

ELECTROCHEMICAL OZONE GENERATION FOR PALM OIL MILL WASTEWATER TREATMENT USING NICKEL/ANTIMONY DOPED TIN OXIDE ANODES

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Abstract

Ozonation have been employed in organic matter degradation and discoloration process of wastewater. In this study, nickel-antimony doped tin oxide (NATO) anode was employed to generate ozone for palm oil mill effluent (POME) wastewater treatment. NATO was synthesized varying Ni concentrations and calcination temperatures. The materials were characterized by X-ray Diffraction (XRD), Scanning Electron Microscopy (SEM), Energy Dispersive X-ray Spectroscopy (EDX) and X-ray Photoelectron Spectroscopy (XPS) techniques. All materials showed rutile structure. The electrode displayed a smooth cracked mud surface morphology. Regarding the oxidation state, the binding energies of the Sb 3d_{3/2} peak were observed at 540.62 eV and 541.51 eV corresponding to Sb³⁺ and Sb⁵⁺, respectively. The key findings show that increasing calcination temperature increases ozone current efficiency obtained from the absorbances of dissolved ozone in liquid phase and current density, which decreases with increasing Ni content. The highest current efficiency and current density (i.e. ca. 30% and 0.18 Acm⁻² in 1 M H₂SO₄ at 2.7V) was achieved at 2% mole ratio Ni content calcined at 650°C. Regarding POME treatment, discoloration and degradation efficiency increased with electrolysis time from the initial chemical oxygen demand (COD) and total organic carbon (TOC) of 1,780 and 96 mgL⁻¹, respectively under the aforementioned conditions. The highest removal efficiency of 80% was achieved within 10 min discoloration and 15 min for TOC and COD. The electrochemical ozone generation using NATO anode have shown high efficiency in the POME treatment due to [•]OH radicals and O₃. NATO is a promising electrocatalyst candidate for wastewater treatment.

Keywords: Electrocatalyst; Electrochemical oxidation; Nickel/antimony doped tin oxide; Ozonation; Palm oil mill wastewater

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Introduction

Elimination of organic substances, as well as pollutants that are hazardous and toxic in water and wastewater treatment, have been made possible by the application of Advanced Oxidation Processes (AOPs). These processes generally include ozonation, UV/H₂O₂, photocatalytic oxidation, electro-oxidation and Fenton's oxidation (Alhaji *et al.*, 2016). The Process of AOPs is based on the production of hydroxyl radicals at a high oxidation potential (2.87 eV) (Muharam *et al.*, 2019). On the industrial scale, different methods such as Cold Corona Discharge (CCD) and the electrochemical generating of ozone are employed to generate ozone. However, the electrochemical method presents several advantages over the CCD method such as low operation voltage, high concentrations of ozone in the gas and liquid phases with high current efficiency (Christensen *et al.*, 2016) as well as being cost-effective.

Ozone is a strong oxidation agent with a redox potential of 2.07 V. It thus has potential application in a wide range of industrial, domestic and health environments including water treatment (Zakaria and Christensen, 2014), the chemical combustion of resistant organics (Silva *et al.*, 2003a). Ozone is employed due to its bactericidal qualities, does not leave harmful residuals or "taste" and requires relatively short contact times. As a result of these advantages, ozone is used as a disinfectant in water purification processes for the pharmaceutical, brewery, bio-technology and electronic industries, where high standards of water are required in the manufacturing process. Another ozone application is the degradation of refractory organic and inorganic compounds. Ozone can be generated at an anode by the electrolysis of water (Silva *et al.*, 2003b). Ozone current efficiency can be influenced by many factors including: cell configuration, anode material, electrode morphology, current density, electrolyte composition, concentration and temperature (Wang *et al.*, 2013).

Several electrode materials have been investigated over the years including graphite, glassy-carbon, platinum, boron-doped diamond (BDD) and Ni/Sb-SnO₂ (NATO) (Christensen *et al.*, 2013) with respect to ozone activity and selectivity. NATO electrodes was reported for the degradation of refractory pollutant, decolourisation, microorganisms removal and decomposition of organic compound (Wang *et al.*, 2006; Zakaria and Christensen, 2014). Wang and co-workers (2006) reported via electrochemical process, the degradation of 4-chlorophenol (4-CP) using NATO anode. It has been suggested that Ni co-dope Sb-SnO₂ could be

responsible of producing 'OH radicals which can be formed from ozone in the absence of organic molecules.

Zakaria and Christensen, (2014), and Maneelok *et al.* (2018) reported the complete decolourisation of Reactive Blue 50 (RB50) Dye solution by NATO at 2.7V due to the destruction of the anthraquinone moiety. Further, Sun *et al.* (2015) reported alternative materials such as co-doping of Ce-Ni into Sb-SnO₂ that enhanced a phenol degradation and TOC removal. The electrocatalytic activities and the service life were attributed to the changed in oxygen vacancies concentration in the lattice of SnO₂ and hence the generation of reactive oxygen species (ROS) in the solution for the indirect oxidation. With respect to previous studies, NATO could enhance the degradation of toxic organic compounds. However, most of the aforementioned studies were investigated on wastewater, with simple composition. Whilst in real life, wastewater always contained organics, more complex inorganics solid and microorganisms (Parsa and Abbasi, 2007). Thus, the applicability of NATO anodes in real wastewater as Palm oil mill effluent (POME) was investigated herein.

Palm oil mill wastewater treatment is important. The composition of POME is complex, and consists of macromolecules, such as polysaccharides, lipids, proteins, and a number of monocyclic and polymeric aromatic molecules with typically the chemical oxygen demand (COD) of 15,000-100,000 mg/L, biochemical oxygen demand (BOD) of 10,000-43,750 mg/L, pH of 3.4-5.2, oil and grease of 8 g/l, suspended solids of 20 g/l and total solids of 40 g/l (Cheng *et al.*, 2016). Different POME treatment methods have been proposed such as adsorption, electrocoagulation, photocatalytic oxidation, ultrafiltration alongside the conventional method of aerobic and anaerobic treatments (Mohammad *et al.*, 2021 and Al-mahbashi *et al.*, 2022). Unfortunately, these methods are land-intensive while others require long hydraulic and long retention time.

The electrochemical approach was considered as a superior alternative to overcome the drawbacks of the aforementioned wastewater treatment technologies due to its simplicity in operation, no chemical adding required and less sludge production (Ng *et al.*, 2015) and is eco-friendly. This research focused on the palm oil mill wastewater treatment using electrochemical ozone generation method at NATO electrodes. The effect of Ni doping and calcination temperature on the performance of the NATO anodes were investigated with respect to

electrocatalytic activity and the active site for ozone generation. The performance of the NATO anodes with respect to electrocatalytic activity i.e., the ozone generation by electrochemical process for POME treatment was evaluated. Therefore, the work provided an important benchmark in studying the ozone generation via electrochemical process for palm oil mill wastewater using NATO electrode.

Materials and Methods

Electrode preparation and characterisation anodes

The NATO anodes preparation was adapted from Maneelok *et al.* (2018). The coating solutions contained $\text{SnCl}_4 \cdot 5 \text{H}_2\text{O}$, SbCl_3 and $\text{NiCl}_2 \cdot 6 \text{H}_2\text{O}$ (Sigma Aldrich), with the mole ratio Sn:Sb:Ni of 500: 8: x, where x is Ni content between 0.5 and 5. The titanium foil was employed as a substrate which was dipped in the precursors solution. The electrodes were then calcined in a furnace with different temperatures at 450°C, 550°C and 650°C for 1 hour and were allowed to cool in air, as presented in Figure 1. The physical-chemical properties of anodes were observed. The crystal structure was obtained by X-ray diffraction (XRD). The morphology and chemical composition were characterised by Scanning Electron Microscope (SEM) and Energy Dispersive X-ray Spectroscopy (EDX), respectively. X-ray photoelectron spectroscopy (XPS) was performed to analyze the chemical state of the surface.

Ozone measurement

Ozone was generated by Electrochemical oxidation process. A 0.64 cm² Pt-Ti mesh and 1M H_2SO_4 were employed as a cathode and electrolyte, respectively. The electrodes were positioned in a cuvette cell filled with electrolyte and placed in an UV spectrophotometer. The potential of 2.7V for 30s was used to collect a reference spectrum. The ozone current efficiency obtained from the absorbance at 258 nm of dissolved ozone in the liquid phase, assuming an extinction coefficient of 3000 M⁻¹cm⁻¹ (Christensen *et al.*, 2013a). The current efficiency of ozone (%CE) can be calculated from equation 1:

$$\eta = 58 * A_{O_3} / Q \quad (1)$$

where A_{O_3} is the ozone absorbance and Q is the charge passed.

Wastewater collection and characterisation

POME samples were collected from an aeration pond of Palm Oil Company, Nakhon Si

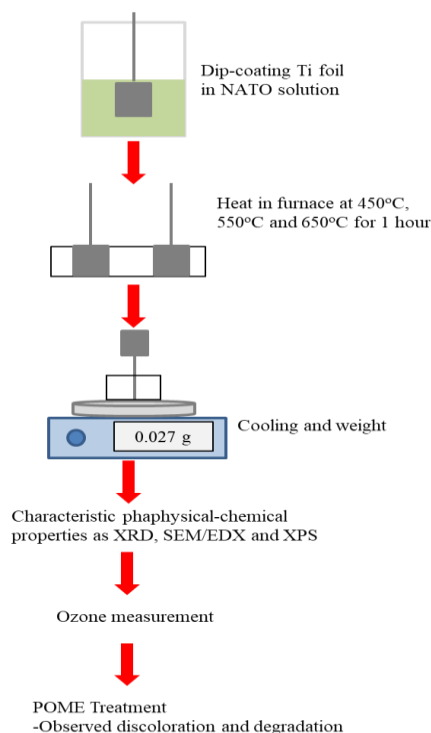


Figure 1. The electrode preparation, ozone measurement and POME treatment process

Thammarat, Thailand. The collected samples were stored in the fridge with the temperature less than 4°C until use.

The experiments of discoloration and degradation were analyzed by the appropriate NATO anodes with the potential of 2.7V as a function of electrolysis times (0.5, 5, 10, 15 and 30 min). The electrochemical cell experiment was the aforementioned conditions. The current and absorbance were recorded during the experiment. Discoloration was measured from the absorbance of the 383 nm band (Sontaya *et al.*, 2013) in term of the ratio A/A_0 . Degradation was observed for chemical oxygen demand (COD) and total Organic Carbon (TOC) using the Closed Reflux, Titrimetric Method (5220C) and the high-temperature combustion method (5310B), respectively (Eaton *et al.*, 1998).

The Palm oil mill wastewater remediation was calculated from the measurements of COD and TOC as follows equation 2:

$$\text{COD, TOC (\%)} = (100 \times (C_i - C_t)) / C_i \quad (2)$$

where C_i is the concentrations of COD and TOC before treatment and C_t the COD and TOC after a time t (Parsa and Abbasi, 2007).

Results and Discussions

Characterisation of anodes

XRD patterns of NATO with different Ni doping concentration are shown in Figure 2. The result of XRD patterns show crystalline single phase of rutile structure. Regarding the peak position of the XRD patterns, the peaks were attributed to (110), (101), (020), (121), (220), (002), (310) and (112) planes of SnO_2 which is in good agreement with crystallographic data base ICSD (Q: S; 00-041-144). No secondary phase was obtained (Sun *et al.*, 2015; Maneelok *et al.*, 2018). With respect to the effect of Ni concentration, it was shown that the crystal structure of NATO sample which contains Ni up to 5 % did not change, suggesting that Ni and Sb substituted Sn ion in the lattice of SnO_2 . It can be elucidated that the ionic radius of Sb^{5+} (0.60 Å) and Ni^{2+} (0.69 Å) have comparable ionic radius of Sn^{4+} (0.69 Å) whilst Sb^{3+} (0.76 Å) and Ni^{3+} (0.72 Å) ions might be placed at the surface (Yang *et al.*, 2012; Sun *et al.*, 2015). The particle sizes of NATO samples were calculated using Scherrer equation. The particle size decreased as a function of Ni doping in the range of ca. 67.42-22.18 nm.

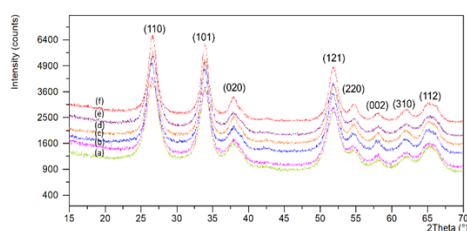


Figure 2. XRD patterns of NATO samples calcined at 650°C with difference Ni content of (a) 0.5%, (b) 1%, (c) 2%, (d) 3%, (e) 4% and (f) 5%

Figure 3 shows SEM image and EDX spectra of the 2NATO anode calcined at 650°C. It can be seen that the surface morphology exhibited the smooth cracked mud morphology which can be resulted from mechanical tension and stress inside the coating caused by the plasticity of the coating, the dopant metals, the cooling process and the difference in the thermal expansion coefficients of the Ti substrate and the coating (Li *et al.*, 2019). In Addition, Li *et al.* (2019) reported that the smooth surface with a smaller amount of cracks may inhibit the penetration of electrolyte. This could protect the Ti substrates from oxidation process, hence electrode was still conductive to improve the electrode stabilization and service life. For the EDX

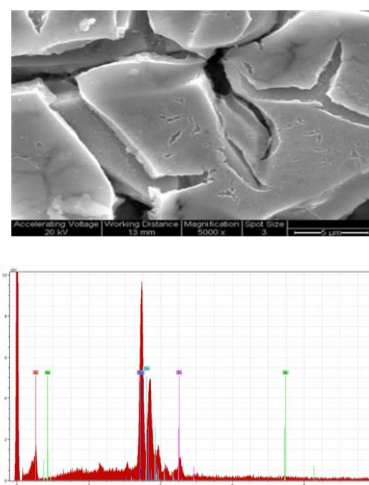


Figure 3. A typical SEM image and EDX spectra of 2NATO anode calcined at 650°C

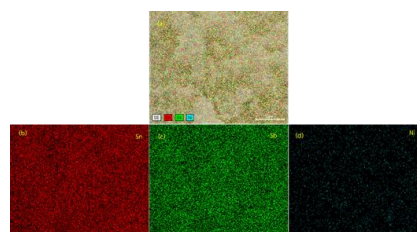


Figure 4. A typical EDX Mapping image of 2NATO anode calcined at 650°C; (a) all elements, (b) Sn (red dot), (c) Sb (green dot) and (d) Ni (blue dot)

spectra in Figure 3 have shown that Sn, Sb and O were detected but not Ni. This might be due to the small amount of Ni doping. Furthermore, EDX mapping analysis was employed to confirm that Ni are still present in the NATO samples. Figure 4 presents the elemental mapping of representative sample of 2NATO. It can be seen that the red, green and blue dots denote Sn, Sb and Ni, respectively. The results show that nickel elements was found in the NATO sample however the Ni peak was not obtained in EDX spectra. Moreover, all elements in Figure 4 are well-dispersed in the NATO sample, supporting the single phase observed using XRD.

The existence of dopants on the surface of material and their chemical oxidation state was conducted by XPS as shown in Figure 5. Figure 5(a) shows the Sn 3d peak splits into Sn 3d_{3/2} and Sn 3d_{5/2}. The peaks were fitted at binding energy (BE) of 495.62 and 487.7 eV, respectively. With respect to chemical oxidation state, the BE was assigned the state of Sn^{4+} in the form of Ni/Sb- SnO_2 (Yang *et al.*, 2012). The chemical shift of Sn 3d state was not detected after doped Sb and Ni dopants. The

separation distance between Sn 3d_{3/2} and Sn 3d_{5/2} peaks of both samples is 8.4 eV. The XPS spectrum in Figure 5(b) show an asymmetric two peaks which was Sb 3d_{5/2} and Sb 3d_{3/2}. The broad asymmetric Sb 3d_{5/2} peak overlaps with O 1s peak, suggesting contributions from other oxygen species i.e. lattice oxygen species (MO_x), bonded oxygen metals (M-O) and metal hydroxides (M-OH). It was reported that the BE around 532.11 eV attributed to adsorbed oxygen (O_{ads}) (Li *et al.*, 2019) plays an important role in electrochemical oxidation to reveal with O₂ and O₃. Furthermore, the Sb 3d_{3/2} peak is generally employed to determine the valence state of Sb. The BE of Sb 3d_{3/2} was observed at 540.62 eV and 541.51 eV corresponding to Sb³⁺ and Sb⁵⁺, respectively, corresponding to other research (Yang *et al.*, 2012).

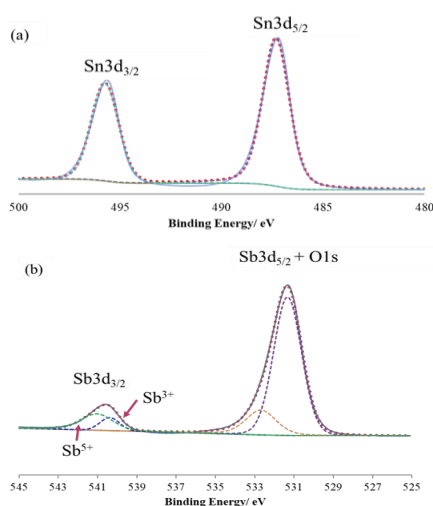


Figure 5. XPS spectra of 2NATO sample calcined at 650°C: (a) the fitted XPS spectra of Sn 3d state and (b) the fitted XPS spectra of Sb 3d state

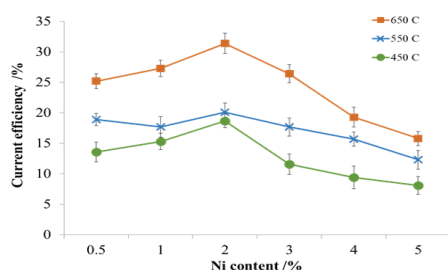


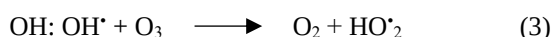
Figure 6. Current efficiency of 2NATO anodes calcined with various calcination temperatures as a function of Ni concentration in 1M H₂SO₄ at 2.7V for 30s

Electrochemical ozone generation

Effect of the concentration of Nickel dopant

The NATO anodes were active to generate ozone due to the absorption peak at $\lambda < 258$ nm which attribute to ozone. With respect to ozone generation as a function of Ni content in Figure 6, the current efficiencies were in the range ca. 10% - 30% for Ni content between 0.5% and 5%. Interestingly, the highest current efficiency was achieved ca. 30% with the nickel concentration of 2%, presented in Figure 6. It can be seen that the current efficiency of all NATO anodes showed similar behaviour that the current efficiencies increased gradually with increasing Ni concentration up to 2%Ni after which decline when loading Ni dopant to 5%. These results might be due to increase the resistance because of increasing Ni dopants (Christensen *et al.*, 2013b). Additionally, the current efficiency decreased because the ratio of O₃ and O₂ active sites decreased with increasing Ni content (Christensen *et al.*, 2013b). Regarding the current density in Figure 6, it decreased linearly with increasing Ni content due to segregation of Ni to the surface. The highest current density was obtained ca. 0.18 Acm⁻² in 1M H₂SO₄ at 2.7V

Moreover, Yang *et al.* (2012) found that high Ni doping levels of around 5 - 7 % in Sb-SnO₂ resulted in decreasing electrocatalytic activity and electrical conductivity. That may be due to side reactions between OER and EOP, and O₂ evolution of OH radicals (Chan *et al.*, 2005), for example, reaction with (equation 3).



Effect of calcination temperature

It can be seen in Figure 6 and Figure 7 that the current efficiency and current density of various Ni contents of NATO anodes calcined at 450°C, 550°C and 650°C show the appropriate calcination temperature. The current efficiency and current density increased as a function of calcination temperature. This might be due to high active site of NATO at high temperature.

Characterisation of POME

The raw palm oil mill wastewater was characterized as shown in Table 1. The properties of POME presented have an initial COD and TOC of 1,780 mgL⁻¹ and 96 mgL⁻¹, respectively. This POME was employed to investigate the degradation and discoloration.

In this work, the 2NATO anode calcined at 650°C was used to remove colour and organic matter in POME wastewater. As shown in Figure 8, the discoloration and degradation of POME

increased as a function of electrolysis time. The colour of POME was removed ca. 80% within 10 min and reached ca. 90% within 15 min. While the COD and TOC removal efficiency required 15 min for 80% and 25 min for 90%. These results showed better performance compared with other research (Alhaji *et al.*, 2016; Li *et al.*, 2019). Moreover, the NATO material give a good performance for the degradation and colour removal in a short period that might be attributed to the generation of hydroxyl radicals ($\text{OH}^\bullet$) and ozone as follow equation 4-5:

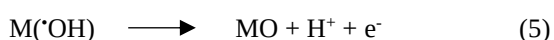


Table 1. Characteristics of raw palm oil mill wastewater

Parameter	Value
pH	6.12
Temp. ($^\circ\text{C}$)	33
COD (mg L^{-1})	1,780
TOC (mg L^{-1})	96

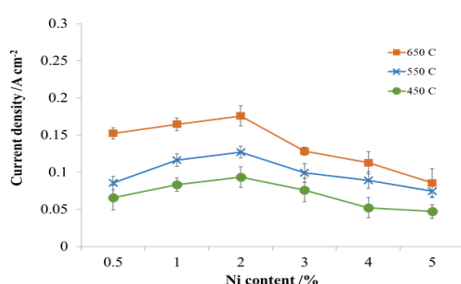


Figure 7. Current density of 2NATO anodes calcined with various calcination temperatures as a function of Ni concentration in 1M H_2SO_4 at 2.7V for 30s

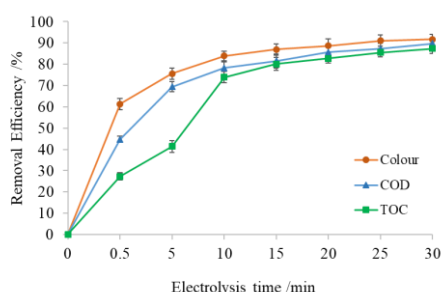


Figure 8. Removal efficiency of colour, COD and TOC using 2NATO anode calcined at 650°C with different electrolysis times at 2.7V

In term of Ti electrode, the reaction between the adsorbed hydroxyl radicals on the anode surface and the active oxygen species are generated the absorbed hydroxyl radical on the surface of Ti anode (Li *et al.*, 2019).

The key finding showed NATO electrodes is a good candidate for electrocatalytic material for ozone generation via an electrochemical process. The NATO anodes were employed to examine the electrocatalytic activities for decolorisation and degradation of organic pollutants in palm oil mill wastewater. Furthermore, the ozone is not only applied for wastewater treatment, but also employed to remove odour. Hence, the NATO was found to be efficient to generate ozone and remove the organic matter in wastewater. However, the ozone generation technology still need further investigation with respect to electrode performance in order to achieve higher active and selective ozone current efficiency.

Conclusions

Nickel and antimony doped tin oxide is a highly active catalyst to generate ozone in order to remediate the organic pollution and undesirable colour of palm oil mill wastewater. NATO catalyst was employed to investigate the surface properties associated with ozone activity. The XRD characterisation showed that all NATO materials which contained Ni concentration up to 5% mole ratio presented a single phase of rutile structure. Sb^{3+} ions located on the surface of the crystallites which was related to possible oxygen active site. The surface morphology exhibited a smooth cracked mud structure caused by mechanical tension. The XPS spectrum of NATO materials confirmed that chemical oxidation state of Sn and Sb exhibited Sn^{4+} , Sb^{3+} , and Sb^{5+} . Remarkably, the ozone current efficiency increases with increasing calcination temperature, whereas decreases with increasing Ni content. The highest ozone current efficiency ca. 30% and current density 0.18 A cm^{-2} was achieved at 2% mole ratio Ni content calcined at 650°C . Regarding POME treatment, discoloration and degradation efficiency increased as function of electrolysis time. 80% removal efficiency of discoloration and degradation was achieved within 10 min and 15 min, respectively. The electrochemical ozone generation using NATO anode have shown high efficiency in the POME wastewater treatment due to $\text{OH}^\bullet$ and O_3 .

Acknowledgments

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