Application Note #000392

KeyWords
XPS, Food, Dairy Products, Measurements, Surface Analysis, Charge Compensation

EnviroESCA™

XPS surface analysis of Italian hard cheese with EnviroESCA

This application note presents how EnviroESCA can be used to analyze the surface of food samples, e.g., Italian hard cheese. Charge compensation of this insulating natural product is accomplished by Environmental Charge Compensation enabling X-ray photoelectron spectroscopy on such 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.

Fig. 1 Italian hard cheese that was used a sample in this analysis

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. 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.

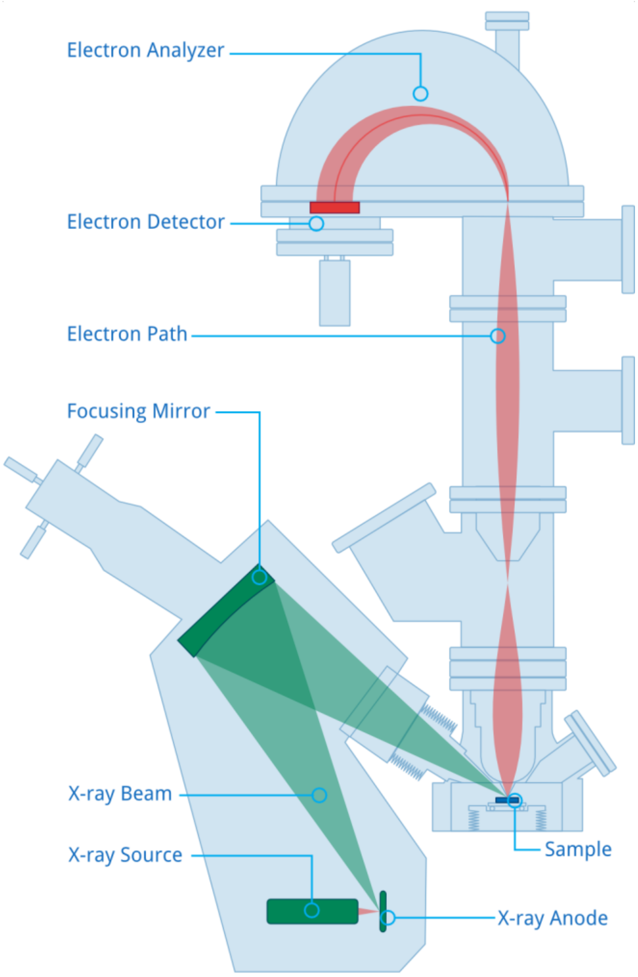

Fig. 2 XPS with EnviroESCA

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

The presence of fat, oil, flavor, and other volatile compounds makes it difficult to investigate dairy food 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 as illustrated in Fig. 3.

In this study the surface of an organic produced hard cheese from Italy was investigated without any further pre-treatments with the EnviroESCA.

Because of fat/oil, volatile compounds, and charge up of the surface in vacuum a working pressure of 1 mbar was chosen for this study.

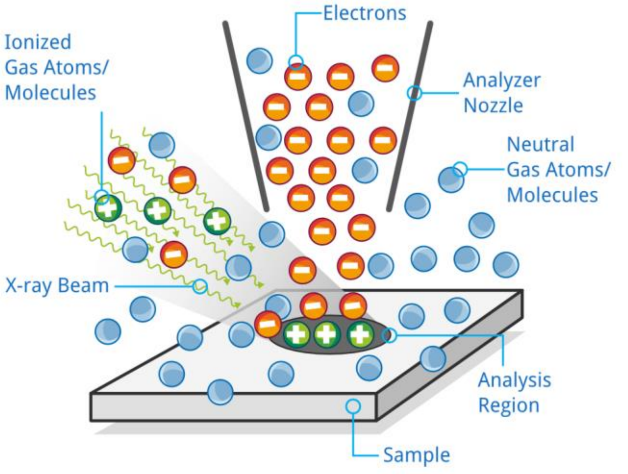

Fig. 3 Environmental Charge Compensation

Results

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

A thin piece was cut from a bigger part of an Italian hard cheese (cf. Fig. 1), put in a petri dish, and placed on the sample plate. No additional masking or electrical contacting was performed.

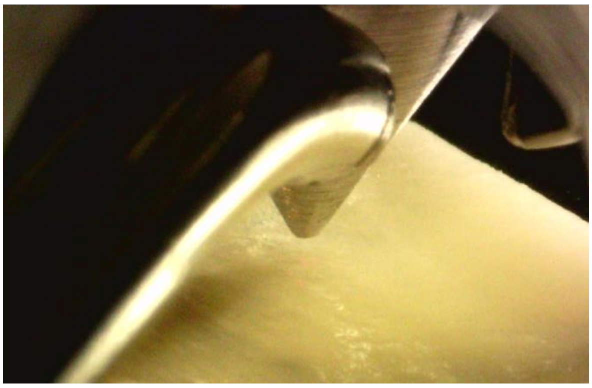

Fig. 4 Camera view on the analyzed area of a piece of hard cheese.

First of all a wide scan (2 Scans, 1 eV step width) was acquired in less than three minutes after starting the pump down of the Sample Environment to 1 mbar. Figure 5 displays the resulting survey spectrum showing oxygen and carbon as main elements together with a nitrogen contribution originating from a mixture of ambient air and pure nitrogen gas used for venting.

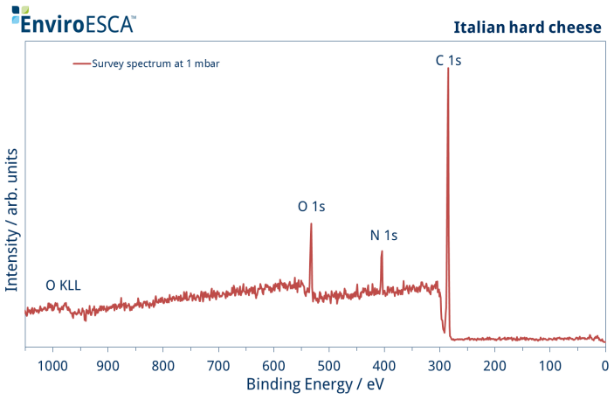

Fig. 5 XPS survey spectrum of a piece of Italian hard cheese

An overall shift of the spectrum by about 1 eV to higher binding energies can be assumed when comparing the measured C 1s and O 1s peak positions of 285.9 eV and 533.8 eV to reported literature values.[1]

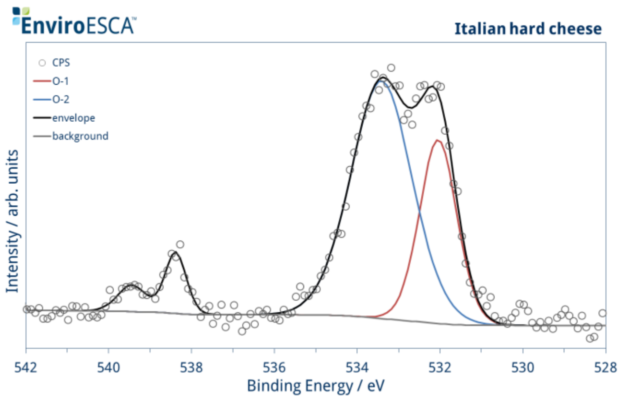

Fig. 6 High-resolution O 1s XP spectrum of a piece of Italian hard cheese measured at 1 mbar. Open circles represent experimental data and the black line shows the fitted curve. Colored lines correspond to different oxygen peak components O-1 and O-2.

Further insights into the surface chemistry could be provided by detail scans of the O 1s and C 1s regions. The O 1s core-level spectrum exhibits two major peak components O-1and O-2 located at 532.0 eV and 533.4 eV, respectively (cf. Fig. 6). Amongst others those components can be assigned to oxygen atoms in *O*=C (O-1) and *O*–C (O-2) moieties from the main cheese components fat and carbohydrates.[1] The minor feature at 538 eV originates from oxygen of the residual ambient air.

Figure 7 displays a detail spectrum of the carbon 1s region measured on the thin piece of hard cheese. The spectrum was shifted by 1.2 eV to locate the main C 1s peak component at 285.0 eV.

The quantitative surface composition is unknown so far but the cheese's C 1s score-level spectrum reveals typical triacylglyceride components as hydrocarbons (*C*C/*C*H, 285.0 eV), *C*-C=O at 285.6 eV, carbon atoms bound to oxygen by a single bond (*C*-O, 286.4 eV) as well as carbon atoms originating from carbonyl/acetal(*C*=O, O-*C*-O) and carboxyl (*C*OO) moieties located at 287.2 eV and 289.4 eV, respectively.

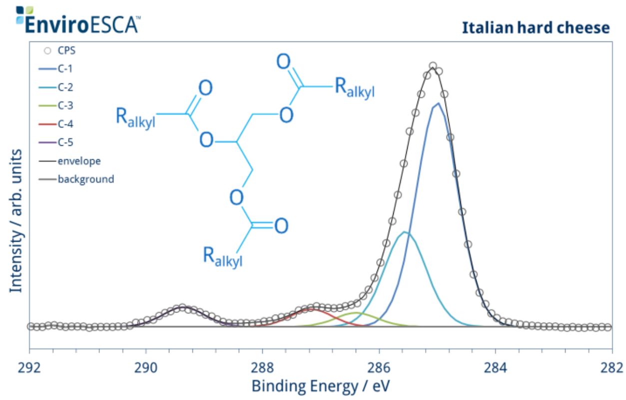

Fig. 7 High-resolution C 1s XP spectra of a piece of Italian hard cheese measured at 1 mbar. Open circles represent experimental data and the black line shows the fitted curve. Colored lines correspond to carbon atoms located in *C*C/*C*H, *C*-C=O, *C*-O, O-*C*-O / *C*=O, and *C*OO moieties. The inset shows a general structure of a triacylglyceride (fat).

Conclusion

EnviroESCA has proven to be a powerful tool to investigate with XPS the surface of sensitive food samples. Because of the intrinsic charge compensation of the gas environment high resolution and high quality spectra are recordable. Even the surface analysis in the presence of such components as fat and oils or volatiles is no problem for EnviroESCA. Moreover, the Environmental Charge Compensation is enabling XPS of insulating materials at near ambient pressures (p ≥1 mbar).

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