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Iron sulphates production being polarized by the direct and alternating currents

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Abstract. The process of iron oxidation in the sulfate electrolytes was studied by the method of electrochemical polarization by the steady and transient currents. Initially, in the first electrolyzer, the iron electrodes were oxidized under the influence of alternating current. The results of iron oxidation in the first electrolyzer were monitored by determining the weight loss of the iron electrodes. An almost rectilinear increase in the mass of iron is established to be observed, which passed into the solution in the form of Fe^{2+} ions when the current density changes in the range of 80-400 A / m². The current efficiency is close to 100%, and sometimes exceeds 100%, since the chemical dissolution of iron in sulfuric acid also occurs simultaneously. The iron sulfate (II) can be obtained by evaporation of the solution. After that, the sulfate solution containing iron ions (II) was sent to the second electrolyzer, in which the oxidation of iron (II) to iron (III) was carried out under the direct current. The electrode spaces were separated by an MA-40 anion exchange membrane. Over 90% of iron (II) was demonstrated to be transfers into the trivalent state within 1 hour in the second electrolyzer at a current density of 120 A / m². A change in the current density to 600 A / m² leads to an increase in the oxidation state to 97.5%, i.e. iron ions (II) are almost completely oxidized under the specified conditions. As a result of the experiments, an electrochemical method for producing iron sulfate (III) was developed.

Key words: iron, electrolyte, sulfate, electrolysis, alternating current.

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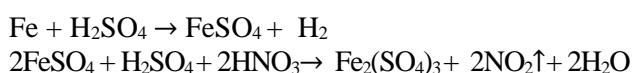
Introduction

Iron forms many compounds being an active element. Its compounds are widely used in various branches of technology and industry. Specifically, there is a research devoted to the development of a

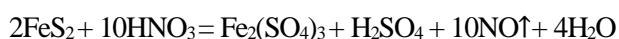
method for producing sulfate and iron oxide nanoparticles: when silica gel and aluminum oxide are impregnated with Fe (II) sulfate solutions, ferric hydroxide and oxysulfate nano-particles are formed on the surface of silica gel, and iron oxide nanoparticles on the surface of aluminum oxide [1, 17,

18]. To say more, iron compounds, namely sulfates, are used in water purification as a coagulant [2-4]. In medicine, various compounds containing both ferrous and ferric iron are also used [5,6]. Iron sulfate (III) can be used as a mordant for dyeing wool and as a background for the enrichment of sour coal. Acidic solutions are used as an oxidizing medium in the extraction of useful components out of ores [7]. Iron ions (III), being active oxidizing agents, are widely used in the technology of subsurface leaching of uranium, although the authors of [8] note that the use of sulfate solutions of ferric iron is associated with the problem of its regeneration. Nevertheless, the researchers [9] used a solution of ferric iron along with the ammonium sulphate and sodium peroxoborate in the study of the comparative sulfuric acid catalytic oxidation of uranium (IV) to uranium (VI).

An existing and frequently used method for producing ferric sulfate is that, first, metallic iron is chemically dissolved in sulfuric acid when heated to produce ferrous ions [10]. Further, the iron ions (II) are oxidized in the presence of nitric acid, while the following reactions proceed:



Grinding of iron to a state of sawdust is noted to be required for a more efficient process, in addition, nitrogen dioxide, a substance that is uncommon for the environment is released as a result of the reaction. This is result in the method has certain disadvantages. Iron sulfate (III) is also obtained by dissolving iron oxide (III) in 75-80% sulfuric acid. In addition, iron sulfate (III) solutions can be obtained by oxidation of pyrites with nitric acid:



There is also a method for producing iron sulfate (III), which serves as a coagulant, by treating pyrite cinder with hot sulfuric acid. The sifted cinder must first be treated with concentrated nitric acid (in the amount of 8-10 kg per 1 g of coagulant). The oxidized cinder is mixed and kept before cooking the coagulant for 5-6 hours. All these methods are described in the monograph of Pozin M.Y. [7].

In order to increase the degree of conversion of ferrous iron to ferric, the authors of [11] propose adding a catalyst in the form of iron tetrasulfophthalocyanine to the solution. The essence of the method lies in the fact that the catalyst is added to the sulfuric acid solution of iron sulfate (II) on carbon fabric and the mixture is stirred in a stream of oxygen. The degree of conversion reaches 83%. Using an anode made of graphite or platinum, it is

proposed to oxidize iron sulfate (II) electrochemically at a temperature of 80°C [12]. The current yield of iron oxidation (II) to iron (III) reaches 95.8%. Considering that, iron sulfates are most widely used in various branches of technology and industry, as well as in medicine, but the existing methods of their producing are imperfect and complex, we believe that the development of new methods for producing iron compounds is an urgent problem. In this regard, the aim of our study is to develop a two-stage method for producing iron sulfate (III) by polarizing iron electrodes, first with alternating current of industrial frequency and then anodic oxidation of the resulting solution on a lead electrode.

Testing method

A set was assembled to conduct electrolysis with alternating and direct current, the circuit diagram of which is provided in Figure 1.

The set consists of 1 and 2 electrolyzers (conventionally named “the first” and “the second” electrolyzers). The first electrolyzer and the cathode space of the second electrolyzer are filled with sulfate electrolyte (4). Iron (3) was used as electrodes in the first electrolyzer, a lead electrode (5) was used as the anode, and titanium (6) was used as the cathode. In the second electrolyzer, interelectrode spaces are separated by an MA-40 anion exchange membrane (7). We used LATR (8) to obtain alternating current, and as a constant current source - Matrix MPS - 3003D ”(9). An alternating current ammeter E 538 (10) and a direct current ammeter (11) were used to measure the current strength.

A titanium plate cut from titanium sheets of the VT-1-0 brand was used as the electrodes in the experiments, iron of St. 3 brand and lead of C-1. Before electrolysis, the electrodes were cleaned with emery, thoroughly washed with distilled water, wiped with filter paper, and degreased with ethyl alcohol. A sulfuric acid of “chemically pure” was used for an electrolyte solution to be prepared. In the first electrolyzer, the concentration of sulfuric acid was equal to 250 g / dm³. Iron electrodes were polarized by industrial alternating current with a frequency of 50 Hz at various current densities. After dissolution of the iron electrodes by alternating current, the electrolyte from the first electrolyzer was fed into the anode space of the second electrolyzer, in which ferrous iron is oxidized to a trivalent state during anodic polarization. At the end of the experiment, the resulting solution was evaporated, the precipitated crystals were dried and identified by chemical, X-ray phase and X-ray fluorescence methods.

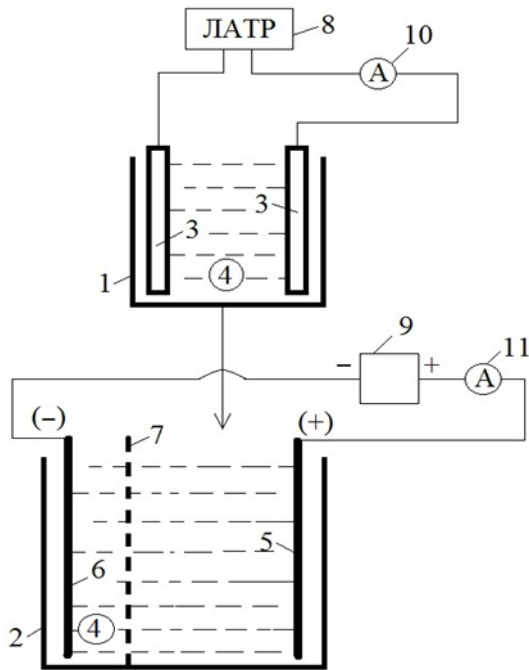
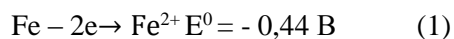


Figure 1 A set flow sheet for producing iron sulfate (III) by two-stage electrolysis: 1 and 2 - the first and the second electrolyzers; 3 - iron electrodes; 4 - sulfate electrolyte; 5 - lead electrode; 6 - titanium electrode; 7 - MA-40 anion exchange membrane; 8 - an alternating current source - LATP; 9 - DC source - current rectifier; 10 - alternating current ammeter; 11 - DC ammeter.

Results and discussion

When the electrodes are polarized in a sulfuric acid solution by alternating current in the first electrolyzer (1), the iron oxidation reaction takes place:

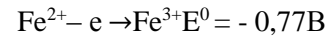


This reaction proceeds in the anode half-cycle of the alternating current.

As the previous studies have established [13, 14], being polarized by alternating current, a targeted course of electrode processes is possible despite the fact that the current direction changes with a frequency of 50 Hz, i.e. within a second, each electrode 50 times becomes either the anode or the cathode. But unlike those processes that occur during polarization by direct current, in this case, the iron manages to oxidize in the anode half-cycle only to the divalent state. In the cathode half-cycle of alternating current, due to the low value of the overvoltage of ion recovery, hydrogen gas is released on the surface of the iron electrodes. Moreover, due to the negative value of the potential of the $\text{Fe}(\text{II}) \leftrightarrow \text{Fe}^0$ system, the reverse cathodic reduction of iron ions (II) to the state of elements does not occur. The processes are repeated at a frequency of 50 Hz in cycle. Thus, in

each anode half-cycle, iron electrodes dissolve according to the reaction (1).

A sulfate solution containing ferrous ions formed in the first electrolyzer (1) was fed into the anode space of the second electrolyzer (2), in which an MA-40 anion exchange membrane separated the electrode spaces. The membrane serves to prevent the reverse reduction of iron ions (III) at the cathode. On the surface of the anode from lead, ferrous ions are oxidized to a trivalent state:



The results of iron oxidation in the first electrolyzer were monitored by determining the weight loss of the iron electrodes. In the second electrolyzer, the amount of iron ions (III) formed was determined by the photocolometric analysis method [15]. The dependences of the decrease in the mass of the iron electrodes upon polarization by alternating current, and the current efficiency of their dissolution on the current density in the first electrolyzer (Fig. 2) and the dependence of the oxidation degree of iron ions (II) to iron ions (III) on the current density upon polarization with the direct current in the second electrolyzer are established (figure 3).

When the current density changes within 80–400 A / m^2 , an almost rectilinear increase in the mass of iron is observed, which transferred to the solution as Fe^{2+} ions (Fig. 2, curve 1). The current efficiency (curve 2) is close to 100%, and sometimes exceeds 100%, since the chemical dissolution of iron in sulfuric acid also occurs simultaneously:

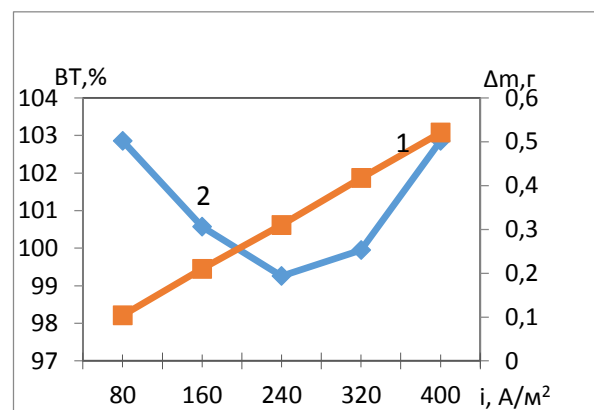
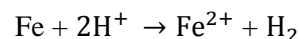


Figure 2 The effect of the current density on the mass of the iron electrodes decrease (curve 1) and the current efficiency (curve 2) when polarized by alternating current ($\tau = 1$ hour; $\text{H}_2\text{SO}_4 = 200 \text{ g} / \text{dm}^3$).

Additional experiments were performed without current polarization with the same

electrodes. It was found that at room temperature in sulfuric acid with a concentration of 200 g / dm³, the iron electrodes are dissolved chemically and within 30 minutes, the mass loss of the two electrodes does not exceed 0.0012 g, i.e. 1.2 mg. In this case, the concentration of sulfuric acid has a slight effect on the change in mass of the electrodes (Table).

A table Change in the mass of iron electrodes when dissolved in the sulfuric acid depending on its concentration with no current imposing

H ₂ SO ₄ , g/dm ³	50	100	150	200
Δm,mg (1-electrode)	0,3	0,4	0,5	0,6
Δm,mg (2-electrode)	0,3	0,4	0,5	0,6

The sulfate solution obtained in the first electrolyzer is noted to be subjected to evaporation in order to obtain iron sulfate (II) as may be required. The testing carried out in the second electrolyzer with the same mass of Fe²⁺ ions provided that even at a current density of 120 A / m² for 1 hour more than 90% of iron (II) goes into the trivalent state. A change in the current density to 600 A / m² leads to an increase in the oxidation state to 97.5%, i.e. iron ions (II) are almost completely oxidized under the indicated conditions (Fig. 3). This is apparently due to the fact that, with an increase in the current density at the electrodes, according to the Tafel equation, the anode potential shifts in a more positive direction, i.e. more favorable conditions are created for the oxidation of iron (II).

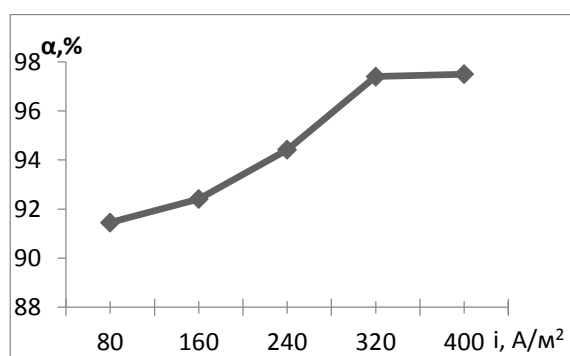


Figure 3 Influence of the tocan density on the degree of iron oxidation (II) being polarized by direct current ($mFe^{2+} = 104,1 \text{ mg}, \tau = 1 \text{ hour}$)

As the studies provide, an increase in the density of the token electrodes leads to an increase in the rate of the reaction, and accordingly, the efficiency of the process is intensified. We note that

by adjusting the current density in the first electrolytic cell, respectively, in the second electrolytic cell, it is possible to control the rate of formation of the target product.

The sulfate solution containing iron ions (III) was evaporated after electrolysis to form a viscous pasty mass (according to the procedure described in [10]), while its temperature reaches 120°C. The mass was cooled to 45-50°C, the precipitated crystals were sucked off on a Buchner funnel and dried at a temperature of 65°C. The obtained crystals were identified by X-ray phase analysis (Fig. 4), X-ray fluorescence (Fig. 5) and chemical analysis. It was found that the composition of the compound corresponds to the formula: Fe₂(SO₄)₃ · 9H₂O.

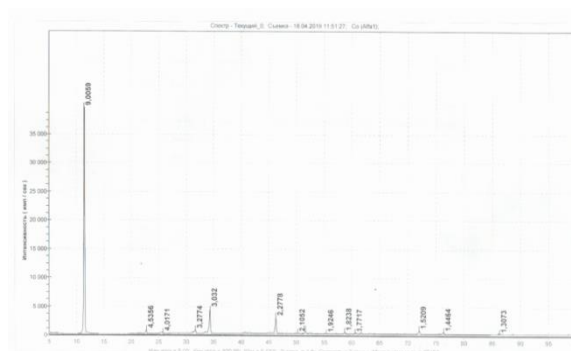


Figure 4 X-ray diffraction pattern of iron sulfate (III)

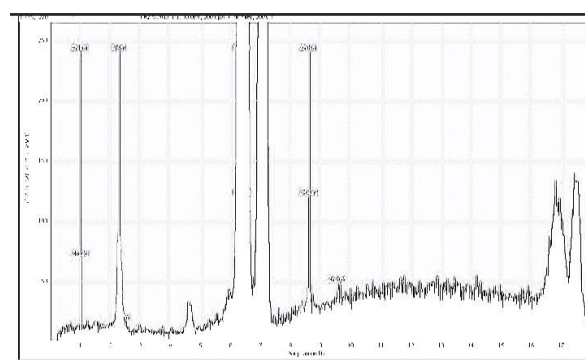


Figure 5 X-ray fluorescence analysis of iron sulfate

Summarizing the results of testing, when the iron electrodes being polarized with the alternating current, the iron was found to be oxidized to a divalent state, and iron sulfate (II) is formed in the solution. With further polarization of the resulting solution by direct current, the iron ions (II) are oxidized to a trivalent state. Based on the results of the study, an electrochemical method for producing iron sulfate (III) was developed [16]. A patent of the Republic of Kazakhstan for a utility model defends the method. The differences and the advantages of the proposed method are that debris, scraps of iron, which are wastes from various technological

processes are used as the feedstock. There is no need to grind metal, making the process easier. The process takes place at room temperature; working conditions are improved, because there is no electrolyte vapor. In addition, there is no need to use nitric acid, respectively, the harmful effects of toxic gas - nitrogen dioxide on the environment are eliminated, and the target product is not contaminated with the nitrate anions.

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Стационарлы және стационарлы емес токтармен поляризациялау кезінде темір сульфаттарының түзілуі

Баешова А. К., Баешов А., Жұмабай Ф. М., Шакенова М. Ш.

Түйіндеме. Темірдің тотығу процесі стационарлы және стационарлы емес электрохимиялық поляризация әдісімен күкірт қышқылды электролиттерде зерттелді. Ең алдымен темір электродтарының тотығуы бірінші электролизерде айналымы ток әсерімен жүргізілді. Темірдің бірінші электролизерде тотығуының нәтижелері темір электродтарының массаларының азаюымен бақыланды. Ток тығыздығы 80-400 А/м² аралығында өзгерген кезде, ерітіндіге Fe²⁺ түрінде өткен темірдің массасы тік сызықты тәуелділік бойынша өзгеретіні анықталды. Ток бойынша шығым 100% -ке жақын, ал кейбір кезде 100%-тен асады, себебі бір уақытта темірдің күкірт қышқылында еруі де орын алады. Бұл ерітіндіні буландыру арқылы темір (II) сульфатын алуға болады. Осыдан кейін құрамында темір (II) иондары бар күкіртқышқылды ерітінді екінші электролизерге жіберілді. Бұл электролизерде темір (II) иондарын темір (III)-ке дейін тұрақты ток әсерімен тотықтыру жүргізілді. Электродтық кеңістіктер МА-40 мембранасымен бөлінді. Екінші электролизерде ток тығыздығы 120 А/м² болғанда 1 сағат ішінде темір (II) иондарының 90%-і үш валентті күйге өтетіні көрсетілді. Ток тығыздығын 600 А/м²-ге дейін өзгерткенде, тотығу дәрежесі 97,5%-ке жетеді, демек, бұл жағдайда темір (II) иондары толық дерлік тотығады. Жүргізілген эксперименттер нәтижесінде темір (III) сульфатын алудың электрохимиялық әдісі жасалды.

Түйінді сөздер: темір, электролит, сульфат, электролиз, айналымы ток.

Формирование сульфатов железа при поляризации стационарными и нестационарными токами

Баешова А. К., Баешов А., Жұмабай Ф. М., Шакенова М. Ш.

Аннотация. Методом электрохимической поляризации стационарными и нестационарными токами изучен процесс окисления железа в серноокислых электролитах. Вначале в первом электролизере проводили окисление железных электродов под действием переменного тока. Результаты окисления железа в первом электролизере контролировали путем определения убыли массы железных электродов. Установлено, что при изменении силы тока в пределах 0,1-0,5 А наблюдается практически прямолинейное увеличение значения массы железа, перешедшего в раствор в виде ионов Fe²⁺. Выход по току близок к 100%, а иногда превышает 100%, так как происходит параллельно и химическое растворение железа в серной кислоте. Выпариванием раствора можно получить сульфат железа (II). После этого серноокислый раствор, содержащий ионы железа (II), отправляли во второй электролизер, в котором проводили окисление железа (II) до железа (III) под действием постоянного тока. Электродные пространства были разделены анионитовой мембраной МА-40. Показано, что во втором электролизере при силе тока, равной 0,1 А, в течение 1 часа более 90% железа (II) переходит в трехвалентное состояние. Изменение силы тока до 0,5 А приводит к увеличению степени окисления до 97,5%, т.е. ионы железа (II) практически полностью окисляются в указанных условиях. В результате проведенных экспериментов разработан электрохимический способ получения сульфата железа (III).

Ключевые слова: железо, электролит, сульфат, электролиз, переменный ток.

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