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RESEARCH ARTICLE

INFLUENCE OF ELECTRODEPOSITION POTENTIAL ON COBALT(II) NITRATE-DOPED POLY(3,4-ETHYLENEDIOXYTHIOPHENE) SURFACE PROPERTIES FOR SENSING APPLICATIONS

Nursyamimi Nayli Mohd Shahri[1], Zainiharyati Mohd Zain[1,2], Umi Zulaikha Mohd Azmi[3], Rossuriati Dol Hamid[1,2,*]

[1]*Faculty of Applied Sciences, Universiti Teknologi MARA, Shah Alam, 40450 Shah Alam, Selangor, Malaysia.*
[2]*Electrochemical Material and Sensor (EMaS) Research Group, Universiti Teknologi MARA.*
[3]*Malaysian Nuclear Agency, Jalan Bangi, 43000 Kajang, Selangor.*

Abstract. Fabrication of cobalt (II) nitrate-doped poly (3,4-ethylenedioxythiophene) (Co-PEDOT) on SPCE via electrochemical deposition is necessary to enhance the sensitivity of the modified electrode for sensing applications. The 3,4-ethylenedioxythiophene (EDOT) monomer was prepared in aqueous solution with sodium dodecyl sulfate (SDS) and cobalt (II) nitrate to improve its dispersion and solubility in water. Electrochemical deposition of Co-PEDOT was carried out using cyclic voltammetry (CV), where the applied potential induces oxidation of EDOT into radical cations, leading to polymerization and PEDOT formation. In this study, the effect of CV upper potential limits (+1.0 to +2.0 V) on Co-PEDOT surface properties, overoxidation behaviour, and electrochemical performance toward the $[Fe(CN)_6]^{3-}/[Fe(CN)_6]^{4-}$ redox system was investigated. The formation of PEDOT was confirmed by Raman spectroscopy through characteristic bands. The spectra also suggested that the doped Co appeared as cobalt (II) oxide (CoO), although several CoO peaks could not be conclusively identified due to overlapping with PEDOT peaks. Morphologically, all films exhibited a cauliflower-like structure, confirming successful Co-PEDOT growth on SPCE. Increasing the upper potential from +1.0 to +1.2 V promoted PEDOT formation, as evidenced by the highest sulfur content and branch-like structures. At +1.6 V, these structures fragmented into leaf-like features due to overoxidation, and they disappeared at +2.0 V, consistent with reduced sulfur content and surface roughness. This is further supported by the decreased C=C stretching of thiophene rings at +1.6 V, indicating onset of overoxidation, while presence of sulfone (SO_2) bands at +2.0 V confirming further degradation. The film prepared at +1.2 V, prior to overoxidation, exhibited the roughest surface and highest redox activity toward the $[Fe(CN)_6]^{3-}/[Fe(CN)_6]^{4-}$ probe. These findings demonstrate that applied potential plays a critical role in tuning Co-PEDOT properties and optimizing sensor performance.

Keywords: Co-PEDOT, electrodeposition potential, SPCE, morphology.

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***Corresponding author: rossuriati2996@uitm.edu.my**

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1. INTRODUCTION

Although screen-printed carbon electrodes (SPCEs) are widely used in electrochemical sensing due to their suitability for on-site applications, the carbon working electrode often limits their sensitivity, necessitating surface modification with conductive materials [1]. Among the available modifiers, conducting polymers such as poly(3,4-ethylenedioxythiophene) (PEDOT) are particularly attractive due to their high conductivity, good chemical stability and ease of deposition onto carbon substrates [2]. PEDOT is commonly fabricated on SPCEs via electrochemical deposition, where the morphology and composition of the polymer layer can be conveniently controlled by adjusting the applied potential or the number of deposition cycles. In this process, PEDOT is formed through the electrochemical polymerization of its monomer, 3,4-ethylenedioxythiophene (EDOT), a thiophene-based compound with electron-rich ethylenedioxy groups that facilitate oxidation and polymer growth [3]. However, the low solubility of EDOT in aqueous media often necessitates the use of organic solvents during electrodeposition [4]. Therefore, surfactants [5] and dopants [6] can be introduced to improve EDOT solubility and enable the formation of PEDOT films in more environmentally friendly aqueous systems.

Dopants play multiple roles during polymer deposition such as enhancing stability and solubility, but their primary function is to increase the conductivity of the conjugated polymer. Inorganic dopants are among the most common due to their small ionic size which allows for efficient diffusion into the polymer matrix and effective charge balance within PEDOT conjugated backbone. Among them, transition metal-based dopants are chosen because they can significantly enhance the conductivity and electrochemical activity of PEDOT [6]. Although noble metals such as gold, platinum and palladium are effective, their high cost limits large-scale application [7]. Consequently, low-cost transition metals such as tungsten, manganese, nickel and cobalt have emerged as attractive alternatives. In this study, cobalt (II) nitrate was selected as the dopant for PEDOT electrodeposition in aqueous media because it is cost-effective and can both facilitate analyte redox reactions and improve electron transfer at the electrode surface.

Among electrochemical deposition methods, cyclic voltammetry (CV) is widely used due to its ability to precisely control monomer oxidation and PEDOT film formation. However, the applied potential, particularly the upper potential limit, can significantly influence the surface properties and may induce overoxidation of Co-PEDOT deposited on SPCEs, thereby affecting its sensing performance. Although higher positive potentials can lead to overoxidation, they may also induce beneficial structural changes that enhance selectivity and sensitivity [8]. To date, the influence of CV upper potential limits on the surface properties and electrochemical behaviour of Co-PEDOT/SPCE has not been systematically investigated. Therefore, this study aims to examine the effect of varying the CV upper potential limit (+1.0 to +2.0 V, with a constant lower limit of 0 V) on the surface properties and overoxidation of Co–PEDOT films, as well as to evaluate the electrochemical performance of the modified electrodes using the $[Fe(CN)_6]^{3-}/[Fe(CN)_6]^{4-}$ redox system.

2. MATERIALS AND METHODS

2.1 Material, Chemicals and Reagents

All purchased chemicals were analytical grade and used directly without any purification. Phosphate buffer solution (PBS) was purchased from R&M, Malaysia. Cobalt (II) nitrate hexahydrate ($Co\ (NO_3)_2 \cdot 6H_2O$), sodium dodecyl sulfate (SDS) and 3,4-ethylenedioxythiophene (EDOT) were obtained from Sigma Aldrich, USA. The SPCE (refs C110) was obtained from Metrohm Dropsens, Spain.

2.2 Cobalt (II) Nitrate Doped-PEDOT Electrodeposition on SPCE

The pretreatment of SPCE was carried out by using chronoamperometry mode at +1.40 V for 300 s in PBS solution at pH 7.20 [1]. Then, the SPCE was cleaned with deionized water and air dried before modified with Co-PEDOT. The Co-PEDOT electrodeposition was carried out using a compact potentiostat/galvanostat (Autolab PGSTAT101) from Metrohm (Spain). The EDOT electropolymerization was carried out using cyclic voltammetry at a fixed lower potential limit (0 V) but various upper potential limit (+1.0, +1.2, +1.6 and +2.0 V) with a fixed number of cycles (30 cycles) and a scan rate of 100 mV/s in solution containing 0.01 M EDOT, 0.10 M ($Co(NO_3)_2$•$6H_2O$) and 0.05 M SDS. The Co-PEDOT electrodeposited on SPCE obtained was washed with deionized water, followed by air dried and kept in a desiccator before use.

2.3 Material Characterization

Raman spectrometer (Renishaw Centrus 0JQP25) at 532 and 785 nm lasers were used to measure Raman spectra of Co-PEDOT. The morphology and elements on the electrodes were captured and recorded using field-emission scanning electron microscopy with energy-dispersive X-ray spectroscopy (FESEM-EDX, FEI Versa 3D Dual Beam). The roughness measurement was performed using atomic force microscopy (AFM, Park System NX10). The thickness of electrodeposited Co-PEDOT on SPCE cannot be measured using FESEM-EDX or AFM due to the fragility of the SPCE substrate and the central position of the working electrode. Attempts to obtain cross-sectional or step-height measurements were unsuccessful, as cutting or cleaving the substrate caused deformation and damage to the electrode surface, preventing reliable thickness determination.

2.4 Detection of Ferric/Ferrocyanide Couple

A 0.9 mM potassium ferrocyanide solution in 0.1 M KCl was used to study the redox behavior of the ferric/ferrocyanide couple ($[Fe(CN)_6]^{3-}$/$[Fe(CN)_6]^{4-}$) on the bare SPCE and Co-PEDOT electrodeposited on SPCE. The sensing capabilities of these electrodes were evaluated based on the redox response of the $[Fe(CN)_6]^{3-}$/$[Fe(CN)_6]^{4-}$ couple observed by cyclic voltammetry in the potential range of -0.4 to +0.6 V at a scan rate of 100 mVs^{-1}.

3. RESULTS AND DISCUSSION

3.1 Cyclic Voltammetry (CV)

In this study, CV was selected as the electrodeposition method because it enables real-time monitoring of the electroactivity of the growing Co-PEDOT electrodeposited on the SPCE. The CVs of the Co-PEDOT measured at a various upper potential limit (between +1 and +2 V) but at a constant lower potential of 0 V is shown in Figure 1. The influence of the upper potential limit on EDOT oxidation was negligible at ≤ +1.6 V (Figure 1(a-c)) as the oxidation peak potential remained unchanged with increase number of CVs cycles (~ +0.7 V), consistent with previous reports [9,10]. However, at an upper potential of +2.0 V, the EDOT oxidation shifted to ~ +0.9 V (Figure 1(d)). Notably, during the first and second cycles, the oxidation peak appeared at ~ +0.7 V before progressively shifting to ~ +0.9 V with increasing cycles. This behavior is attributed to predominant PEDOT overoxidation at +2.0 V, which introduces electron-withdrawing groups (e.g., SO_2) and reduces the conductivity of the polymer backbone [11]. Consequently, subsequent EDOT oxidation requires higher potential due to slower electron transfer on the less conductive surface.

A sharp increase in current following the EDOT oxidation is evidence of PEDOT growth [9]. Above +0.68 V, the currents at all upper limit potentials studied showed decrease in current with number of cycles. While below +0.68 V, the currents at all upper potential studied increase with number of cycles, except at +1.6 V the current behave oppositely with number of cycles. Nevertheless, at all upper potential limits the current remained unchanged between 25th and 30th cycles. Therefore, 30 cycles were chosen for the electrodeposition in this study. Interestingly, CV feature of Co-PEDOT electrodeposited on SPCE at +1.0 V was closely resembled to +1.2 V. Meanwhile the CV feature at +1.6 V was comparable to +2.0 V with appearance of an additional broad band (between +1.2 and +1.5 V) at 5th cycle. These CV feature differences probably due to PEDOT overoxidation that occurred at a higher upper potential limit (i.e. +1.6 and +2.0 V), where Ismail et al. [10] observed PEDOT overoxidation at upper potential limit ≥ +1.5 V.

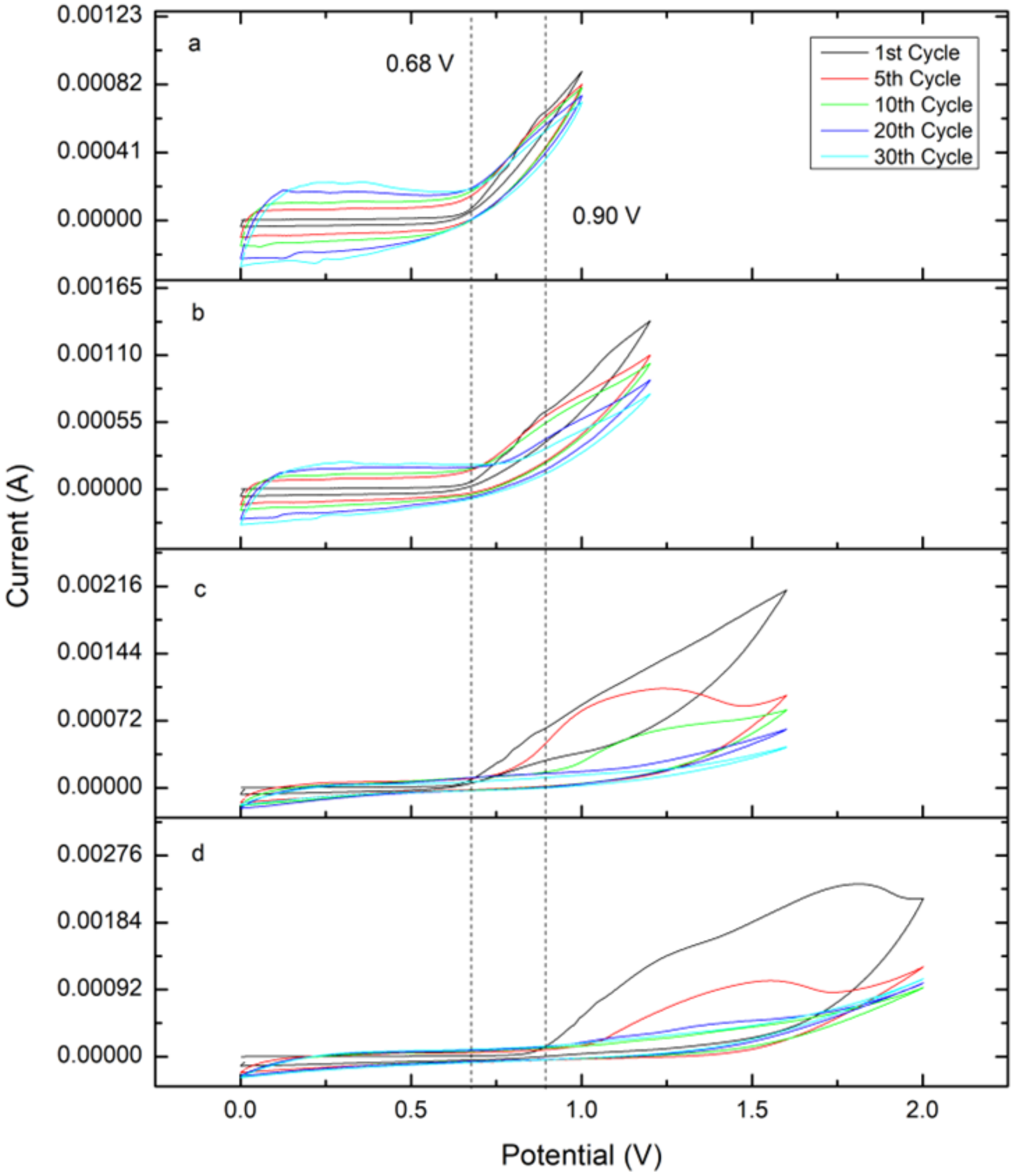

Figure 1: CV of Co-PEDOT electrodeposited on SPCEs collected at various upper potential limits of PEDOT electrodeposition and cycles, (a) +1.0 V (b) +1.2 V (c) +1.6 V and (d) +2.0 V

3.2 Raman Spectroscopy

The Co-PEDOT electrodeposited on SPCE can be confirmed by the Raman spectra shown in Figure 2. The general features of electrochemically grown PEDOT were associated with C-H stretches (2868 cm^{-1} and 2950 cm^{-1}), oxythylene groups (567 cm^{-1}, 988 cm^{-1}), C-O-C deformation (1094 cm^{-1}), C-S-C deformation (699 cm^{-1}, 851 cm^{-1}), C_α-$C_{\alpha'}$ inter-ring stretch (1256 cm^{-1}) and thiophene rings of C_β=C_β stretches (1367 cm^{-1}), C_α=C_β symmetric stretches (1431 cm^{-1}) and C_α=C_β asymmetric stretches (1503 cm^{-1}) [2]. These PEDOT characteristic features were observed at all the upper potential limits applied during the Co-PEDOT electrodeposition. The position of the symmetric C_α=C_β stretching band is commonly used to distinguish whether PEDOT adopts a quinoid or benzoid structure [12]. In this study, the electrodeposited PEDOT at all upper potential limits was predominantly quinoidal, as evidenced by the symmetric C_α=C_β peak appearing at approximately 1430 cm^{-1}.

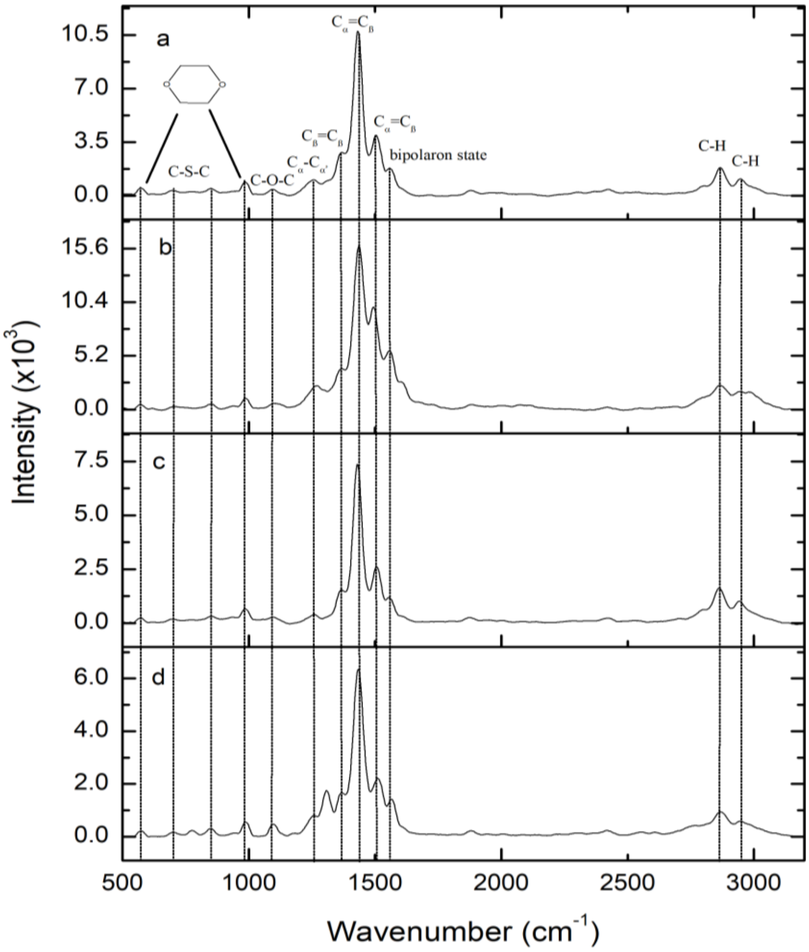

Figure 2: Raman spectra of Co-PEDOT electrodeposited on SPCEs at (a) +1.0 V, (b) +1.2 V, (c) +1.6 V and (d) +2.0 V. Spectra obtained at 532 nm excitation laser

The appearance of a peak at 1563 cm^{-1} suggests Co-doped PEDOT associated with the bipolaron state, regardless of the upper potential limit applied [12]. The bipolaron state arises when two polarons combine into a delocalized dication, indicating a formation of highly doped and conductive PEDOT structure. Raman spectra were also recorded using a 785 nm excitation laser. Regardless of the electrodeposition voltage applied, the spectral features of Co-PEDOT remained similar and are therefore represented by the spectrum obtained at +1.0 V electrodeposition (Figure 3). The use of a 785 nm laser enhances the intensity of bands in the lower wavenumber region, which is particularly important for probing the cobalt species formed during Co-PEDOT electrodeposition. The peaks at 315 and 441 cm^{-1} in Figure 3 may indicate the presence of cobalt-related species originating from cobalt(II) nitrate, possibly associated with the formation of cobalt oxide (CoO) [13]. However, this assignment cannot be conclusively confirmed because other characteristic CoO peaks at 530, 675 and 1060 cm^{-1} cannot be clearly distinguished due to overlap with PEDOT bands.

All Co-PEDOT electrodeposited between +1.0 and +1.6 V (Figure 2(a), 2(b) and 2(c)) showed no appearance of detectable peaks at 1176 cm^{-1} and 1312 cm^{-1} but these peaks present at + 2.0 V (Figure 2(d)). These vibrations are attributed to sulfone (SO_2) species, which are well-established indicators of PEDOT overoxidation [11]. Although these features are absent at +1.6 V, a noticeable decrease in the intensity of the C_β=C_β stretching mode together with the C_α=C_β symmetric and asymmetric stretching vibrations suggests a reduction in the thiophene conjugation length, indicating the onset of early-stage overoxidation [14]. This observation aligns with the mechanism proposed by Barsch and Beck [11], in which disruption of π-electron delocalization occurs prior to the formation of sulfoxide (S=O) and sulfone (SO_2) groups on the PEDOT backbone. In addition, no evidence of PEDOT overoxidation is observed in the Raman spectra of Co-PEDOT recorded using a 785 nm excitation laser.

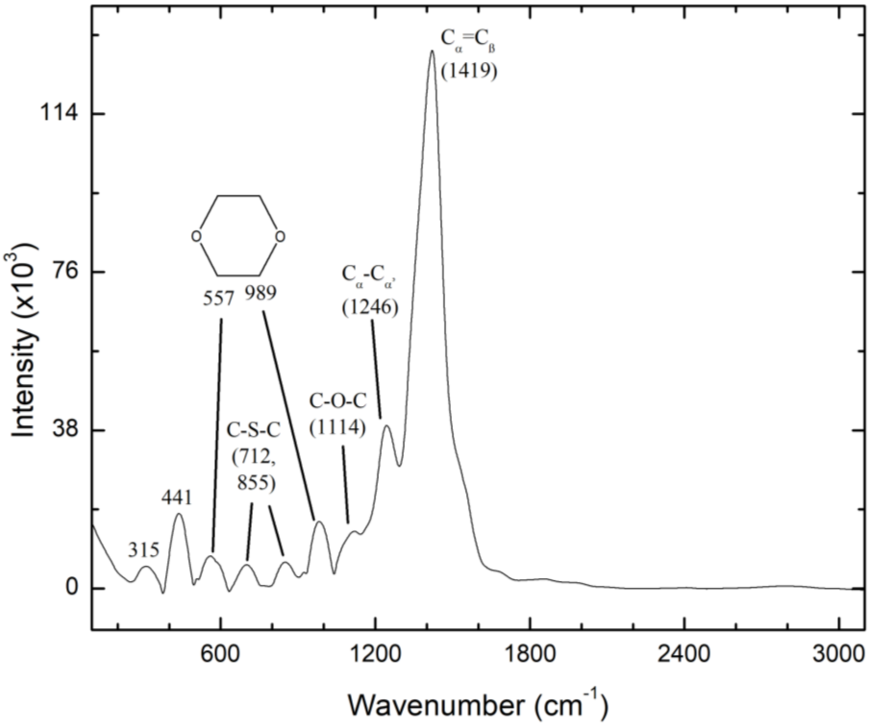

Figure 3: Raman spectrum of Co-PEDOT at +1.0 V measured at 785 nm excitation laser

3.3 Field Emission Scanning Electron Microscopy with Energy-Dispersive X-ray Spectroscopy (FESEM-EDX)

Electrodeposition of Co-PEDOT on SPCE can be evidenced by the morphological changes in bare SPCE before and after the electrodeposition of Co-PEDOT at various upper potential limits (Figures 4 and 5). The bare SPCE (Figure 4(a) and 5(a)) showed irregular flake-like morphology, a typical for unmodified carbon-based electrodes which has been reported in previous study [15]. In contrast, the electrodeposition of Co-PEDOT on SPCE regardless the upper potential limits associates with cauliflower-like globular structures (Figures 4(b-e) and 5(b-e)), a common morphology for PEDOT [16]. The formation of these globular structures suggests the successful growth of PEDOT chains doped with cobalt ions on the SPCE. The cauliflower PEDOT globular size becomes larger with increase the upper potential limit (i.e. from +1.0 to +1.2 V) indicating progressive PEDOT growth (Figure 4(b) and 4(c)). These results are in good agreement with Zubair et al. [17], who observed that higher electrodeposition potential promotes the growth of larger PEDOT globular structures. The PEDOT growth promoted at +1.2 V is also supported by the appearance of PEDOT additional morphology (branch-like extensions) within the cauliflower globules (Figure 5(c)). When overoxidation occurred at +1.6 V, the PEDOT branch-like extensions seem to degrade and fragmented and appeared as leaf-like structures (Figure 5(d)). The probability of branch-like extensions and leaf-like structures as CoO was eliminated by the EDX mapping (Figure 6). The EDX showed sulfur and cobalt were uniformly distributed throughout the Co-PEDOT film indicating that both the cauliflower-like globular structures and the branch-like extensions/leaf-like structures originate from the same material. However, these additional morphologies disappeared, and only cauliflower-like globular structures were observed at upper potential limit of +2.0 V (Figure 4(e)) with the cauliflower globules size remain unchanged between +1.2 and +2.0 V (Figure 4(c-e)). Therefore, overoxidation does not affect the PEDOT cauliflower-like globular structure but causes degradation of additional morphology, i.e. branch-like extensions which degrade into smaller fragments (leaf-like structures) at +1.6 V (Figure 5(d)) and finally disappeared at +2.0 V (Figure 5(e)).

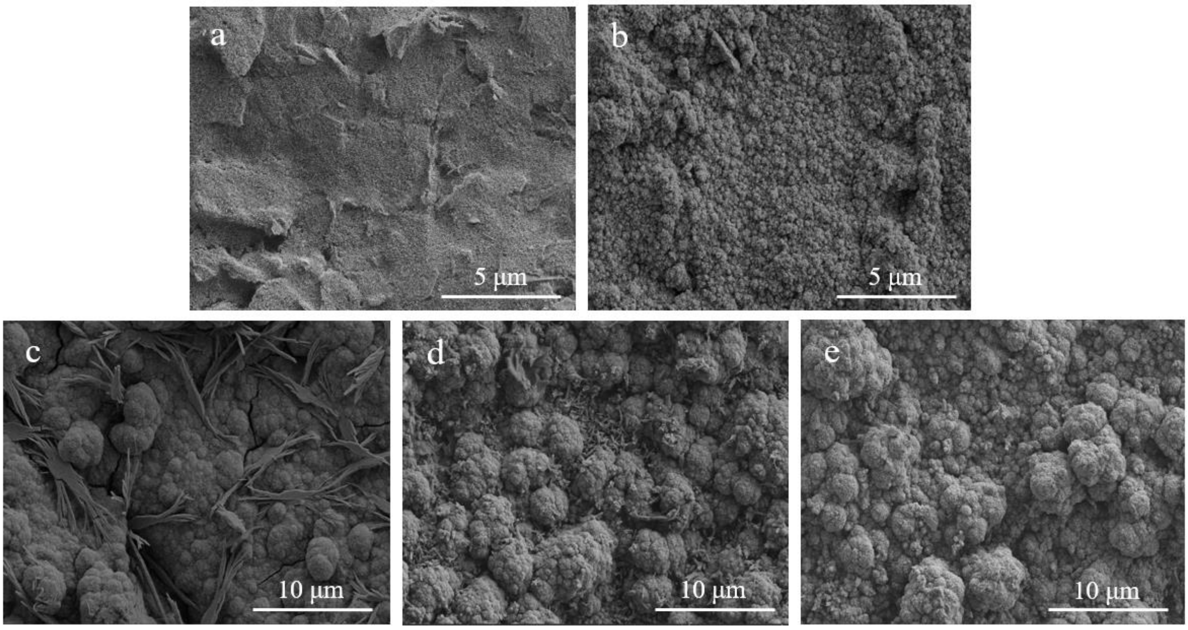

Figure 4: FESEM images (at 10k magnification) of (a) bare SPCE and Co-PEDOT electrodeposited on SPCE at upper potential limits of (b) +1.0 V, (c) +1.2 V, (d) +1.6 and (e) + 2.0 V

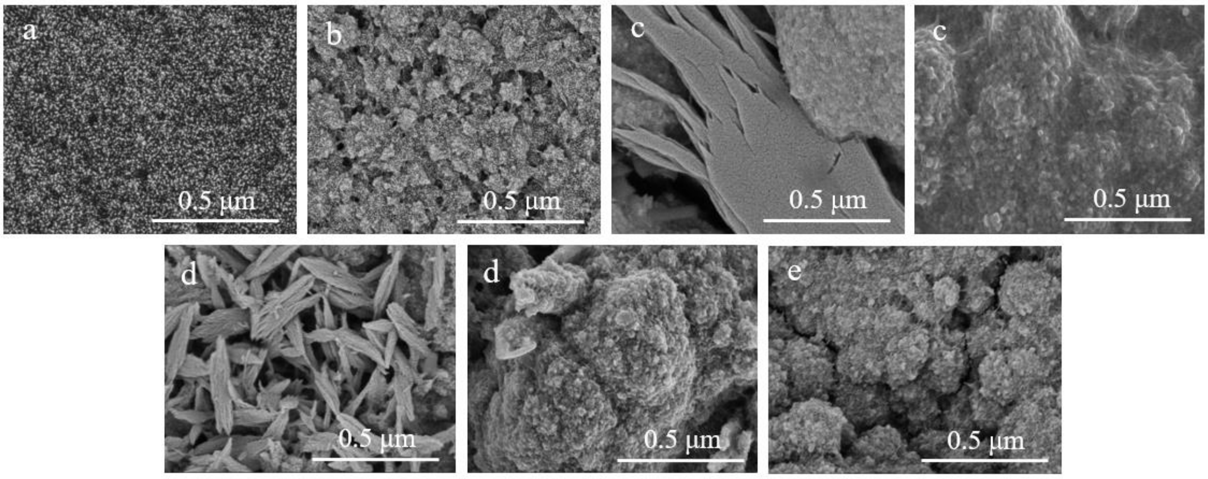

Figure 5: FESEM images (at 100k magnification) of (a) bare SPCE and Co-PEDOT electrodeposited on SPCE at upper potential limits of (b) +1.0 V, (c) +1.2 V, (d) +1.6 and (e) +2.0 V

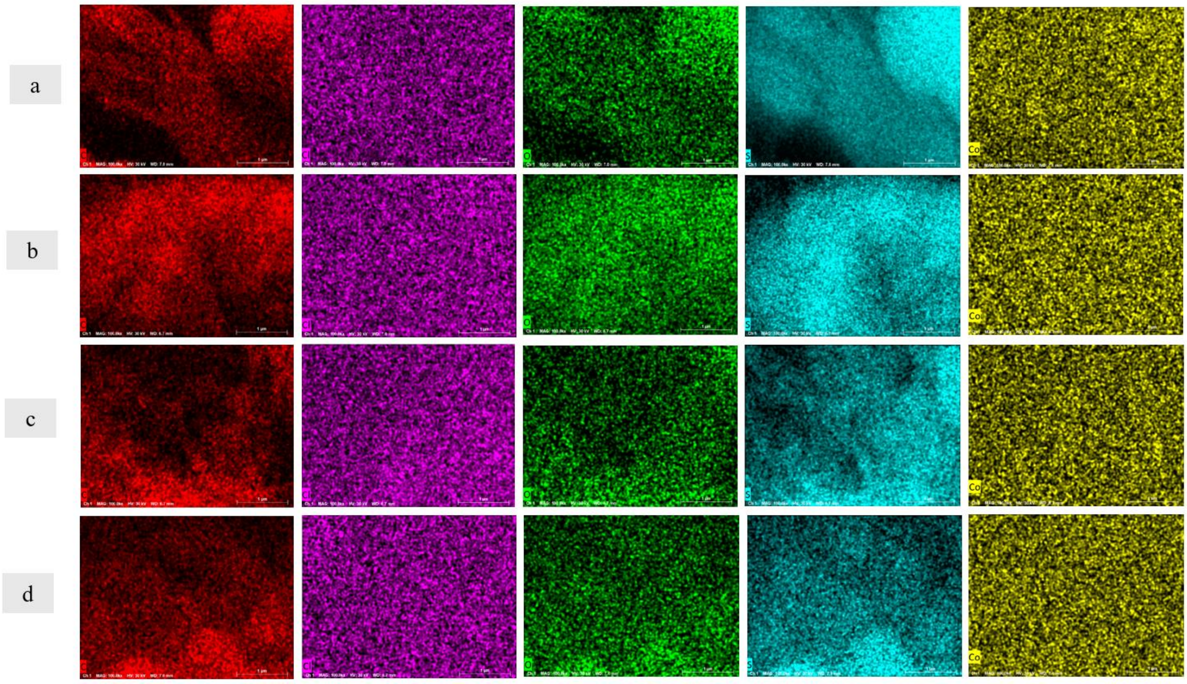

Figure 6: EDX elemental mapping of Co-PEDOT electrodeposited on SPCE at different potentials: (a) +1.2 V (branch-like morphology), (b) +1.2 V (globular morphology), (c) +1.6 V (branch-like morphology) and (d) +1.6 V (globular morphology)

The elemental composition of Co-PEDOT at different upper potential limits were confirmed by EDX (Table 1) at 10k magnification, which provides a more representative view of the surface compared to 100k magnification. From the EDX data, carbon (75.2 - 79.3 %) was the most dominant element on Co-PEDOT followed by oxygen (12.7 - 16.9 %) and sulfur (5.9 - 7.8 %). These EDX results reflect the expected elemental composition based on EDOT's structure (though not in exact stoichiometric ratio) with carbon, sulfur and oxygen arising from the PEDOT backbone. The upper potential limits varied at +1.0 to +2.0 V does not significantly affect the percent cobalt incorporated into the PEDOT structure (cobalt content was between 0.05 and 0.11 %). The presence of chlorine (0.8 - 2.4 %) on the Co-PEDOT was resulted from the chemical residues of cobalt (II) nitrate hexahydrate (containing trace chloride < 0.005 %), sodium dodecyl sulfate (SDS, < 0.01 % chloride) and phosphate-buffered saline (PBS) used during the electrodeposition. The increase in sulfur content (Table 1) from +1.0 to +1.2 V indicates that a higher upper potential enhances PEDOT polymerization before overoxidation begins [18]. This is consistent with the intensity of PEDOT characteristic Raman peaks (699 cm^{-1}, 1094 cm^{-1}, 1503 cm^{-1}, 567 cm^{-1} and 988 cm^{-1}) in Figure 2 at +1.2 V, which were higher than the one at +1.0 V. The decrease in sulfur content between +1.6 and +2.0 V also supports the degradation of PEDOT branch-like extensions into smaller fragments (leaf-like structures) and finally disappeared at +2.0 V (Figures 4(c-e) and 5(c-e)).

Table 1: Energy-Dispersive X-ray Spectroscopy (EDX) analysis of Co-PEDOT electrodeposited on SPCEs at +1.0 V, +1.2 V, +1.6 V and +2.0 V at 10k magnification.

Sample	C (%)	Cl (%)	O (%)	S (%)	Co (%)
Co-PEDOT +1.0 V	76.02	1.03	16.85	6.02	0.08
Co-PEDOT +1.2 V	75.15	0.75	16.17	7.82	0.11
Co-PEDOT +1.6 V	78.13	2.44	12.73	6.64	0.06
Co-PEDOT +2.0 V	79.31	2.06	12.69	5.89	0.05

3.4 Atomic Force Microscopy (AFM)

The effect of upper potential limits on the surface texture of Co-PEDOT electrodeposited on SPCE were evidenced by the AFM images in Figure 7. As seen in the AFM images, the surface at +1.0 V (Figure 7(a)) appeared smooth while at +1.2 V (Figure 7(b)) it became noticeably rougher with pronounced protrusions. With further increase in potential to +1.6 V (Figure 7(c)) and +2.0 V (Figure 7(d)), the surface became progressively denser and smoother. These observations are consistent with the root-mean-square (RMS) roughness values, which increased from 2.363 nm at +1.0 V to 4.736 nm at +1.2 V, before decreasing to 3.106 nm and 2.165 nm at +1.6 V and +2.0 V, respectively. The Co-PEDOT electrodeposited on SPCE prepared at +1.2 V showed the roughest surface may be attributed to the formation of branch-like PEDOT structures [17]. Increasing the potential from +1.6 V to +2.0 V led to a smoother surface as these branches fragmented and disappeared (Figures 4 and 5).

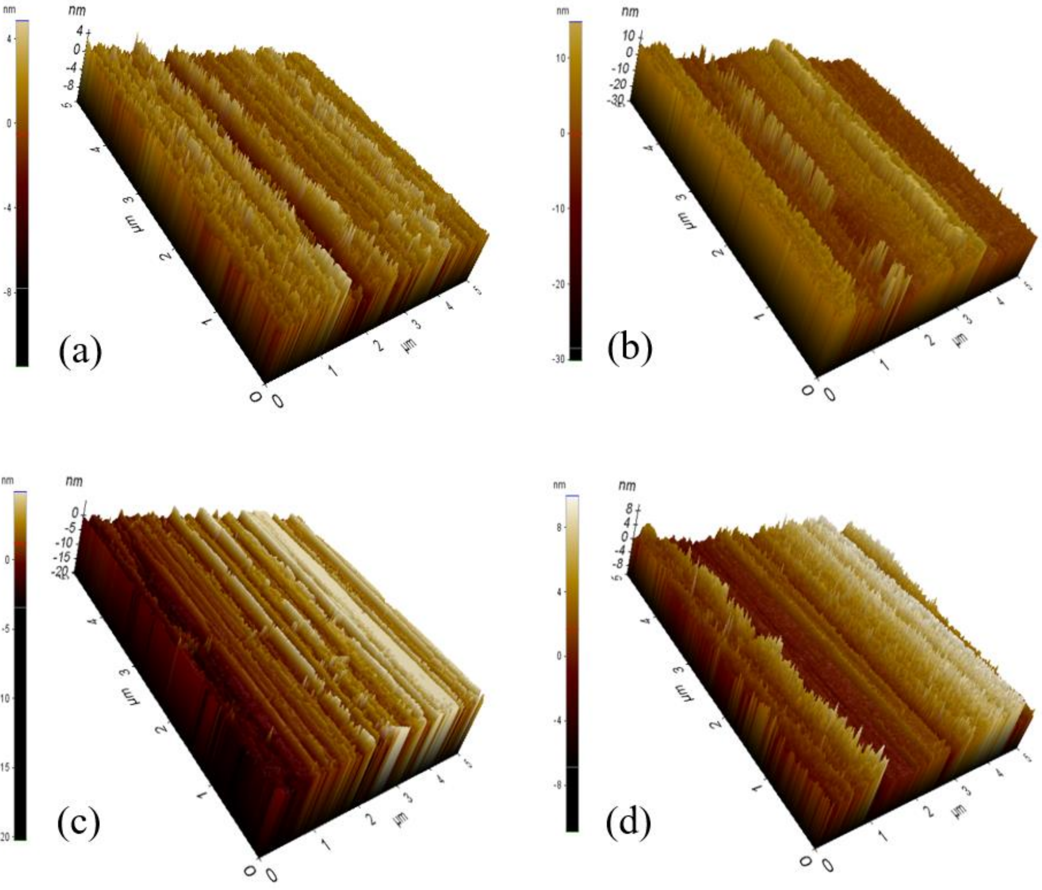

Figure 7: AFM images of Co-PEDOT electrodeposited at upper potential limits of (a) +1.0 V, (b) +1.2 V, (c) +1.6 V and (d) +2.0 V

3.5 Potential of Co-PEDOT film as a sensing material

The $([Fe (CN)_6]^{3-}/[Fe (CN)_6]^{4-})$ redox reaction was used to assess the potential of Co-PEDOT electrodeposited on SPCE as electrochemical sensing material. The capability of this material electrodeposited at various upper potential limits was also compared with bare SPCE as shown in Figure 8. The bare SPCE (inset Figure 8) showed a pair of well-defined redox peaks with anodic-to-cathodic peak current ratio (I_{pa}/I_{pc}) of 1.1 as expected for a reversible redox reaction for $[(Fe (CN)_6]^{3-}/[Fe (CN)_6]^{4-}$. Deposition of Co-PEDOT between +1.0 and +1.2 V promotes the $[Fe (CN)_6]^{3-}/[Fe (CN)_6]^{4-}$ redox reaction, with maximum I_{pa} value observed at +1.2 V. Where the I_{pa} value at +1.2 V increases by 5 times when compared with bare SPCE. However, the I_{pa} value decreased significantly with broad and poor defined peak at higher upper potential limits, i.e. +1.6 and +2.0 V. The I_{pc} also showed a similar feature as the I_{pa} because of the effect of upper potential limit. The Co-PEDOT electrodeposited on SPCE at +1.6 and +2.0 V showed a poor electrochemical performance as a $[Fe\ (CN)_6]^{3-}/[Fe\ (CN)_6]^{4-}$ sensor where the redox peaks were of the analyte cannot be distinguished from the background signal. Overoxidation occurred at +1.6 and +2.0 V resulted with smooth surface and partial structural degradation of PEDOT that reduced the electrochemical performance of the Co-PEDOT electrodeposited on SPCE as a sensor. This is consistent with Zubair et al. [17], who observed maximum redox activity of $[Fe\ (CN)_6]^{3-}/[Fe\ (CN)_6]^{4-}$ at approximately +1.2 V, followed by a decrease in current at a higher potentials resulting from overoxidation and loss of electrochemical activity.

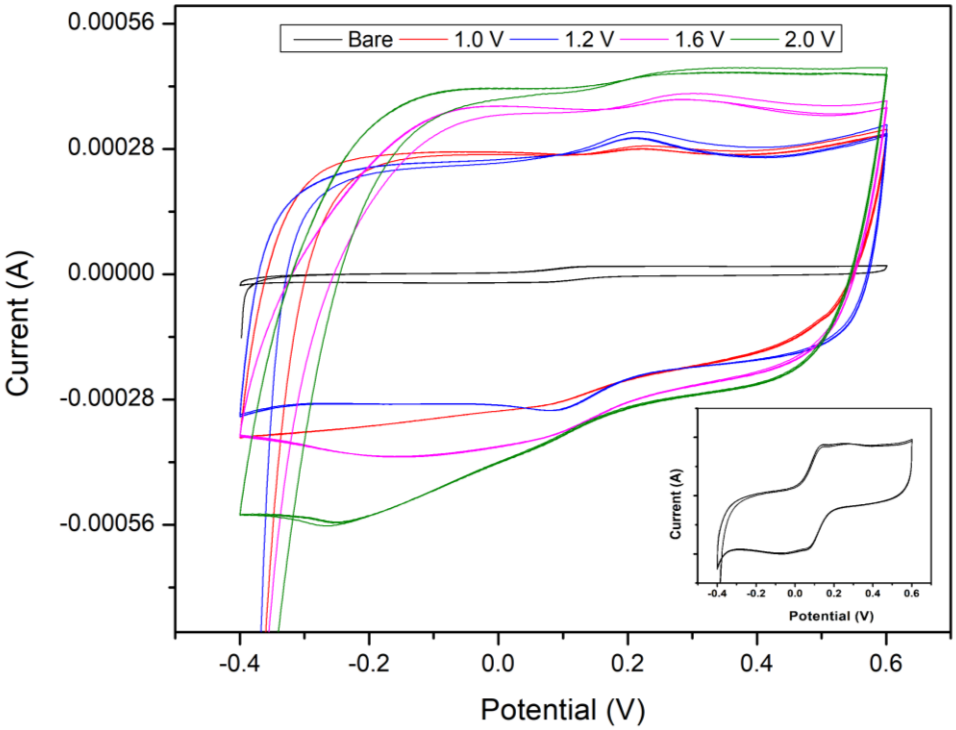

Figure 8: Redox reaction of $(Fe\ (CN)_6]^{3-}/[Fe\ (CN)_6]^{4-})$ on bare SPCE and Co-PEDOT eletrodeposited at upper potential limits of +1.0 V, +1.2 V, +1.6 V and +2.0 V. Inset: redox response of the bare SPCE

4. CONCLUSIONS

Upper potential limit in cyclic voltammetry applied during Co-PEDOT electrodeposition on SPCE significantly affect the surface properties and electrochemical performance of Co-PEDOT as electrochemical sensing material. Increase in upper potential from +1.0 to +1.2 V promotes PEDOT growth on the Co-PEDOT and results with rougher surface. However, when the potential increase to ≥ +1.6 V, overoxidation occurred and caused partial degradation of PEDOT structure and resulted with smoother surface. The changes in the surface properties influenced the electrochemical performance of the Co-PEDOT as a $[Fe\ (CN)_6]^{3-}/[Fe\ (CN)_6]^{4-}$ sensor. The Co-PEDOT at +1.2 V provides the highest electrochemical activity for $[Fe\ (CN)_6]^{3-}/[Fe\ (CN)_6]^{4-}$ redox reaction due to highest degree of PEDOT chains and associated with the roughest surface compared at other upper potential limits.

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Author Contributions

All authors contributed toward data analysis, drafting and critically revising the paper and agree to be accountable for all aspects of the work.

Disclosure of Conflict of Interest

The authors have no disclosures to declare.

Compliance with Ethical Standards

The work is compliant with ethical standards.

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