



Spontaneous electrochemical treatment for sulfur recovery by a sulfide oxidation/vanadium(V) reduction galvanic cell



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ABSTRACT

Sulfide is the product of the biological sulfate reduction process which gives toxicity and odor problems. Wastewaters or bioreactor effluents containing sulfide can cause severe environmental impacts. Electrochemical treatment can be an alternative approach for sulfide removal and sulfur recovery from such sulfide rich solutions. This study aims to develop a spontaneous electrochemical sulfide oxidation/vanadium(V) reduction cell with a graphite electrode system to recover sulfide as elemental sulfur. The effects of the internal and external resistance on the sulfide removal efficiency and electrical current produced were investigated at different pH. A high surface area of the graphite electrode is required in order to have as less internal resistance as possible. In this study, graphite powder was added (contact area >633 cm²) in order to reduce the internal resistance. A sulfide removal efficiency up to 91% and electrical charge of more than 400 C were achieved when using five graphite rods supplemented with graphite powder as the electrode at an external resistance of 30 Ω and a sulfide concentration of 250 mg L⁻¹.

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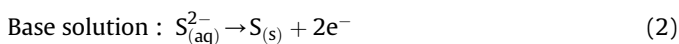
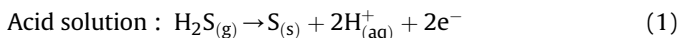
1. Introduction

Sulfide is present in many domestic and industrial wastewaters (Dutta et al., 2008; Pikaar et al., 2012, 2011) as well as in the effluent of sulfate reducing bioreactors (100–250 mg L⁻¹) (Kijjanapanich et al., 2013). This sulfide is toxic and gives offensive odor problems and sewer pipe corrosion (Vincke et al., 2001). Sulfide can also interfere with the iron-phosphate precipitates in soils and sediments due to the formation of iron sulfides and associated release of phosphorous, ultimately leading to eutrophication of water bodies (Smolders and Roelofs, 1993). Therefore, sulfide removal from wastewaters or biological sulfate reducing reactor effluents is required from both an environmental and economic point of view (Dutta et al., 2008).

Common sulfide removal processes are precipitation as metal sulfide, particularly iron sulfide (Firer et al., 2008; Nielsen et al., 2008; Zhang et al., 2009), or biological sulfide oxidation to elemental sulfur (S⁰) (Lens et al., 2002; Sahinkaya et al., 2011). Conversion of sulfide to elemental sulfur either in acid or base conditions is an oxidation reaction (Equations (1) and (2)) where an electron acceptor is required to fulfill the redox reaction. Either chemical or biological processes can be applied for sulfide oxidation to elemental sulfur (González-Sánchez and Revah, 2007). Nowadays, biological sulfide oxidation using oxygen as electron acceptor and sulfide oxidizing bacteria as a catalyst is a popular system (González-Sánchez and Revah, 2009; Henshaw and Zhu, 2001; Krishnakumar et al., 2005; Sahinkaya et al., 2011). However, this system requires energy for oxygen supply (Syed et al., 2006; van den Ende et al., 1996), complicated operation protocol (Syed et al., 2006) and the pH conditions of these biological systems are usually mildly or strongly acidic (Gabriel and Deshusses, 2003; Kraakman, 2003). Oversupply of oxygen also yields low sulfur removal efficiencies, since most sulfide is then converted to sulfate instead of elemental sulfur (Janssen et al., 1995).

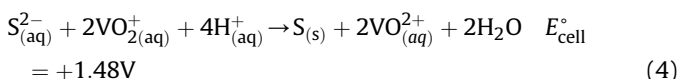
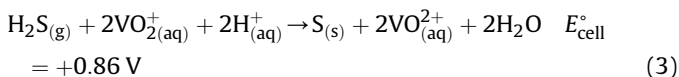
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Electrochemical treatment of sulfide rich wastewaters can be an alternative treatment that offers several advantages, including good energetic efficiency, environmental compatibility, versatility, selectivity and cost effectiveness (Ángela et al., 2009; Dutta et al., 2009; Rajeshwar et al., 1994). The ideal electron acceptor is the one which can provide a spontaneous reaction or produce a galvanic cell. Thus, not only the sulfide is removed, but the produced elemental sulfur can be recovered and electricity will be generated when the oxidation and reduction reactions occur in separated chambers.

Very few galvanic cells based on this principle have been developed for treating sulfide containing wastewater. One cell used hexacyanoferrate (III) ion ($\text{Fe}(\text{CN})_6^{3-}$) as an electron acceptor (Dutta et al., 2008, 2009). In this study, vanadium with oxidation state 5+, i.e. VO_2^+ , was selected, as it has been thermodynamically shown that VO_2^+ is able to perform a spontaneous redox reaction with sulfide/sulfur oxidation as illustrated in Equations (3) and (4). Referring to these equations, the sulfide presented in the ion form (S^{2-}) gives a higher standard cell potential (E_{cell}^0) than H_2S . This means that electricity generated by alkaline sulfide wastewater treatment cells is higher than that by acidic cells.



Whenever oxidation and reduction chambers are connected with an external resistance (R) (Fig. 1), electrons will transfer from the oxidation (anode) to the reduction (cathode) part. Thus, direct electric current (I) occurs. Moreover, the amount of sulfide converted to elemental sulfur varies in accordance with the amount of

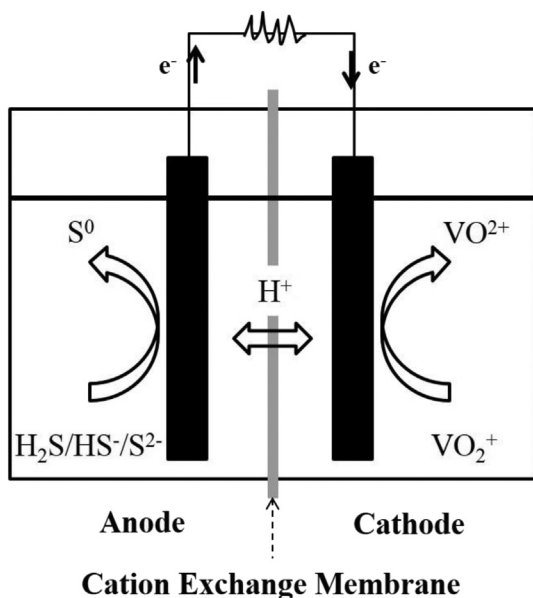


Fig. 1. Schematic representation of the electrochemical sulfide oxidation/vanadium(V) reduction reactor (side view).

electrons flowing through the cell circuit. From Equation (1), two moles of electrons have to be transferred for the production of one mole of elemental sulfur. This means that the rate of electron transfer, i.e. electric current, determines the rate of sulfide oxidation or elemental sulfur production. Therefore, the higher the electric current is produced, the higher the sulfide removal efficiency will be achieved.

The electrical charge value was determined by extrapolating the area under the current/operating time curve. This value can be used for the calculation of the amount of sulfur produced or sulfide removed as Equation (5):

$$\text{Elemental sulfur production, } g = \frac{\text{Electrical charge} \times 32}{2F} \quad (5)$$

where F is the Faraday constant (96485 C mol^{-1}).

Theoretically, current production depends on both the external and internal cell resistance (r) as shown in Equation (6). The highest current production will be obtained when minimum resistances, both external and internal, are employed. However, if the external resistance is too low, the sulfide oxidation rate will be high and a lack of electron transfer can occur because most of the power output of the voltage source is dissipated as heat inside the source itself (Fitzpatrick, 2007). Meanwhile, the sulfide oxidation rate will be slow when a high external resistance is applied. There are many factors affecting the internal cell resistance, for example, type and surface area of the electrode, surface area of the cation exchange membrane, concentration of ions in the solution, etc. If other factors are fixed, electrical current will depend directly on the internal cell resistance.

$$I = \frac{E_{\text{cell}}}{R + r} \quad (6)$$

Some thermodynamic spontaneous reactions proceed at very slow rates at ambient temperature and pressure. Pre-testing prior to this study with mixing of a sulfide solution with a meta-vanadate solution showed that yellow precipitates of elemental sulfur are formed immediately (data not shown). This shows that the redox reaction is a spontaneous reaction both from a thermodynamic and kinetic point of view. Therefore, this research was conducted to treat the effluent of sulfate reducing bioreactors by using a spontaneous electrochemical sulfide oxidation/vanadium(V) reduction cell in a graphite electrode system. The performance of the sulfide removal efficiency at different pH values was evaluated. Different numbers and types of electrodes were studied in order to investigate the effect of the internal resistance of the cell on the sulfide removal efficiency and electrical current produced.

2. Material and methods

2.1. Sulfide wastewater samples

2.1.1. Synthetic sulfide wastewater

Sulfide in buffer solutions of pH 7 and 10 was used as synthetic wastewater. The pH 10 buffer solution was prepared by dissolving a carbonate buffer, i.e., 5 g NaHCO_3 + 1 g NaOH in anoxic water (boiled and cooled to ambient temperature demineralized water), then 1872 mg washed crystals of sodium sulfide ($\text{Na}_2\text{S} \cdot 9\text{H}_2\text{O}$) were added and dissolved. The final volume was made up to 1 L with anoxic water. The concentration of this sulfide solution was 7.8 mM (250 mg L^{-1}). For pH 7 buffer solution, phosphate buffer, i.e., 4 g Na_2HPO_4 + 5 g KH_2PO_4 + 1 g NaCl , were dissolved instead of the carbonate buffer.

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