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RESEARCH ARTICLE

OPTIMIZATION OF ELECTROCHEMICAL PROCESS FOR REMOVING SULPHATE FROM DRINKING WATER BY TAGUCHI MODEL

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ABSTRACT

Consumption of drinking water with high concentration of sulphate was resulted in osmotic diarrhoea, especially in infants. The aim of this applied-analytical study was to investigate sulphate removal from urban drinking water using batch reactor electrochemical. I examined sulphate removal efficiency in different operating cases of the variables current density (1 to 8 mA/cm²), electrodes material (aluminium, copper, iron, steel, and zinc), pH (6 to 8), and time (5 to 40 min). I determined sulphate according to procedure detailed in standard methods. Batch experiments showed that the maximum sulphate removal obtained using zinc-copper as the anode-cathode electrode arrangement. The increase in current density from 1 to 8 mA / cm² in at optimum electrode and pH increased sulphate removal from 71 to 100%. The increase in time from 5 to 40 min at optimum electrode and pH increased sulphate removal from 67 to 100%.

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INTRODUCTION

The sulfate anion is a polyatomic anion with the empirical formula SO_4^{2-} , a molecular mass of 96.06 daltons (96.06 g/mol), and valance of -2. The most important form is sodium sulfate which is applied in different industrial products. Sulfate could be found in natural water at concentration ranging from a few tenths to several thousand milligrams per liter. The highest concentration is usually found in groundwater source and is considered to be a combination of sulfates from atmospheric (about 30%), biological, and geochemical sources. High sulfate concentration in water has been reported for example a zinc mine in Idaho (63000 mg/L). Natural sources of sulfate may emit from dissolution of sulfate minerals, oxidation of pyrite, oxidation of organic sulphur by microorganisms. Anthropogenic sources of sulfate may emit from mining industry (the most hazardous source), reverse osmosis reject water, Kraft pulp and paper mills, and textile mills (Davies, 2007). High sulfate concentration in water draining mining operations has been reported for example mines in British Columbia (BC) (10- \geq 2000 mg/L).

The detergent and glass industries are the most significant consumer of sodium. The glass industry in Europe, as the greatest consumer of sodium sulphate, has used about 110,000 t annually from 1970 to 2006 (Suresh and Yokose, 2006). Sulfate toxicity is related to chloride and hardness concentrations therefore water quality chemistry and characteristics are calculated when setting the sulfate standard throughout the USA. Sulfate is not a toxicant, but rather is a common salt necessary for life at a few amounts. Sulfate is noted as being less toxic than chloride or bicarbonate (Lasier and Hardin, 2010). Sulfate salts are famous to have laxative properties, osmotic diarrhoea, especially in infants (Backer, 2000); metabolic acidosis; ulcerative colitis (Magee *et al.*, 2000); renal failure (Cole and Evrovski, 2000); and hyperthyroidism. Sodium sulfate do not appear to be tumorigenic. Increasing sulfate concentrations has the potential to result in enhancing phosphorus (P) mobilization rate in riverine sediment (Zak *et al.*, 2006), and groundwater (Geurts *et al.*, 2009). Increasing sulfate leads to rising P mobilization and may potentially take part to eutrophication in surface water. Sulfate additions increase methyl mercury production.

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Methyl mercury is a bio-available form of mercury which can biomagnify in the food and produce severe health disorders in humans (Kainz *et al.*, 2008). Health Canada (2011) recommends an aesthetic objective for sulfate in drinking water of no more than 500 mg/L, based on taste considerations (Health Canada, 2011). The US Environmental Protection Agency (EPA) records that about 3% of the public drinking water systems in the country may have sulfate amounts of 250 mg/L or higher (US EPA, 2011). The EPA secondary maximum contaminant level (SCML) is 250 mg/L for sulfate taste and laxative effects. Two point-of-use (POU) methods for removing small quantities of water contain distillation and RO. The most usual point-of-entry (POE) of treating all of the water used in home is ion exchange (IE). However, brine generation and regeneration are some of the disadvantages of IE adsorbent processes (O. Skipton *et al.*, 2010). The most usual method for sulfate removal performs addition of lime, as a chemical precipitant agent, particularly when acidic water is being treated. This requires permanent monitoring and management. The costs of RO and membrane methods are generally higher than the chemical precipitation methods and biological processes. Electrochemical removal is an alternative method to remove sulfate from ground water. The advantages of electrochemical technology are simple reactor arrangement, the lack of the requirement for chemicals post-treatment. This process is selective for sulfate removal (Szpyrkowicz *et al.*, 2006). The products formed during electrolysis, such as $M(OH)_n$, H_2 , H^+ , depend on experimental conditions including cell arrangement, coexisting ions, electrode materials, and pH. Girczy and Kupich also investigate important performance for sulfate removal in the presence alkaline condition (Girczy and Kupich, 2006). The main purpose of this study is to investigate the effects of the primary operational factors on the efficacy of the batch electrochemical reactor in treating sulfate-contaminated urbane drinking water. These factors are included current density, electrode material, pH, and time.

MATERIALS AND METHODS

Preparation of water sample

Sulfate-contaminated water samples used for electrochemical experiments are obtained from urbane distribution system situated the site of a laboratory in Islamic Azad University, Tehran Medical Sciences Branch in Tehran city. The samples are tested for the main physicochemical characteristics. The mean values of these water characteristics are presented in Table 1.

Table 1. The main physicochemical characteristics of sulfate-contaminated urbane water

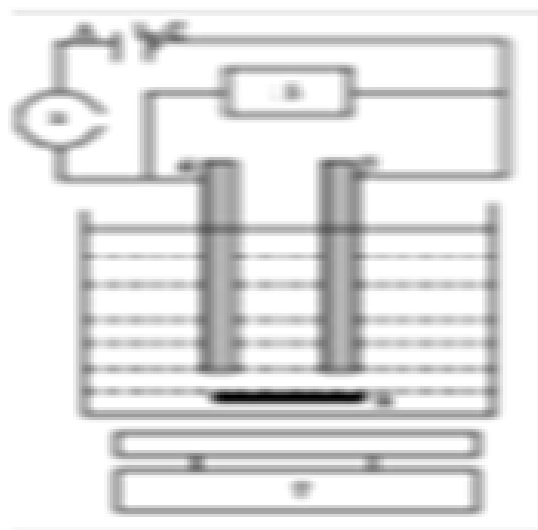
Parameter	Unit	Value
Calcium	mg/L as $CaCO_3$	162
Dissolved oxygen	mg/L	8.05
Nitrate	mg/L	9.5
ORP	mV	279
pH	-	7.19
Sulphate	mg/L	93.8
Temperature	$^{\circ}C$	20
Total Alkalinity	mg/L as $CaCO_3$	122

Preparation of electrodes

The weight of electrode is measured after washing with distilled water. The electrode is pre-treated by washing with detergent, tap water. The cleaned electrode is dried before immersing in the reactor in water (Moussavi *et al.*, 2011).

Experimental set up

The batch electrochemical reactor is a 360 ml glass vessel ($10 \times 6 \times 6$ cm) Figure 1c. Various materials of aluminium (Al), copper (Cu), iron (Fe), steel (As), and zinc (Zn) are used as anode and cathode electrodes. The area of each electrode is 36 cm^2 ($9 \times 4 \times 0.1$ cm). The distance between electrodes is adjusted to 2 cm. The AC electrical source has maximum electrical power of 60 W. To evaluate the effect of electrolysis, on the sulfate removal process, samples undergo with different current density ($1-8 \text{ mA/cm}^2$), electrodes material (Al, Cu, Fe, As, and Zn), pH (ca. 6-8), and different times (5-40min). Magnetic stirrer (AiKa, Germany) is used for homogeneous mixing of water samples (Table 2). For each test, 200 mL of sample water is poured into the reactor. All tests are performed at laboratory temperature ($20 \text{ }^{\circ}C$). Chloride acid and sodium hydroxide solutions (0.1 N) are used for pH adjustment.



1. Power supply, 2. Current density ($1-8 \text{ mA/cm}^2$), 3. Voltage volume ($1-60 \text{ V}$), 4. Cathode electrode, 5. Anode electrode, 6. Magnet, 7. Magnetic stirrer

Fig. 1. The batch electrochemical reactor

Analytical methods

All tests are performed in triplicate, and the mean data values are reported. The water samples are tested for sulfate, ORP, pH, and temperature after electrochemical by using spectrophotometer (DR 2010, America), ORP-meter (CG, Malesia), pH-meter (Hack, America), respectively. Sulfate is determined according to procedure detailed in standard methods (AWWA, 2012). The percentage sulfate removal is calculated according to the following Eqn (1):

$$R(\%) = 1 - \frac{C_e}{C_0} \quad \dots \dots \dots (1)$$

Where the percentage of sulfate removal (R , percentage) and the sulfate value before and after treatment (C_0 and C , mg/L) expressed.

Kinetics reaction models are calculated according to the following Equns (2) and (3):

$$\ln C_t = \ln C_0 - K_{1t} \dots\dots\dots (2)$$

$$\frac{1}{C_t} = K_{2t} + \frac{1}{C_0} \dots\dots\dots (3)$$

Where C_0 and C_t are the concentration of sulfate at the beginning and after time t of the reaction, respectively. K_1 and K_2 are the first, and second order reaction constants, respectively. Values of K_1 and K_2 can be calculated from the slope of the plots $\ln C_t$ versus t , and $1/C_t$ versus t , respectively. Electrodes are rinsed with distilled water after conducting all tests.

RESULTS

The results obtained from this study were shown below. The effects of current density, electrodes material, pH, and different times on the performance of electrochemical reactor in sulfate removal from sulfate-contaminated urbane drinking water were investigated.

Effect of electrode materials

Fig. 2 showed the effects of anode and cathode electrode materials on the sulfate removal at the experimental conditions given in Table 2. As found in Fig. 2, AS-AS as anode-cathode electrodes produced the mean of the lowest sulfate removal (0.0%), while Zn-Cu as anode-cathode electrodes produced the mean of the highest nitrate removal (100%) at similar experimental conditions Table 2.

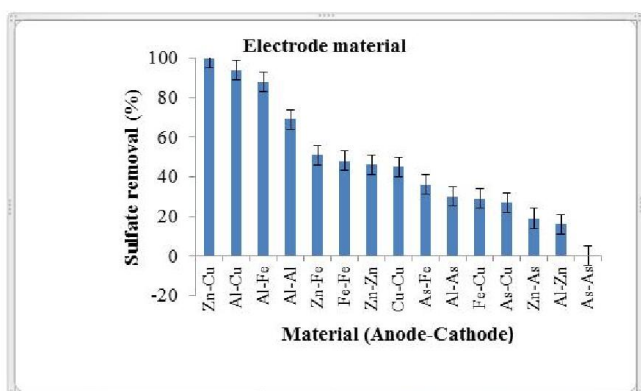


Fig. 2. The effect of electrodes material on the sulfate removal in the batch electrochemical reactor

Sulfate removal in the electrochemical reactor was seriously affected by the electrode material. Therefore, the material which would be produced an oxidation reduction potential just sufficient to transform water (H_2O) to hydrogen (H_2 gas) would be the preferred material.

Arslan-Alaton selected aluminum and stainless steel electrodes for the study of color removal. With stainless steel electrodes only 700 g/m^3 sludge production was obtained under the experimental conditions employed (Arslan-Alaton *et al.*, 2009). The higher removal with aluminum electrodes might be due to their finer bubble producing capacity. El-Nass *et al.* reported a higher sulphate removal from petroleum refinery wastewater with Al electrode than with Fe, and As electrodes in a reactor (El-Nass *et al.*, 2009). Jing-welet *al.* defined a higher sulphite removal from tannery wastewater with As, and Fe electrodes than with Al electrode in a reactor (Jing-welet *al.*, 2007).

Effect of water pH

Electrochemical experiments were carried out an initial pH values in the range of 6 to 8 at the experimental conditions given in Table 2. The results were indicated in Fig. 3. In the case of the Zn-Cu as anode-cathode electrodes, the mean sulphate removal decreased from 100% to 80% when the pH increased from 6 to 8. Sulfate removal in the electrochemical reactor was mainly influenced by the water pH. The mean sulfate removal slowly decreased when the pH was increased from 6 to 7. The mean sulphate removal sharply decreased when the pH was increased from 7 to 8. This finding supported that electrochemical efficiency was a function of pH. Canizares *et al.* concluded that pH had an overall effect on the treatment efficiency for electrocoagulation for synthetic oil-in-water emulsion and effluent from a door manufacturing facility (Canizares *et al.*, 2009). The possible electro reduction mechanism for sulfate removal was described as follows: in the electrochemical reactor, the main reaction at the anode was oxygen evolution and cathions evolution:

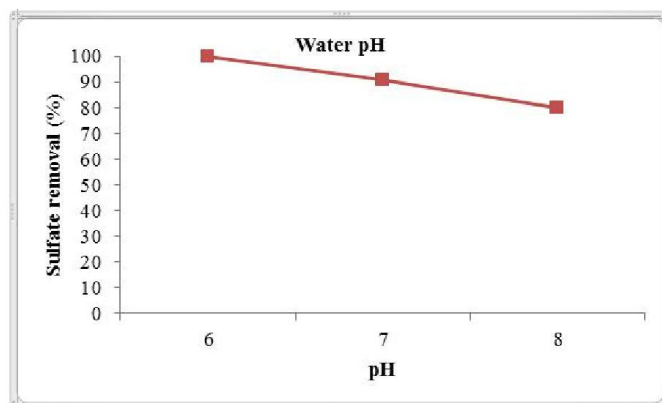
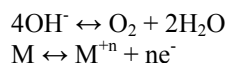
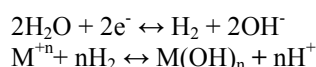


Fig. 3. The effect of water pH on the sulfate removal in the batch electrochemical reactor



The main reaction at the cathode was hydrogen evolution



Reaction of sulphate reduction led to increasing water pH and temperature. Canizares *et al.* also reported the similar results (Canizares *et al.*, 2009).

Liu *et al.* reported that Eriochrome Black T reduction was degraded under the most efficient of experimental conditions employed (current density: 5 mA/cm² and pH 5.5) (Liu *et al.*, 2009).

Effect of current density

Electrochemical experiments were carried out at a current density levels in the range from 1 to 8 mA/cm² at the experimental conditions given in Table 2. The efficiency of sulfate removal increased as the current density and time increase. The results were indicated in Fig. 4. In the case of the Zn-Cu as anode-cathode electrodes, the mean sulfate removal increased from 71% to 100% when the current density increased from 1 to 8 mA/cm². Performance and economy of sulphate removal in the electrochemical reactor was influenced by the current density, pH, and reaction time. Bazrafshan *et al.* also reported the similar results. The increase of mean sulfate removal as a result of increase in the current density was related to the finer bubble producing in cathode electrode (Cu) (Bazrafshan *et al.*, 2006). Therefore, finer bubble evolution was enhanced, increasing the reduction conditions based on the mechanisms given. The sulphate removal as a function of current density was proportional to the sulphate concentration in water. Mahviet *et al.* also reported the similar results (Mahviet *et al.*, 2009). The increase of mean sulphate as a result of increase current density and reaction time was related to the rate increase of the oxidation indirect effect. Priya *et al.* also reported the similar results (Priya *et al.*, 2011).

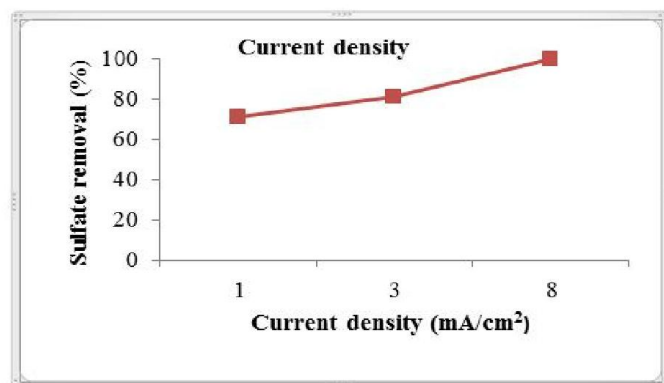


Fig. 4. The effect of current densities on the sulfate removal in the batch electrochemical reactor

Anode weight loss

Fig. 5 showed the value of anode weight loss in the electrochemical reactor at the experimental conditions given in Table 2. As found in Fig. 5, the anode consumed mass increased with increasing current density. In the case of the Zn-Cu as anode-cathode electrodes, the mean anode weight loss increased from 2 mg to 22 mg when the current density increased from 1 to 8 mA/cm². The increase of mean sulphate removal as a result of increase in the current density was related to the rate increase of the consumed anode (Zn). Del Angle *et al.* also reported the similar results (Del Angle *et al.*, 2014). It could be concluded that the mean anode weight loss from 2 to 8 mg as a result of increase in the current density from 1 to 3 mA/cm² followed linear equation.

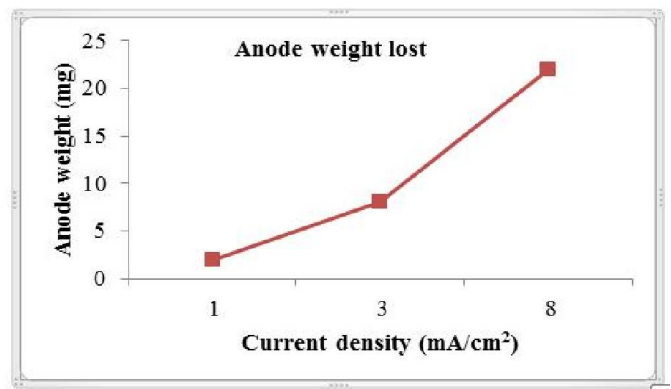


Fig. 5. The anode weight lost in the electrochemical reactor as a function of current densities

The mean anode weight loss from 8 to 22 mg as a result of increase in the current density from 3 to 9 mA/cm² never followed linear equation.

Effect of time

Electrochemical experiments were carried out as a function of the time levels in the range of 5 to 40 min at the experimental conditions given in Table 2. The efficiency of sulfate removal increased as the time increased. The results were indicated in Fig. 6. In the case of the Zn-Cu as anode-cathode electrodes, the mean sulfate removal increased from 67% to 100% when the time increased from 5 to 40 min. The increase of mean sulphate removal as a result of increase in the current density was related to the amount decrease of time that could be attributed to increasing opportunity for adsorption / desorption reactive material species and blocking current. Similar effects of reaction time the electrochemical were also reported for treatment nitrate contaminant by other researchers when using other reaction times (Alizadeh *et al.*, 2013; Polatieds *et al.*, 2005; Li *et al.*, 2009; Li *et al.*, 2010; Li *et al.*, 2010; Ding *et al.*, 2010).

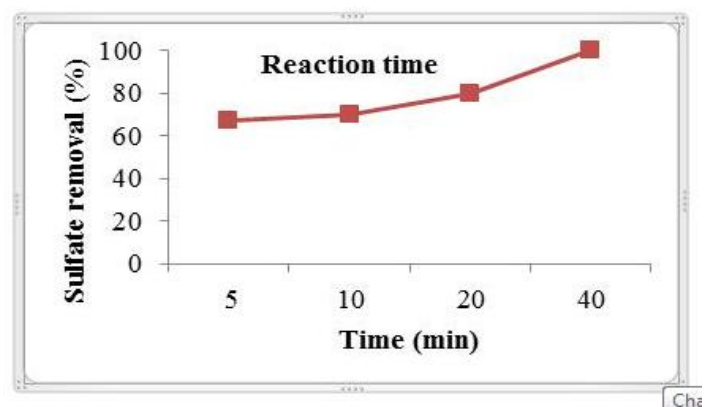


Fig. 6. The effect of time on the sulfate removal in the batch electrochemical reactor

Oxidation reduction potential (ORP)

Fig. 7 showed the effects of anode and cathode electrode materials on the ORP value at the experimental conditions given in Table 2.

As found in Fig. 7, As-As anode-cathode electrodes produced the mean of the lowest ORP value (+8 mV), while Zn-Cu as anode-cathode electrodes produced the mean of the highest ORP value (-338 mV) at similar experimental conditions (Table 2). It seemed that under high reducing environment (ORP -338 mV) with Zn-Cu as anode-cathode electrodes sulfate rapidly converted to products such as hydrogen gas.

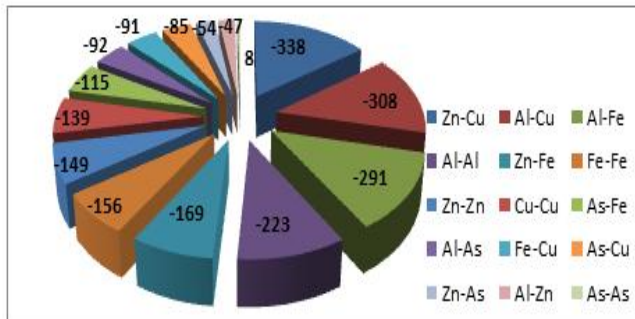


Fig. 7. The effect of electrodes material on the ORP value in the batch electrochemical reactor

Kinetic studies

Fig. 8 showed the plots of the kinetics first, and second order reaction models fitted with the sulfate removal experimental data in batch electrochemical reactor at the experiments conditions given in Table 2.

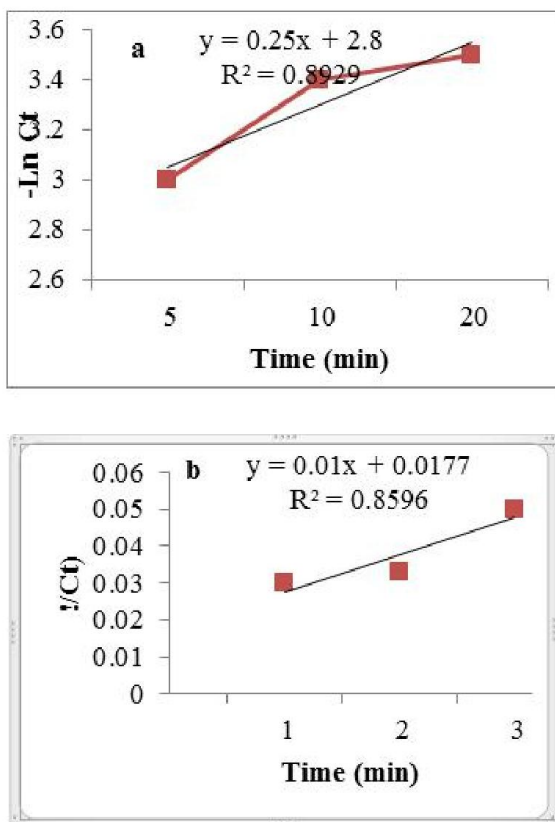


Fig. 8. The plots of first, and second order reaction models fitted with the sulfate removal experimental data in batch electrochemical reactor (experimental conditions: 20 °C, pH: 8, reaction time: 5-40 min)

The experimental data fitted better to the first order reaction. The results of sulfate removal efficiency by Taguchi model showed that reaction time was the most important variable. As expected, for the correlation coefficient (R^2) increased, accordingly the experimental data fitted better were increased. The increase of electrical current intensity as a result of increase in reaction time was related to the rate increase of the electrical conductivity. The decrease of current density led to increasing demand reaction time in order to obtaining alike performance.

Conclusions

The chemical reduction of sulfate from urbane drinking water was investigated in an electrochemical reactor in batch and monopolar electrodes connection mode. Several operational variables were examined for the effects on process reduction efficiency. The following conclusions were obtained from the experiments

- The highest removal of sulfate was given with applying Zn-Cu as anode-cathode electrodes (100%).
- The reaction time significantly effected on sulfate reduction during the process; reduction of sulfate enhances with increasing reaction time, for given experimental conditions.
- The pH had a significant effect on sulfate reduction, with the highest reduction obtained at pH 6.
- The 8 mA/cm² current density achieved the highest removal of sulfate during the reactor with applying Zn-Cu as anode-cathode electrodes (100%).
- Sulfate reduction followed a first-order rate equation during reactor.
- The removal mechanism was attributed to increasing reduction conditions as result of hydrogen evolution at the cathode.
- The electrochemical reactor, in batch and monopolar electrodes connection mode, was showed to be an efficient and viable process for meeting a high degree of sulfate reduction from drinking water and be considered as a promising technology for treating sulfate-polluted drinking water.
- There was necessity for new investigations about other removal mechanisms were not considered in the reports.

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