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Treatment of Wastewater by Anodic Oxidation Using SnO_2 Rotating Cylinder Electrode

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Abstract

Tin oxide is a promising anode for the oxidation of refractory organic compounds, with advantages of low cost and absence of secondary pollutants. In this study, a Cu/SnO_2-Sb_2O_5 anode was fabricated by cathodic deposition in a rotating system. The effect of electrode rotation speed on the structural properties of the synthesised anode (Cu/SnO_2-Sb_2O_5) was investigated in the range of 50–250 rpm. The structure and morphology of the prepared anode were examined via SEM and XRD. Results showed that the Cu/SnO_2-Sb_2O_5 anode prepared at 250 rpm possessed a compact, multi-layer coating structure without cracks. The electrochemical performance of the Cu/SnO_2-Sb_2O_5 anode was studied by examining its activity in degrading methylene blue (MB) at a concentration of 100 mg/L by anodic oxidation at 20 mA/cm^2 for 4 h. The anode prepared at 250 rpm was able to degrade MB with an efficiency of 93.6%, which was higher than that obtained by the anode prepared at 50 rpm (85.18%). The pseudo-first-order rate constant at 250 rpm was 0.01258 min^{-1}, which was 1.6 higher than that observed for the anode prepared at 50 rpm, confirming the importance of rotation in improving mass transfer and facilitating the deposition of tin oxide with high catalytic activity. The Cu/SnO_2-Sb_2O_5 anode fabricated at 250 rpm exhibited an enhanced service life of 18 h (200 mA/cm^2; 0.5 M NaOH), whereas that prepared at 50 rpm had a reduced service life of 12 h. Compared with the Cu/SnO_2-Sb_2O_5 anode prepared by thermal decomposition, the present anode exhibited a service life of 1.5 times greater than that of the thermal method, confirming the efficacy of the electrodeposition approach in fabricating the Cu/SnO_2-Sb_2O_5 anode.

Keywords: *Anodic oxidation; Alectrodeposition; Rotating cylinder electrode; Cu/SnO_2-Sb_2O_5; Methylene blue*

1. Introduction

Electrochemical advanced oxidation processes (EAOPs) have recently been utilised for the treatment of wastewaters containing refractory organic compounds because of its many benefits such as mild operational conditions, superior oxidation performance, ease of handling and eco-friendly processes [1–4]. Nevertheless, the application of EAOPs on an industrial scale is limited by the low current efficiency of the used anodes, their limited service life and high expense associated with their fabrication [5],[6]. EAOPs can be improved with the development of cost-effective anodes with high efficiency, which is a crucial consideration for researchers. Hence, various anodic materials have been developed, including boron-doped diamond and metal oxides [7]. Among them, antimony-doped oxide tin (SnO_2-Sb) is the most promising anodic material because of its low cost, ease of preparation, inert surface and absence of heavy metal pollution [8–10]. However, the limitation of this anode, represented by its brief operational lifespan, remains a challenge [7], [8], [11]. Antimony-doped oxide tin anodes can be prepared by various methods, including sol-gel, dip-coating, spray pyrolysis and electrodeposition [12]. Among them, electrodeposition is a facile, cost-

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effective method for generating thick films at low temperatures, with potential for industrial-scale production [8].

The SnO_2-Sb film can be used for cathodic deposition either on a stationary titanium substrate using $SnCl_2$, followed by calcination at 550 °C [7],[8],[12–17], or on a stationary copper substrate from $SnCl_4$ with HNO_3 [10],[18–21]. However, no previous work has been conducted on SnO_2-Sb film electrodeposition using a rotating cylinder electrode (RCE).

The RCE has been used successfully in wastewater treatment because of its uniform potential and current distributions, as well as enhanced mass transfer resulting from turbulent flow conditions at moderate rotational speeds [22]. In addition, it is readily expandable and may be operated with a compact design [23]. RCE was applied in various industrial sectors, including heavy metal removal [24–26], anodic oxidation of wastewaters [27–30] and cathodic deposition of metals and alloys [31]. Therefore, the present work aimed to investigate the effect of rotation speed (50–250 rpm) on the structure, service life and performance of the Cu/SnO_2-Sb_2O_5 anode in degrading methylene blue (MB). The selection of MB as a model organic contaminant is based on recent studies demonstrating that tin oxide anodes have significant catalytic activity for the anodic oxidation of organic pollutants, especially MB [32],[33]. Additionally, MB is classified as a cationic dye, which is used in different applications, such as dyeing and medical sciences, where long-term exposure to MB may lead to anaemia, nausea, hypertension and vomiting [34]. The Cu/SnO_2-Sb_2O_5 anode was prepared by cathodic deposition and then calcined at 550 °C.

2. Experimental Work

2.1. Chemicals

Tin chloride ($SnCl_2 \cdot 2H_2O$; 99% purity) and antimony chloride ($SbCl_3$; 99% purity) were sourced from Sisco Research Laboratories. Sulphuric acid (H_2SO_4; 98%), citric acid (99%) and sodium hydroxide pellets (NaOH; 98%) were acquired from Laboratory Reagent. Na_2SO_4 (99%) was obtained from Central Drug House. MB ($C_{16}H_{18}N_3SCl$; 97%) was purchased from BDH Chemical Ltd., Poole, England. All chemical reagents used in this work were of analytical grade; hence, no further purification was performed.

2.2. Electrode Preparation

An anode composed of antimony tin oxide was prepared by cathodic deposition of a tin film on a copper cylinder, followed by calcination at 550 °C. The copper cylinder had an outer diameter of 1.8 cm, a thickness of 0.5 mm and a length of 1 cm. It was used as a substrate. It was initially polished with 400-grit sandpaper, followed by 800-grit sandpaper, rinsed with distilled water and ethanol and soaked in 1 M HCl solution for 15 min under sonication.
The cathodic deposition of a tin film on a copper substrate was performed in a cylindrical electrochemical cell with a capacity of 300 mL, as shown in Fig. 1. It was composed of a copper rotating cylinder as the cathode and four Sn strips acting as anodes located in a cell body. Each Sn strip had a length of 10 cm, a width of 1 cm and a thickness of 0.5 cm. The cell body had a length of 12 cm and a diameter of 8 cm. The four Sn strips were uniformly arranged inside the cell body via a Teflon ring located at the top of the cell body. The copper cylinder was fixed on a stainless steel rod with an external diameter of 1.7 cm and attached to an electric motor.

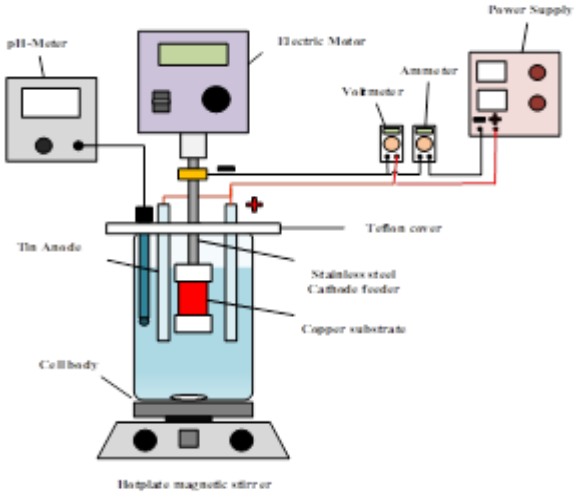


Fig. 1. Schematic of the electrodeposition cell

About 150 mL of an aqueous solution containing 67.50 g/L tin chloride, 2.25 g/L antimony chloride and 57.50 g/L citric acid was used as an electrolyte for the deposition of Sn-Sb film. This electrolyte was selected based on research conducted by Sun et al. [7]. Cathodic deposition commenced with a current density of 10 mA/cm^2 applied to the cathode via a DC power source (UNI-T: UTP3315TF-L) for 30 min at 50 °C [7]. Upon completion of deposition, Cu cylinder coated with Sn and Sb was detached from the stainless steel rod, washed several times

with distilled water and dried at 50 °C for 1 h. The copper cylinder coated with tin and antimony was calcined at 550 °C for 3 h to produce a Cu/SnO_2-Sb_2O_5 anode. The above procedure was employed to prepare anodes at different rotational speeds (50, 100, 150 and 250 rpm).

2.3. Characterisation of Electrode Performance

2.3.1. Physicochemical Characterisation

The morphology and crystal structure of the Cu/SnO_2-Sb_2O_5 anode were analysed by SEM (FEI Company, the Netherlands). It was operated at a high voltage of 25 kV, bias of 1400 V, zero bias and spot of 8.0. The XRD of the Cu/SnO_2-Sb_2O_5 anode was identified by X-ray diffraction (XRD 6000, Shimadzu, Japan). It was operated at 30 mA and 40 kV. The 2θ range was tested between 20° and 80° at a step size of 0.2° and scan step time of 1.2 s. The pH of the solution was measured using a digital pH meter model (PH211, HNNA Instrument Inc., Romania).

2.3.2. Electrochemical Decolourisation Test

The oxidation capacity of the Cu/SnO_2-Sb_2O_5 anode was assessed by the electro-oxidation of an aqueous solution containing 100 mg/L MB, supplemented with 35.5 g/L sodium sulphate as a supporting electrolyte [32]. Anodic oxidation was performed in an electrochemical cell similar to that used in section 2.2, where Cu/SnO_2-Sb_2O_5 was used as an anode, and a graphite cylinder (L=8 cm and D=7 cm) was used as a cathode. Anodic oxidation was conducted at a current density of 20 mA/cm^2 for 4 h under various rotational speeds for the anode. During oxidation, 1 mL of solution was extracted every 1 h and then diluted 10 times. The concentration of MB was measured by determining the absorbance of the sample at $\lambda_{max.}$= 665 nm using a Shimadzu UV-spectrophotometer (Japan). The MB concentration was derived from the calibration curve depicted in Figure 2.
The MB removal efficiency was evaluated using Eq. 1 [32]:

$$RE\% = \frac{C_o - C_f}{C_o} \times 100 \quad \ldots (1)$$

where C_o represents the initial MB concentration (100 mg/L), and C_f represents the concentration of MB (mg/L) at the end of each experiment.

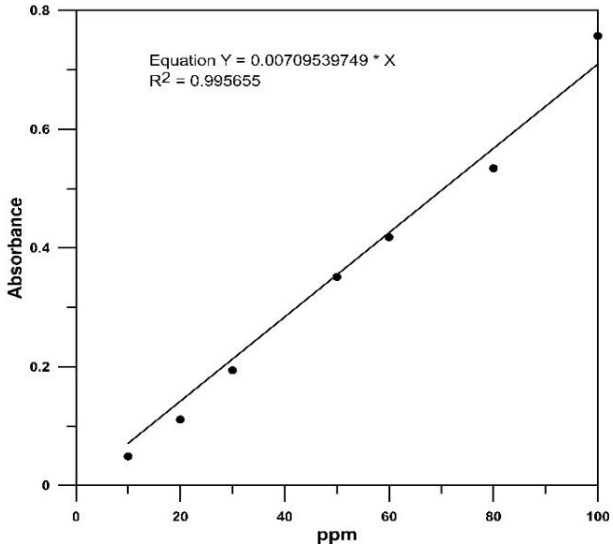


Fig. 2. Calibration curve for methylene blue

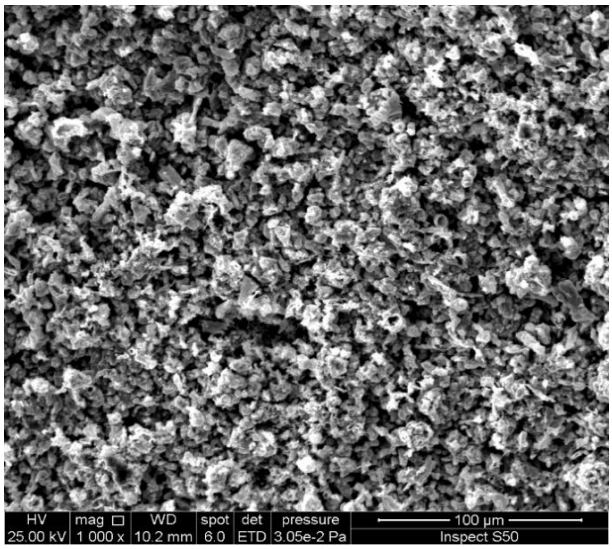
2.3.3. Accelerated Service Life Test

In the literature, the service life of the tin oxide anode can be achieved using either 0.5 M H_2SO_4 or 0.5 M NaOH at a current density of 200 mA/cm^2 [35]. The acceleration of service lifetime depends on the method of tin oxide anode preparation, current density and the concentration of the acidic or alkaline medium. Elevated concentrations and current densities result in a reduced acceleration of service lifetime [7]. The accelerated lifetime test in the present work was performed using the same cell described in section 2.3.2, using 100 mL of 0.5 M NaOH solution and a current density of 200 mA/cm^2. We favoured an alkaline medium to prevent the direct corrosion of the substrate (copper) by acid that may occur during the test. The cell voltage was monitored during testing. The anode was considered active as the voltage increased by 2 V above the initial value [36].

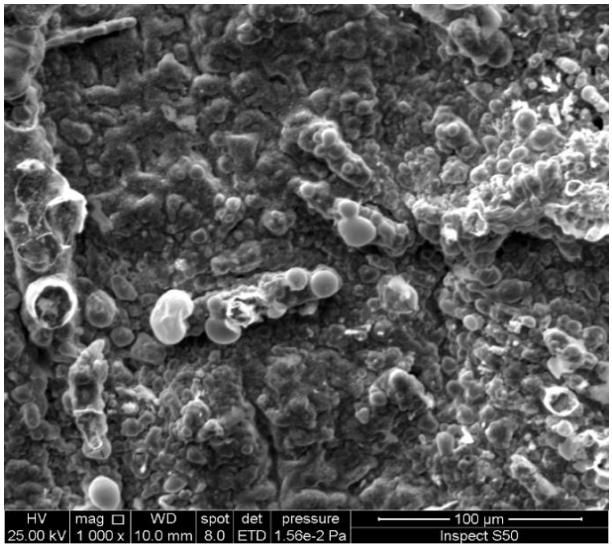
3. Results and Discussion

3.1. Physicochemical Characterisation

Figure 3 represents the SEM images of the prepared anodes via electrodeposition under two rotational speeds: 50 and 250 rpm. The structures appeared to be multilayer coatings without cracks at both rotational speeds. This morphology may happen as a result of existing citric acid that has the ability to form a stannous citrate complex on the Sn film surface [7],[8]. Small particle sizes were observed as the rotational speed increased, accompanied with a compact and uniform surface.



(a)

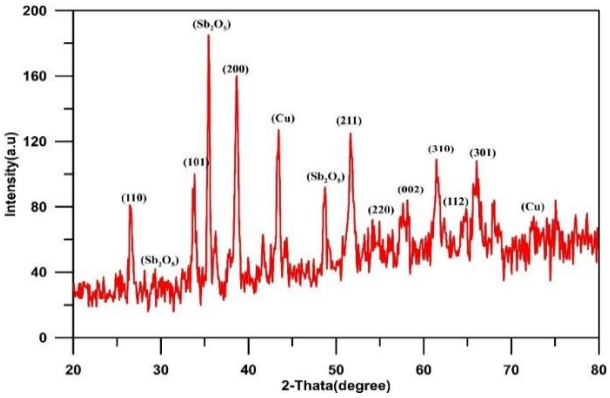


(b)

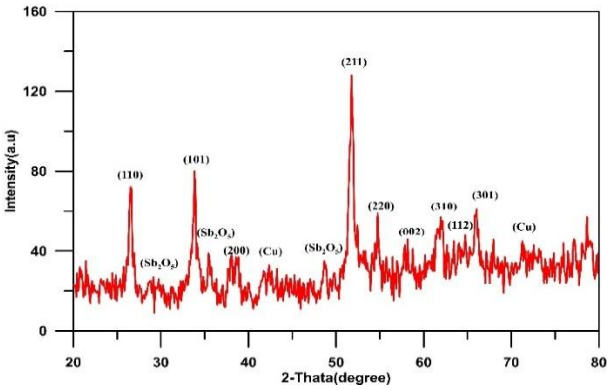
Fig. 3. SEM results for Cu/SnO_2-Sb_2O_5 anodes at two rotational speeds: a) 50 and b) 250 rpm

Honorata Kazimierczak et al. [37] reported similar phenomena, indicating that an increase in rotational speed leads to reducing th particle size of films formed by deposition of tin, zinc and bismuth on a Cu substrate. Furthermore, Chun-Wei Wu et al. [31] found that an increase in rotation leads to a decrease in electrode surface roughness during the electrodeposition of cobalt–manganese–phosphor coating on copper. A similar result was observed regarding the roughness of the copper/tin alloy film prepared by electrodeposition [38].

Figure 4 presents the XRD results for anodes fabricated by electrodeposition at two rotational speeds (50 and 250 rpm). Strong diffraction peaks related to SnO_2 with tetragonal rutile structure appeared at 2θ= 26.6º (110), 33.9º (101), 37.9º (200), 51.5º (211), 54.8º (220), 57.8º (002), 61.9º (310), 64.7º (112) and 65.9º (301) for both cases. These peaks corresponded to 'PDF no. 41-1445'. The observing a strong peak at the (211) plane is an indication of preferential crystallographic orientation along the (211) direction at 50 rpm. However, at 250 rpm, the orientation favoured the (200) direction due to the formation of a strong peak [7,39]. In all cases, Cu peaks were identified at 43.3° and 74.7° [18], whereas antimony oxide peaks were recognised at 2θ=28.4°, 30°, 35.2° and 48.7°, corresponding to the cubic crystallographic phase (COD1010505) [40]. Tin was converted completely to its oxide as no peaks were observed [7].



(a)



(b)

Fig. 4. Results of XRD related to Cu/SnO_2-Sb_2O_5 anodes at two rotation speeds. a) 50rpm, b) 250rpm

3.2. Electrochemical Decolourisation of MB

Figure 5 shows the degradation of MB by electro-oxidation using the prepared Cu/SnO_2-Sb_2O_5 anodes via cathodic deposition at different rotational speeds. The results showed that optimal performance was achieved for the anode prepared at 250 rpm, resulting in removal 61.54% MB after 1 h, which increased to 93.6% at 4 h.

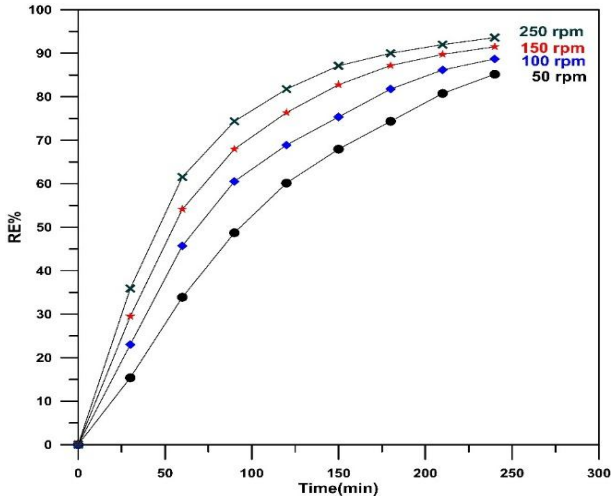


Fig. 5. Decolourisation of methylene blue during anodic oxidation via the Cu/SnO_2-Sb_2O_5 anode

The degradation of MB can be expressed as a pseudo-first-order kinetic based on Eq. 2 [32]:

$$Ln\left(\frac{C_f}{C_o}\right) = -kt \quad \dots (2)$$

where k represents the kinetic rate constant (h^{-1}).

Figure 6 illustrates the plot of $\ln(C_f/C_o)$ versus time for the prepared anodes at various rotational speeds. Anodic oxidation followed a pseudo-first-order reaction, as all lines exhibited R^2 values beyond 0.99 (0.991–0.999).

As shown in Figure 6, the k value at 250 rpm was 0.01258 min^{-1} greater than that at 50 rpm by 1.6. This behaviour may be due to the rotational effect on the enhancement of mass transfer towards the cathode during electrodeposition, which also led to the formation of crack-free deposits. These results confirmed that the prepared Cu/SnO_2-Sb_2O_5 anode at 250 rpm exhibited excellent activity and performance. Additionally, the k-values in this work were double those reported for tin oxide prepared by the sol-gel method (0.006 min^{-1}) [41], thereby confirming that tin oxide prepared by cathodic deposition had good activity.

To compare the performance of the Cu/SnO_2-Sb_2O_5 anode formulated at 250 rpm with other anodes prepared under stationary conditions, we calculated the amount of MB degraded per Coulomb applied (Y) based on the following formula [7]:

$$Y\left(\frac{mg}{coulomb}\right) = \frac{C_o \times V \times RE\%}{I \times s \times t} \quad \dots (3)$$

where C_o is expressed in mg/L, V is expressed in L, I is expressed in A/cm^2, s is expressed in cm^2 and t is expressed in s.

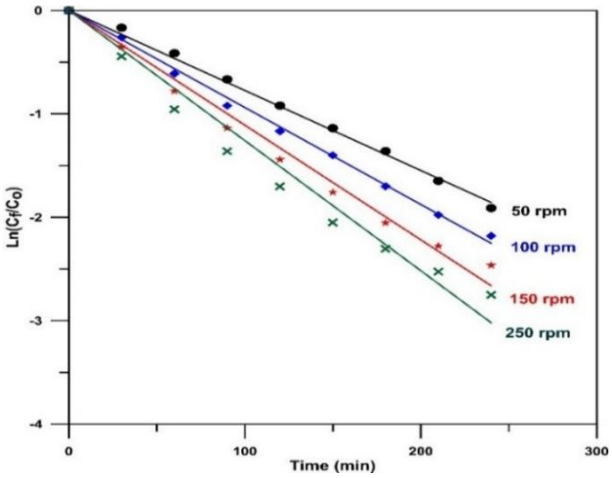


Fig. 6. Ln(C_f/C_o) versus time for degrading methylene blue by anodic oxidation

Table 1 represents the values of Y for different anodes in comparison with the present work. Rotation improved the performance of the anode, where Y of the present anode was 1.8 times that observed in sol-gel, confirming that electrodeposition was the superior preparation method.

Table 1,
Comparison of the activity of Cu/SnO_2-Sb_2O_5 anode relating to anodes used in previous studies

Anode	Method of preparation	C_o $\frac{mg}{L}$	V (ml)	I $\frac{mA}{cm^2}$	S (cm^2)	Time (min)	RE%	Y (10^{-3}x mg/C)	Ref.
Ti/SnO_2-Sb stationary	Sol-gel	50	50	20	2	100	43.8	4.56	41
Ti/SnO_2-Sb stationary	Pulsed electrodeposition	50	50	20	2	120	65.1	5.65	42
Ti/SnO_2- Sb stationary	DC-electrodeposition	100	100	20	4	240	89.6	8.07	8
Cu/SnO_2-Sb_2O_5 Rotating cylinder anode	DC-electrodeposition	100	150	20	6	240	93.6	8.15	Present work

Y of the present work exhibited a 10% improvement over the findings of Sun et al. [8] due to the effect of rotation. Furthermore, the fabricated anode was less expensive than the anode prepared by Sun et al. [8] due to the utilisation of copper as a substrate and ease of scaling up the rotating cylinder electrode.

3.3. Accelerated Service Life

Figure 7 illustrates the effect of rotation speed on the accelerated service life of the Cu/SnO_2-Sb_2O_5 anodes. During the test, the voltage of the cell remained constant until a sharp rise occurred, reaching 10 V, which indicated the deactivation of the anode. Figure 7 shows that the Cu/SnO_2-Sb_2O_5 anode prepared at 250 rpm exhibited the maximum accelerated service life (18 h), whereas the prepared anode at 50 rpm had the lowest accelerated service life (12 h). This indicates that rotation could enhance the structure of the tin film, making it more compact and free of cracks due to the uniform deposition resulting from the hydrodynamic effects of rotation on mass transfer towards the cathode surface [27].

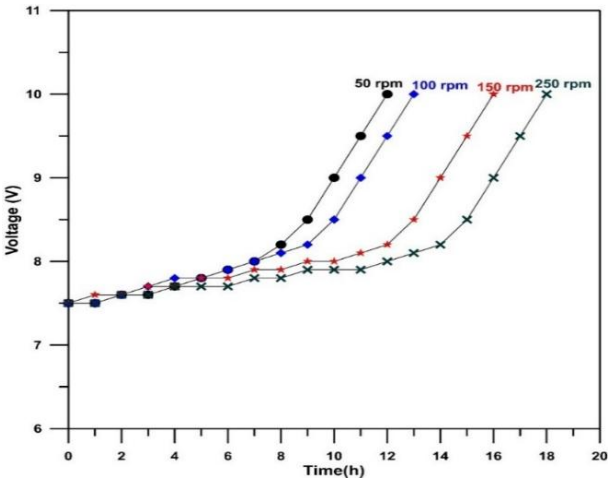


Fig. 7. Accelerated service life curves for Cu/SnO_2-Sb_2O_5 anodes (0.5 M NaOH; 200 mA/cm^2)

Table 2 shows the accelerated service life time of the anode compared with anodes fabricated by different methods and conditions in the literature. Comparing the present anode with Ti/SnO_2–Sb_2O_5 produced by thermal decomposition [36] revealed that the present anode demonstrated a high service life time. Furthermore, the prepared anode exhibited an extended service life under accelerated conditions compared with the same anode produced via electrodeposition at a stationary anode [43]. This indicates that a compact deposit formed on the surface of the present anode due to the rotational effect, resulting in enhanced performance. The stability of the anode depends on the morphology of its surface, as elevated current densities may modify the crystalline structure of the anode, leading to its deactivation. Moreover, the development of cracks in the coating permitted O_2 (formed by the electrolysis of H_2O) to penetrate through the surface of the substrate, causing the production of an insulating layer on the anode [11]. In addition, a passive layer may be fabricated on the surface of anode, resulting in charge transfer failure with increase in cell voltage [44].

Table 2,
Accelerating service life of the present Cu/SnO_2-Sb_2O_5 anode against related anodes

Anode	Preparation method	Test conditions	Service life time(h)	Ref.
Ti/SnO_2–Sb_2O_5	Thermal decomposition	200 mA/cm^2; 0.5 M NaOH	12	36
Ti/SnO_2-Sb	Thermal decomposition	500 mA/cm^2; 1.0 M NaOH	0.6	45
Ti/SnO_2-Sb	Thermal decomposition	100 mA/cm^2; 0.1 M H_2SO_4	4	46
Ti/SnO_2-Sb-4%TiN	Dip coating	100 mA/cm^2; 0.25M Na_2SO_4	9.65	11
Ti/SnO_2-Sb	Dip coating	100 mA/cm^2; 0.25M Na_2SO_4	5.92	11
Ti/SnO_2-Sb	Dip coating	100 mA/cm^2; 0.25M Na_2SO_4	5.0	47
Ti/SnO_2-Sb	Sol-gel	100 mA/cm^2; 0.25 M Na_2SO_4	6	42
Ti/SnO_2-Sb	Sol-gel	100 mA/cm^2; 0.5 M H_2SO_4	0.4	48

$Ti/SnO_2–Sb$	Electrodeposition	100 mA/cm^2; 1.0 M NaOH	12.1	49
$Ti/SnO_2–Sb_2O_5$	Electrodeposition	100 mA/cm^2; 0.5 M H_2SO_4	15	43
Ti/SnO_2-Sb	Electrodeposition	100 mA/cm^2; 0.1 M H_2SO_4	25	46
Cu/SnO_2-Sb_2O_5 Rotating anode	Electrodeposition	200 mA/cm^2; 0.5 M NaOH	18	Present work

Further work should be conducted to enhance the results of the present work via the application of pulsed electrodeposition and doping of other metal oxides to increase the activity and surface lifetime of tin oxide anodes.

4. Conclusion

In this work, a rotating anode composed of Cu/SnO_2-Sb_2O_5 was fabricated by a simple, direct electrodeposition method. The impact of speed of rotation was investigated in the range of 50–250 rpm. The surface of the anode prepared at 250 rpm was examined by SEM, showing compact deposits without cracks. The prepared anode at 250 rpm demonstrated high electrochemical activity for the degradation of MB, with a removal percentage of 93.6% over 4 h at a current density of 20 mA/cm^2. The pseudo-first-order rate constant at 250 rpm was 0.01258 min^{-1} greater than that at 50 rpm by 1.6, confirming the role of rotation in improving the cathodic deposition process.

The prepared anode has an accelerating surface life of 18 h at 250 rpm , which was superior to those prepared by thermal decomposition, dip coating and sol-gel methods. Additionally, it surpassed that produced by cathodic deposition at a stationary anode, confirming the role of anode rotation in obtaining uniform deposits with high electrolytic activity. The revised anode configuration could be easily scaled to a prototype level.

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Conflicts of Interest

The authors declare that there is no conflict of interest regarding the publication of this paper.

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معالجة المياه العادمه بالأكسدة الأنودية باستخدام أقطاب أسطوانية دوارة من أكسيد القصدير

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المستخلص

يعد أكسيد القصدير بمثابة أنود واعد لاكسدة المركبات العضوية المقاومة للأكسدة؛ وذلك كونه رخيص ولا ينتج تلوث ثانوي. في هذه الدراسة، تم تصنيع أنود Cu/SnO_2-Sb_2O_5 عن طريق الترسيب الكاثودي في نظام دوار. تم دراسة تأثير سرعة دوران القطب على الخواص التركيبية للأنود المحضر في المدى من ٥٠ إلى ٢٥٠ دورة في الدقيقة. تم فحص بنية وشكل الأنودات المحضرة عبر تقنيات SEM وXRD . أظهرت النتائج أن أنود-Cu/SnO_2 Sb_2O_5المحضر عند ٢٥٠ دورة في الدقيقة له هيكل طلاء مدمج ومتعدد الطبقات بدون أي تشققات. تم فحص الأداء الكهروكيميائي لأنود-Cu/SnO_2 Sb_2O_5عن طريق تحلل ١٠٠ ملجم/لتر من الميثيلين الأزرق عند ٢٠ مللي أمبير/سم^2 لمدة ٤ ساعات. أظهرت النتائج أن الأنود المحضر عند ٢٥٠ دورة في الدقيقة لديه القدرة على اكسدة صبغة الميثيلين الزرقاء بكفاءة ٩٣,٦٪ وهي أعلى من تلك التي تم الحصول عليها بواسطة الأنود المحضر عند ٥٠ دورة في الدقيقة (٨٥,١٨٪). علاوة على ذلك، كان ثابت التفاعل من الدرجة الأولى ($0.01258\ min^{-1}$) وهو أعلى بمقدار ١,٦ من المعدل الملحوظ للأنود المحضر عند ٥٠ دورة في الدقيقة مما يؤكد أهمية الدوران في تعزيز انتقال الكتلة مما يؤدي إلى ترسب أكسيد القصدير مع النشاط التحفيزي العالي. أكدت النتائج أن الأنود Cu/SnO_2-Sb_2O_5 المحضر بسرعة ٢٥٠ دورة في الدقيقة له عمر خدمة متسارع يبلغ ١٨ ساعة (0.5 مولاريNaOH ؛ ٢٠٠ مللي أمبير/سم^2) بينما الأنود المحضر بسرعة ٥٠ دورة في الدقيقة له عمر خدمة أقل يبلغ ١٢ ساعة. وبالمقارنة مع الأنود Cu/SnO_2-Sb_2O_5 المحضر بالتحلل الحراري، فإن الأنود الحالي له عمر خدمة يبلغ ١,٥ مرة مما لوحظ عند النهج الحراري مما يؤكد نجاح تبني نهج الترسيب الكهربائي في تحضير الأنود-(Cu/SnO_2 Sb_2O_5).