

Electrochemical Passivation and Morphological Characterization of Tungsten Anodic Films in Different pH Environments

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Abstract

This study investigates how electrolyte pH and applied anodic potential influence the qualitative electrochemical response and surface-visible morphology of tungsten anodic films. Four aqueous electrolyte conditions that represented strongly acidic, neutral, mildly alkaline, and strongly alkaline conditions were used to anodically polarize the tungsten working electrodes (0.25 x 20 mm exposed dimension): 0.1 M H₂SO₄ (pH 1), phosphate buffer (pH 7), phosphate buffer (pH 9), and 0.1 M NaOH (pH 13). An anodic response region was identified using potentiodynamic polarization, and scanning electron microscopy (SEM) was used to examine the resulting surface morphology at selected potentials. The results of the SEM analysis indicate that pH and applied potential jointly influence surface-observable characteristics. Rougher, porous-appearing regions of the surface were more apparent in strongly acidic and strongly alkaline media. Approximate surface-opening sizes included 125-135 nm at pH 1 and 0.75 V, 126-300 nm at pH 9 and 0.275 V, 200-700 nm at pH 9 and 0.4 V, and 105-600 nm at pH 13 and selected potentials. However, the interpretation of polarization curves is based solely on qualitative considerations because the current data do not permit Tafel analysis, electrochemical impedance spectroscopy, gas-sensing tests, oxide-thickness measurements, X-ray Diffraction, or X-ray Photoelectron Spectroscopy. Thus, the results can be interpreted as indicating pH- and potential-dependent tungsten surface modification, but not as a direct validation of the presence of methane in the air. The research is relevant because the ability to control tungsten surface morphology is an essential preliminary step for applications that require surface activity, such as future tungsten-based sensing and electrochemical-device optimization.

Categories: Advanced Materials, Catalysis and Reaction Engineering, Materials Engineering

Keywords: potentiodynamic, polarization, morphological, tungsten, anodic films

Introduction

Materials containing transition metals are significant in sensing, electrocatalysis, energy storage, optoelectronics, and photoelectrochemical systems because their surface chemistry and morphology can be tuned through electrochemical processing [1-3]. The behavior of non-noble metals in aqueous media is particularly sensitive to the composition of the

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electrolyte, the pH, applied potential, and exposure time. Oxidic, passivistic, local dissolution, roughening, cracking, or the development of porous-looking surface features may occur on a metal surface under anodic conditions [4-7].

Tungsten is a technologically relevant transition metal due to its high thermal stability, electrochemical activity, and the ability to form surface oxide species in an anodic environment. It has been studied using tungsten- and tungsten oxide-based materials as gas sensors or other surface-dependent devices [8]. But the existence of a surface morphology that can be useful for a sensor is not identical to gas-sensing performance. The gas-sensing validation needs to be exposed to target gases, with measurements of response and recovery, selectivity analysis, repeatability, and sensitivity testing. These experiments were not part of the current study.

The gap addressed here is thus rather narrow and well-defined: the research paper discusses the effect of electrolyte pH and applied potential on the electrochemical response and the subsequent surface morphology of anodically treated tungsten, as observed by scanning electron microscopy (SEM). Past literature has examined tungsten oxide and electrochemical surface modification, whereas less has addressed the gas-sensitive tungsten surface and morphology under anodic polarization in acidic, neutral, mildly alkaline, and strongly alkaline electrolytes.

This work aims to correlate the qualitative polarization behavior of tungsten with the morphology observed by SEM after anodic treatment in the presence of the following electrolytes at pH 1, 7, 9, and 13. The objectives are as follows: (i) to prepare tungsten specimens under specified pH conditions of electrolytes; (ii) to record potentiodynamic polarization responses under a scan rate of 1 mV s^{-1} ; (iii) to examine selected polarized surfaces using SEM; and (iv) to identify pH- and potential-dependent morphology trends and clearly state the limitations of SEM-only and qualitative electrochemical evidence. The practical significance of the research is that controlled surface modification is an early-stage need for designing tungsten surfaces for utilization later in sensing, catalytic, and electrochemical applications.

Materials And Methods

Materials and electrode configuration

As the working electrode/anode, Tungsten wire was used; as the counter electrode/cathode, platinum was wire. The dimensions of the tungsten electrode, which were exposed in the electrochemical figures, were follows: $0.25 \times 20 \text{ mm}$. The word specimen is constantly used throughout this manuscript to denote each surface of a tungsten electrode that has been subjected to electrochemical treatment. Before anodic testing, samples were polished before being rinsed in deionized water. Since deep parallel striations were observed in the SEM images, this interpretation treats such linear marks with caution, treating them as possible polishing or preparation features rather than features of anodic crack formation. When discussed as either "cracked" or "fissured," the term refers only to surface-visible features in the SEM images, not to established through-thickness cracks.

Electrolyte preparation and pH selection

The electrolyte set was chosen to span the major aqueous pH regimes of interest for tungsten surface stability: strongly acidic, neutral, mildly alkaline, and strongly alkaline. The pH 1 electrolyte was $0.1 \text{ M H}_2\text{SO}_4$ and was used to investigate tungsten behavior in a highly acidic sulphate environment. Phosphate buffers were prepared to give near-neutral conditions and mildly alkaline conditions with more stable pH control: pH 7 and pH 9. The electrolyte used was 0.1 M NaOH (pH 13.0) to study strongly alkaline behavior. These various reagents were thus utilized to generate controlled pH conditions and not to measure reagent-specific effects. All the solutions were purged with nitrogen at approximately 21.9°C to remove dissolved oxygen before electrochemical analysis.

Reference electrode and electrochemical limitations

The electrochemical cell consisted of a tungsten working electrode, a platinum counter electrode, and an Ag/AgCl reference electrode prepared in the laboratory. The electrode used was Ag/AgCl, prepared by anodizing silver in 0.1 M HCl . The potentials reported are expressed in V vs Ag/AgCl, as shown on the figure axes. One limitation is that the reference electrode system did not use a saturated KCl salt bridge, in which chloride activity was fixed. Thus, the absolute

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potential values could be influenced by reference-potential drift in sulphate/phosphate/hydroxide media. To this end, the revised manuscript will not over-compare absolute potentials across different pH solutions and will utilize the polarization curves to assign qualitative areas of anodic response based on features observed on the surface by SEM.

Potentiodynamic polarization and SEM analysis

Anodic modification of tungsten at a scan rate of 1 mV s^{-1} was induced by potentiodynamic polarization. Specific samples were taken off following polarization at the given potentials and analyzed using SEM. The surface morphology was observed using a HITACHI S-540 scanning electron microscope at an accelerating voltage of 20 kV and a spot size of 3. SEM is used only to obtain information about the topography visible on the surface. It is not a direct determination of oxide thickness, subsurface structure, crystallographic phase, chemical composition, or gas-sensing behavior. The discussion, thus revised, does not assert any methane detection performance, nor does it treat the polarization curves as providing any quantitative performance for methane detection.

Sample preparation for SEM and instrumentation

A SEM (HITACHI S-540) was used to determine the surface topography of anodically processed tungsten specimens. The samples were placed on conductive stubs and cleaned with a stream of nitrogen before imaging. Their introduction was into a vacuum chamber, which was electrically grounded to remove charging effects, and the observations were made at an accelerating voltage of 20 kV with a spot size of 3 (see Figure 1).

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FIGURE 1: HITACHI S-540 SEM instrument used for surface imaging of anodically treated tungsten specimens

SEM, scanning electron microscopy

Results And Discussion

Overall pH-dependent morphology trends

Across the range of pH values examined, tungsten surface morphology depended on both the electrolyte pH and the anodic potential. The interpretation of the SEM observations is based on qualitative surface-visible morphology rather than on evidence of oxide phase, film thickness, or gas-sensing behavior. This difference is noteworthy, as it has been noted that gas-sensing claims should include gas exposure, sensitivity, selectivity, and response-recovery measurements, which were not included in the present work.

When polarized at lower potentials in the pH 1 sulfuric acid electrolyte, specimens exhibited relatively compact surface appearances. The surface then became more irregular, with shallow depressions or openings due to the electric field occurring in the region of about 125-135 nm. Figures 2 and 3 support these observations by showing the images of pH 1 SEM at 0.2 V, 0.4 V, 0.55 V, and 0.75 V, respectively. Since the photographs also reveal deep parallel striations, the new interpretation does not consider each linear element as a crack anodic. Part of these marks can be due to mechanical polishing or preparation of a specimen (Figures 4 and 5).

The tungsten surface in phosphate buffer at pH 7 was largely smooth or compact across the series of potentials tested. Local discontinuities and roughened areas were observed at selected potentials associated with passivation, and the apparent feature dimensions were reported to be approximately 200-700 nm. These pH 7 changes are associated with Figures 6 and 7, which show the cyclic voltammetric response and the corresponding SEM observations at 0.2, 0.4, 0.8, and 1.0 V.

The surface tended to be smoother in the phosphate buffer (pH 9) than in either the strongly acidic or strongly alkaline electrolytes. The pH 9 electrochemical features were identified at 0.275, 0.4, 0.70, and 1.0 V as indicated in Figure 8. The plateau-like section of the curve is viewed with caution, as it may reflect the instrument's current levels of saturation and compliance rather than a true kinetic plateau.

Morphology was more heterogeneous in the electrolyte sodium hydroxide (pH 13). The presence of surface openings with a mean of about 105-600 nm was observed at the anodic potentials of interest, and their distribution was not uniform. The highly alkaline environment thus seemed to favor irregular surface modification in contrast to neutral and mildly alkaline environments. It was also found in Figure 9 of the original manuscript that there was an unnatural step-like polarization response; this has been treated as a limitation, as the shapes might be due to instrument range/compliance artifacts rather than true electrochemical events. Table 1 summarizes the pH and potential-dependent morphology.

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Electrolyte condition	Potential(s) discussed	Main SEM-observed morphology	Approximate feature size	Interpretive limitation
0.1 M H ₂ SO ₄ , pH 1	0.2, 0.4, 0.55, 0.75 V	Compact to roughened; shallow depressions/openings at higher potential	125-135 nm at 0.75 V	Parallel striations may include polishing marks
Phosphate buffer, pH 7	0.2, 0.4, 0.8, 1.0 V	Mostly smooth/compact with local roughened areas	About 200-700 nm in selected regions	Qualitative SEM interpretation only
Phosphate buffer, pH 9	0.275, 0.4, 0.70, 1.0 V	Generally smoother; selected openings at lower/intermediate potentials	126-300 nm at 0.275 V; up to 200-700 nm at 0.4 V	The voltammogram plateau may include an instrument artifact
0.1 M NaOH, pH 13	-0.075, 0.10, 0.25, 0.80 V	Heterogeneous surface with irregular openings	About 105-600 nm	Step-like curve features interpreted cautiously

TABLE 1: Summary of pH- and potential-dependent morphology

SEM, scanning electron microscopy

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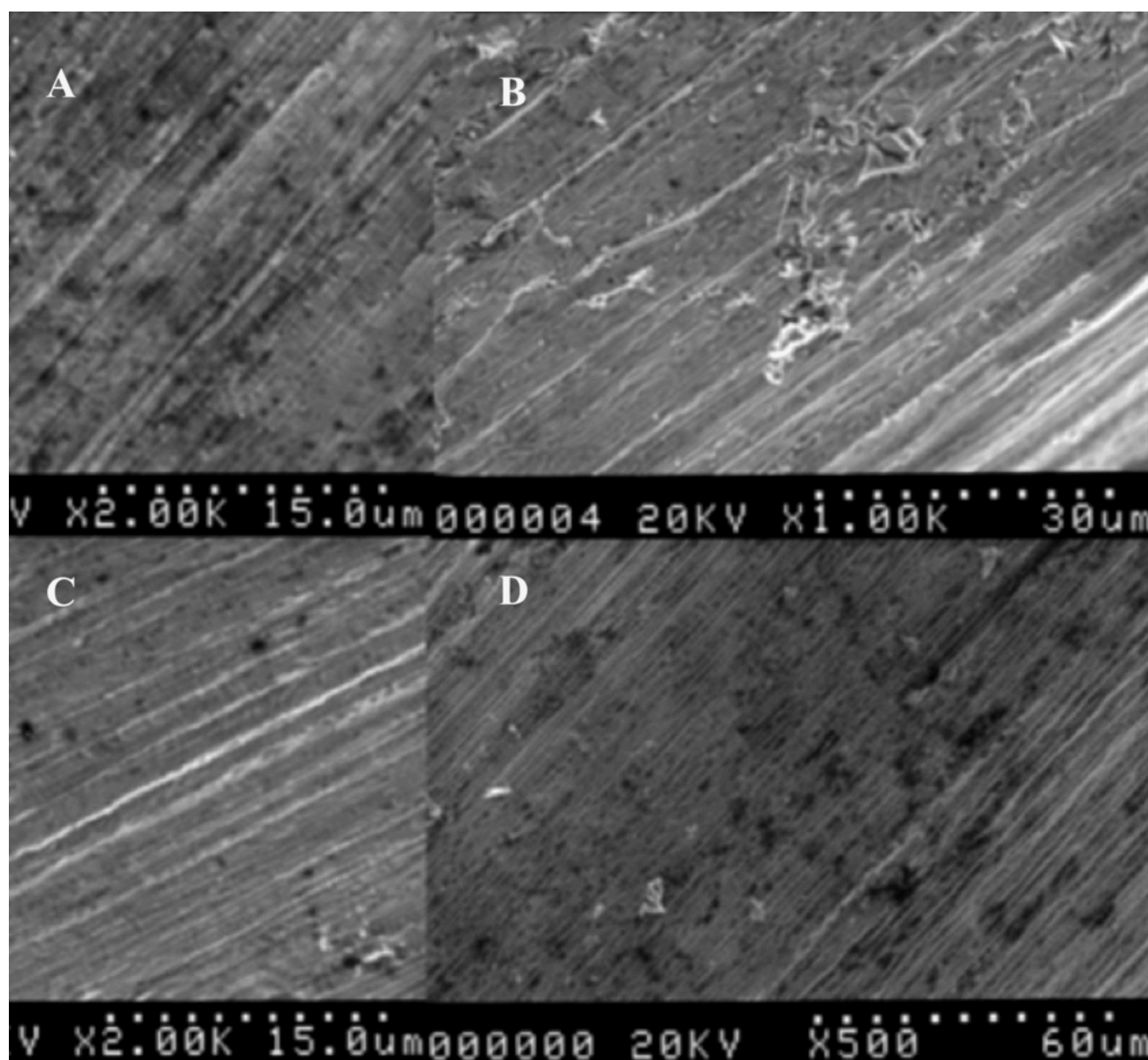


FIGURE 2: SEM images showing surface morphology of anodically treated tungsten in 0.1 M H₂SO₄ (pH 1) at applied potentials of 0.2 V (A), 0.4 V (B), 0.55 V (C), and 0.75 V (D). Images are interpreted qualitatively; the visible parallel striations may include polishing-related marks and should not all be assigned to anodic cracking

SEM, scanning electron microscopy

The discussion of pH 1 is supported by Figure 2, which shows a shift from relatively compact regions to more irregular surface areas as the anodic potential increases. This figure also shows why one should be careful: some of the linear striations can be observed across several panels and perhaps indicate the presence of preparation marks rather than electrochemical cracking alone.

Figure 3 illustrates SEM images of anodically treated tungsten in 0.1 M H₂SO₄ (pH 1) at applied potentials of 0.2 V (A), 0.4 V (B), and 0.55 V (C). The panels illustrate surface-visible roughening and local irregularities under acidic conditions. SEM observations do not provide phase, oxide-thickness, or chemical-composition information.

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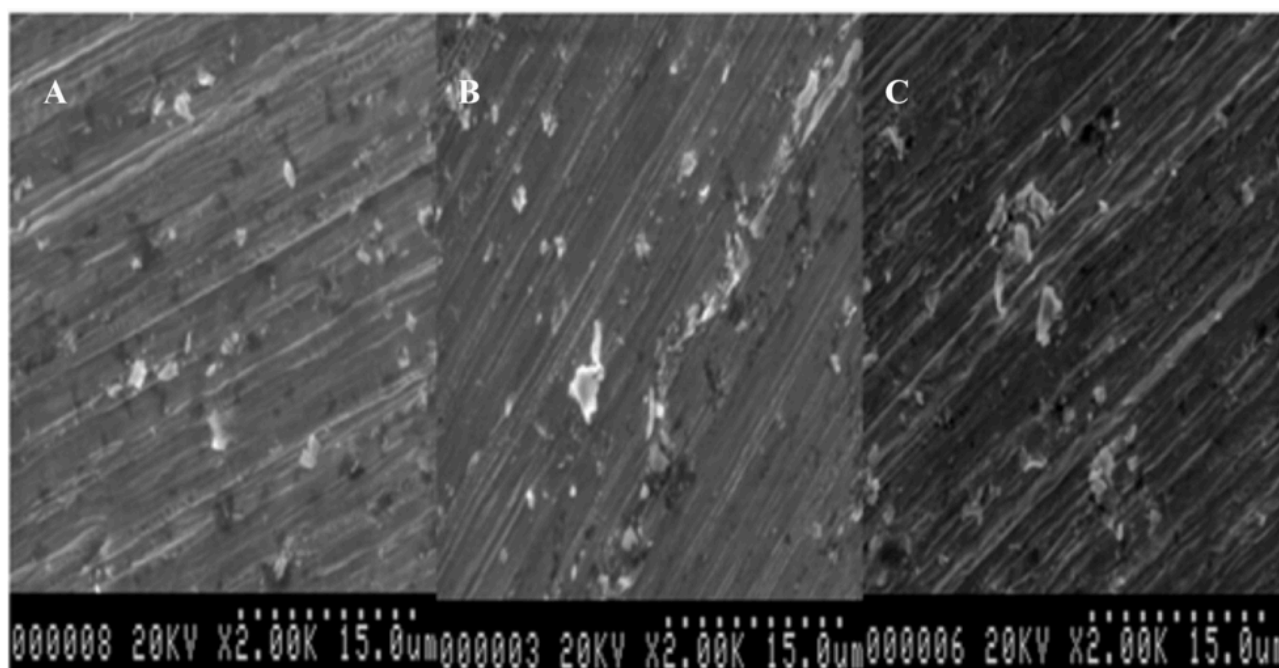


FIGURE 3: SEM images of anodically treated tungsten in 0.1 M H₂SO₄ (pH 1) at applied potentials of 0.2 V (A), 0.4 V (B), and 0.55 V (C). The panels illustrate surface-visible roughening and local irregularities under acidic conditions. SEM observations do not provide phase, oxide-thickness, or chemical-composition information

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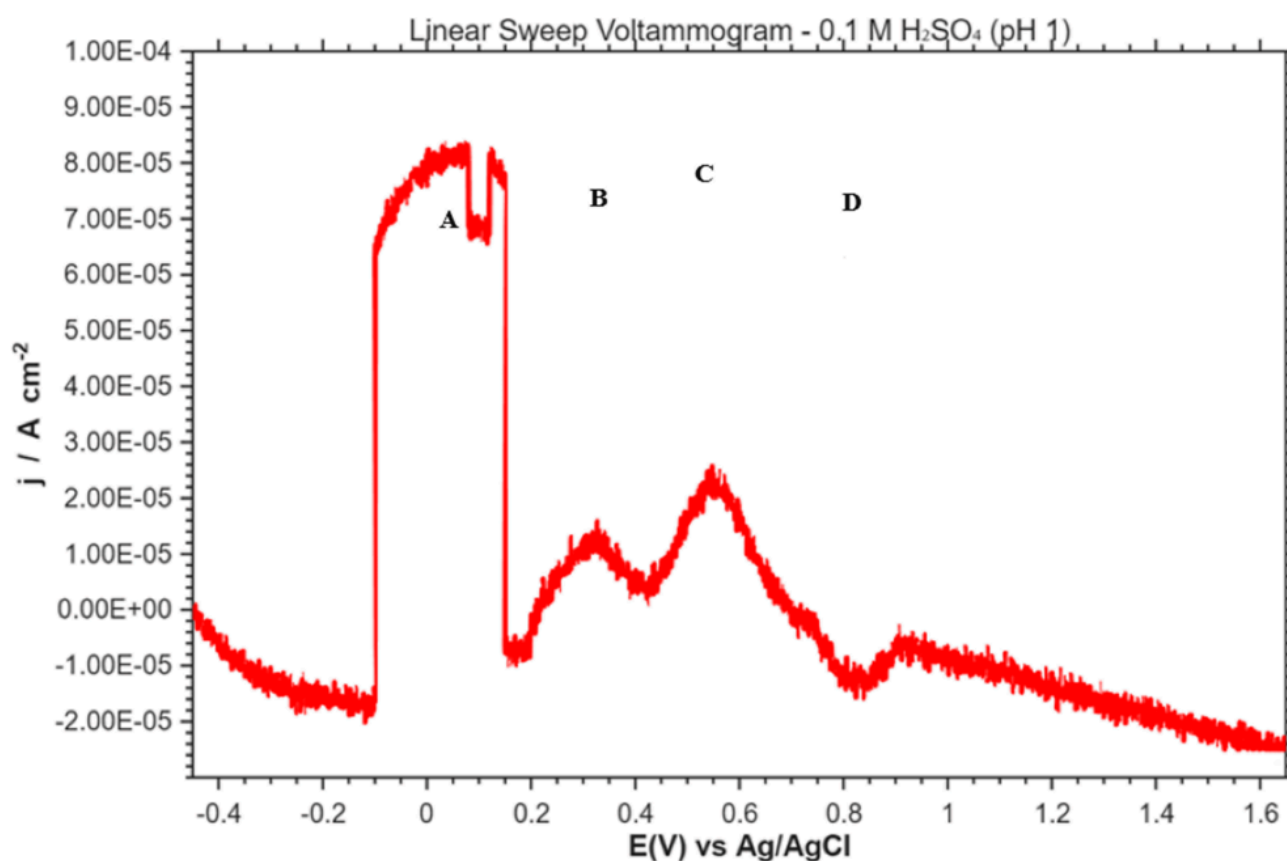


FIGURE 4: Linear sweep voltammogram of a freshly polished tungsten electrode (0.25 x 20 mm) recorded in 0.1 M H₂SO₄ (pH 1) at 1 mV s⁻¹. Features are used only as qualitative indicators for selecting SEM observation potentials; the rectangular/flat response region may include instrument compliance or current-range artifacts

SEM, scanning electron microscopy

Figure 4, pH 1 voltammogram, shows that during the anodic sweep, the current density changes, but Tafel slopes, corrosion current density, passivation current density, and oxide-growth kinetics are not extracted. The sharp and flat areas of the curve are handled with caution; it may indicate a potential compliance or clipping artifact.

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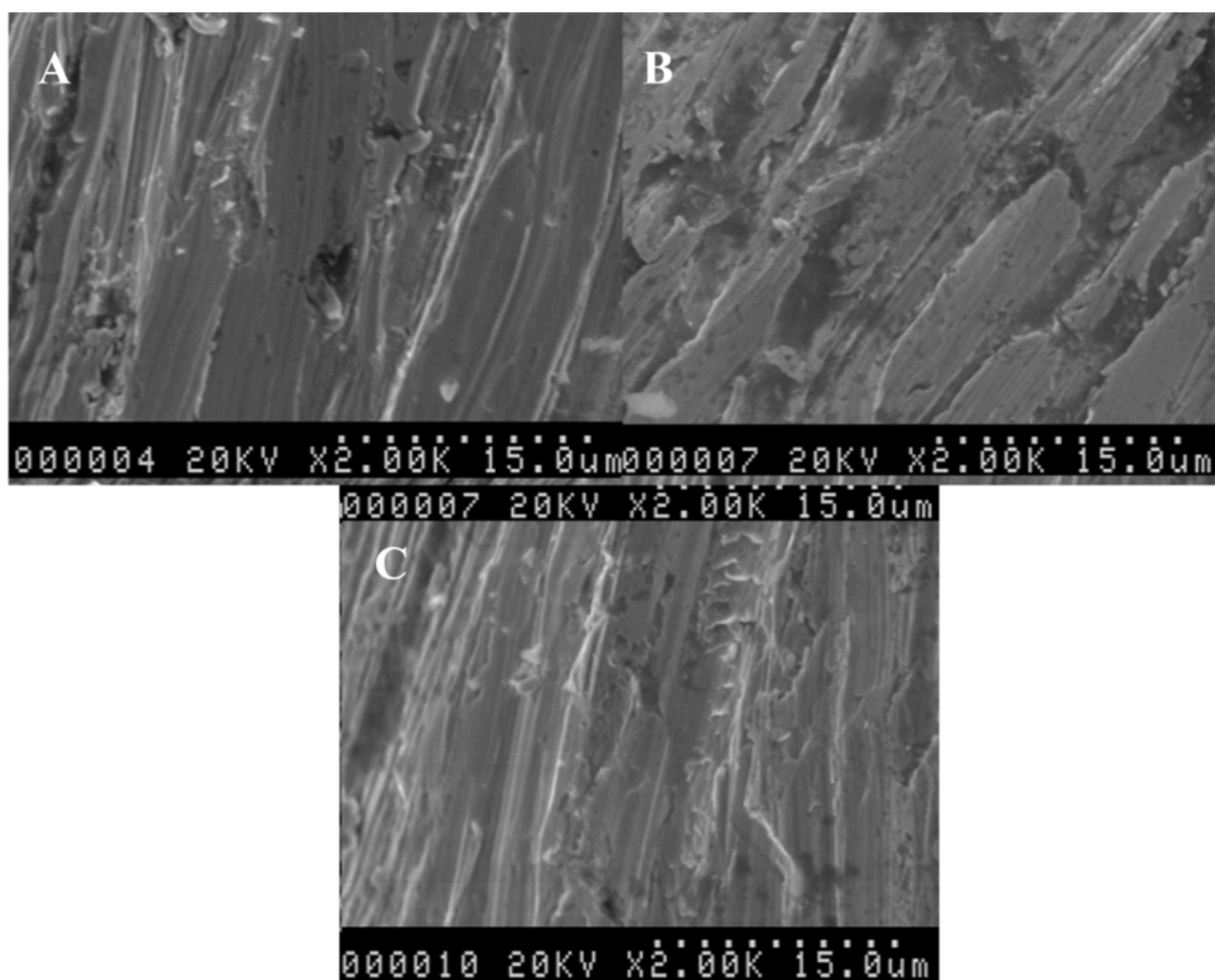


FIGURE 5: SEM images of anodically treated tungsten in 0.1 M H₂SO₄ (pH 1) at 0.2 V (A), 0.4 V (B), and 0.55 V (C), arranged clockwise from the top left. The panels show surface-visible morphology after selected acidic polarization conditions; interpretation is limited to SEM-observable topography

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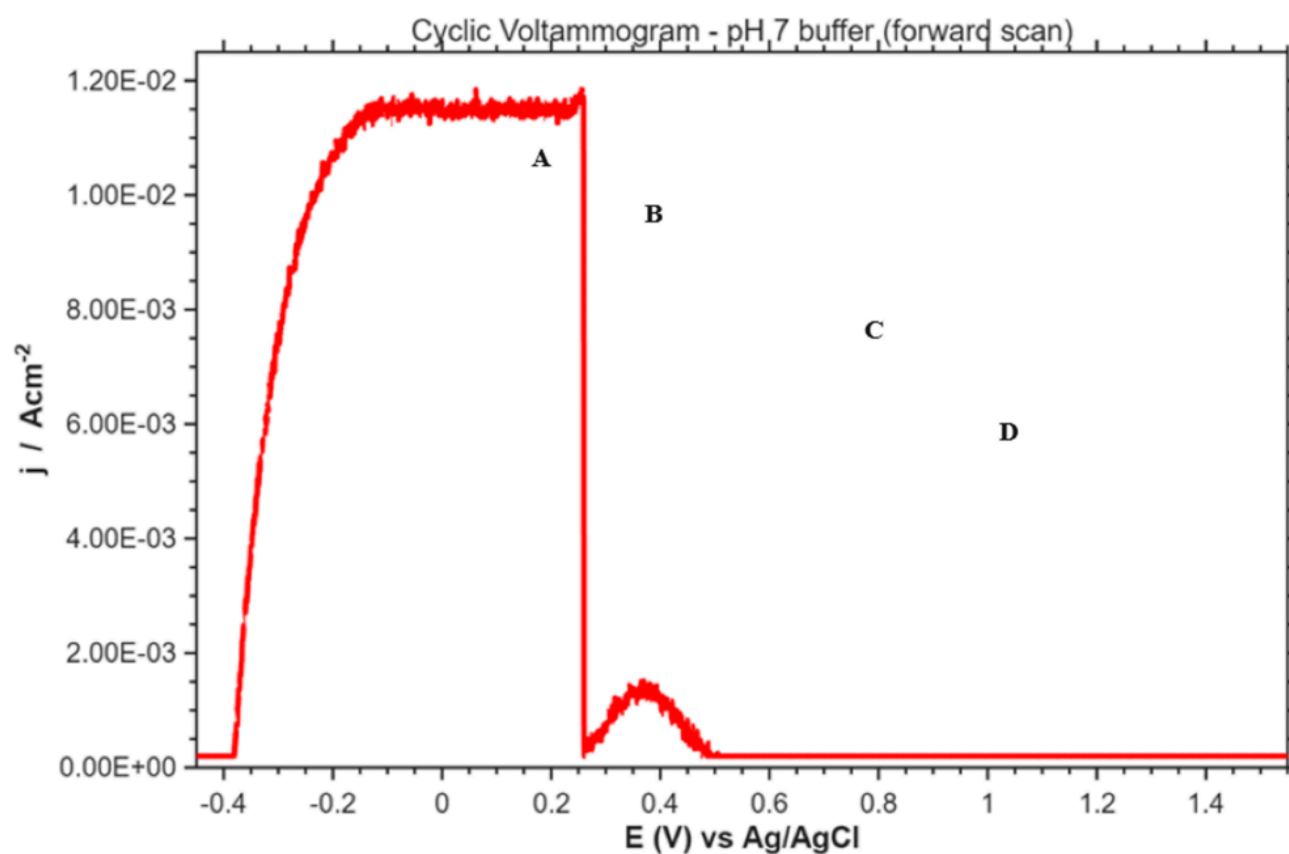


FIGURE 6: Cyclic voltammogram of a freshly polished tungsten electrode (0.25 x 20 mm) in phosphate buffer (pH 7; 0.1 M $\text{H}_2\text{NaO}_4\text{P} \cdot \text{H}_2\text{O}$ + 0.1 M $\text{HNa}_2\text{O}_4\text{P} \cdot 2\text{H}_2\text{O}$) at 1 mV s^{-1} . Labels I-IV correspond to 0.2, 0.4, 0.8, and 1.0 V, respectively, and are qualitatively associated with SEM-observed surface changes

SEM, scanning electron microscopy

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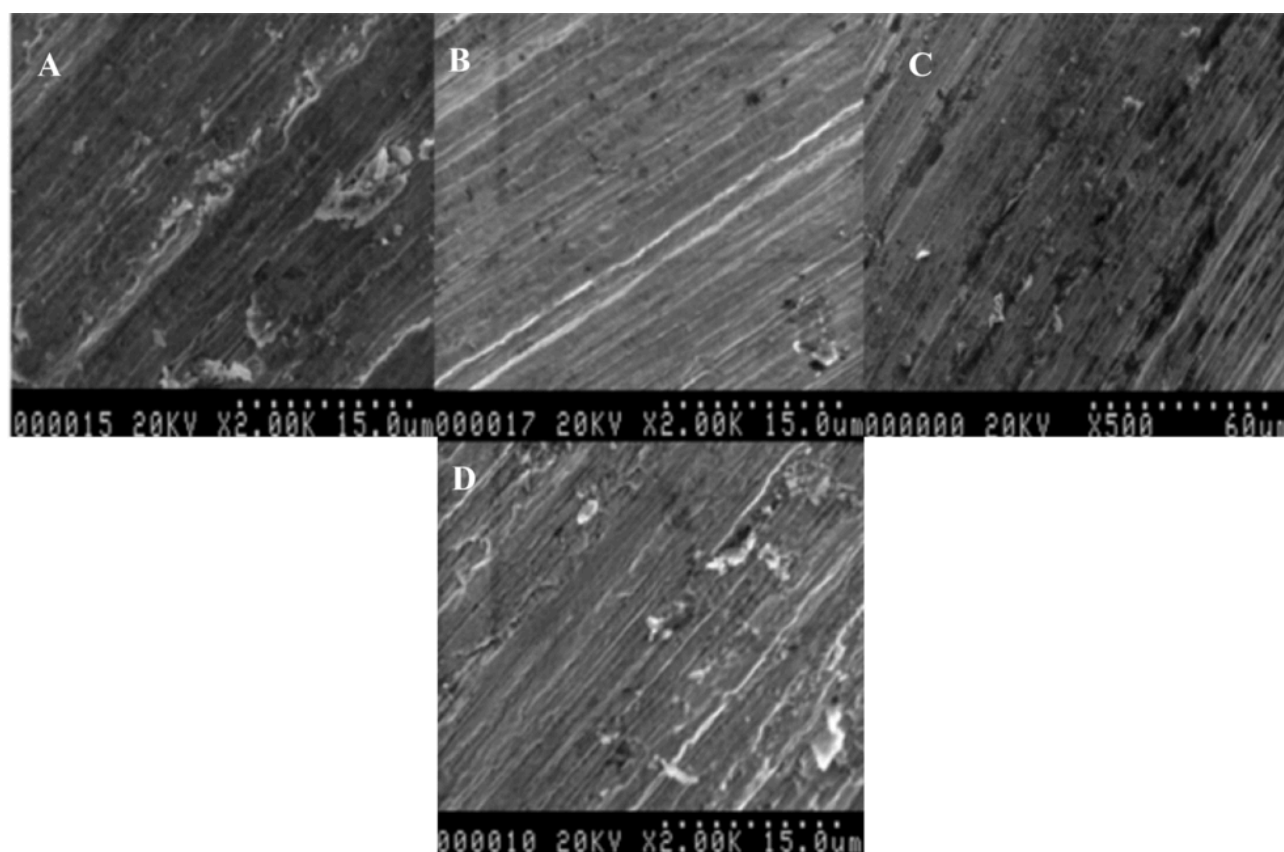


FIGURE 7: SEM images of anodically treated tungsten in phosphate buffer at pH 7, recorded at 0.2 V (A), 0.4 V (B), 0.8 V (C), and 1.0 V (D). The panels show mainly compact/smooth morphology with localized surface irregularities; all comparisons are qualitative because not all original images were acquired for strict same-magnification comparison

SEM, scanning electron microscopy

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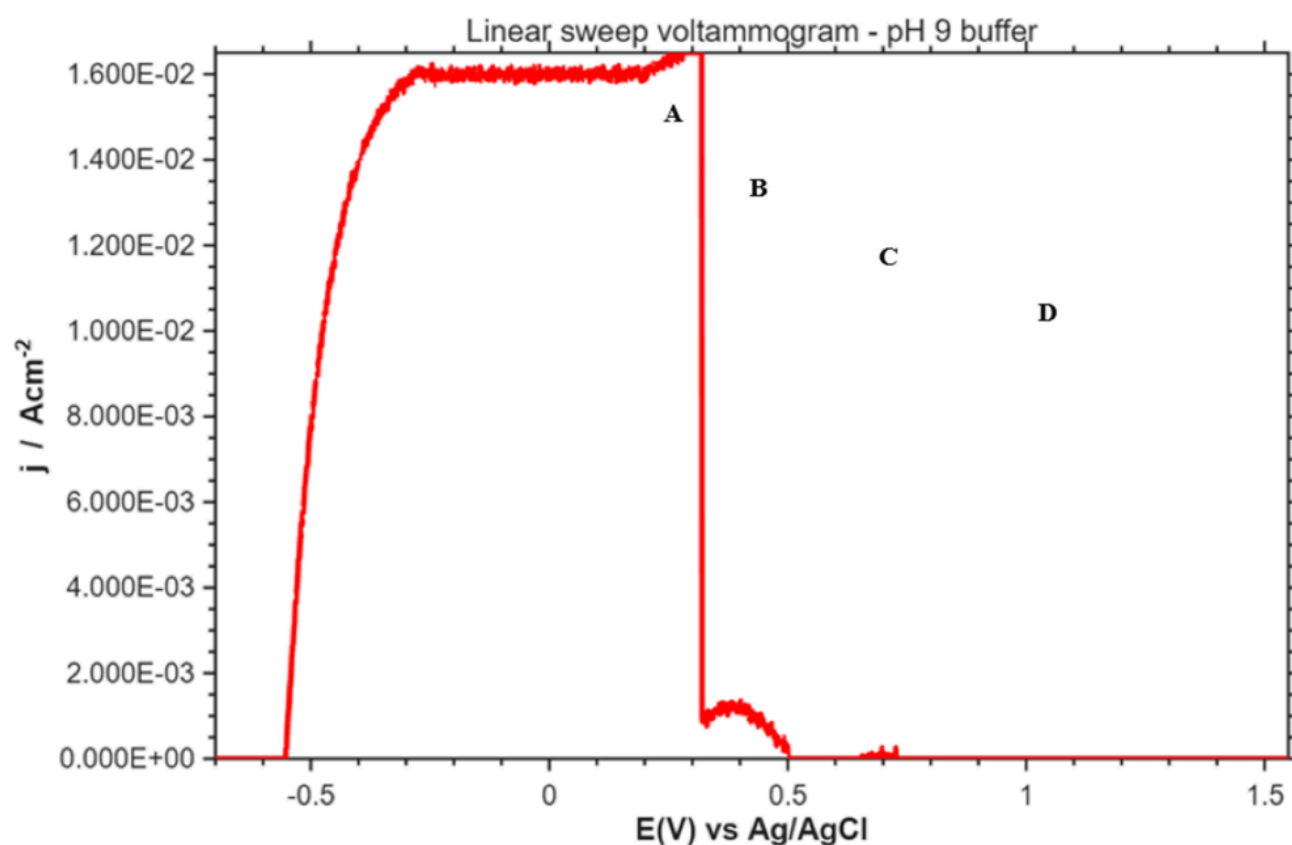


FIGURE 8: Linear sweep voltammogram of a freshly polished tungsten electrode (0.25 x 20 mm) in phosphate buffer (pH 9; 0.1 M $\text{HNa}_2\text{O}_4\text{P}_2\text{H}_2\text{O}$ + 0.1 M $\text{Na}_3\text{O}_4\text{P}_2\text{H}_2\text{O}$) at 1 mV s⁻¹. Labels I-IV correspond to approximately 0.275, 0.4, 0.70, and 1.0 V. The plateau-like region is interpreted cautiously as a possible instrument-range artifact

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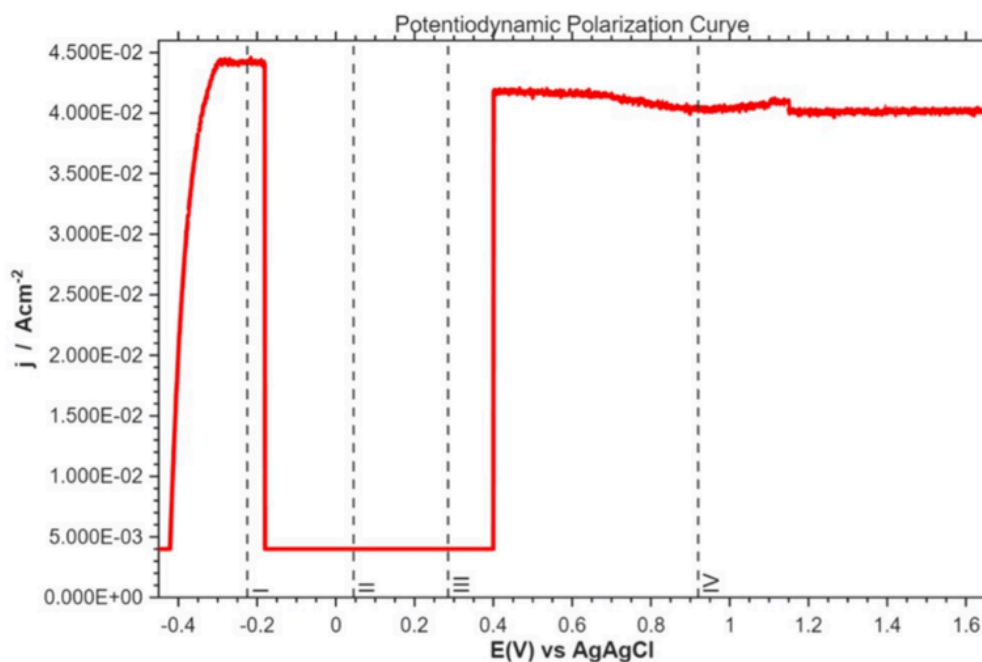


FIGURE 9: Linear sweep voltammogram of polished tungsten in 0.1 M NaOH (pH 13) showing electrochemical features associated with surface morphology changes.

Relationship between electrochemical response and surface morphology

These findings indicate that tungsten surface appearance in anodic polarization depends on pH and applied potential. The surface tended to exhibit more irregular and porous-looking structures at the chosen potentials in strongly acidic and strongly alkaline environments. Smoother or compact surfaces were more common in neutral or slightly alkaline circumstances. These tendencies can be attributed to the general assumption that the local pH and applied potential jointly influence oxide formation, passivation, and dissolution. Nevertheless, the current evidence is insufficient to determine the exact oxide-growth mechanism, dissolution pathway, crystallographic phase, or chemical state of the anodic film because XRD, XPS, oxide thickness measurements, and chemical state measurements were not performed.

The polarization curves are thus taken as qualitative indicators rather than quantitative kinetic indicators. No reported Tafel analysis, corrosion potential, corrosion current density, passivation current density, impedance spectrum, or statistical reproducibility analysis was performed. This eliminates the need for vigorous mechanistic assertions about corrosion/passivation kinetics. The new meaning is intentionally restricted to the correlation between general areas of anodic responses and the morphology visible on the surface in SEM images.

The practical importance of the work is that controlling surface morphology is significant for technologies whose functioning is sensitive to the active surface area, surface defects, adsorption sites, and charge-transfer behavior. To develop future methane or hydrocarbon sensors, even tungsten surfaces would have to be converted to known oxide phases and tested under controlled gas exposure. Therefore, the current study presents some initial surface-modification data which could be used to optimize subsequent sensor-material development, but does not represent an operational methane sensor.

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Limitations

The study is limited to qualitative potentiodynamic polarization and SEM-based surface morphology. It does not involve testing gas-sensing properties, sensitivity, selectivity, response/recovery time, limit of detection, repeatability, or long-term stability.

Possible drift across electrolytes may be caused by the use of a laboratory-prepared Ag/AgCl reference electrode and no saturated KCl salt bridge was used. Hence, no over-comparison should be made between pH 1, 7, 9, and 13 solutions in terms of absolute potentials.

SEM images depict visible linear striations, which may result from mechanical polishing or preparation. Because of this, there is a cautious discussion of the crack-like features, without necessarily providing them as purely anodic in nature.

The electrochemical curves contain flat or stepped profiles that could indicate instrument conformity, current saturation, or range-resolution artifacts. This is why the curves cannot be used for quantitative kinetic analysis.

The morphology will not determine tungsten oxide phase, crystallinity, film thickness, or chemical composition. Such claims would require X-ray Diffraction (XRD), X-ray Photoelectron Spectroscopy (XPS), Raman spectroscopy, cross-sectional SEM/Transmission Electron Microscopy (TEM), and thickness measurements to support such claims.

Made clear that no Tafel analysis, corrosion/passivation constants, EIS, oxide-thickness measurement, XRD, XPS, or gas-sensing metrics were measured; conclusions are limited accordingly.

Conclusions

This revised work demonstrates that electrolyte pH and the applied anodic potential influence the SEM-observable surface morphology of tungsten after electrochemical treatment. Experiments at both low and high potentials, under acidic pH 1 and strongly alkaline pH 13 conditions, respectively, showed more heterogeneous and porous-looking features at selected potentials. The approximate ranges of feature size varied with pH and potential, with 125-135 nm at pH 1 and 0.75 V, and larger, scattered features such as 126-300 nm at pH 9 and 0.275 V. These data indicate that the electrochemical environment can affect tungsten surface morphology. Nevertheless, such conclusions can be drawn only from qualitative morphology and general trends in the electrochemical response. The work does not support claims of methane detection, does not quantify corrosion or passivation kinetics, and does not determine the phase or composition of oxide layers. Before any company makes any claim regarding the performance of methane sensors, controlled reference electrodes should be used, repeated specimens should be used, same-magnification SEM comparison should be done, Tafel and impedance analysis, XRD/XPS phase and chemical analysis, oxide-thickness measurements, and direct gas-sensing tests should be performed before any performance claims are made.

Future work

To enhance quantitative reliability, prepare duplicate tungsten specimens, and report mean feature dimensions with standard deviations.

The use of a commercial Ag/AgCl reference electrode with saturated KCl and a salt bridge is recommended to reduce reference-potential drift between different electrolytes.

Record SEM micrographs at matched magnification, working distance, and imaging conditions for all pH values at similar potentials.

Perform Tafel analysis, electrochemical impedance spectroscopy, and repeat scans to obtain reliable corrosion/passivation parameters.

To identify the tungsten oxide phase, chemical state, and film structure, use XRD, XPS, Raman spectroscopy, or TEM.

Measure oxide thickness and surface roughness using cross-sectional SEM, profilometry or Atomic Force Microscopy.

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Perform exposure tests with methane and interfering gases to determine response, recovery, sensitivity, selectivity, reproducibility, and stability.

Additional Information

Author Contributions

All authors have reviewed the final version to be published and agreed to be accountable for all aspects of the work.

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Acquisition, analysis, or interpretation of data: Abdulhakim A. Shittu, Akaninyene C. Offong, Sunday A. Onikanni

Drafting of the manuscript: Abdulhakim A. Shittu, Akaninyene C. Offong, Sunday A. Onikanni

Critical review of the manuscript for important intellectual content: Abdulhakim A. Shittu, Sunday A. Onikanni

Disclosures

Human subjects: All authors have confirmed that this study did not involve human participants or tissue. **Animal subjects:** All authors have confirmed that this study did not involve animal subjects or tissue. **Conflicts of interest:** In compliance with the ICMJE uniform disclosure form, all authors declare the following: **Payment/services info:** All authors have declared that no financial support was received from any organization for the submitted work. **Financial relationships:** All authors have declared that they have no financial relationships at present or within the previous three years with any organizations that might have an interest in the submitted work. **Other relationships:** All authors have declared that there are no other relationships or activities that could appear to have influenced the submitted work.

Data Availability Statements

All data generated or analyzed during this study are included in this published article and/or its appendices.

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