Extended Abstract of PSA-19 (review)
O-36

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

Dhananjay I. Patel,[1] Paul Dietrich,[2] Andreas Thißen,[2] and Matthew R. Linford[1*]
[1]*Brigham Young University, Provo, UT, USA.*
[2]*SPECS Surface Nano Analysis GmbH, Berlin, Germany.*
*Corresponding author's email: mrlinford@chem.byu.edu

(Received: June 6, 2019; Accepted: July 9, 2019)

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].

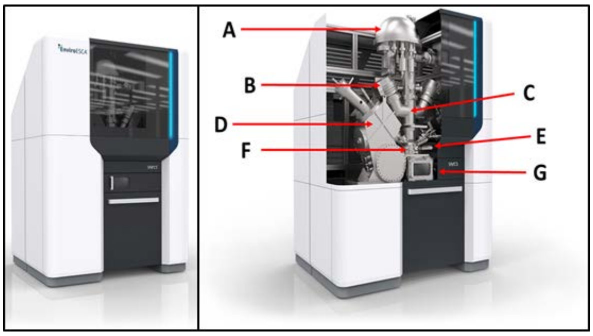

Fig. 1. The EnviroESCA NAP-XPS system: (left) exterior and (right) interior showing the hemispherical analyzer (A), a turbo pump for differential pumping (B), the analysis column with multiple pumping stages (C), the X-ray generation system (D), the analysis chamber (F), digital microscopes (E), and the sample environment/loading port (G).

(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.

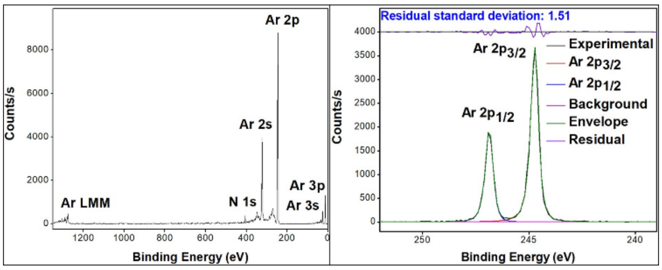

Fig. 2. NAP-XPS analyses: (*left*) Ar survey spectrum, (*right*) Ar 2p narrow scan.

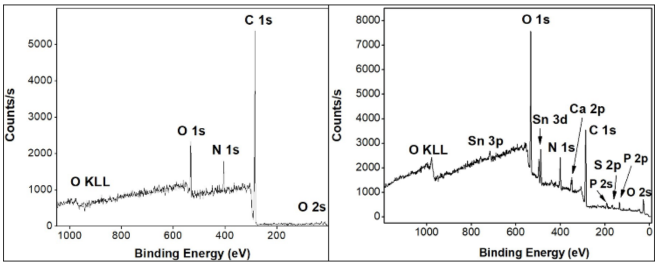

Fig. 3. NAP-XPS analyses: (*left*) a survey spectrum of a hard Italian cheese, (*right*) a survey spectrum of a human tooth

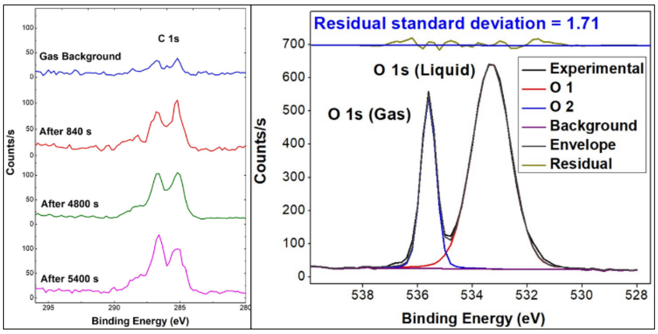

Fig. 4. NAP-XPS analyses: (*left*) C 1s narrow scans obtained in real time of a sample of Coca-Cola as the water in it evaporates, (*right*) O 1s narrow scan of Coca-Cola showing a narrower, higher energy peak from gas phase water. Peak fitting here was with Gaussian-Lorentzian sum (SGL) functions. [9]

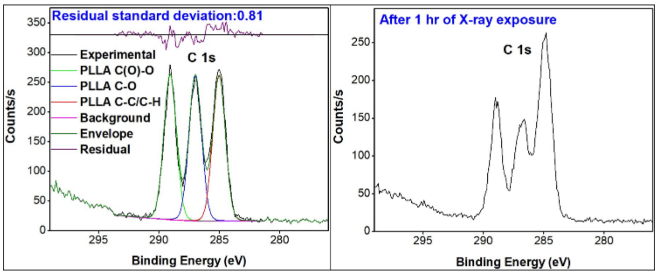

Fig. 5. NAP-XPS analyses: (*left*) a C 1s narrow scan of PLLA polymer at a fresh spot on the sample (*right*), a C 1s narrow scan of PLLA after 1 h of X-ray exposure. The fit on the left here was with SGL synthetic peaks, and a uniqueness plot [10] was used to confirm the absence of fit parameter correlation.

References

[1] M. R. Linford, T. Roychowdhury, D. Shah, *Vac. Tech. Coatings* **August**, 26 (2018).

[2] D. Shah, D. I. Patel, T. Roychowdhury, G. B. Rayner, N. O'Toole, D. R. Baer, and M. R. Linford, Journal of Vacuum Science & Technology B **36**, 062902 (2018).

[3] D. I. Patel, S. Bahr, P. Dietrich, M. Meyer, A. Thißen, M. R. Linford, Argon, by Near-Ambient Pressure X-ray Photoelectron Spectroscopy (NAP-XPS). *Surf. Sci. Spectra* (2019), Submitted.

[4] T. Roychowdhury, S. Bahr, P. Dietrich, M. Meyer, A. Thißen, and M. R. Linford, *Surf. Sci. Spectra* **26**, 014015 (2019).

[5] D. Shah, T. Roychowdhury, S. Bahr, P. Dietrich, M. Meyer, A. Thißen, and M. R. Linford, *Surf. Sci. Spectra* **26**, 014016 (2019).

[6] D. I. Patel, T. Roychowdhury, V. Jain, D. Shah, T. G. Avval, S. Bahr, P. Dietrich, M. Meyer, A. Thißen, M. R. Linford, Introduction to near-ambient pressure x-ray photoelectron spectroscopy (NAP-XPS) characterization of various materials. *Surf. Sci. Spectra* (2019), Accepted. https://doi.org/10.1116/1.5109118

[7] D. Shah, C. V. Cushman, P. Dietrich, M. Meyer, A. Thißen, M. R. Linford, M. R., Coca-Cola, by near-ambient pressure XPS. *Surf. Sci. Spectra* (2019), Submitted.

[8] D. I. Patel, S. Noack, C. D. Vacogne, H. Schlaad, S. Bahr, P. Dietrich, M. Meyer, A. Thißen, M. R. Linford, Poly L-Lactic Acid (PLLA), by near-ambient pressure XPS. *Surf. Sci. Spectra* (2019), Submitted.

[9] V. Jain, M. C. Biesinger, and M. R. Linford, *Appl. Surf. Sci.* **447**, 548 (2018).

[10] B. Singh, A. Diwan, V. Jain, A. Herrera-Gomez, J. Terry, and M. R. Linford, *Appl. Surf. Sci.* **387**, 155 (2016).