Application Note #000383

KeyWords
XPS, Medical and Biomaterials, Hydrogel,
Contact Lens, Measurements, Quality Control

Investigation of Hydrogel Contact Lenses with EnviroESCA

This application note presents how EnviroESCA can be used to analyze the surface of water filled hydrogels, such as contact lenses like in the present study as an example for medical and biomaterials. The advantage of such investigations under non ultrahigh vacuum conditions is demonstrated and results from wet and dry samples are compared.

Motivation

Hydrogels are being widely used in medical and biomaterial products. For example they find application as contact lens material, for pharmaceuticals in drug delivery applications or implants in cosmetic surgery. As the surface of these gels gets in contact and interacts with the human body it is essential to understand the surface chemistry and the surface interaction in different environments.

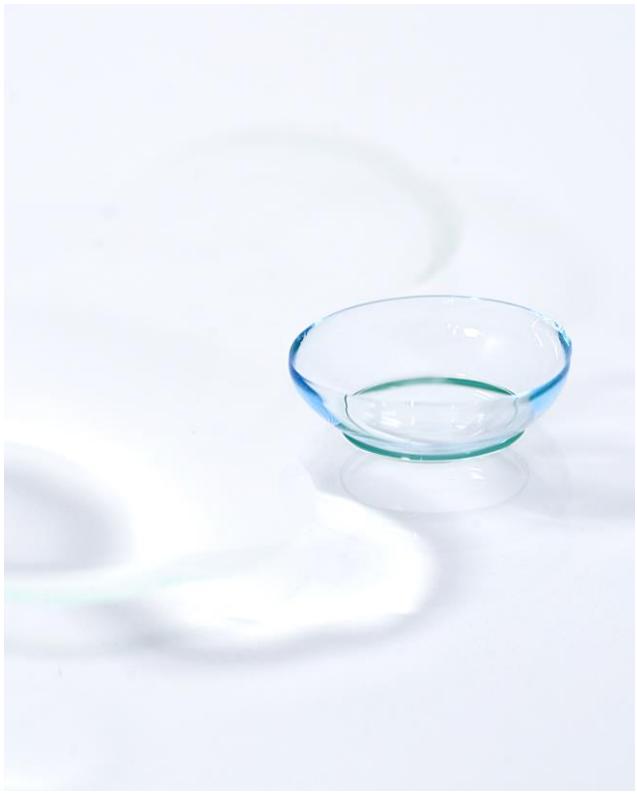

Fig. 1 A hydrogel based contact lens used in this study

Method

EnviroESCA utilizes X-ray Photoelectron Spectroscopy (XPS) as its main analytical technique.

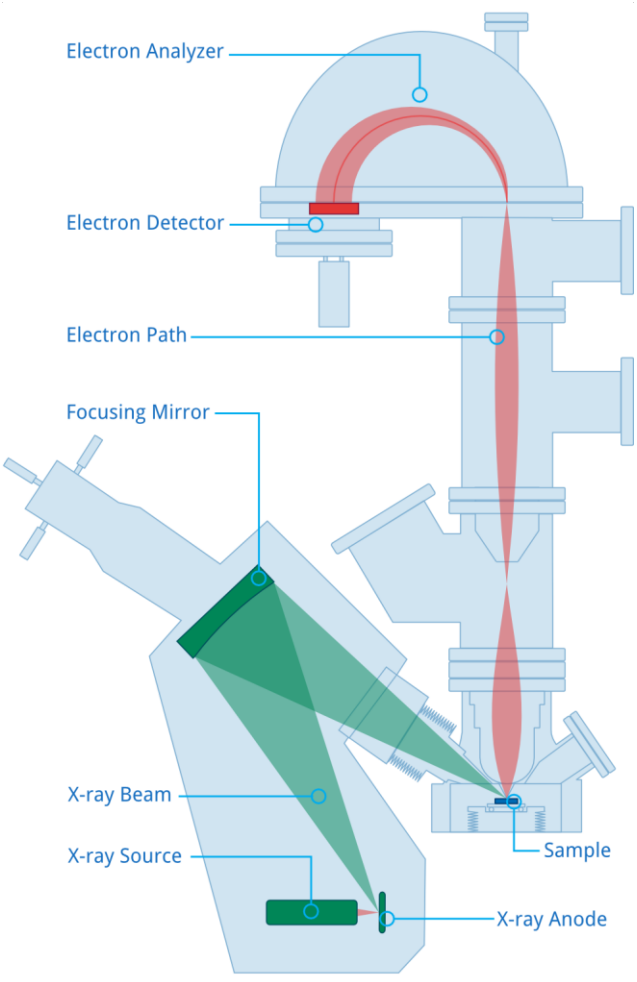

Fig. 2 XPS with EnviroESCA

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. 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 determined.

Experimental Section

Hydrogels are highly adsorbent and can contain a very high amount of water (up to more than 90%). This large amount of water normally does not allow hydrogel products to be analyzed with surface sensitive methods that require ultra-high vacuum (UHV) conditions.

For the analysis of hydrogel based contact lenses in classical XPS systems the lenses need to be dehydrated before being introduced into the analysis system. Several methods for the dehydration are common including drying in air, in nitrogen or freezing with subsequent freeze-drying [1]. Also the dried lenses tend to charge up when illuminated with soft X-rays during the analysis, therefore in classical XPS systems low energy electron and ion sources are being used to circumvent the charging effects, like a shift of the binding energy and a broadening of the peak distribution.

With EnviroESCA investigations of wet hydrogel samples is possible and easy to perform. EnviroESCA can work in pressures up to several dozens of mbar, also in water atmosphere, which makes dehydration of the lenses unnecessary. An intrinsic charge compensation method which we call Environmental Charge Compensation makes additional electron or ion sources for charge compensation like being used in classical XPS systems 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).

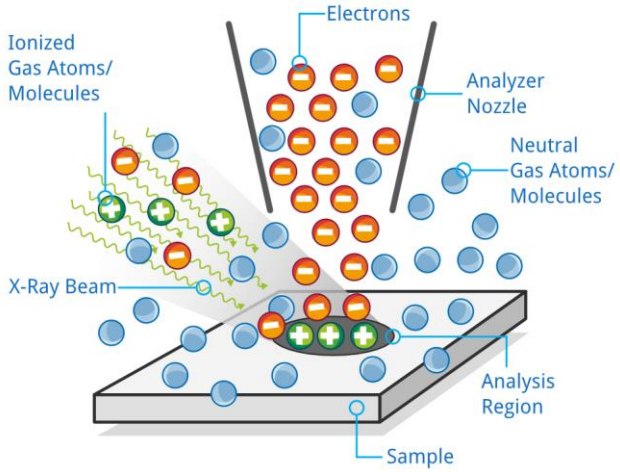

Fig. 3 Environmental Charge Compensation

In this study EnviroESCA was used for surface investigation of two hydrogel based contact lenses taken from the same batch.

The first lens was pre-dried on filter paper in ambient air over 8 hours before being loaded in EnviroESCA.

The second lens was taken directly from its sale packaging. The edge of the second lens was carefully touched with a filter paper to remove additional liquids from its surface. Because a water filled hydrogel is very flexible a special sample plate was used which resembled the curvature of the lens. The time frame between removing the second lens from the sales packaging and

measuring of the first XPS spectrum was less than 5 min.

Because the lenses would charge up in vacuum conditions a working pressure of 1 mbar was chosen.

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.

1. Hydrogel contact lens dehydrated by drying in ambient air

The dehydrated sample was fixed with carbon tape to the EnviroESCA sample holder. Figure 4 shows a photo taken with one of the three digital microscopes at the analysis compartment. Please note that the lens deformation is due to the ex-situ dehydration process.

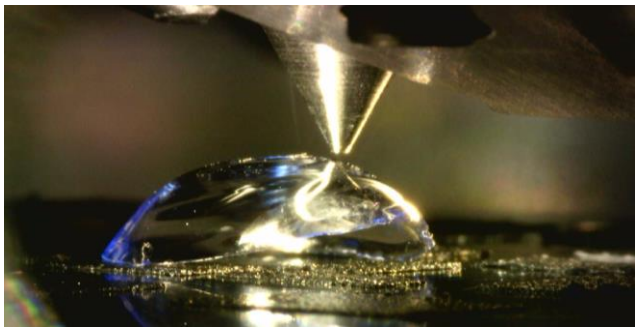

Fig. 4 Dehydrated contact lens in front of the analyzer nozzle

Figure 5 shows a survey scan taken with EnviroESCA right after pump down of the SampleEnvironment to a pressure of 1 mbar.
The survey clearly shows peaks of the Auger and the 1s core level peaks of Oxygen together with the 1s peaks of Nitrogen and Carbon. The peak position of the C 1s in this survey is 288.6 eV.

A detail spectrum taken of the Oxygen is shown in figure 6.

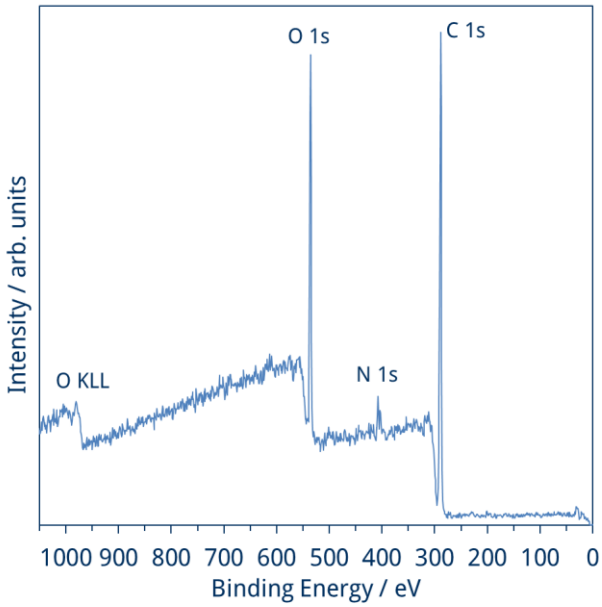

Fig. 5 Survey spectrum of the dehydrated contact lens

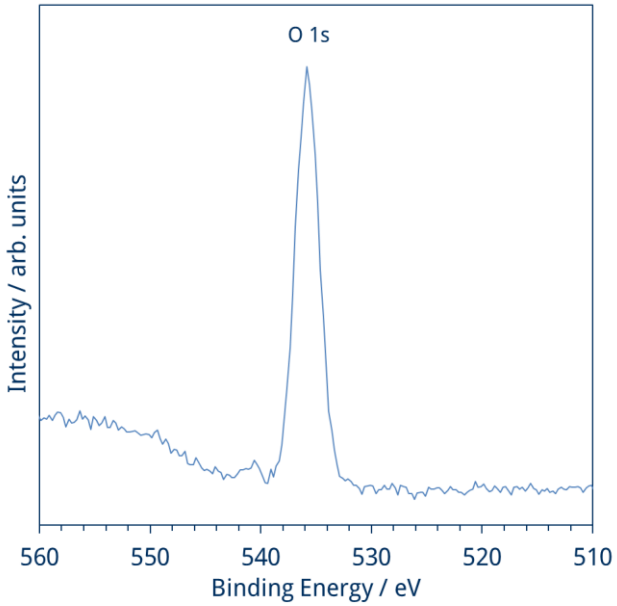

Fig. 6 A detail spectrum of the O 1s region of the dehydrated contact lens

A high resolution scan of the Carbon 1s reveals three different components. Each component results from electrons escaping Carbon atoms that have different binding configurations with their nearest neighbors in the molecule. The spectrum shown in figure 7 was

recorded with a step width of 0.1 eV in 4 minutes and 18 seconds.

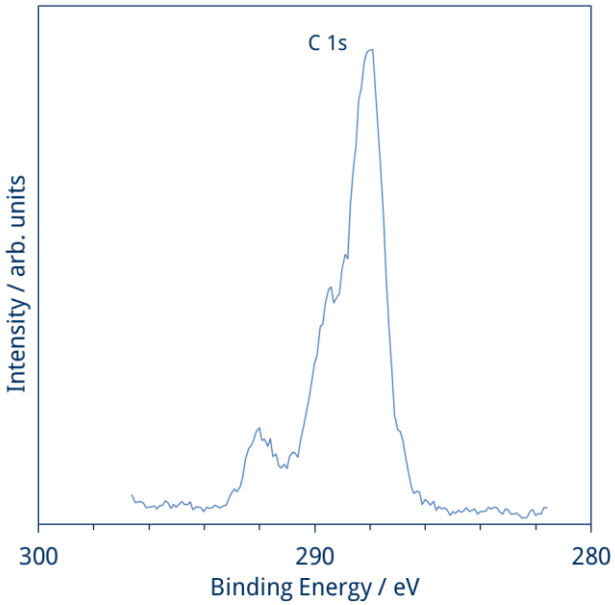

Fig. 7 The C 1s region of the dehydrated contact lens in higher resolution

2. Hydrogel contact lens taken directly from its sales packaging

The survey spectrum taken on the “wet” contact lens is shown in figure 8.

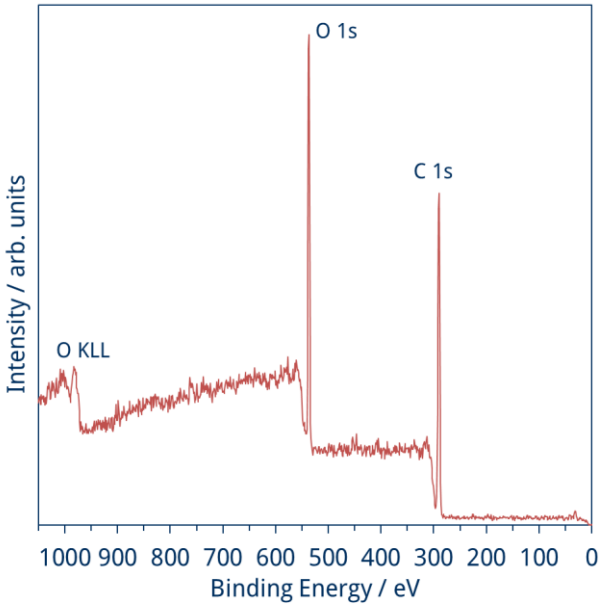

Fig. 8 Survey spectrum of the "wet“ contact lens

A detail scan of the Carbon 1s region also reveals three components but with different ratios compared to the one taken on the dehydrated lens.

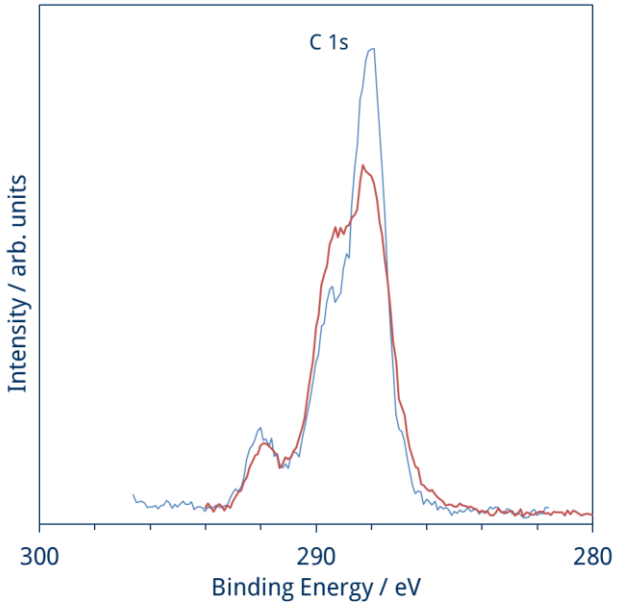

Fig. 9 The C 1s region of the wet (red) lens and the dehydrated lens (blue). Please note the red spectrum was shifted by -2.6 eV for comparison)

Conclusion

The results shown in this application note clearly demonstrate that EnviroESCA is capable of performing XPS even on water filled hydrogels without the need to dehydrate the sample before introducing them into the analysis system.

Furthermore, the comparison of the C 1s region of both contact lenses reveals clear differences in the ratio of the components in the Carbon peak. The study of the "wet" contact lens resembles the conditions of the real application of hydrogels much better and can provide valuable insights.

[1] C. C.S. Karlgard et al., Applied Surface Science 230 (2004) 106-114