Near Ambient Pressure X-ray Photoelectron Spectroscopy (NAP-XPS). Characterization of Non-Traditional Materials with the SPECS EnviroESCA Instrument

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Near ambient pressure X-ray photoelectron spectroscopy (NAP-XPS) is an advanced version of traditional XPS. NAP-XPS works at relatively high pressures, which allows many types of samples to be analyzed. Here we show NAP-XPS analyses of various non-traditional materials and describe advantages and unique features of the technique.

NAP-XPS (see Fig. 1) has the following advantages and features. [1]

1. High quality data are obtained at relatively high working pressures. For example, Fig. 2 shows high quality survey [2] and 2p narrow scans from Ar gas [3]. In general, higher working pressures result into higher backgrounds due to increased inelastic scattering of photoelectrons.

2. NAP-XPS allows analysis of gases, liquids, and solids, including biological and food samples that outgas. For example, Fig. 3 shows survey spectra of a hard Italian cheese [4] and a human tooth [5].

3. NAP-XPS allows very fast sample introduction and pump down due to its higher working pressure. Additionally, it does not require a load lock because samples are placed directly into the analytical chamber. These qualities make NAP-XPS a high throughput analysis technique [6].

4. NAP-XPS does not require external charge compensation. This intrinsic charge compensation is a result of X-ray induced ionization of gas molecules present in the chamber [6].

5. Real time monitoring of samples is possible with NAP-XPS. For example, Fig. 4 shows C 1s narrow scans of Coca-Cola as a function of time under vacuum [7]. Here, we observe an increase in oxidized carbon as a function of time because of evaporation of water from the sample, which increases the concentration of sugar in it.

6. NAP-XPS collects signals from a liquid or solid sample and the gas surrounding it. For example, Fig. 4 shows the O 1s narrow scan of Coca-Cola [7], which includes signals from both the liquid sample and water vapor.

7. The SPECS EnviroESCA instrument collects and records every scan it acquires. A retrospective analysis of this type of data can reveal sample damage. For example, Fig. 5 shows the C 1s narrow scans of poly L-lactic acid (PLLA) before and after 1 h of X-ray exposure [8]. We observed significant...
sample damage here.

Fig. 2. NAP-XPS analyses: (left) Ar survey spectrum, (right) Ar 2p narrow scan.

Fig. 3. NAP-XPS analyses: (left) a survey spectrum of a hard Italian cheese, (right) a survey spectrum of a human tooth.

Fig. 4. NAP-XPS analyses: (left) C 1s narrow scans obtained in real time of a sample of Coca-Cola as the water in it evaporates, (right) O 1s narrow scan of Coca-Cola showing a narrower, higher energy peak from gas phase water. Peak fitting here was with Gaussian-Lorentzian sum (SGL) functions. [9]

Fig. 5. NAP-XPS analyses: (left) a C 1s narrow scan of PLLA polymer at a fresh spot on the sample (right), a C 1s narrow scan of PLLA after 1 h of X-ray exposure. The fit on the left here was with SGL synthetic peaks, and a uniqueness plot [10] was used to confirm the absence of fit parameter correlation.

References