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Special Collection: Reproducibility Challenges and Solutions II with a Focus on Surface and Interface Analysis

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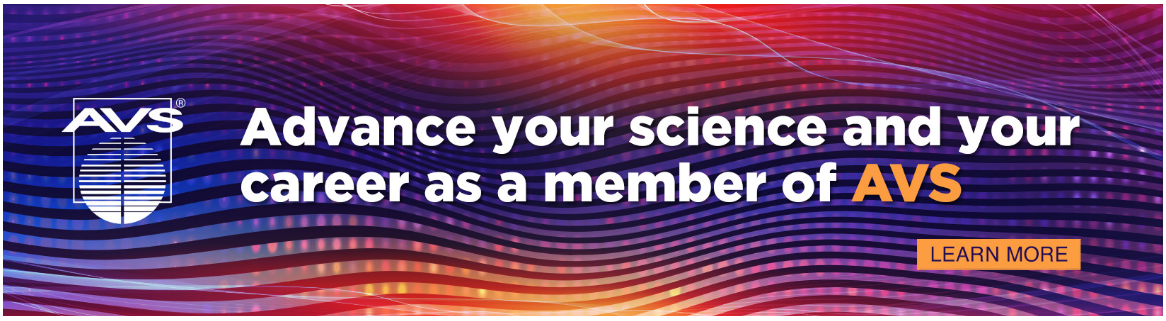

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Critical examination of the use of x-ray photoelectron spectroscopy (XPS) O 1s to characterize oxygen vacancies in catalytic materials and beyond

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ABSTRACT

In 2021, Idriss published a manuscript outlining that an assignment commonly utilized in catalyst research is not feasible. Despite this, the idea that you can measure oxygen vacancies (O_V) with *ex situ* x-ray photoelectron spectroscopy (XPS) persists. A review of 427 manuscripts citing Idriss's work found that for the citations related to catalysis (55%), those with an interest in O_V were more likely to avoid using XPS to identify O_V. Regardless, many catalysis researchers continue to rely on erroneous application of XPS data. Of the citations not related to catalysis research (45%), these were more likely to utilize the more obscure approaches. Surprisingly, two thirds of the citations showed no apparent interest in the assignment, an observation shared between both groups. We believe that many of these citations have failed to understand the original publication and/or are using it as a general citation for O 1s assignments. When the data were viewed as a function of publication year, three observations were made: (1) The number of citations using XPS for O_V is increasing, (2) those utilizing it as a direct measure are decreasing, and (3) one alternative method is gaining popularity. Herein, we provide evidence that most approaches are generally not applicable, or if they are, can only be successfully performed in-operando using highly specialized equipment with carefully planned methodologies; less than 3% of the citations reviewed met these criteria. It is envisaged that this publication will assist in improving the quality of XPS data interpretation in relation to XP O 1s spectroscopy.

I. INTRODUCTION

Development of new heterogeneous catalysts is a critical area of research as these materials have the potential to provide improvements in environmental outcomes for key industries, such as through Greenhouse gas mitigation by CO_2 methanation[1] and NO_X reduction,[2] and generation of green hydrogen by photocatalytic water splitting.[3] As such, these materials will likely play a pivotal role in the global transition to a green economy.

Catalytically active sites of interest tend to be on the surface of the material that forms an interface with the reactant in question. X-ray photoelectron spectroscopy, or XPS, is then an excellent candidate for the characterization of these materials, as it provides information regarding surface chemistry and is the most widely used technique for this purpose.[4] Considering the potential of XPS for characterizing this type of material and the continued increase in the global publication output,[5] the data presented in Fig. 1 are

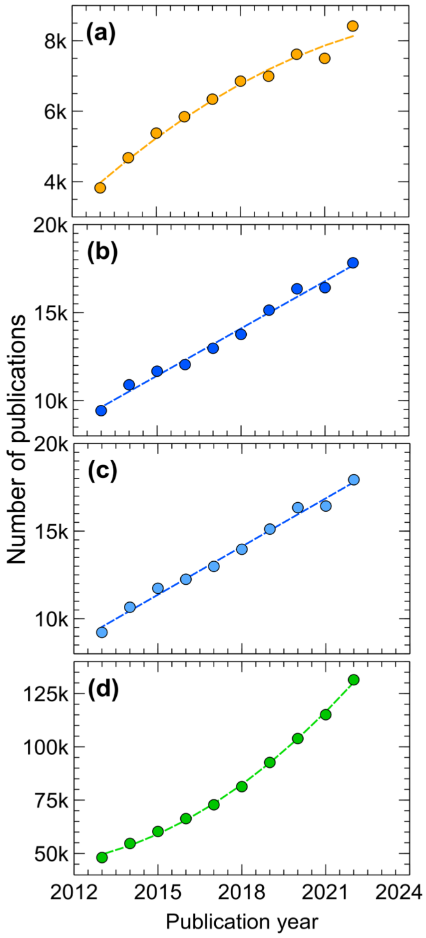

FIG. 1. (a) "Catalyst" and "x-ray photoelectron spectroscopy" with a second order polynomial trendline. (b) "Catalyst" and "x-ray diffraction" with a linear trendline. (c) Catalyst and "electron microscopy" with a linear trendline. (d) Catalyst and material with a second order polynomial trendline. Data obtained via multiple Scopus Web of Knowledge searchers. Briefly, the number of papers utilizing XPS in catalyst research appears to be decreasing while the number of catalyst papers, in general, is increasing.

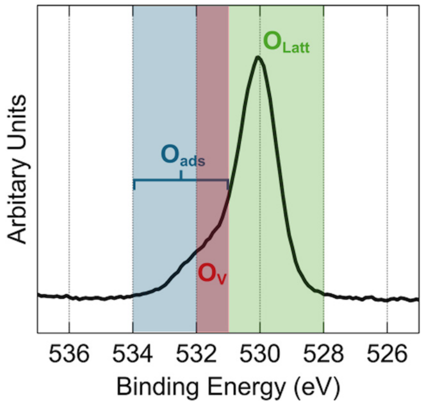

FIG. 2. High-resolution O 1s from a common metal oxide catalysis support (m-ZrO2) with the different nomenclature utilized in the literature (O_{Latt}, O_{ads}, and O_V) and their typical BE region labeled for these assignments.

surprising. Four plots present the number of publications per year containing the key word "catalyst" together with "x-ray photoelectron spectroscopy," and two other techniques commonly employed in characterizing these materials, specifically "x-ray diffraction" (i.e., XRD) and "electron microscopy" (i.e., EM techniques including transmission EM and scanning EM), with plot (d) using the keyword "material" as a means to capture the total number of papers involving catalysis in materials science. First, for XPS, the rate of change is decreasing and is potentially approaching a steady state, while for the other two techniques, the trend for the rate of change is steady for the 10-year period explored here. Second, the absolute values for XPS are less than half of the other two core characterization techniques. These observations suggest that XPS is not utilized nearly as much as the other characterization techniques in catalysis research, and that, as the rate of utilization is dropping relative to the overall research output, researchers are relying less on this critical technique.

One potential reason for this difference in utilization is a general lack of knowledge within the field regarding how to apply this technique and how to interpret the resultant data. It has been one author's experience that while many researchers in this area typically have a solid understanding of XRD and EM techniques, their knowledge of XPS has been limited. Unfortunately, this is not a problem unique to catalysis research[6] or XPS, but of characterization, more broadly.[7] Of the publications surveyed on this topic by one of the authors prior to this current study, the vast majority contain significant errors in relation to their interpretation of the XPS data, and anecdotal evidence suggests at a higher rate than that stated in a recent examination of XPS in the literature.[8]

In addition to the types of errors reported previously,[8] one uniquely relied upon in catalysts research is the assignment in the binding energy (BE) region of 531–532 eV of the O 1s region to oxygen vacancies (O_V), often utilizing the figure or fit component label "O_V." In addition to O_V, assignments to O 1s spectra found in the literature include O_{Latt} for lattice oxygen (O^{2-}), and O_{abs}, O_{org}, or O_{ads} for various adsorbed organic and (carbon–)oxygen species, including hydroxyls. Some authors have attempted to relate the O_{ads} component to O_V, making the effective BE region for O_V identification larger. Figure 2 provides a visual representation of the location of these assignments for a typical O 1s metal oxide spectrum. The examination of O_V is of importance to catalysis researchers as it is used as a metric to demonstrate improvements in the catalytic activity of their candidate when compared to their control materials. Authors will typically use an increase in intensity within this BE range as an indicator of increased performance, thus assigning the material as a superior candidate. It is important to note that while authors will typically fit the O 1s spectra to show—graphically or numerically—an increase in the O_V component compared to a control sample(s) of their choice, there is no standardized test, and direct comparisons between published works are rarely, if ever, made. Thus, while many in the field will claim a significant improvement in the catalytic activity based on O 1s fitting, the result is overall of little value as the relevant context required to understand the significance of this increase is not provided.

In 2021, Idriss published an important paper discussing the assignment of O_V in XPS O 1s.[9] Using the existing published data from other authors and examination of the basic principles of physical chemistry, Idriss clearly demonstrated that it is not possible to use XPS O 1s as a direct measure of O_V. Despite this publication, the authors have found that this approach to characterizing O_V persists, and, in some cases, either ignoring or misrepresenting Idriss's work.

Herein, we provide a brief outline of the terminology used in this manuscript (Sec. II), the importance of O_V to the catalysis community (Sec. III) and the potential contributions to the XPS O 1s region (Sec. IV) that have been incorrectly associated with O_V. The core messages of the 2021 Idriss publication are discussed in Sec. V. A recent review article concerning O_V in catalysts published after Idriss's work is examined (Sec. VI) to gain a greater understanding of how researchers in this field are applying XPS to their materials. The validity of one of the approaches identified from the literature is examined (Sec. VII). The general peak fitting practices applied to O 1s are examined and compared to best practice (Sec. VIII). To gain a broader view of the literature and to understand the impact of this important paper on the catalyst community, a review of papers that have cited Idriss's publication (Sec. IX) is outlined. Publications of interest are identified and discussed, while trends in the citation data are presented.

Throughout the manuscript, when a characterization approach from the literature is introduced, it is given as designator "Group X" where X is a numerical value starting from 1. From that point forward, when this type of approach is discussed in the main text, it is accompanied by its designator, e.g., Group 1. A full list of these different approaches is provided in Sec. X, and recommendations for each approach are provided.

II. TERMINOLOGY USED IN THIS MANUSCRIPT

A. *Ex situ*, *in situ*, and in-operando XPS

Ex situ XPS refers to any sample that has been exposed to air after any relevant processing or treatment, but prior to loading into the XPS instrument and subsequent analysis. Both *in situ* and in-operando XPS involve treatment of the sample within the instrument, typically under vacuum. For *in situ* XPS, the treatment and analysis occur at different times, for example, treatment of the sample in a different vacuum chamber than used for analysis. A current example of such instrumentation is the gas reaction cell offered by Kratos Analytical (Shimadzu) on their Supra+ instrument, where this cell is isolated from the main instrument.

An important distinction between these two types of XPS is under what conditions the analysis is performed. For in-operando XPS, the data are collected while the sample is held at or exposed to the environmental conditions of interest. A current example of this instrumentation is the near ambient pressure (NAP) XPS offered by SPECS (SPECSGROUP). Care should be taken to ensure that the approach is truly in-operando, as such instrumentation does not guarantee success if the analysis environment does not replicate benchmark conditions.[10]

B. Oxygen vacancy (O_V) versus oxygen defect (O_{def})

O_V in the context of catalysis are defined in Sec. III and by Idriss[9] and are highly reactive states that require significant energy to effectively maintain. Importantly, the discussion within this manuscript does not relate to oxygen defects that may occur in a sample, including in the subsurface and bulk, such as point defects that are stoichiometrically stable. We note that these terms have been used interchangeably within the literature.

III. WHAT IS AN O_V AND WHY IS IT IMPORTANT IN CATALYSIS RESEARCH?

Certain types of catalysts rely on the generation of defects, where they can significantly alter the reactivity of a material through the provision of highly reactive, unsaturated coordination sites for a reaction.[11] Clearly, defective oxygen sites fit this description and, consequently, both surface and subsurface oxygen vacancies and their dynamics have been subjected to considerable research,[12] with CO oxidation,[13] water-gas-shift reaction,[14,15] and automotive catalytic converters[16,17] being common examples.

Catalytic reactions on metal oxides typically follow a Mars van Krevelen (MvK)[18] or Eley Rideal[19] type mechanism, where oxygen vacancies are pivotal. By means of examples, we will briefly discuss the MvK mechanism of CO oxidation over a metal oxide. The reaction has two steps: (1) the adsorption to and, hence, oxidation of CO by lattice oxygen and (2) regeneration of the metal oxide by gas phase oxygen.

For example, the MvK oxidation of CO over Fe_2O_3 is as follows:

$$CO + xFe_2O_3 \rightarrow CO_2 + xFe_2O_{(3-1/x)},$$

$$\frac{1}{2}O_2 + xFe_2O_{(3-1/x)} \rightarrow xFe_2O_3.$$

Here, CO is oxidized by Fe_2O_3, leading to a partial reduction of oxide, which is then reoxidized by O_2. For a fraction of time, there is an oxygen-deficient lattice due to oxygen atoms being lost when forming CO_2, but this is quenched by the dissociation of O_2. These sites are clearly highly reactive and, therefore, readily react with the gas phase to form oxides or hydroxides in moist air and would not be measurable.

IV. CONTRIBUTIONS TO XP O 1s SPECTRA AND PRIOR EXPERIENCE ANALYZING CATALYSTS

The O 1s region can be challenging to interpret, with several potential contributions to the spectrum over a relatively small BE region. Most discrete contributions to the O 1s region occur within or near the region of interest, specifically 531–532 eV. A good summary of most of these contributions can be found in Mark Biesinger's XPS reference page for O 1s.[20] Contributions include hydroxyls and organic oxygen in the form of ketones and amides, as well as silicon–oxygen groups from silica, silanes, and silicone; the former can be used as a filler while the latter is a common lab contaminant. Adventitious carbon (AdC) can be found on most materials that have been exposed to the atmosphere, including inorganic materials such as metal oxides used in catalysis. AdC usually consists of a significant hydrocarbon component and typically has a contribution at ∼ +4 eV away from the main C 1s peak, assigned to acid or ester groups and can be influenced by the local environment.[21,22] While most authors in the catalysis field tend to ignore the contributions discussed above, we have found that they can account for some, if not all, of the intensity in the O 1s region, and can explain the changes in intensity when comparing samples. Even in circumstances where a change in the 531–532 eV O 1s region cannot be explained in full by a change in the oxygen–carbon or –other heteroatom groups, one cannot discount the potential of hydroxyl groups and/or bound water on the surface when performing an *ex situ* analysis.

Other considerations when examining the reported values for O 1s are related to data handling such as BE correction and instrumental artifacts such as differential charging.[23] The influence on the resultant data and interpretation can be difficult to determine if sufficient data and/or details are not provided, which is often the case. Incorrect BE correction can mean that the assignment(s) provided may not be sitting within the region of interest, while differential charging can result in the intensity at a lower BE shifting to a higher BE (or the opposite direction), inadvertently inflating intensity at the region of interest.

V. IDRISS'S 2021 SURFACE SCIENCE PAPER

Idriss's publication used existing studies within the literature to provide a legitimate interpretation to the region of O 1s spectra typically assigned to O_V by catalyst researchers. Examples demonstrating the application of *ex situ* XPS analysis to measure O_V were provided by Idriss in the first 33 citations, giving sufficient evidence that the stated problem exists. Using past studies of reduced and nonreduced thin films and single crystal metal oxides together with an understanding of basic physical chemistry, in context of these samples, Idriss provided five points of contention regarding the use of XPS to determine O_V, specifically:

(1) "A binary or mixed metal oxide material or catalyst containing surface oxygen vacancies will be oxidized instantaneously in ambient conditions":[9] this is certainly true but would also be true for other high-energy surface vacancies on other, perhaps nonoxide, materials.
(2) It is not possible to measure an XP O 1s signal from a missing oxygen atom.
(3) In the case of an indirect measure of O_V, specifically assigning intensity in the 531–532 eV region to oxygen atoms adjacent to an oxygen vacancy due to a change in the coordination number is also incorrect.
(4) Furthermore, it is unlikely that the kinetic energy of a photoelectron escaping from an oxygen atom will differ if adjacent to an O_V.
(5) In a scenario where either molecular oxygen or water dissociatively adsorbs on the surface, this will regenerate O_V.

Unfamiliarity with the concept described in point (1) could be perceived as a lack of critical thinking by some in the field of catalysis. As noted by Idriss in relation to a computational study of metal oxides, "The removal of an oxygen atom from the surface is costly, and therefore once exposed to water, oxygen, or an oxygen-containing compound such as CO_2, will be healed non-catalytically, unless a considerable amount of energy is given." In general, the oxidation of surfaces is extremely rapid; so once the (post-treated) sample is exposed to air during transfer to the XPS instrument, i.e., any *ex situ* experiment, any O_V on the surface will no longer be present. Related to this is point (5), as rapid healing of O_V can occur even in instances where the local environmental composition is not strictly air. If oxygen is available, oxidation can occur even in UHV environments.[24]

Regarding point (2), it is understandable how a catalysis scientist not possessing a solid understanding of XPS fundamentals could unintentionally ignore this basic principle. The explanation for point (3) is rather straightforward as "the removal of an oxygen atom does not alter the coordination number of the adjacent oxygen atoms…." Idriss states doubt, but does not outright reject another related interpretation that "the kinetic energy of an oxygen atom adjacent to an oxygen vacancy is different from that of an oxygen atom in a stoichiometric compound." Recent papers demonstrated, using density functional theory (DFT) calculations, that there is no significant influence on the BE position of the emitted photoelectrons from the remaining oxygen atoms near an O_V.[25,26] Based on this work, it is fair to now outright reject this alternative interpretation.

Overall, Idriss's work outlined that the three different interpretations used for XP O 1s spectra by the catalyst community have no basis. One would assume that the generation and perpetuation of these assignments are the result of confirmation bias as there is no apparent scientific basis, and they are merely convenient for telling a particular story. In addition, it is a timely reminder that researchers must think more carefully about the context of their samples throughout the characterization process rather than focus on the ideal scenario in which they will be used. It should not come as a surprise that generating a surface with groups that are highly reactive toward oxygen, then this surface will react when exposed to air, healing the vacancy.

VI. REVIEW PAPER IN J. MATER. CHEM. A 2022

A 2022 review paper in J. Mater. Chem. A[27] provides details regarding different approaches for the characterization of oxygen vacancies. The paper contains only a single paragraph on XPS (Sec. III A), grouped with three other techniques (XRD, electron paramagnetic resonance, and Raman), while alternatives such as electron microscopy (Sec. III E) are not grouped. This is likely related to the importance of the technique to the community and supports the observations made in the introduction regarding usage. Several examples of use are provided together with five citations, where these can be placed into broadly three groups: (1) measuring BE shifts, (2) identification of a particular oxidation state, and (3) relying on a peak for O_{ads} in the high-resolution O 1s fit, including the ratio of O_{ads} to O_{latt}, and correlating O_{ads}/O_{latt} and O_2-temperature-programmed desorption (O_2-TPD) results with catalytic performance. The paragraph ends stating that XPS is suitable for "qualitative and semi-quantitative analysis but not for accurately quantifying surface defects." Herein, the examples and citations of the J. Mater. Chem. A review paper are examined, providing a basis for the first three groups (i.e., Groups 1–3) for categorizing the different approaches utilizing the XP O 1s spectrum in the study of oxygen vacancies. None of the examples presented in this section are considered acceptable and should be disregarded. A full list of these groups can be found in Sec. X.

The first example is related to observing changes in the BE position of the metal and oxygen peaks (**Group 1**—BE shifts), with two references provided. The first reference[28] examines MnO_2 nanowires, where an increasing shift to a lower BE for the O_{latt} component is associated with an increase in oxygen vacancies; no citation is provided for this assertion. This observed shift is likely the result of poor charge correction, as every photoelectron peak presented shows the same trend to lower BE, meaning the O_{latt} components are likely sitting at a similar BE for all samples. The second reference[29] suffers from similar problems, where all spectra for the candidate sample, including the O 1s and Fe 2p spectra, are shifting to a lower BE. The authors also state that a large amount of Co(II) is evidence of O_V (**Group 2**—identification of a specific oxidation state). The citation for this statement[30] makes a similar statement but provides no citation. Unless performed in-operando with carefully planned experimentation, an O_V will not exist during data collection and, thus, will not be responsible for the observed oxidation state. The work presented by Rogala *et al.*[31] is an excellent example of in-operando XPS.

The next example relies on a comparison of O_{ads}/O_{latt} values (**Group 3**—utilization of O_{ads}/O_{latt}). Depending on the publication, the component O_{ads} can, but not always, include all O 1s intensity above the O_{latt} peak, where surface hydroxyls can be formed via dissociation of water at the O_V site. Some authors state that O_{ads} is important in the design of catalysts as it is believed to facilitate oxidation reactions of organics.[29,32,33] One of the citations provided for this approach in the J. Mater. Chem. A review paper, Ref. 20,[32] presents values for O_{ads}/O_{latt} for a series of samples with their candidate sample having the highest value. The authors claim that previous studies have "proven that abundant O_{ads} species could promote oxidation reaction…" with two citations.[34,35] Neither citation contains XPS data but rather was measured using *in situ* techniques including O_2-TPD, meaning no connection is provided between the oxygen measured by these two techniques undertaken in very different environments. It is shown in Sec. VII that there is poor correlation between these two techniques (**Group 3**). References 82[36] and 83[37] also rely on O_{ads}/O_{latt} to determine that a particular sample had more oxygen vacancies.

A summary of the different approaches that researchers in the catalyst community use to demonstrate oxygen vacancies from XPS data is presented in Sec. X. It was found that the review article analyzed in this section did not encompass all examples used by researchers, with additional examples found during the research undertaken in Sec. IX. It is interesting to note that the authors of the J. Mater. Chem. A review article did not provide an example of using XPS to directly measure O_V (**Group 4**—Direct measure of O_V).

VII. IS O_{ads} AN INDICATOR OF CATALYTIC PERFORMANCE?

Further investigation into the approach of using O_{ads} from O 1s spectra as a direct indicator of catalytic performance was considered necessary. One objective was to determine the relationship between O_{ads} from XPS and O2-TPD as inferred to by the examples referenced above. O_2-TPD is an *in situ* technique that measures the amount of desorbed oxygen as a function of temperature. These two approaches examine adsorbed oxygen, but using different methods and under different conditions.

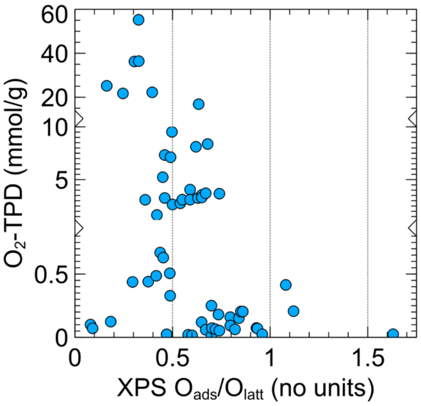

FIG. 3. Is there a link between *in situ* O desorption and *ex situ* O_{ads} measurements? A very low coefficient of determination ($R^2 = 0.135$) indicates poor correlation between O2-TPD and XPS O_{ads}/O_{latt} (n = 60 from 18 references). Note the use of broken *Y* axis to better visualize O2-TPD values between 0–0.8 and 1.2–10 mmol/g.

Several searches within Web of Knowledge were undertaken to find additional papers that used both XPS and O_2-TPD to characterize catalysts. A considerable number of publications have used both techniques, but did not present the quantified results from O_2-TPD, choosing instead to compare the intensities qualitatively, e.g., Refs. 38–41. Of the manuscripts that did quantify both the XPS and O_2-TPD results, the most commonly reported units for O_2-TPD were mmol/g or umol/g. However, not all data were comparable due to the different units used for O_2-TPD, such as "normalized peak area."[36] A plot of O_{ads}/O_{latt} versus O_2-TPD (mmol/g) is provided in Fig. 3. A single reference is responsible for all O_2-TPD values >10 mmol/g,[42] while for several references, the reported values were <1 mmol/g; it is not clear whether these large values are an error in the reported values. A very low coefficient of determination indicates poor correlation between the results from O_2-TPD and XPS O_{ads}/O_{latt} (n = 60 from 18 citations[36,37,42–57]). Therefore, there is no evidence to support the assertion that O_{ads} is a reliable indicator of catalytic performance. This is unsurprising, considering the incompatibility between the two approaches.

VIII. PEAK FITTING PRACTICES USED IN THE EXAMINED PAPERS COMPARED WITH BEST PRACTICE

From the examples provided within the J. Mater. Chem. A review article and further searches of the literature, it was observed that there was no consistency with the number of components used to fit the O 1s spectrum, and, thus, what portion of the photopeak was associated with O_{ads}. Undoubtedly, some of these variations in the applied peak fitting can be traced to the poor use of line shapes, with many analysts utilizing purely Lorentzian shapes, or peaks with unreasonable FWHMs.[58–60] For the papers examined, an O 1s fit featuring a two-component[28,36,61] and a three-component[29,32,37,47] system were both common, and an example of a four-component[62] fit was found, but when it came to this specific approach of using O_{ads}, the two-component fit was prevalent. A two-component fit for a region spanning in some cases more than 5 eV (∼529 to >544 eV), the FWHM of the O_{ads} component, in particular, is so wide that it includes intensity from the O_{latt} region. This fitting approach also ignores other contributions to O, including organic species. Overall, this would mean that the contribution to O_{ads} is severely overstated.

Of course, it may not be necessary to fully interpret the O 1s spectrum and, equally, there are plenty of instances where the O 1s spectrum may be overinterpreted. Generally, transition metal oxide surfaces, depending on their reactivity, comprise of lattice oxygen (ca. 529.5–531 eV), hydroxide (ca. 531–532 eV), and carbonate (ca. 530.5–532.5 eV); the latter can be confirmed using the high-resolution C 1s spectrum. At a higher BE, trapped (e.g., intercalated) or adsorbed water, a loss peak (discussed below), or peak asymmetry (discussed below) (ca. 534–536 eV) may be present. For the authors, absorbed water that is still present on the surface under ultrahigh vacuum conditions is a contentious issue. If feasible, it would only account for an extremely small fraction of the total oxygen present in the case of an oxide. Clearly, there is an overlap in these O1s binding energies, and this is also not accounting for any adventitious organic material which may be present.

Based on the measurements of pure oxides, hydroxides, and carbonates, it has been observed that the FWHM of the oxidic species varies, with typical values of 1.2–1.6 eV for lattice oxide and carbonates, while hydroxides vary between 1.6 and 2.0 eV. Of course, these values are dependent on the degree of successful charge compensation and the spectrometer resolution. Nevertheless, these values are good guides and, consequently, anything significantly different from these values should be a cause for concern.

With reference to the binding energies of C–O functional groups, taking the work of Beamson and Briggs,[63] their binding energies values for polymers (adjusted to a C 1s reference energy of 284.8 eV) generally fall in the range 532.8–533.4 eV; hence, any structure above ca. 533 eV, and assuming the C 1s spectrum reveals C–O based species, should be attributable to organic species.

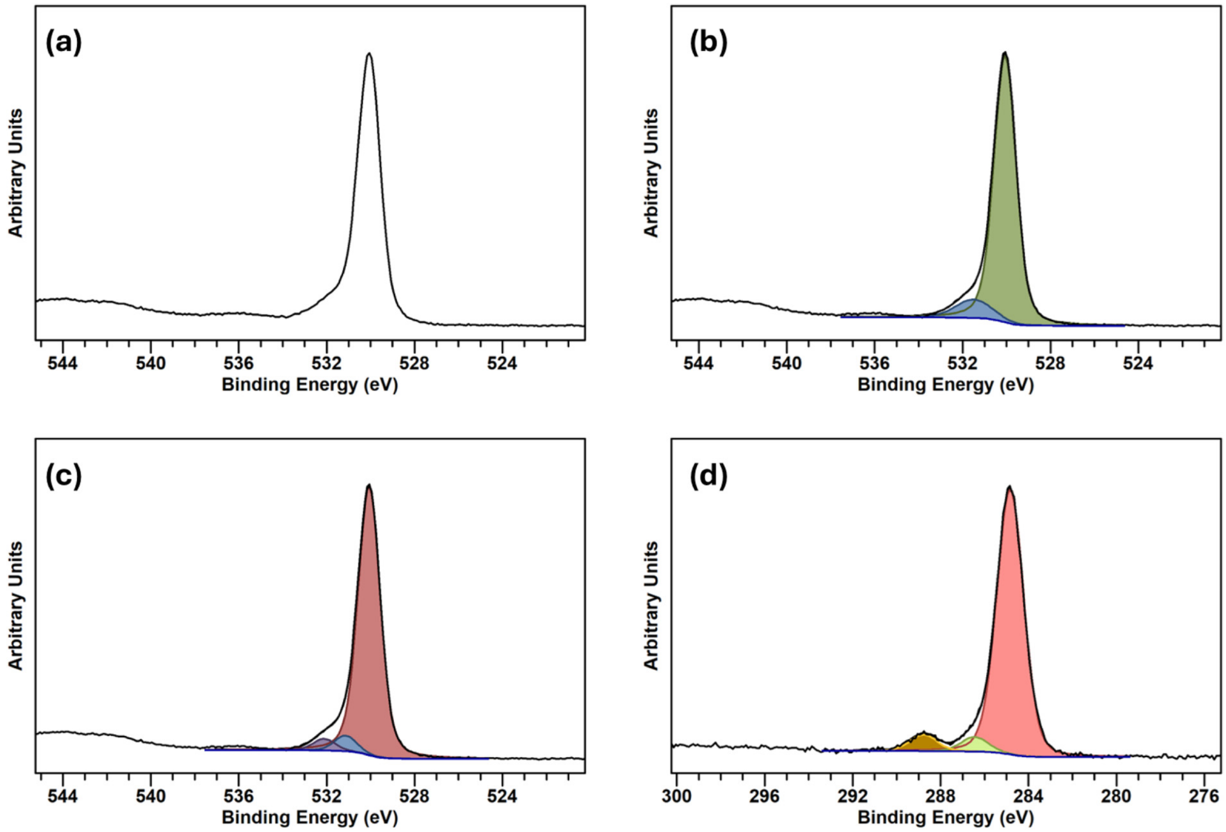

FIG. 4. Core level spectra for an as-received anatase sample: (a) raw O 1s spectrum, (b) spectra in (a) fitted with two components with constrained FWHM, (c) spectra in (a) fitted with three components, and (d) the corresponding C1s spectrum showing the C–O containing species. These conflicting 2- and 3-peak fit protocols highlight the potential shortcomings of peak fitting O 1s, as discussed in the main text.

By example, let us consider TiO_2 (Fig. 4), herein presented as the anatase form, but similar arguments are valid for other polymorphs or mixtures such as P25. First, we note the small broad structure centered at ca. 536 eV: given the sample is Na-free, this is an oxygen loss feature as noted by reflected electron energy loss spectroscopy measurements rather than a Na KLL Auger peak and should not be confused with some high-energy species.[64]

The peak has structure to the high binding energy side of the main photopeak indicating the presence of species such as hydroxides and organics; hence, for our discussion, we have fitted the O 1s spectrum two ways, with two and three component models. The C 1s spectrum reveals that the C–O functional groups are relatively low in concentration and, using the relationships derived by Henderson *et al.*,[65] we find that the 3-peak model tends to under/overestimate the peaks assigned in Fig. 2(c) as OH (blue) and organic (purple). The O_{latt} to Ti ratio in this case is ∼2.1, a value slightly over the expected stoichiometry but within the uncertainties associated with XPS quantification. The two-peak model, Fig. 2(b), also yields an O_{latt} to Ti ratio of ∼2.1, but, instead, we model the hydroxide, and potentially any organic as the peak (blue) at 531.5 eV, with a FWHM of 1.9 eV, in line with our bulk measurements.

From even this simple example, it is clear that there are significant complications to an in-depth interpretation of the O 1s spectra; it is our viewpoint, therefore, that it is sufficient to fit the O 1s envelope with features ascribed to lattice, adsorbed oxygen (OH, CO_3 etc.), and organic species alone.

It should be noted that for materials such as IrO_2,[66] which is a conducting oxide, the lattice oxide peak is asymmetric, and failing to account for this will lead to erroneous oxygen ratios or assignment of chemical states. IrO_2, in particular, has a screened state which, if the analyst was to assign peaks based on the overall O 1s spectrum envelope, could easily be mistaken for OH.[67] The asymmetry on conducting oxides arises from a finite O partial density of states in the vicinity of the Fermi level, which allows excitation of the O 1s electron to induce a continuum of final states through electron-hole pair excitation. Such effects have been noted for chemisorbed oxygen (and other elements) in surface science studies of oxygen adsorption on Fe(110),[68] while for bulk oxides, this has been noted for RuO_2,[69] OsO_2,[70] MoO_2,[71] and PbO_2.[72] Hard x-ray photoelectron spectroscopy studies of the conducting oxide fluorine doped tin oxide also reveals an asymmetric structure attributed to the combination of O–Sn bonds and plasmon loss.[73]

IX. WHAT HAS HAPPENED SINCE THE IDRISS PUBLICATION—WORKS CITING IDRISS'S PAPER

A. Publications of interest

At the time of writing (February 3, 2025), the 2021 publication by Idriss had 427 citations (Web of Knowledge). In this section, we explore the impact that this publication has had on the field by examining how authors have applied Idriss's recommendations.

Five notable publications include the work by Frankcombe and Liu.[26] The authors use *ab initio* methods to calculate the binding energies of O 1s (photo) electrons from different ions originating from a model ZnO slab presenting subsurface oxygen vacancies. Keeping in mind the limited scope of this approach in part due to the computational resources required for modeling, the authors found (1) "The resulting O 1s binding energies show no signature for oxygen defects in bulk regions," and (2) "Furthermore, the 531 eV binding energy feature often ascribed to oxygen vacancies or oxygen deficient regions can instead be readily explained by the O 1s electrons from water molecules strongly bound to the exposed ZnO surface (i.e., chemisorbed, as distinct from more loosely bound water) or surface oxygen passivated with hydrogen." Point 1), but for surface oxygen vacancies (rather than subsurface defects), was reported earlier in 2020 by Posada-Borbón and Grönbeck[25]—"Furthermore, we do not find any clear spectroscopic signature originating from oxygen vacancies." Taken together, we now have evidence from *ab initio* calculations that oxygen vacancies are not expected to have sufficient influence on the BE positions such that their contributions can be directly observed from the XPS spectra.

The work by one of the authors[74] is an excellent example of using all available information from XPS, specifically, the elemental quantification together with the fitting of high-resolution spectra, and knowledge of the sample, to confirm your hypothesis. In this case, it is demonstrated that the peak often assigned to Ov in damaged or reduced CeO_2 is not associated with Ov. Intentionally damaging a sample in a controlled manner is one option available for examining defect chemistry.

The work by Wong,[75] published as a Comment in ACS Applied Materials & Interfaces, provides an alternative assignment and interpretation to the data presented in an earlier publication in the same journal. The authors of the original publication relied on a direct measure of O_V using O 1s (**Group 4**) and claimed that these O_V were responsible for device improvement. Wong, however, correctly identifies the peak as associated with hydroxyl groups and provides an alternative theory for device enhancement, one that does not rely on O_V. While not a catalysis paper, this is an important example of how progress in research can and is being stifled by incorrect data processing and interpretation. There are direct parallels between the scenario outlined by Wong and many papers that rely on the erroneous identification of O_V via XPS and the subsequent association with performance improvement, where the real reason is left unidentified.

The fourth manuscript appears to be in direct response to Idriss's publication, as the authors provide "three approaches for oxygen vacancies with XPS…," claiming that "these three alternative approaches offer increased reliability by evaluating the true 'fingerprints' of oxygen vacancies."[76] This manuscript, referred to as the JECS paper herein, does cite some in-operando works that provide a convincing argument for the positive identification of O_V via XPS, such as the work by Rogala *et al.*[31] However, the authors of this manuscript believe that the JECS paper is potentially misleading as it fails to acknowledge an important point made by Idriss, specifically that *ex situ* XPS of O_V is not feasible due to the healing of these surface states. For example, the manuscript only refers to XPS, making no distinction regarding in-operando or *ex situ* XPS, or that researchers must avoid any source of oxidation during experimentation. It is only in the Conclusion statement that the authors address other potential causes for the apparent O_V observed using the stated three approaches. In our opinion, insufficient attention is given to these points, and an inexperienced researcher would be misled into believing that these options are the exception. The authors should have only used citations that relied on in-operando XPS and clearly stated that *ex situ* XPS is not applicable. The three approaches presented by the authors were previously identified during the review of citations and their suitability is addressed in Sec. X.

The fifth and final noteworthy work citation is unique as it is potentially the first manuscript reviewed that combines *in situ* XPS and DFT calculations to further support the hypothesis that XPS is unable to distinguish oxygen vacancies.[77] Samples of In_2O_3 were transferred between the analysis chamber and a high-pressure gas cell for analysis and treatment, respectively. The experiments presented are similar to the examples detailed by Idriss.[9] It is unfortunate that the work suffers from some poor choices related to analysis and experimental design that are not internally consistent, such as an inclusion of a component assigned to oxygen vacancies when fitting the O 1s spectra. The authors state that "In addition, DFT calculations, after optimizing the alignment to the calibrated XPS assignments, further find that the presence of oxygen vacancy among the lattice oxygen does not induce new feature to O1s binding energy but results in a non-uniform and slight shift (0.1–0.3 eV) to the higher energy, yielding a small unsymmetrical shoulder peak denoted as O(II) of lattice oxygen." However, in all of the figures presented, rather than use an asymmetric peak for the oxide contribution, the authors use a distinct component, O (II), and this component is shifted by 0.9 eV, i.e., three times larger than expected, based on their own DFT calculations. While the samples remain under vacuum, treatment and analysis does not occur simultaneously and the authors do not consider the possibility of oxidation within the analysis chamber,[24] healing any O_V via a stoichiometric reaction, nor do they explain why this O(II) component is the largest for the "as prep" sample that is prepared *ex situ*. Considering the BE of the component and their own DFT results, it is likely associated with hydroxyls rather than oxygen atoms adjacent to an O vacancy (**Group 6**). This example highlights that performing *in situ* XPS does not guarantee success, and that consideration should be made as to the local environment of the sample during the entire experiment. The paper would have benefited from a more careful and complete analysis, together with additional experiments such as angle-resolved XPS to provide evidence for their assignment of certain oxygen groups. The authors would also need to acknowledge that some oxidation and carbon deposition would have likely occurred during sample transfer and analysis.

B. Complete review of citations that cited the Idriss 2021 paper—Examining the results

At the time this review was performed, all 427 manuscripts citing the Idriss 2021 manuscript were examined to identify how each manuscript handled the analysis of O 1s data in response to Idriss's conclusions. Four groups were created in addition to Groups 1 through to 3 that were previously identified. The complete list of these groups can be found in Sec. X. In addition to grouping by their interpretation, they were further filtered by whether they were interested in O_V [O_V(Y)] or not [O_V(N)], and whether the material type was a catalyst [either C(Y) or C(N), for yes and no, respectively]. The interest in O_V was judged by whether it featured in any discussion, e.g., if it was assigned but not discussed, then the paper was judged as not interested in O_V. The results of this examination are presented in Table I and Table S1 in the supplementary material. Note that only 13 citations or 3.1% of the citations employed in-operando XPS; this includes NAP-XPS, which, as explained in Sec. II, does not guarantee experimental success at examining the sample in the intended state. There is evidence that the field is beginning to acknowledge the limitations of this approach.[10]

TABLE I. Examination of 427 journal articles that have cited the Idriss 2021 paper as of February 2025 (Refs. 26 and 74–499), grouped by how they interpreted XP O 1s data, and expressed as a heat map (0% = red; 100% = green) (n = 426). Definitions for each "Group" can be found in Sec. X. The full results can be found in Table S1 in the supplementary material. Citations that did not utilize XPS for Ov identification, i.e., Group 7 row, represent the largest total for any individual group. Overall, however, more manuscripts citing Idriss relied on XPS to identify Ov than those that did not. Further observations and details are provided in the main text.

Group	Interpretation	Total (%)	Intersection (%)			
			O_V(N)C(N)	O_V(N)C(Y)	O_V(Y)C(N)	O_V(Y)C(Y)
1	BE shifts[a]	3.5	40.0	20.0	13.3	26.7
2	Oxidation state[a]	9.2	48.7	23.1	5.1	23.1
3	O_{ads} approaches[a,b]	18.5	53.2	20.3	6.3	20.3
4	Direct measure of O_V[b]	21.4	24.2	19.8	13.2	42.9
5	O atoms adjacent of O_V[b]	2.3	10.0	30.0	20.0	40.0
6	Comparing O/metal or O_{latt} conc.	0.9	75.0	25.0	0.0	0.0
7	Does not rely on XPS for O_V	41.8	16.9	18.5	25.3	39.3
Other	—	2.3	10.0	10.0	20.0	60.0
Total		100.0	29.1	19.7	16.4	35.0

[a] Discussed in the J. Mater. Chem. 2022 review manuscript.
[b] Discussed in the Idriss 2021 manuscript.

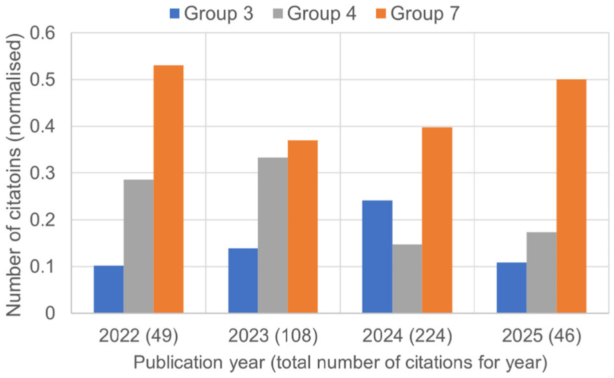

FIG. 5. From the review of papers citing the Idriss paper, the number of citations (normalized by total citations per year) for each year since publication, for the three main groups (Group 3—O_{ads} approaches, Group 4—Direct measure of O_V, and Group 7—Does not rely on XPS for O_V) identified. Trends, including an increase in the number of citations using XPS for O_V, are identified and discussed in the main text.

From the "Total" row in Table I, the largest proportion of manuscripts examined (35.0%) were categorized as catalyst papers where O_V was of interest to the authors, while another 19.7% were catalyst papers but the authors did not discuss O_V. The remaining 45% of the manuscripts reviewed were based on materials other than catalysts, where two thirds were categorized as O_V (N), meaning unless they were assigned to **Group 7** (citation does not rely on XPS for O_V), then XPS was used to identify O_V, but the result was not utilized.

The significance of these results becomes clear when observing the intersection results of the Group 7 row, where it is revealed that the noncatalyst papers that did not utilize O_V accounted for the smallest relative fraction for this group, while catalyst papers that were interested in O_V had the largest proportion. The results for these two categories are discussed below.

The former result is somewhat unexpected—why is there an appreciable amount of citations utilizing XPS to identify O_V papers that are (1) noncatalysis papers and (2) have no apparent interest in O_V? Considering all manuscripts reviewed have cited Idriss's work, there is no apparent motivation for these works to effectively ignore the message in the aforementioned paper. During the review, it was noted that some authors have adopted Idriss's work as a general citation for O 1s assignments. If these authors have not fully engaged with the content, whether that is because they are not familiar with the field due to language barriers or some other reason, then, perhaps, they have mistaken Idriss's warnings as a valid assignment. Another potential reason could be related to a desire to obtain as much information as possible for their characterization, in effect, overinterpreting the data simply for the sake of bulking the findings reported in the manuscript.

Back to the latter result, the category representing catalyst papers with an interest in O_V representing the largest relative fraction of citations that do not rely on XPS for O_V may represent a positive outcome where researchers in this area are engaging at a greater level with Idriss's paper. However, considering the substantial values observed in Table I in cells that represent catalyst researchers utilizing XPS to characterize O_V, most researchers in the field still need to change their approach. For example, authors in this category represented the largest relative fraction for Group 4 (direct measure of O_V). Taken together, there is a subset of catalyst researchers with an interest in O_V, which is the most persistent group in applying this particular erroneous interpretation, and this category of researcher is less likely to replace it with one of the alternative approaches found during the review. This category of researcher has the most to gain from using this interpretation, as it provides a straightforward and accepted means to explain performance improvement for their material. These authors can then quickly move onto their next publication without spending additional time properly investigating their material.

As the publication date of the citations ranges from 2022 to 2025, it was possible to arrange the number of citations by year, as shown in Fig. 5 for the three major groups (Groups 3, 4, and 7). Three observations can be made regarding the number of papers relying on (1) XPS to identify and/or measure O_V is increasing (Group 7 is decreasing over time), (2) XPS as a direct measure of O_V is decreasing (Group 4 is decreasing over time), and (3) an O_{ads} approach is increasing (Group 3 increasing over time). Considering these are papers that have cited Idriss's work, it is alarming that the number of papers relying on XPS to measure O_V is increasing. While there appears to be a shift away using XPS as a direct measure for O_V over time, indicating that there is potentially an acknowledgement regarding the main argument made by Idriss, it is being replaced by alternatives such as the O_{ads} approaches that are also flawed, as detailed above.

During the review, it was noted that the journal that featured the most was a surface science journal, specifically Applied Surface Science, with a total of 36 manuscripts. From the journal's website, "A Journal Devoted to Applied Physics and Chemistry of Surfaces and Interfaces," it is a fair assumption then that those involved in these publications (authors, editors, and reviewers) should have a firm understanding of a core surface-specific characterization technique such as XPS. This would include the physics/quantum mechanics that underpin the technique and, thus, be aware that you cannot measure a photoelectron from a missing atom (Group 4). Awareness of the Idriss paper should not be necessary to critically assess publications, as the concepts described, such as the reactivity of oxygen vacancies to ambient air, should be fundamental knowledge to those working in catalysis and surface science. With these points in mind, there are some noticeable differences in this subset of data (Table II), and not necessarily intuitive if your position is that a surface science journal with a high impact factor (6.9 at the time of writing) should set a higher standard for surface science-related content. Comparatively, a greater fraction of the citations relied on XPS to identify O_V (72.2% vs 58.2%). A larger fraction relied on XPS as a direct measure of O_V (30.6% vs 21.4%), and a much greater fraction of these manuscripts were catalyst papers (90.9% vs 62.7%).

Previous studies[8] have indicated that a journal's impact factor is likely not a predictor of quality for the XPS data published. There is, however, a universal expectation that these journals publish research of the highest quality. Considering the observation

TABLE II. Examination of journal articles published in Applied Surface Science that have cited the Idriss 2021 paper as of February 2025 (Refs. 77, 81, 88, 92, 98, 110, 117, 126, 131, 153, 170, 171, 184, 188, 208, 214, 218, 235, 239, 266, 267, 279, 281, 300, 317, 326, 329, 342, 393, 396, 405, 408, 422, 459, 461, and 498), grouped by how they interpreted XP O 1s data and expressed as a heat map (0% = red; 100% = green) (n = 36). Compared to the parent dataset (Table I), Group 7 no longer represents the largest total for any individual group, with a greater fraction of papers relying on XPS for O_V identification.

			Intersection (%)			
Group	Interpretation	Total (%)	O_V(N)C(N)	O_V(N)C(Y)	O_V(Y)C(N)	O_V(Y)C(Y)
1	BE shifts[a]	2.8	0.0	100.0	0.0	0.0
2	Oxidation state[a]	19.4	28.6	28.6	14.3	28.6
3	O_{ads} approaches[a,b]	13.9	0.0	100.0	0.0	0.0
4	Direct measure of O_V[b]	30.6	9.1	54.5	0.0	36.4
5	O atoms adjacent of O_V[b]	5.6	0.0	0.0	50.0	50.0
7	Does not rely on XPS for O_V	27.8	20.0	50.0	0.0	30.0
Total		100.0	13.9	44.4	5.6	36.1

[a] Discussed in the Idriss 2021 manuscript.
[b] Discussed in J. Mater. Chem. A 2022 review manuscript.

made above that the continued use of XPS as a direct measure of O_V (Group 4) may persist as it is a convenient time saver, certainly, these authors are also benefiting by publishing in more prestigious journals, at least in the surface science field. One potential reason for this is regarding perception—assigning a performance improvement to O_V appears to be more impressive in the field than to another physical property such as structure.[500] Authors, in particular, early career researchers, can rely on existing publications as a template, including the use of current buzz words in their particular field. This result could also provide an explanation for the observation made above that the number of papers relying on XPS to identify O_V is increasing—researchers are relying more on papers in relatively high impact factor journals that, at least in the case of the journal examined herein, have a higher relative fraction of manuscripts using XPS to identify O_V, thereby legitimizing these approaches. Irrespective of the real reasons, this outcome should be a call to surface science journals to do a better job at addressing obviously flawed interpretation. It is worth noting that we did not examine papers that have not cited the Idriss paper, where it is highly likely that a significant fraction of these papers utilize the erroneous practices described herein.

X. DISCUSSION REGARDING THE DIFFERENT APPROACHES

The categories (groups) for using XPS to measure O_V within the catalyst community and their flaws are detailed below. Note that for the interpretations listed, typically, samples were exposed to air prior to analysis, meaning that the highly reactive surface species would have healed prior to loading the samples into the instrument. In addition, no convincing evidence has been presented to support the (unsubstantiated) approaches. As is the case for the original "oxygen defect" assignment for by Fan and Goodenough in 1977,[501] that was later adopted by the catalyst community to identify and quantify O_V, these interpretations are based on hypotheses that have never been independently verified and do not withstand scrutiny.

A. Specific approaches and recommendations

Importantly, O_V, as defined in Sec. II, are only present during XPS analysis if performed in-operando and approximately 97% of the citations reviewed relied on *ex situ* XPS. Below, only two approaches are recommended in addition to not relying on XPS to demonstrate O_V; these require complex instrumentation that is not readily available to all researchers and multistep experimentation to succeed.

Our general recommendation would be to avoid fitting O 1s where possible and only use the spectrum to do a qualitative assessment of the chemical environment of oxygen atoms. Otherwise, as discussed in Sec. VIII, a fit utilizing components ascribed to lattice, adsorbed oxygen (OH, CO_3, etc.) and organic species, using the survey spectrum to identify heteroatoms with potential contributions to oxygen, and employing tools such as that developed by Henderson *et al.*,[65] is sufficient. No component should be included to represent O_V (indirectly or otherwise). Even in instances where the experiment is performed in-operando and O_V are expected, the expected BE shift relative to the main oxide peak for O adjacent to an O_V (Group 5) is so small that the contribution to O 1s cannot be resolved from the O^{2-} component.

1. List of methods used in the literature for using XPS to characterize O_V and our recommendations

1. *Relying on BE shifts of the photopeaks* (**Not** recommended):
    a. Examples examined (e.g., in Sec. VI) suffer from obvious errors in charge correction.
    b. It is possible that this approach is borrowed from alloying studies, where shifts in BE between the main photopeaks of the two elements denote charge sharing.[502–504] As noted recently by Du and co-workers,[505] this approach is

   controversial as for many examples in the literature: BE shifts are the result of inconsistent charge corrections rather than due to intrinsic properties, as noted above in 1.a.
   c. There are other potential causes for BE shifts including differential charging and band bending, as summarized in the Conclusion of the JECS paper.[76] Even experienced experimentalists can struggle to confirm such cases when the magnitude of the influence is minor, while inexperienced researchers will have difficulty identifying these alternative causes simply by being inexperienced (i.e., not knowing what they do not know). Noting that reported BE shifts can be as low as 0.1 eV (or less), which is within the typical error range associated with BE referencing, the level of uncertainty can be far too high even if carried out under appropriate conditions (i.e., in-operando).
   d. This is one of the recommended approaches in the JECS paper labeled "Probing oxygen vacancies via Fermi level shift analysis." In the examples presented by the authors, the researchers are relying on an indirect measure of the Fermi edge by measuring a related shift of a photopeak. As noted above in 1.a.–c., such peak shifts can be induced by other intrinsic properties, or poor experimentation or data processing.

2. *Identification of a particular oxidation state* (Recommended **only** under in-operando conditions):
   a. Researchers should be mindful that oxidation states can exist for other, more likely, reasons, e.g., Ref. 74.
   b. As recommended in the JECS paper, we only recommend if measured in-operando, applying a carefully planned methodology that prevents unintentional oxidation and can provide evidence of O_V, such as the work presented by Rogala *et al.*[31]
3. *The ratio of adsorbed oxygen (O_{ads}) to lattice oxygen (O_{latt}), (ii) correlating O_{ads}/ O_{latt} and O_2-TPD results (for catalytic performance) or (iii) Relying on O_{ads} (or hydroxyls or water), as an indirect, 1:1 measure of O vacancies* (**Not** recommended):
   a. Inconsistency in the peak fit employed within the literature (see Sec. VIII) makes comparison between different authors essentially impossible. Parameters include:
      i. The number of components used to represent the region in question differs, ranging from 1 to 4.
      ii. Variation in component FWHM and positions.
      iii. Variation in lineshapes.
   b. Peak fits typically do not consider all contributions to this region of the spectrum, including carbon–oxygen species (or any other cation–oxygen species, e.g., SiO_x), hydroxyls, and bound water. Thus, values for O_{ads} are typically inflated.
   c. Herein, we report poor correlation between XPS and O_2-TPD results (Sec. VII).
   d. O_{ads} groups are present on metal oxides where O_V are not expected and, thus, would likely be present irrespective of whether their catalyst has (or had) an abundance of O_V.
   e. Inconsistency in the significance of O_{ads}/O_{latt} or O_{latt}/O_{ads} within literature.
      i. Examples exist where either of the ratios stated above result in a relatively large value for their candidate material is used as evidence of superior catalytic performance.
      ii. No standardized comparison exists. Authors will compare values within a dataset but rarely, if ever, compare with other reported values.
   f. Considering the points above, there is no evidence that a relatively high amount of O_{ads} is correlated with O_V concentration for *ex situ* XPS, and is not recommended.
4. *Direct measure of O_V* (**Not** recommended):
   a. There is no O atom; thus there is no photoelectron ejected that can be measured. This assignment should not be used under any circumstance.
5. *O atoms adjacent to/in the vicinity of an O vacancy or "low coordination oxygen species"* (**Not** recommended):
   a. DFT calculations demonstrate that the shift is nonuniform and too small to differentiate from oxide contribution (Sec. IX A.).
   b. Carefully executed in-operando XPS results indicate that contributions to the region of the O 1s spectrum are not associated with this assignment, e.g., Ref. 31.
6. *Comparison of O/(metal cation) ratios from quantification* (Recommended **only** under in-operando conditions):
   a. This is an acceptable approach if the experiment is carefully designed and performed in-operando, for example, see the work of Kucharski *et al.*[506]
   b. This is one of the recommendations in the JECS paper under "Probing oxygen vacancies via oxygen non-stoichiometry." There are, however, two issues: first, the authors do not explicitly state the need for in-operando experimentation. Second, some of their examples are not recommended, such as relying on normalized peak intensity. Atomic ratios derived from survey data (and supplemented with high-resolution data if required) are necessary to ensure accurate comparison.
7. *Does not rely on XPS to demonstrate O vacancy* (**Only option** if performing *ex situ* XPS—this is most of the literature):
   a. This is the only acceptable approach in all cases where the sample has been exposed to air, or when the equipment required for in-operando measurements is not available. Most authors should rely on this option and utilize other *in situ* methods for identifying O_V rather than attempting to rely on XPS.

B. Closing comments

The authors of the J. Mater. Chem. A review (Sec. VI) identified three approaches for using XPS to identify O_V. [Groups 1 to 3 (ii)], while ignoring the most common approach (Group 4—direct measure). It is possible that the authors were attempting to avoid the most erroneous example and to limit the length of their discussion or were simply not aware of the other approaches. However, one must be impressed at the number and sheer diversity of the available interpretations in the literature to interpret O_V from XPS data. This alone should be a cause for concern for the community as it is not common to have several different ways to analyze a specific piece of data and some of these approaches are mutually exclusive. It is also possible that additional approaches exist within the literature that have not been identified herein, and that new approaches will develop as the application of Idriss's work forces authors to reevaluate their approach.

Reflecting on this study, the two largest or most significant issues observed are that the authors applying the approaches described above (1) are not assessing the validity and applicability of the approaches they are using, and (2) a reluctance to change their approach when faced with an unambiguous assessment demonstrating that the approach is erroneous. Materials science and engineering has become complex in mature fields such as catalysis. It requires the application of many different techniques to demonstrate superior performance of their material in the intended application. Coupled with the "publish or perish" mindset prevalent in academia, one can understand why researchers would take the "easy" approach and follow the methodologies employed by their colleagues in the field. However, the fact that those applying these approaches did not feel it was necessary to confirm their application was experimentally sound is alarming. One could argue that the esteem of the author of the original assignment meant that the authors applying these methods felt confident in the assignment. However, what this and seemingly all authors applying these approaches have failed to recognize is that any material exposed to atmosphere will oxidize and these highly reactive O_V groups will heal. It is disappointing that seemingly a large proportion of the catalyst field has neglected to consider this rather mundane piece of surface science knowledge.

Considering the outcome of the manuscript examined in Sec. IX A,[77] it is unlikely that further *in situ* studies will reveal clear evidence of O_V using XPS. Only measurements performed in-operando have the potential to demonstrate O_V due to the reactivity of these oxygen-deficient states. Considering the (lack of) general availability of such instrumentation and the complexity of the methodology required to achieve a positive result, it is likely that only researchers with sufficient motivation, time, and resources will rely on the recommended approaches outlined above. Catalysis researchers need to cease attempts to find alternative methods to use XPS, *ex situ* or otherwise, for O_V identification without providing the adequate proof of concept. There are other reasons why you should collect (*ex situ*) XPS data of your samples, such as confirming the surface stoichiometry before catalysis and that no contaminants are present before or after catalysis, as this may influence the results of your performance tests. Prior to the overreliance on high-resolution spectra commonly found in papers today, our colleagues utilized atomic ratios from XPS to determine coke distribution on ZSM-5,[507] and the distribution of Sn in aqueous impregnated γ-Al_2O_3 supported catalysts.[508] These reasons alone provide sufficient reasoning for undertaking surface chemical analysis of your catalytic sample without attempting to "overinterpret" the results to draw additional information based on flawed science.

XI. CONCLUSIONS

XPS is an important tool for characterizing the surface chemistry of materials. However, in the most common form, *ex situ* XPS, it cannot be used to directly identify or quantify oxygen vacancies. Indirect observations related to oxygen vacancies are only feasible if performed in-operando and the experiments are correctly designed. Far too many publications are relying on flawed science while citing work that directly contradict their approach. Innovation is thereby stifled as researchers have a false sense of understanding of their materials. Before relying on a particular approach, researchers should undertake due diligence by confirming it has been validated. Herein, we have shown that many of the approaches utilized in the literature are flawed even if performed in-operando. Of the remaining two, it is unlikely that they become a common staple in catalysis papers due to the highly specialized equipment and the complexity of the experimentation required. There are, however, very good reasons why XPS should be utilized in catalysis. Despite the shortfalls identified herein, readers are encouraged to seek help from specialists in the field when applying this surface sensitive technique to their materials.

SUPPLEMENTARY MATERIAL

The supplementary material is comprised of the results of the two tables presented in the main text, specifically Tables I and II, prior to any grouping.

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AUTHOR DECLARATIONS

Author Contributions

Christopher D. Easton: Conceptualization (lead); Data curation (lead); Formal analysis (lead); Investigation (lead); Methodology (lead); Project administration (lead); Visualization (lead); Writing – original draft (lead); Writing – review & editing (lead). **David J. Morgan:** Investigation (supporting); Visualization (supporting); Writing – original draft (supporting); Writing – review & editing (supporting).

Conflict of Interest

There are no conflicts of interest.

DATA AVAILABILITY

The data that support the findings of this study are available within the article and its supplementary material.

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