Applied Surface Science Advances 19 (2024) 100534

Contents lists available at ScienceDirect

Applied Surface Science Advances

journal homepage: www.sciencedirect.com/journal/applied-surface-science-advances

Avoiding common errors in X-ray photoelectron spectroscopy data collection and analysis, and properly reporting instrument parameters

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ARTICLE INFO

Keywords:
X-ray photoelectron spectroscopy
XPS
Reporting
Data collection
Common errors

ABSTRACT

Despite numerous tutorials and standards written to the technical community on X-ray photoelectron spectroscopy (XPS), difficulties with data acquisition, analysis, and reporting persist. This work focuses on common errors in XPS that are frequently observed in the scientific literature and their sources. Indeed, this work covers: (i) XPS data collection, initial data analysis, and data presentation, (ii) Handling XPS backgrounds, (iii) Common errors in XPS peak fitting, and (iv) XPS data presentation and reporting. Graphical examples of errors and appropriate ways of handling data and correcting errors are provided. Additional readings are listed for greater in-depth exploration of the subjects discussed.

Introduction

X-ray photoelectron spectroscopy (XPS) is a core electron spectroscopy that is based on the photoelectric effect. It is the most widely used and important method for chemically analyzing surfaces [1–3]. In its conventional embodiment, it is sensitive to the outermost 5–10 nm of materials. XPS detects and identifies elements at surfaces based on the kinetic energies of the electrons that are ejected upon sample irradiation with X-rays. With care, XPS can be a quantitative spectroscopy [4]. XPS can detect all the elements, except hydrogen and helium, albeit, strictly speaking, XPS can detect H and He, but the cross sections of these elements are very small [5,6]. A significant advantage of XPS is its ability to provide chemical information about materials, i.e., chemical speciation, via its sensitivity to the oxidation states of the elements [7]. That is, the bonding environment of an element often directly affects the binding energies of its core electrons. This is an example of an 'initial state' effect. Accordingly, in an indirect fashion, XPS is sensitive to hydrogen because hydrogen chemically shifts the elements it is bonded to and also affects the valence band spectra of materials [8]. XPS depth profiling, imaging, and operando studies are regularly performed, [9–13] and XPS is increasingly used with hard X-ray sources (HAXPES) [14–16] and as a near-ambient pressure (NAP) technique (NAP-XPS) [17–19]. NAP-XPS is an important branch of XPS because it allows scientists to study materials, i.e. catalysts, under (more or less) operating conditions. NAP-XPS also makes it possible to analyze materials with relatively higher vapor pressures that are not compatible with UHV systems. HAXPES allows materials to be probed at greater depths than is possible with conventional XPS. Data science/chemometrics tools will probably be used more frequently in the future to analyze the large data sets collected in many modern XPS studies [20,21]. Because of its strengths, XPS use and reporting has increased about 1.5 times faster than the growth of the scientific literature in general [22].

In spite of significant advances in many surface and analytical characterization methods, the quality of much of the data, data analysis, and information from these techniques in the current scientific literature is a matter of increasing concern [22–25]. Indeed, a fairly recent survey

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https://doi.org/10.1016/j.apsadv.2023.100534
Received 28 September 2023; Received in revised form 28 November 2023; Accepted 29 November 2023
Available online 6 January 2024