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Fluoride Removal from Zinc Sulfate Solution by Nanopowder Zirconium Oxide in Industrial Electrolysis Process of Zinc Production

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Abstract
Fluoride impurities in industrial zinc sulfate electrolytes adversely affect the zinc electrowinning process by promoting zinc deposition on the aluminum cathode, thereby reducing process efficiency and product quality. In the present study, zirconium oxide (ZrO_2) has been used as an adsorbent to remove fluoride of zinc sulfate solution during the industrial electrolysis process of zinc production. The effects of five operational parameters, including ZrO_2 dosage, temperature, contact time, stirring speed and pH were systematically evaluated to optimize the fluoride removal process. The initial concentrations of zinc sulfate and fluoride in the electrolyte were 210 g/L and 453 mg/L (ppm), respectively. Under the optimum operating conditions (ZrO_2 dosage of 2.0 g/L, temperature of 90 °C, contact time of 20 min, stirring speed of 200 rpm, and also pH 7), more than 90% of the fluoride was removed from the zinc sulfate electrolyte. Moreover, under industrial operating conditions demonstrated that the fluoride concentration could be reduced to below 55 mg/L (adsorption capacity=200 mg/g), significantly alleviating operational problems associated with zinc electrowinning. These findings demonstrate that ZrO_2 nanopowder is an efficient and practical adsorbent for fluoride removal from industrial zinc sulfate electrolytes and has considerable potential for industrial application.

Keywords
Fluoride removal; zirconium oxide (ZrO_2); zinc sulfate; Zinc Production

1. Introduction

Zinc is one of the most important non-ferrous metals worldwide, with more than 75% of its production being achieved through hydrometallurgical or electrolytic processes. Industrial zinc production by the electrolytic process generally consists of four main stages: roasting, acid leaching, solution purification, and electrowinning [1]. Owing to the increasing exploitation of low-grade zinc ores and complex mineral resources, a variety of impurities originating from both metallic and non-metallic minerals are introduced into the processing stream. Among these impurities, fluoride is considered one of the most detrimental contaminants affecting the zinc electrowinning process [2].

During roasting and acid leaching, fluoride-containing minerals dissolve into the process solution, leading to an increase in the fluoride concentration of the zinc sulfate electrolyte. Excessive fluoride causes several operational problems, including corrosion of the anodes, zinc adhesion to the aluminum cathodes, deterioration of current efficiency, and difficulties in stripping the deposited zinc sheets from the electrode surface [3, 4]. In addition, the release of fluoride-containing wastes may pose environmental and occupational health concerns. Therefore, effective control of fluoride concentration in industrial electrolytes is essential for both process performance and environmental protection. In industrial zinc plants, particularly in China, the fluoride concentration in the electrowinning electrolyte is generally maintained below 50 mg/L to ensure stable operation [5].

Several techniques have been employed for fluoride removal from zinc sulfate electrolytes [6] including: volatilization during roasting, Soda washing, chemical precipitation, ion exchange, coagulation, membrane separation, and adsorption [6-12]. Pyrometallurgical treatment requires high-temperature furnaces, resulting in considerable energy consumption and operating costs. Soda washing generates a large volume of wastewater that requires further treatment. Likewise, chemical precipitation often suffers from poor filtration characteristics, whereas ion exchange processes may result in zinc losses, relatively low fluoride selectivity, and the production of secondary fluoride-containing waste streams. Adsorbent

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materials based on aluminum-loaded resins and silica have also been investigated; however, their performance is frequently limited by poor selectivity in highly concentrated electrolyte solutions containing large amounts of competing ions [13, 14]. In recent years, adsorption process has been attracted as one of the most effective and economically attractive techniques for fluoride removal because of its operational simplicity, relatively low cost, high efficiency, environmental compatibility, and minimal generation of secondary pollutants [15–22]. Consequently, a wide variety of adsorbents, including: activated carbon, metal oxides, composite materials, and nanostructured adsorbents have been developed for fluoride removal from aqueous systems [23–27]. Among these compounds, zirconium oxide (ZrO_2) has attracted particular attention because of its strong affinity toward fluoride ions through Lewis's acid-base interactions, high chemical stability, low toxicity, and excellent adsorption performance [28, 29].

Although numerous studies have investigated fluoride removal from synthetic aqueous solutions, but; only limited information is available for fluoride removal from real industrial zinc sulfate electrolytes under practical electrowinning conditions. Furthermore, the application of ZrO_2 nanopowder in highly concentrated zinc sulfate electrolytes and the evaluation of its performance under industrial operating conditions have rarely been reported. Therefore, in the present study, ZrO_2 nanopowder was employed as an adsorbent for fluoride removal from a real industrial zinc sulfate electrolyte obtained from a zinc electrowinning plant. The effects of the main operational parameters including: adsorbent dosage, temperature, contact time and stirring speed, and solution pH were systematically investigated and optimized. The results demonstrate that ZrO_2 nanopowder can effectively reduce fluoride concentration in industrial zinc sulfate electrolytes.

2. Experimental

2.1. Material and apparatus

ZrO_2 nanopowder (20 nm) purchased from Nanosany Corporation (Iran). Other chemicals used were of analytical grade, purchased from Merck and used without any further purification. $ZnSO_4$ solution was obtained from the zinc production line of Azar Shimi Razi Company ((make-up Ardestan mine, Iran). In order to prepare the standard solution of fluoride (1000 ppm), 2.210 g of NaF was dissolved in deionized water and made up to 1000 mL in a flask.

The absorption spectra were recorded on a Shimadzu Model 160-A UV-Vis spectrophotometer with a 1 cm quartz cell at 580 nm. An atomic absorption spectrometer (AAS, Varian AA220) was utilized for the metals analysis in tested solutions. A Horiba pH meter was used for adjusting the acidity of the solution.

2.2. General procedure

The experiments were designed and performed based on simulation of industrial process in semi-industrial scale. The change of reaction conditions at different levels of variables can be probably resulted to form various materials. Therefore, the statistical procedures cannot used to conduct experiments and obtain optimum conditions. The conventional procedure of experimental design (one-at-time) was exploited in this work. In this type of investigation, it is assumed that variables are independent and there is no correlation between them. According to the operational conditions of industrial zinc production process variables (amount of ZrO_2, temperature, reaction time, stirring speed and pH) were selected and investigated. The initial concentrations of zinc sulfate and fluoride in solutions were 210 g/L and 453 mg/L, respectively.

2.3. Fluoride determination

The concentration of fluoride ions was determined by UV-Vis and SPANDS reagent as a standard method (Fluoride-USEPA SPADNS Method) [30]. This method is based on the reaction between fluoride ion and SPANDS reagent (red color) and formation a colorless complex. In fact, the fluoride concentration is followed and determined based on its decolorization reaction on SPANDS reagent. Hence, increasing of fluoride concentration can cause to decrease the color of sample solution. Finally, the absorbance of sample solutions was determined by UV-Vis spectrophotometer in wavelength 580 nm (λ_{max}). According to the Beer-Lambert law, the absorbance of sample solution is directly related to the concentration of fluoride ions. The calibration curve was constructed by measuring the instrumental responses (in 580 nm) to a set of standard solutions of fluoride at a range of concentrations: 0, 1.0, 2.0, 4.0, 6.0 and 8.0 mg/L. The corresponding values of the regression equation were as: $Y=1.1926 - 0.0540\ X$ ($R=0.9989$, X= Fluoride concentration (mg/L)).

3. Results And Discussion

3.1. Preliminary studies

The preliminary investigations of fluoride adsorption were performed. The results showed that the ZrO2 dosage and temperature had the greatest effects on fluoride removal and the contact time, stirring speed and pH were also effective. Therefore, the experimental parameters were investigated in the following order: ZrO_2 dosage, temperature, contact time, stirring speed and pH. For each experiment, 100 mL of electrolyte was

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sampled from the zinc production line of Azar Shimi Razi Company. The industrial investigations obtained during the electrowinning process indicated that fluoride concentrations above 55 mg/L resulted in increased zinc adhesion to the cathode surface, thereby making the subsequent removal of the deposited zinc layer more difficult. Due to necessity and important of controlling Zn solution concentration, the change in number of other elements and interfering compounds such as: Fe, Mn, Cd, Ni, Co, Pb must be also checked during the electrolysis process. Analysis of control solution was done using AAS and UV-vis methods, the results report in Table 1.

Table 1. The measured values (mg/L) of the control solution of zinc sulfate (make-up Ardastan, Iran)

Pb	Co	Ni	Cd	Mn	Fe	Zn	Cl	F
1.35	0.74	0.91	0.42	2.37	0.95	85000	355	453

In order to eliminate the possible interfering substances and ions including: aluminum compounds, chlorine, iron, phosphates, sodium, hexametaphosphate and sulfates, the samples were prepared according to the procedure described in Ref. [30].

The presence of chloride and sulfate ions did not significantly affect in fluoride removal. Similar observations have been reported by Nagaraj et al. [31]. This behavior may be attributed to the lower affinity of these ions for the active sites of the ZrO_2 adsorbent compared with fluoride ions. In contrast, bicarbonate ions in solution had the most effect on fluoride removal. Similar behavior has also been reported in previous studies [29, 31]. The competitive effect of bicarbonate may be associated with the comparable ionic size of fluoride and bicarbonate ions (approximately 0.13 nm), as well as the presence of hydroxide ions in the solution. Yu et al. [29] suggested that the reduced fluoride removal in the presence of bicarbonate may be related to the formation of five-membered complexes between bicarbonate species and Zr^{4+} ions, followed by their adsorption onto the active sites of ZrO_2. Among the investigated anions, phosphates ion also showed a significant inhibitory effect on fluoride removal, and seems that it is due to their similar interaction in capturing the active sites of ZrO_2. Overall, the fluoride removal efficiency decreased with increasing concentrations of competing anions. Shan et al. found a direct correlation between the concentration of anions and their ability to occupy active sites of adsorbent [32].

3.2. Optimizing the factors (variables) affecting on fluoride removal

3.2.1. Effect of ZrO_2 amount

In order to check the effect of adsorbent amount on fluoride removal efficiency, zinc sulfate solution (100 mL, 210 g/L) was poured into a laboratory beaker (200 mL). The mouth of the beaker was sealed, with only two openings provided for adding the material and measuring the temperature. Then, values 0.5 to 2.5 g of ZrO2 (adsorbent) were added to solution and other parameters were constant during investigations (temperature 90 ºC, stirring speed, 200 rpm; adsorption time 20 min; pH = 7; and fluoride concentration 453 mg/L). Finally, the concentrations of fluoride ion in solution were determined using UV-Vis (in 580 nm) method. The obtained results are presented in Fig. 1. According to this figure, the fluoride removal from zinc sulfate solution was increased until 2.0 g of adsorbent and then, the adsorption changes were insignificantly in higher amounts. This trend was due to increase the probability of fluoride adsorption on active sites of adsorbent and then reaching equilibrium [33, 34]. Moreover, in highest adsorbent amounts, ZrO_2 particles may convert to a clot form. As a result, the fluoride removal efficiency was decreased due to reduce the surface area of adsorbent. Therefore, 2.0 g of ZrO_2 was chosen as the optimum amount.

Due to the necessity of controlling changes (control check) in during process, the other elements and compounds such as: Zn, Fe, Mn, Cd, Ni, Co and Pb were measured using AAS. It was found that their concentrations remained constant.

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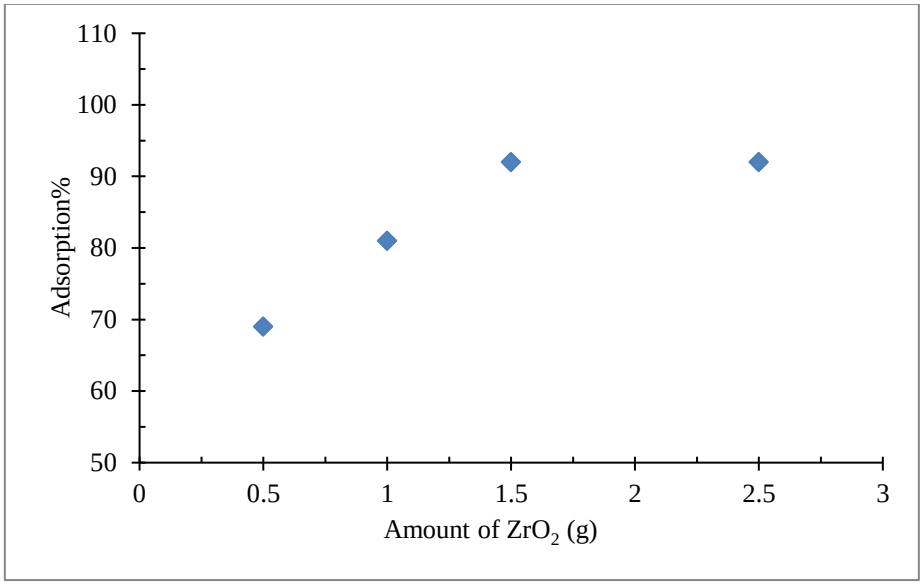

Fig. 1. Effect of ZrO_2 amount. Conditions: fluoride concentration (453 mg/L); temperature, 90 ºC; contact time, 20 min; stirring speed, 200 rpm; pH, 6.

3.2.2. Effect of temperature

The effect of temperature on the fluoride removal was investigated in range 70 to 100 °C (stirring speed, 200 rpm; adsorption time, 20 min; pH, 6; ZrO_2 amount, 2.0 g; and fluoride concentration, 453 mg/L). According to Fig. 2, the removal efficiency of fluoride was increased until 90 ˚C and at higher temperatures; the obtained results did not change significantly. Regard to important energy consumption in production cost, 90 ºC was selected as the optimum value. In these conditions, the fluoride concentration was reduced to the lower than standard level, 50 mg/L. Again, due to the necessity of controlling changes in the number of other elements and compounds, it was checked.

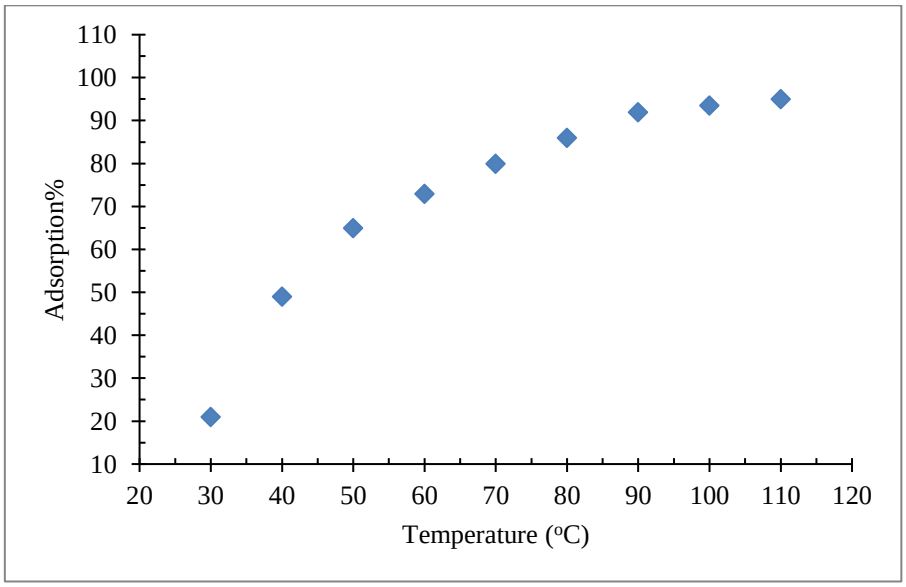

Fig. 2. Effect of temperature. Conditions: fluoride concentration (453 mg/L); ZrO_2 amount, 2.0 g; contact time, 20 min; stirring speed, 200 rpm; pH, 6.

3.2.3. Effect of adsorption time (contact time)

Fig. 3 shows the effect of contact time on fluoride adsorption by ZrO_2 in the optimum adsorbent amount (2.0 g) and temperature (90 ºC). This factor was investigated and tested in range 5 to 25 min. The other conditions were similar to previous section. As shown in this figure, the maximum fluoride removal was obtained in contact time 20 min and then, it was constant and adsorption process was reached to equilibrium**.** Therefore, 20

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min was recorded as the optimum time of fluoride adsorption. Sathish et al. have also reported a similar trend in their studies on fluoride adsorption using coconut sheets containing carbon saturated with Zr [35]. Finally, the control check was also performed that any significant changes did not observe.

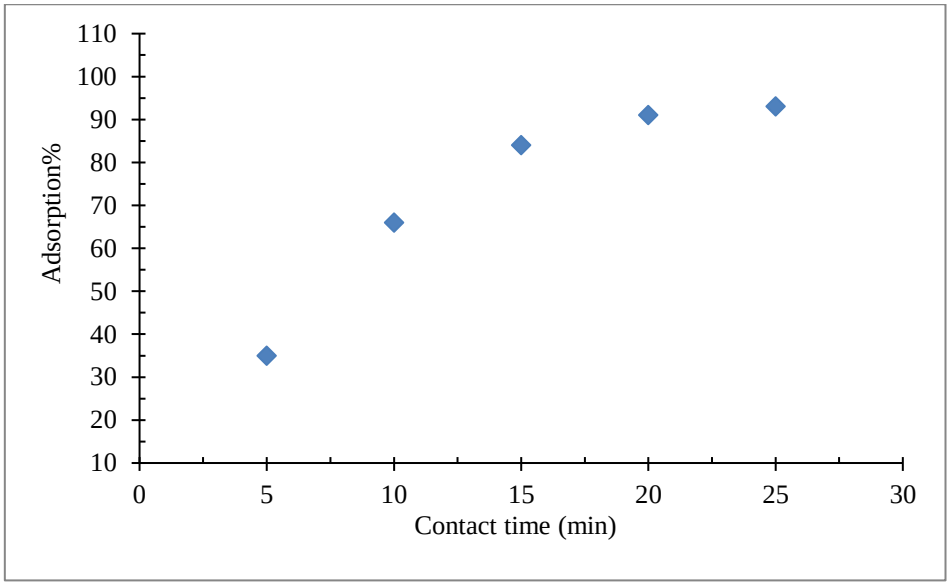

Fig. 3. Effect of contact time. Conditions: fluoride concentration (453 mg/L); ZrO_2 amount, 2.0 g; temperature, 90 ºC; stirring speed, 200 rpm; pH, 6.

3.2.4. Effect of stirring speed

After optimizing the factors including: amount of ZrO_2, temperature and contact time, the effect of stirring speed on the fluoride removal was tested in range 50 to 300 rpm (5 runs); while other conditions were similar to previous section. Fig. 4 illustrates that by increasing the stirring speed up to 200 rpm, the fluoride removal was improved and at higher values, its changes were minor. Thus, 200 rpm was selected to continue tests. Finally, the control check was also repeated and found that the concentration changes of other substances were ignorable.

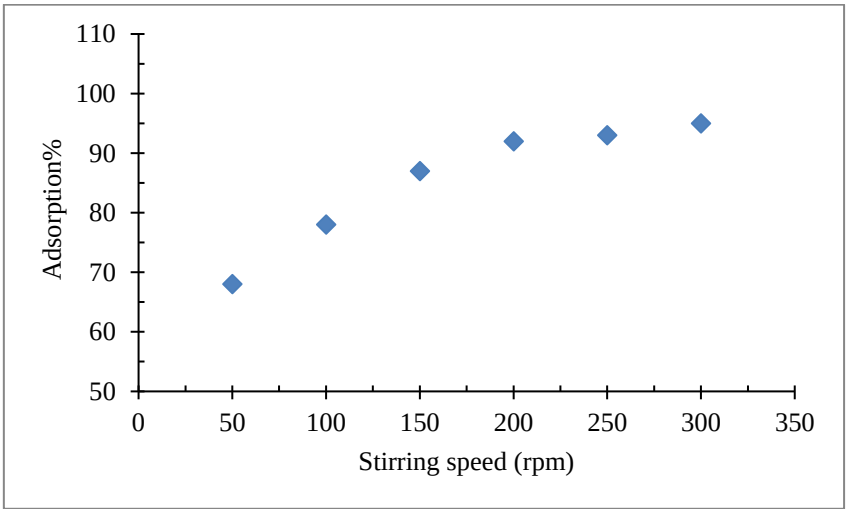

Fig. 4. Effect of stirring speed. Conditions: fluoride concentration (453 mg/L); ZrO_2 amount, 2.0 g; temperature, 90 ºC; contact time, 20 min; pH, 6.

3.2.5. Effect of pH

The pH solution of zinc production is between 6 and 7 (Fig. 5). In the basic media, $Zn(OH)_2$ precipitation was obtained and also in acidic conditions: H^+ ions tend to bind to fluoride and produce hydrofluoric acid which this can decrease the fluoride adsorption on ZrO_2. Therefore, experiments were done in real pH (6-7) of zinc production solution.

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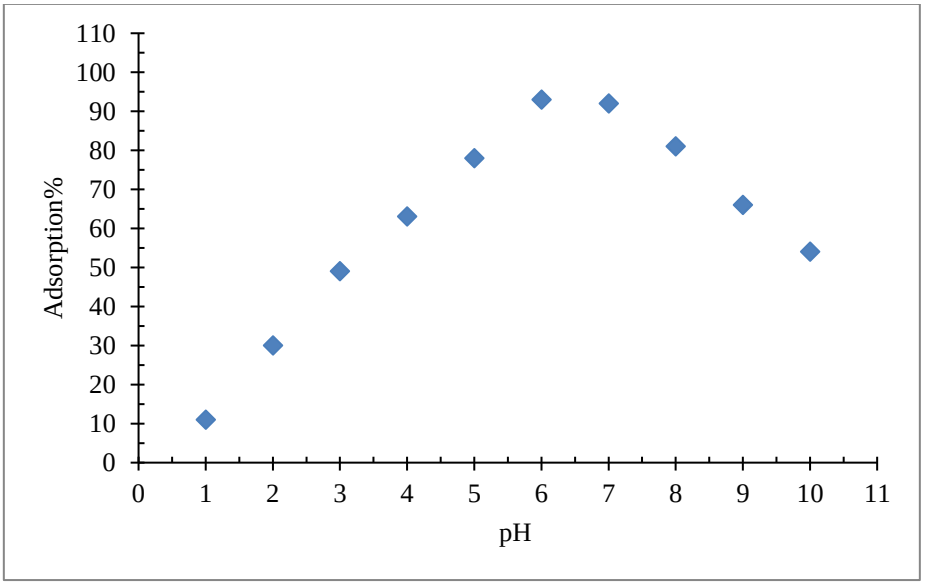

Fig. 5. Effect of pH. Conditions: fluoride concentration (453 mg/L); ZrO_2 amount, 2.0 g; temperature, 90 ºC; contact time, 20 min; stirring speed, 200 rpm.

3.3. Investigation of fluoride removal in real sample (Application)

According to the investigations of adsorption, the optimum conditions were as: amount of ZrO_2, 2.0 g in 100 mL of sample solution; temperature, 90 ºC; contact time, 20 min; stirring speed, 200 rpm; and pH, 6. Under optimum conditions, several experiments were conducted and repeated on semi-industrial and also industrial scales production that fluoride in zinc sulfate solution reached below standard limit (55 mg/L). The adsorption capacity was 200 mg/g. Furthermore, the adhesion problems of zinc to cathode surface were not seen.

3.4. The fluoride removal mechanism

Several researches have been conducted on the fluoride removal field of zinc sulfate solution using metal oxides as adsorbent including: Zr(IV), Al(III), La(III) and Ce(IV). These compounds and also the modified adsorbents with the mentioned metal oxides have a high tendency to adsorb fluoride. Zr-OH is formed due to the absorption of water molecules on zirconium oxide [36]. The fluoride adsorption mechanism on ZrO_2 is an ion-exchange mechanism, which could be expressed as follows:

$$\text{——Zr——OH}_{\text{ads}} + \bar{\text{F}} \longrightarrow \text{——Zr——F}_{\text{ads}} + \text{OHaq}$$

4. CONCLUSIONS

In this work, ZrO_2 was introduced and applied as an adsorbent to remove fluoride from zinc sulfate solutions during the electrolysis process of zinc production. The findings revealed that the amount of ZrO_2 and temperature variables (factors) were more important in the fluoride adsorption process. Also, in optimum ZrO_2 amount, temperature was appeared and played as most important and fundamental factor in the fluoride adsorption. So that, when all other factors were at their proper levels (contact time, 20 min; stirring speed, 200 rpm; and pH, 6), but temperature was not at optimum level, the weak fluoride adsorption results were obtained using ZrO_2. The main conditions were as: 2.0 g of ZrO_2 in 100 mL solution, the extraction temperature 90 ºC. Under optimum conditions, the fluoride in zinc sulfate solution has been reached to lower 55 mg/L with adsorption capacity 200 mg/g. This concentration was suitable for the industrial electrolysis process of zinc production without adhere the zinc layer on surface of aluminum cathode.

Declaration of interest

There are no conflicts to declare. The authors report no conflicts of interest. Also, the authors are responsible for the writing and content of this article.

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چکیده

ناخالصی‌های فلوراید در الکترولیت‌های صنعتی سولفات روی با افزایش رسوب روی روی کاتد آلومینیوم، بر فرآیند الکتروویننگ روی تأثیر منفی می‌گذارند و در نتیجه راندمان فرآیند و کیفیت محصول را کاهش می‌دهند. در مطالعه حاضر، اکسید زیرکونیوم (ZrO2) به عنوان جاذب برای حذف فلوراید از محلول سولفات روی در طول فرآیند الکترولیز صنعتی تولید روی استفاده شده است. تأثیر پنج پارامتر عملیاتی، شامل دوز ZrO2، دما، زمان تماس، سرعت هم زدن و pH، به طور سیستماتیک برای بهینه‌سازی فرآیند حذف فلوراید ارزیابی شد. غلظت اولیه سولفات روی و فلوراید در الکترولیت به ترتیب ۲۱۰ گرم در لیتر و ۴۵۳ میلی‌گرم در لیتر (ppm) بود. تحت شرایط عملیاتی بهینه (دوز ZrO2 2.0 گرم در لیتر، دمای ۹۰ درجه سانتیگراد، زمان تماس ۲۰ دقیقه، سرعت هم زدن ۲۰۰ دور در دقیقه و همچنین pH 7)، بیش از ۹۰٪ فلوراید از الکترولیت سولفات روی حذف شد. علاوه بر این، تحت شرایط عملیاتی صنعتی نشان داده شد که غلظت فلوراید می‌تواند به کمتر از ۵۵ میلی‌گرم در لیتر (ظرفیت جذب = ۲۰۰ میلی‌گرم در گرم) کاهش یابد، که به طور قابل توجهی مشکلات عملیاتی مرتبط با الکتروویننگ روی را کاهش می‌دهد. این یافته‌ها نشان می‌دهند که نانوپودر ZrO2 یک جاذب کارآمد و کاربردی برای حذف فلوراید از الکترولیت‌های سولفات روی صنعتی است و پتانسیل قابل توجهی برای کاربرد صنعتی دارد.

کلیدواژه‌ها

حذف فلئور، زیرکونیم اکساید، روی سولفات، تولید روی.

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