Extended Abstract of PSA-19

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Positive and Negative Contributions of XPS to Reproducibility Issues

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Challenges associated with reproducibility and replication are impacting many areas of science. The reproducible preparation and delivery of nanoparticles is challenging and XPS can help address this problem. However, as the importance and use of XPS grown, there are increasing numbers of publications that include the inappropriate or incorrect use of XPS. Such papers add to reliability and reproducibility issues in the literature, sometimes leading to replication of errors and incorrect analyses. Some of the types of problems with "bad" XPS in publications are identified and the development of a series of guides and tutorials to help address the problem is described.

1. Reproducibility Problems

An increasing number of studies, surveys and editorials highlight experimental and computational reproducibility and replication issues that commonly appear in most areas of modern science [1-5]. In a survey reported in Nature in 2016, 90% of those interviewed indicated that there were reproducibility issues in the scientific literature and more than 50% indicated that the reproducibility problems were significant [6]. An AVS survey conducted in 2018 found that 65% of those responding identified reproducibility as a significant issue [7]. As described by Baer and Gilmore [8], there are multiple and complex causes of what some have called a "reproducibility crisis," which can impact materials, interface/(bio)interphase, vacuum and others sciences of importance to the surface analysis community. Reproducibility challenges are not new, but now appear in both old and new forms requiring innovative solutions. Drivers influencing reproducibility problems include the increasingly multi-discipline, multi-method nature of much advanced science, increased complexity of the problems and systems being addressed, and the large amounts and multiple types of experimental and computational data being collected and analyzed in many studies. Such issues challenge research teams and the review process. Systematic and sustained efforts are needed to address the causes of reproducibility problems that can hinder the rate of scientific progress and lower public and political regard for science [8].

2. XPS as a Solution

As the most commonly applied surface analysis method, use and misuse of XPS contributes positively and negatively to reproducibility challenges. Because of the high impact of the surfaces on the behaviors of materials as well as in biological and environmental systems, the use of XPS has been increasing for at least two decades [9]. In the area of nanoparticles, the lack of surface characterization is one source of material non-reproducibility that XPS can help identify and remedy [10-13]. For nanoparticles XPS can provide a great deal of information related to reproducible production, processing, and delivery of nanoparticles for many important applications. It can identify contamination, monitor changes in surface chemistry due to processing or after synthesis [14, 15], and quantify surface coverages and thicknesses [16-18]. Thus XPS is a highly useful tool for improving the reproducibility of nanoparticles and their use in research and technology.

3. XPS as a Source

However, XPS can also be the source of several types of reproducibility issues. Many researchers lack the experience or expertise needed to obtain the information sought from XPS measurements in useful and reproducible ways. Multiple types of problems appear and many of them can be observed in the literature including: analysis of samples at an inappropriate times or in non-optimal conditions[12]; inappropriate handling and preparation of samples[19]; damage to samples or sample charging during analysis leading to faulty conclusions[20, 21]; incorrect or inconsistent quantification and/or spectral interpretation[22]; lack of calibration and/or incorrect instrument set up and data acquisition[23]; chemically meaningless fitting of data[24]; inadequate reporting of methods, processes and results[25].

In the survey conducted by the AVS in 2018, 2/3 of those who responded thought reproducibility was a significant issue and indicated that guides and best practices could be a resource of help to the community. The AVS is developing a collection of tutorials, guides and best practices to XPS analysis for beginners [24, 25], including basic information about instrument operation, data collection analysis, and reporting. In addition articles outlining best practices in areas of application such as corrosion, catalysis, energy materials and biological samples are planned.

4. References

[1] M. Sené, I. Gilmore, J.-T. Janssen, Metrology *Nature,* 547 (2017) 397-399.

[2] R. Harris, Reproducibility issues, *Chem. Eng. News*, 95 (2017) 2.

[3] A. Belu, K. Maniura, S. McArthur, Biointerphases, 11 (2016) 040201.

[4] R.D. Peng, *Science,* 334 (2011) 1226-1227.

[5] S.L. McArthur, Repeatability, *Biointerphases*, 14 (2019) 020201-020201.

[6] M. Baker, *Nature*, 533 (2016) 452-454.

[7] D.R. Baer, Results for AVS Reproducibility Survey, AVS, New York, 2019. [available online] https://myemail.constantcontact.com/Beneath-the-AVS-Surface--January-2019.html?soid=1101176060676&aid=nQdcrQ2DAyE

[8] D.R. Baer, I.S. Gilmore, *J. Vac. Sci. Technol.* A, 36 (2018) 068502.

[9] C.J. Powell, *Micros. Today,* 24 (2016) 16-23.

[10] D.R. Baer, et al., *J. Vac. Sci. Technol.* A, 31 (2013) 050820

[11] A.S. Karakoti, et al. *Surf. Interface Anal*, 44 (2012) 882-889.

[12] D.R. Baer, et al., *Biointerphases,* 11 (2016) 04B401.

[13] L.-K. Mireles et al., *Data in Brief,* 7 (2016) 1296-1301.

.[14] D.R. Baer, et al., J. *Vac. Sci. Technol.* A, 31 (2013) 050820

[15] D.R. Baer, M.H. Engelhard, *J. Electron Spectros. Rel. Phenomena*, 178-179 (2010) 415-432.

[16] D.J.H. Cant, et al. *Surf. Interface Anal.*, 48. (2016) 274-282.

[17] C.J. Powell, et al. *J. Phys. Chem*. C, 122 (2018) 4073–4082.

[18] S.D. Techane, et al. *Anal. Chem*., 83 (2011) 6704-D. Briggs, J.T. Grant (Eds.) 6712.

[19] D.R. Baer et al., *Surf. Interface Anal.*,50 (2018) 902-906.

[20] D. Baer, *Front. Chem*. 6 (2018) 145.

[21] D.R. Baer, et al., Ch. 9 in *Surface Analysis by Auger and X-Ray Photoelectron Spectroscopy,* ed. by D. Briggs, J.T. Grant, SurfaceSpectra Ltd and IM Publications 2003, pp. 211-233.

[22] D. Shah et al., *J. Vac. Sci. Technol.* B, 36 (2018) 062902.

[23] J.T. Grant, J. Surf. Anal., 14 (2008) 398-405.

[24] P.M.A. Sherwood, *Surf. and Interface Anal.*, 51 (2019) 589-610.

[25] D.R. Baer, et al., *J. Vac. Sci. Technol.* A, 37 (2019).