 285 eV. The main component C-2 (286.7 eV) originates from singly oxygen-bound carbon atoms (C-O) located, e.g., in polysaccharides as cellulose.[1]

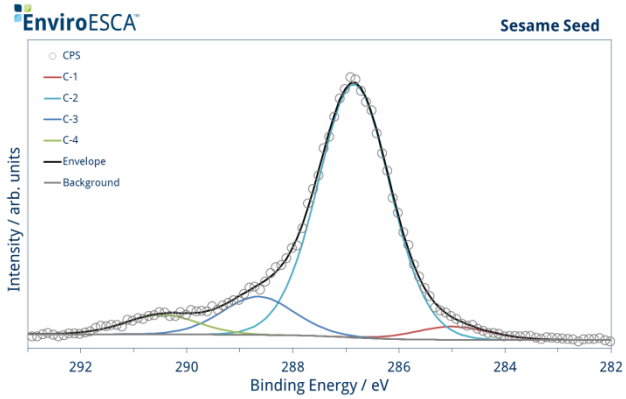

Fig. 8 High-resolution C 1s XP spectrum of sesame sample 1 measured at 1 mbar. Open circles represent experimental data and the black line shows the fitted curve. Colored lines correspond to: C-C (C-1), C-O (C-2), O-C-O / C=O (C-3), and COO (C-4) carbon moieties.

Conclusion

EnviroESCA has proven to be a powerful tool to investigate the surface of food samples even in bulk quantities with XPS. High resolution and high quality spectra are recordable with ease using the Environmental Charge Compensation. For EnviroESCA outgassing of volatiles from the sample is no problem under near ambient pressure conditions, here at 1 mbar.

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