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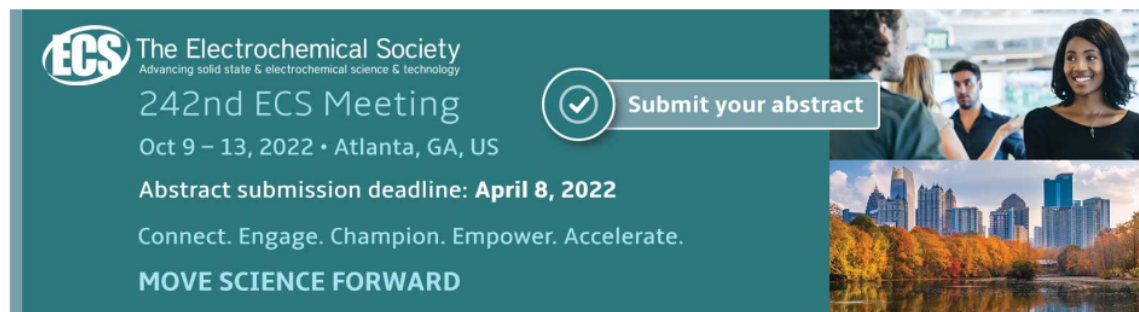
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The effect of calcogenate sulfur on the performance of the S-TiO₂/Ti electrode as a photoelectrocatalytic sensor for phenolic compounds

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Abstract. The TiO₂/Ti and S-TiO₂/Ti electrodes are effectively prepared using the anodizing process. The objective of this analysis is to determine the characteristics of the S-TiO₂/Ti electrode and its efficiency as a photoelectrocatalytic sensor device. The composite electrode of S-TiO₂/Ti was prepared using the Ti plate anodizing method in glycerol and NH₄F 0.27 M with a bias potential of 25 V for 4 h followed by calcination at 500 °C for 90 minutes. Doping S to the TiO₂/Ti electrode was carried out in sol-gel containing sulfur by the dip-coating process then calcined for 10 minutes at 150 °C. The obtained TiO₂/Ti and S-TiO₂/Ti electrodes are characterized by UV-Vis DRS, XRD, SEM-EDX, and FTIR techniques. TiO₂/Ti was shown to be active under UV light while S-TiO₂/Ti was active under visible light. We also observed that the best composition of the S-TiO₂/Ti electrode was 1 M with a current value of 5.71x10⁻⁵ A in the visible area. The S-doped TiO₂/Ti electrode photoelectrocatalytic activity was stronger than the undoped electrodes under visible light area irradiation.

1. Introduction

Phenol is a compound generally used in different industries, so this compound is manufactured in very large quantities. The widespread use of phenols has great potential to be disposed of into the water environment as organic pollutants. The presence of phenol in the aquatic environment can cause serious problems because several harmful phenol derivatives can be formed in the presence of phenol compounds in the environment. Generally, this waste comes into the environment as waste from various industries such as paper, pharmaceuticals, dyes, herbicides, pesticides, plastics, crude oil refining, and hospitals. The average concentrations of phenol in wastewater from various industrial processes varied between 35-8000 mg/L [1–3]. The effects of this compound cause an unpleasant odor, skin irritation, toxicity, respiratory irritation, cause human health problems, damage, and death to aquatic ecosystems [4,5]. Therefore, the development of simple, fast, sensitive, and selective analytical methods is indispensable for phenol detection in aquatic ecosystems.

Currently, several techniques have been used for the determination of phenol pollutants such as High-Performance Liquid Chromatography-Mass Spectrometry (HPLC-MS), [6] and Liquid Chromatography-Mass Spectrometry (LC-MS) [7]. But, these techniques have some weaknesses such as relatively long measurement time, sample preparation is complicated, and high costs analysis [4,8,9].



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Therefore, it is necessary to propose an alternative method for phenol detection that is simple, fast, and easy to apply, one of which is the photocatalyst technique [10]. The photocatalyst method has the advantage of being harmless to the environment because this technique doesn't involve the use of hazardous reagents [11–13]. Even more, electrochemical reactions do not depend on the conditions of the wastewater and can proceed as long as the current is supplied to the electrodes.

Photocatalytic methods have been developed in the last decade using TiO₂ semiconductors. Several of the semiconductors material that has been widely used include TiO₂ [14], SiO₂ [15], ZnO [16], Al₂O₃ [17], BiO₂O₃ [18] and SrTiO₃ [19]. Titanium dioxide (TiO₂) has been attracted attention in recent years to removing organic pollutants like phenolic because of its unique properties, environmentally friendly, inexpensive, non-toxic, corrosion-resistant, and chemically inert [20–22]. Unfortunately, it can only be activated by ultraviolet light, because of the relatively large bandgap (3.20 eV for anatase phase and 3.0 eV for rutile phase), thus limiting its application [23,24]. Thus, several strategies can be implemented to improve TiO₂ performance such as reducing the bandgap energy and increasing the surface area of the photocatalyst.

Some researchers have been reported about photoelectrocatalytic sensors for phenolic compounds such as Syafiuddin et al. [25] reported the result of the degradation of phenolic using titanium dioxide doped with Nitrogen and Sulfur with maximum removal 77.87%. Yunus et al. [26] reported the influence of TiO₂ Co-doped C and S catalysts in the photocatalytic degradation of phenolic compounds. Nam et al. [27] prepared S-TiO₂ composites for the degradation of methylene blue with the thermal hydrolysis method by photocatalytic. Zhu et al. [28] reported a novel method for synthesizing S-doped TiO₂ has been developed to degrade organic pollutants in sunlight and increase photocatalytic operation. However, the reports about the application of S doped TiO₂ as a photocatalytic for phenolic compound degradation are very limited or not reported yet.

In the present work, the determination of phenol using a three-electrode system, a working electrode (S-TiO₂/Ti and TiO₂/Ti electrode), an auxiliary electrode (a platinum wire or sheet), and a reference electrode (Ag/AgCl electrode) was investigated. The Electrode S-TiO₂/Ti was synthesized using the sol-gel technique. Moreover, the sol-gel method can facilitate the control of properties such as morphology, sulfur composition, and in consequence, could improve photocatalytic activity.

2. Materials and methods

The materials used are Titanium tetraisopropoxide (Ti[OCH(CH₃)₂]₄) (TTIP) (Sigma-Aldrich), as the precursor of the TiO₂, sulfuric acid (H₂SO₄) (Sigma-Aldrich) as the S dopant, sodium nitrate (NaNO₃) (p.a) and deionized water as the electrolyte solution, ammonium fluoride (NH₄F) and glycerol 98% (p.a) as the anodizing process, acetylacetonate (Sigma-Aldrich), acetic acid (Sigma-Aldrich), ethanol (Sigma-Aldrich) as a solvent, and 99 percent purity Ti plate used as the electrode manufactured material.

2.1 Electrode preparation

Synthesis of S-TiO₂ using the sol-gel method was carried out by mixing 15 mL of ethanol, 1 mL of glacial acetic acid of 0.1 M and 2 mL of distilled water into a reflux flask containing 4 mL of Titanium Tetra Iso Propoxide (TTIP), 15 mL of ethanol and 0.5 mL of acetylacetonate. The mixture is then refluxed at 50 °C for 3 hours. Furthermore, 4 mL of sulfuric acid with concentrations of 2.0 M, 1.0 M, and 0.5 M was added to obtain TiO₂ sol with sulfur content. The obtained sol was evaporated for 48 hours at room temperature, then heated for 30 minutes at 80 °C. The created sol-gel is put into a beaker. The final stage is the coating process, by immersing the area that has been overgrown with TiO₂ on the titanium plate for 5 minutes. It was then calcined for 15 minutes to form S-TiO₂/Ti electrodes at a temperature of 150 °C.

2.2 Measurement of photocurrent response to the phenolic test compound

Measurement of photocurrent response to phenolic compounds using S-TiO₂/Ti as active electrodes, Ag/AgCl as reference electrodes, and Platina (Pt) as counter electrode were carried out using the Multi Pulse Amperometry (MPA) technique with a bias potential of 0.5 Volt within 60 seconds.

Measurements were made with various concentrations of 0.1, 0.3, 0.5, and 0.7 ppm to see the difference in the resulting photocurrent responses. The determination of a blank solution (0.1 M NaNO₃) as a standard method for the I_{blank} variable, meanwhile the phenolic compound (0.1 M NaNO₃) as a sample to obtain the I_{net} variable.

3. Results and discussion

3.1 Activity test of TiO₂/Ti and S-TiO₂/Ti electrodes using LSV and CV

The photoelectrochemical behavior of electrodes TiO₂/Ti and S-TiO₂/Ti was observed using the Cyclic Voltammetry (CV) and Linear Sweep Voltammetry (LSV) methods in a 0.1 M NaNO₃ electrolyte solution using a 3-electrode portable potentiostat device. Figure 1 shows the reaction of TiO₂/Ti and S-TiO₂/Ti electrodes with UV illumination and without UV illumination.

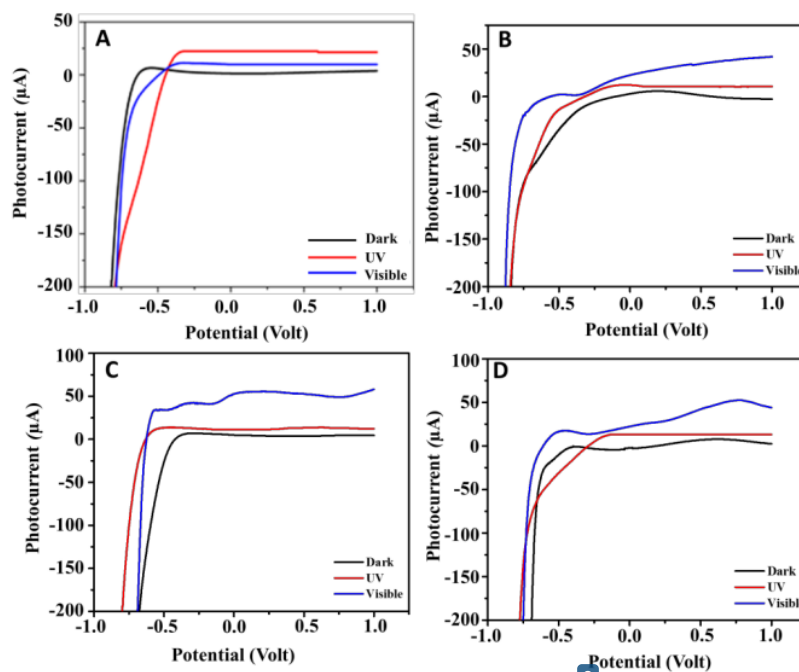


Figure 1. Test of electrode activities by the process of LSV (A) TiO₂/Ti; (B) S-TiO₂/Ti 0.5 M; (C) S-TiO₂/Ti 1 M; and (D) S-TiO₂/Ti 2 M

The light current response of the electrode that is not illuminated by UV light (dark) does not indicate a light current. This is because electrons and holes are not formed as the initiator of oxidation and reduction reactions. When the UV illumination of TiO₂/Ti and S-TiO₂/Ti electrodes shows a high increase in light current, this indicates photocatalytic activity. Figure 1A shows that while exposed to UV light irradiation with a peak current of 2.23×10^{-12} A, the TiO₂/Ti electrode performance is higher. This is consistent with the theory that TiO₂ is active with an energy gap of 3.2 eV only in the UV region with a wavelength of around 388 nm [29–31].

Figures 1 (B), (C) and (D) show that S-TiO₂/Ti electrodes with varying concentrations of sulfur (S) have different photoelectrocatalytic activity. Also, it was observed that the light current produced by the S-TiO₂/Ti electrodes was higher than the TiO₂/Ti electrodes. This is because S doping has decreased TiO₂ bandgap energy and serves as an electron acceptor to increase the electrons and holes that enter the TiO₂ surface. On the other hand, the addition of S dopant to TiO₂ can reduce the energy gap of TiO₂,

so that the electrodes can absorb light at a fairly large wavelength with less energy [32,33]. The peak current from the S-TiO₂/Ti electrode can be found in Table 1.

Table 1. The peak current of the S-TiO₂/Ti electrode

The variations concentration of S-TiO ₂ /Ti electrode	Peak Light Current (μA)	
	UV	Visible
0.5 M	1.22×10 ⁻⁵	4.13×10 ⁻⁵
1 M	1.37×10 ⁻⁵	5.71×10 ⁻⁵
2 M	1.29×10 ⁻⁵	5.23×10 ⁻⁵

It can be shown, based on Table 1 that the results of measurements using S-TiO₂/Ti electrodes with a concentration of 1 M produce a peak value of light current of 1.37.10⁻⁵ μA using UV light and the peak value of light current using visible light is 5.71.10⁻⁵ μA. The S-TiO₂/Ti electrode with a concentration of 1 M is the best because it produces a higher photocurrent response when compared to other electrodes.

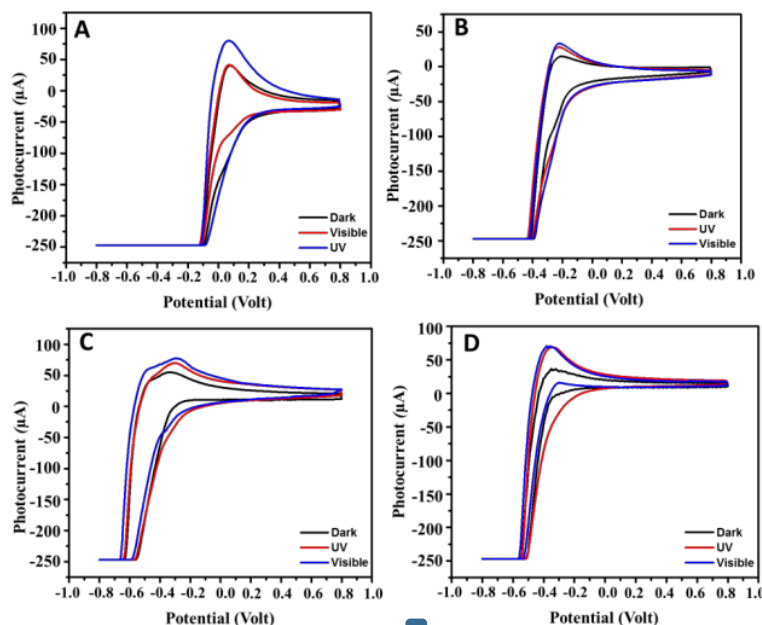


Figure 2. Test of electrode activities by CV method (A) TiO₂/Ti; (B) S-TiO₂/Ti 0.5 M; (C) S-TiO₂/Ti 1 M; and (D) S-TiO₂/Ti 2 M

Furthermore, cyclic voltammogram measurements of the TiO₂/Ti and S-TiO₂/Ti electrodes were carried out to determine the performance of the electrodes in detecting oxidation-reduction reactions that occur in the sensor technique. A cyclic voltammogram is a cyclic depicting the movement of electrons resulting from a redox reaction that occurs on the surface of the working electrode. Figure 2 shows the performance of TiO₂/Ti and S-TiO₂/Ti electrodes using UV illumination and without UV illumination with the Cyclic Voltammetry (CV) method.

3.2 Photocurrent response of the phenol compounds

Photocurrent response measurement of TiO₂/Ti electrodes and S-TiO₂/Ti electrodes to phenol compounds was carried out using the Multi Pulse Amperometry (MPA) method. The test was carried

out on the test solution with a respective concentration of 0.1, 0.3, 0.5, and 0.7 ppm. The amperogram of TiO_2/Ti and S- TiO_2/Ti electrodes against phenol compounds are shown in Fig.3.

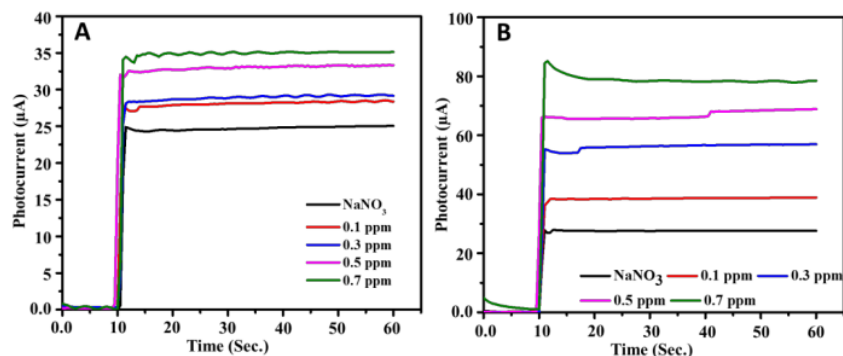


Figure 3. The photocurrent response of phenolic compound (A) Amperomogram of TiO_2/Ti electrode; (b) Amperomogram of S- TiO_2/Ti electrode

Based on Figure 3A, B, it is visible that the photocurrent value increases with increasing solution concentration. The resulting light current is the sum of the oxidation currents of phenol compounds and the oxidation currents of electrolyte solutions [34,35]. The presence of this electrolyte solution increases the conductivity of the solution which is directly proportional to the current of light. According to Nurdin et al. [36], the light current of the solution containing the analyte will coincide with the blank solution light current, which indicates that the degradation process is complete. However, the picture above does not show the light current curve of the analyte coincides with the light current of the blank solution, this is due to the too large volume of the phenolic compound solution so that the oxidation process is imperfect within 60 seconds.

The performance of the two different electrodes, namely TiO_2/Ti and S- TiO_2/Ti , will be known by observing the closeness of the charge value produced by the two electrodes to the theoretical produced charge value. Figure 4 shows Q_{net}'s relationship with phenolic compounds.

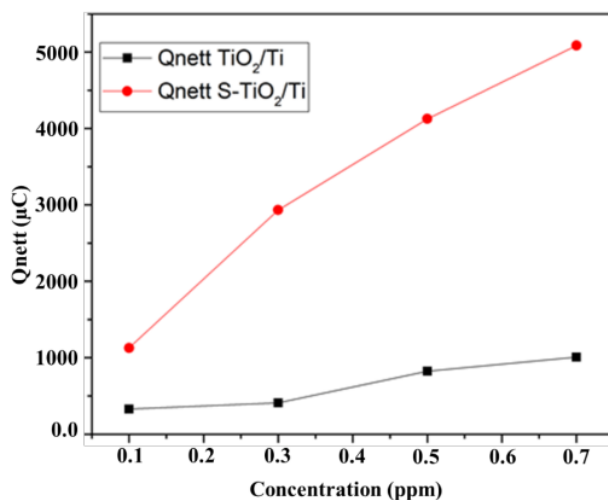


Figure 4. The relation between Q_{net} and concentration of phenolic compound

In general, S-TiO₂/Ti electrodes have better accuracy than TiO₂/Ti electrodes. The performance of the S-TiO₂/Ti electrode shows the highest level of accuracy in phenolic compounds. This is due to the simple molecular structure of phenol which makes it easy to mineralize. Also, the resulting charge is directly related to the test compound's concentration. This is in line with the law of Faraday, which states that the greater the solution concentration, the greater the charge received. This is due to the interaction of the analyte molecule and the surface of the electrode. The strong interaction with the surface of the catalyst causes an increase in the rate of oxidation of organic compounds so that the resulting load is greater.

4. Conclusion

In summary, a novel TiO₂/Ti and S-TiO₂/Ti electrode were prepared using the anodizing process. The result demonstrated that the TiO₂/Ti electrode was only active under UV light irradiation, whereas the S-TiO₂/Ti electrode was active in the visible light region. We also observed that the S-TiO₂/Ti electrode with the best composition of sulfur was 1 M with a current value of 5.71 x 10⁻⁵ A in the visible area. The S-doped TiO₂/Ti electrode photoelectrocatalytic activity was stronger than the undoped electrodes under visible light area irradiation.

Acknowledgement

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