Вивчено особливості рециклінгу маткового розчину в технології одержання фератів (VI). Встановлено, що головним чином на ступінь очистки від баластних аніонів впливають температура та концентрація лугу. Вивчено розчинність K₂FeO₄, KCl, K₂SO₄, KNO₃ в присутності великого надлишку OH⁻ йонів. Запропоновано технологічні рішення щодо рециклінгу концентрованих лужних розчинів для різних способів синтезу фератів (VI)

Ключові слова: одержання сполук Fe(VI), гіпохлорит, електросинтез, рециклінг, матковий розчин

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Изучены особенности рециклинга маточного раствора в технологии получения ферратов (VI). Установлено, что основное влияние на степень очистки от балластных анионов оказывают температура и концентрация щелочи. Изучена растворимость K₂FeO₄, KCl, K₂SO₄, KNO₃ в присутствии большого избытка OH⁻ ионов. Предложены технологические решения по рециклингу концентрированных щелочных растворов для различных способов синтеза ферратов (VI)

Ключевые слова: получение соединений Fe(VI), гипохлорит, электросинтез, рециклинг, маточный раствор

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1. Introduction

Ferrates(VI) are new "green" oxidants, which due to their unique properties are used in many spheres, such as industrial ecology [1-4], organic and inorganic synthesis [5, 6], batteries production [7], etc. The widespread use of ferrates and their large-scale production are largely cut down by the high price that has currently developed in the market of iron(VI) compounds. It should be noted that the price for ferrates of reactive purity ($\geq 97 \%$ Fe(VI)) for scientific purposes in some degree has speculative character (100–150 USD per 1 g), because now there is a rush demand caused by the increased attention of many scientists from different countries to ferrate technologies [2-4, 7]. At the same time, the price of commercial ferrates (90 % Fe(VI)) for the industry is much lower. Nevertheless, for successful competition with traditional reagents, it is still high. This stimulates further research on the modernization of ferrates(VI) technologies aimed at reducing the cost of these compounds.

As a result of this, actual studies are devoted to the search of ways to improve the recycling technology of concentrated alkaline solutions, which accumulate in large volumes after crystallization and recrystallization of potassium ferrate. During the development of the appropriate processUDC 661.872:544.6

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USAGE OF MOTHER LIQUOR RECYCLING FOR THE OBTAINING OF Fe(VI) COMPOUNDS

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es, it is necessary to take into account that the composition of the mother liquor is broadly determined by the method of ferrates(VI) production. Therefore, for the chemical (hypochlorite) or electrochemical methods, technological decisions for recycling will have their own peculiarities according to the differences in the composition of the liquid phases in which the base product is synthesized.

2. Literature review and problem statement

One of the ways to reduce the cost of Fe(VI) compounds production is the recycling of concentrated alkaline solutions, which are used in large quantities to create conditions that ensure the stability of Fe(VI) derivatives in an aqueous medium. However, this issue is not given enough attention. In the papers [8, 9], devoted to the technologies of obtaining ferrates, a special role is assigned to the purity of the used alkali metal hydroxides, which is due to the use of very concentrated alkaline solutions (14–16 M OH⁻) for Fe(VI) synthesis. The requirements for the initial alkaline solutions used in various technological operations (synthesis, crystallization, recrystallization, etc.) are virtually identical regardless of the method of Fe(VI) compounds production. Solutions must be purified as much as possible from heavy metal compounds (especially cobalt, nickel, chromium, etc.), organic compounds and reducing agents [5, 10]. Otherwise, the yield and purity of the target product will be significantly reduced. The treatment of concentrated alkalis with a flow of direct electric current, offered in [11] and adapted for Fe(VI) technology [9], allows reducing the content of heavy metal impurities to a safe level. To eliminate the negative effect of inorganic and organic compounds, which are capable of interacting with the target product, it has been offered to use strong oxidizers (O_3 , H_2O_2), which do not contaminate the alkaline solution with the products of redox reactions [2, 5].

On the basis of the prepared on a special technology alkali, a hypochlorite solution is prepared at first, into which solid salts of Fe (more often the crystalline hydrate of iron(III) nitrate) are added [2, 5, 8]:

$$2 \text{Fe}(\text{NO}_{3})_{3} \cdot 9 \text{H}_{2} \text{O}_{(s)} + 3 \text{ClO}^{-} + 10 \text{OH}^{-} \rightarrow \\ \rightarrow 2 \text{FeO}_{4}^{2-} + 3 \text{Cl}^{-} + 6 \text{NO}_{3}^{-} + 23 \text{H}_{2} \text{O}, \qquad (1)$$

followed by precipitation of solid potassium ferrate [5, 8]:

$$2\text{KOH}_{(s)} + \text{FeO}_4^{2-} \rightarrow \text{K}_2\text{FeO}_{4(s)} + 2\text{OH}^-.$$
(2)

After the technological cycle in the mother liquor, due to chemical reactions ballast anions and impurities are inevitably accumulated, the sources of which are iron-containing raw materials as well as hypochlorite. As we can see from the equation of chemical reaction (1), the main impurities are NO_3^- and Cl^- anions. It is reported in [2, 8] that if spent alkali is used for Fe(VI) synthesis without any purification, so the purity of the obtained ferrate falls to 12 % due to the constant accumulation of impurities. Therefore, impurities should be removed before reusing the mother liquor. The authors [8] noted that specially purified from chlorides and nitrates alkali wastes can be reused to prepare K₂FeO₄ with a purity and yield above 90 and 60 %, respectively after ten recycles. Therefore, such purification is recommended in order to reduce the cost of potassium ferrate. It should be noted that these recommendations refer only to the synthesis of ferrates from iron(III) nitrate and do not affect other cheaper iron compounds, in particular, chlorides, sulfates or hydroxides:

$$2\text{FeCl}_{3(5)} + 3\text{ClO}^{-} + 10\text{OH}^{-} \rightarrow 2\text{FeO}_{4}^{2^{-}} + 9\text{Cl}^{-} + 5\text{H}_{2}\text{O},$$
 (3)

$$Fe_{2}(SO_{4})_{3(s)} + 3ClO^{-} + 10OH^{-} \rightarrow \rightarrow 2FeO_{4}^{2-} + 3Cl^{-} + 3SO_{4}^{2-} + 5H_{2}O,$$
(4)

$$2\text{FeO(OH)}_{(3)} + 3\text{ClO}^- + 4\text{OH}^- \rightarrow 2\text{FeO}_4^{2-} + 3\text{Cl}^- + 3\text{H}_2\text{O}, \quad (5)$$

whose use in the economics may be more practical [12].

In addition, the use of 11 M potassium hydroxide solutions for recycling, proposed in [8], does not meet the requirements for modern technologies, in which it is recommended to use solutions with a higher KOH concentration [2, 5, 10]. Moreover, it can be assumed that such low values of the yield of the target product, obtained in [8] are also due to the accumulation of soluble and insoluble heavy metal compounds in the system, the formation of which is justified in [10, 13].

There is no information in the literature on the recycling of mother liquors after electrochemical synthesis.

The collection of described above facts predetermines the need for continuing research in this direction and efforts concentration in increasing the profitability of obtaining Fe(VI) compounds by introducing a recycling operation into the technological cycle for the production of ferrates(VI).

3. The aim and tasks of the study

The aim of this study is to investigate the recycling of the spent mother liquor to improve indexes of ferrates synthesis and to reduce the cost of releasing the target product.

To achieve this aim, it was necessary to solve the following tasks:

– to determine the solubility of the target product K_2FeO_4 and some salts (KCl, K_2SO_4 , KNO₃), accumulating as ferrates are synthesized in the mother liquor, in the presence of a large excess of KOH;

to investigate the influence of the main factors (temperature and concentration of OH anions), which determine the efficiency of ballast salts removal and, ultimately, recycling in general;

to consider the practicability of recycling of concentrated alkaline solutions for various methods of obtaining ferrates(VI).

4. Materials and research methods for the purification of concentrated alkaline solutions from impurities of different nature

4. 1. Materials, substances and devices used for the synthesis of ferrates by chemical and electrochemical methods

For the preparation of base alkaline solutions, we used KOH (reagent grade), NaOH (especially pure) and twice distilled water. Ferrates solutions were prepared in two ways - by hypochlorite and electrochemical methods represented in [5, 7, 9, 12]. To implement chemical synthesis, we used $Fe(NO_3)_3 \cdot 9H_2O$, $Fe_2(SO_4)_3 \cdot 9H_2O$, $FeCl_3 \cdot 6H_2O$, FeO(OH) reagents with a purity degree not lower than reagent grade. For transpassive dissolution, we used rectangular shape samples (S=32 cm²), made of cast iron and steel of various grades, which composition is determined by ARL 3460 Optical Emission Spectrometer (Switzerland). The polarization of the electrodes was carried from direct current source B5-47 (Russia) to control the amount of electricity at constant current integrator IPT-1 (Belarus). The process was carried out in reactors of polymethylmethacrylate and polytetrafluoroethylene. The temperature of the working solutions was monitored and maintained using ultra thermostat U-2 (Germany) with an accuracy of ± 0.1 °C.

4. 2. The determination of ballast salts solubility and cleaning mother liquors from impurities

The solubility of KCl, K₂SO₄, KNO₃ (all reagents with the reagent grade purity) in KOH was investigated by the method recommended in [14, 15]. Solutions purification from precipitation and suspended particles was performed under vacuum using a Buchner funnel and Bunsen flask with filters made of polymer materials that are stable in solutions, which contain ferrates of alkali metal hydroxides. If necessary, mother liquors were treated with a flow of direct electric current to remove heavy metal compounds [9, 11].

4.3. The analysis of the mother liquor components

The ferrates analysis was carried out by the amperometric titration method given in [16] with one indicator electrode. The concentration of other anions in the mother liquor was adjusted by using standard procedures recommended in [17].

Electronic spectra of solutions absorption were prepared by spectrophotometer Specord M 40 (Germany) in sealed quartz cuvettes with an absorbing layer thickness of 1 cm.

5. The results of the recycling studies of concentrated alkaline solutions

Mother liquor, which remained after the redox reactions and the recovery of solid K_2FeO_4 , was the main object of investigation in this work. It is natural to assume that the method of ferrates(VI) synthesis must influence the composition of spent mother liquor. Obviously, it will determine the need and technological decisions for recycling.

5. 1. The determination of the solubility of potassium ferrate in an alkaline medium

As a rule, the spent mother liquor contains some of FeO_4^{2-} , which has not passed in the solid phase in the process (2). To reduce the unproductive losses of the target product, actions must be taken that the amount of dissolved potassium ferrate in the filtrate was minimal. There are not enough data on K_2FeO_4 solubility in an alkaline medium, they are contradictory and are obtained for disparate conditions [8, 14, 15], so at the first stage of the investigations its solubility has been studied in conditions, which are identical in composition used for the synthesis of Fe(VI) compounds. The results of these researches are presented in Fig. 1.



Fig. 1. K_2 FeO₄ solubility dependence on the temperature in KOH solutions, M: 1 - 12.0; 2 - 14.3; 3 - 16.1

As it is clear from Fig. 1, near-equilibrium concentration of potassium ferrate does not depend only on the temperature of the solution, but also on the concentration of background electrolyte. On the qualitative level, the obtained dependences correspond to the data of [8, 14, 15], some of the discrepancies may be due to the differences in the purity of the K_2 FeO₄ samples used.

5. 2. Recycling of mother liquors resulting from chemical synthesis of Fe(VI) compounds

At the next stage of the research, under the same conditions four options of hypochlorite synthesis were performed from the various iron(III) compounds: $Fe(NO_3)_3 \cdot 9H_2O$, $Fe_2(SO_4)_3 \cdot 9H_2O$, $FeCl_3 \cdot 6H_2O$, FeO(OH), which are frequently used for the preparation of Fe(VI) compounds. After the reactions 1, 3-5 and room-temperature filtration of the formed precipitation, a complex analysis of spent mother liquors was carried out, the results of which are presented in Table 1.

Table 1

The content of basic and ballast anions in mother liquors at 20 °C

Precursor	Concentration, M					
	OH⁻	FeO_4^{2-}	Cl-	NO_3^-	SO_4^{2-}	
$Fe(NO_3)_3 \cdot 9H_2O$	13.0	4.3·10 ⁻³	0.166	0.191	0	
$Fe_2(SO_4)_3 \cdot 9H_2O$	13.5	3.8·10 ⁻³	0.163	0	2.9·10 ⁻³	
$FeCl_3 \cdot 6H_2O$	13.4	3.9·10 ⁻³	0.165	0	0	
FeO(OH)	14.3	3.3·10 ⁻³	0.157	0	0	

As it follows from the data of Table 1, in the mother liquor, apart from OH^- anions, there are chlorides (CIO^- reduction products), as well as some ballast anions (nitrates, sulfates or chlorides). As we should expect, the nature of ballast anions is determined by the type of used starting iron(III) compounds.

It is advisable to purify the mother liquor for reuse in the production cycle by using the effect of salting out various substances in the presence of large amounts of alkali metal hydroxides. The solubility of some salts in concentrated alkaline solutions is shown in Fig. 2.



Fig. 2. Solubility of ballast salts in the mother liquor at: a - 0 °C; b - 20 °C; $1 - KNO_3$; 2 - KCI; $3 - K_2SO_4$

As it is seen from the data in Fig. 2, the solubility of ballast salts is significantly affected by the temperature and concentration of potassium hydroxide. In order of decreasing solubility, the salts can be arranged in the following phenomenological series: $KNO_3-KCl-K_2SO_4$. It should be noted that the solubility of the target product is comparable only to the solubility of potassium sulfate (Fig. 1, 2), so the mother liquor will be purified during the salting-out process as a result of co-crystallization of both salts.

The content of other ballast salts in the system after the first stage of salting out is sufficiently large (Table 1), therefore, it is obvious that for deeper purification it is necessary to reduce the temperature and increase the concentration of alkali. Therefore, for the purification of solution from nitrates and chlorides, it is expedient to carry out two stages of salting out – one at room temperature and the other at low temperatures.

The solutions obtained after the reaction (5) (the precursor of FeO(OH)), are most suitable for recycling because they contain a minimum amount of ballast salts (only chlorides). Moreover, in the process of synthesis based on other precursors, the consumption of OH⁻ anions is approximately 2.5 times as large (Table 1).

5.3. Recycling of mother liquors resulting from electrochemical synthesis of Fe(VI) compounds

In the process of transpassive dissolution of iron in alkaline medium (concentrated NaOH or KOH solutions):

$$Fe+8OH^{-}-6e^{-} \rightarrow FeO_4^{2-}+4H_2O, \tag{6}$$

along with the target product, extraneous anions accumulate in small amounts, which is due to the presence of heavy metal impurities in used electrode materials. It has been established that the main impurities in the liquid phase are manganese and chromium oxoanions (MnO_4^{2r}, CrO_4^{2r}) (Table 2).

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The content of manganese and chromium oxoanions in the mother liquor after electrosynthesis

Na		ω, %		C·10 ⁵ , M	
NO.	Electrode material	Mn	Cr	MnO_4^{2-}	$\mathrm{CrO}_4^{2\text{-}}$
1	Low-carbon steel	0.38	0.032	0.2	0
2	Open-hearth iron	0.88	0.137	0.9	0.1
3	Low alloyed steel	0.95	0.143	1.3	0.15
4	Foundry iron	0.88	2.83	1.1	3.4

Note: current density is 5 mA/cm^2 , electrolysis time is 10 hours

These results do not go against the works [6, 10, 13], which inform about the inclusions of manganese and chromium in the crystalline ferrates prepared by electrochemical and chemical methods.

In addition, due to the uneven dissolution of the electrodes, the formation of so-called anodic sludge takes place, accelerating decomposition of ferrates. Therefore, in order to eliminate the negative effect of insoluble Co, Ni, Cr, Mn compounds, etc., contained in the sludge [10, 13] and the transition of some its components (Cr, Mn) into the liquid phase, it is necessary to reduce the contact time of the latter with the mother liquor, for example, by means of periodic or continuous filtration.

As a rule, in order to reuse KOH solutions after separation of the target product, it is only sufficient to correct the electrolyte by the concentration of potassium hydroxide. However, if the initial solution was NaOH, then after the processes (6) and (2) the mother liquor, which is an aqueous solution of sodium and potassium hydroxides, it can not be separated into individual components, so, it can not be recycled.

6. Discussion of the results of the mother liquor recycling research

It should be noted that the practicability of recycling is much higher when cleaning is based on physical methods, because its implementation costs are minimal. That is why for the purification of mother liquor, the salting-out operation has been chosen as the main technological decision, which effectiveness is regulated by the changing of temperature. Low-temperature salting-out is well suited for this system, as for removing of ballast salts (or target product) from the mother liquor there is no need to add a special salting-out agent – its role is performed by the main component of the solution – potassium hydroxide, which cleaning is the main task of recycling.

As it follows from Fig. 1, for the fuller extraction of the target product after synthesis from the mother liquor, it is necessary to use low temperatures. However, it must be taken into account that the solubility of ballast salts also reduces with increasing KOH concentration and decreasing temperature (Fig. 2). Therefore, the first stage of recovering solid K_2 FeO₄ is expedient to carry out at room temperature, when the main part of the target product will precipitate. During the second stage of salting out (low-temperature area), the precipitation will mainly contain ballast salts. Such technological decision, unlike those proposed in [5, 8], will simplify the purification of the target product from ballast salts. The experiments showed that as a result of low-temperature salting out, the potassium sulfate (its content after purification does not exceed 0.3 g/l can be removed from the mother liquor, in addition to ferrates(VI). At the same time, such complete removal of nitrates and chlorides was not achieved even at 0 °C. However, the residual concentration of these anions of ~ 10 g/l in the mother liquor after the second stage of purification is also an acceptable result. With such content in the system, nitrates and chlorides do not have a noticeable negative effect on the synthesis.

It is important to note that the use of iron(III) oxohydroxide FeO(OH) instead of crystalline hydrates of salts has several advantages. In the synthesis, this precursor does not form additional ballast anions (reactions 1, 3–5). Besides, it is characterized by the lowest consumption of OH⁻ anions, so only 2 moles of KOH are consumed to form one mole of the target product, while for Fe(III) salts, 5 moles are required. Therefore, these facts allow us to consider that FeO(OH) is the best iron-containing precursor for the synthesis of ferrates.

It is necessary to emphasize that in [8] it is proposed to purify alkaline solutions containing 11 M potassium hydroxide, which are formed as a result of the mixing of the spent mother liquor with more dilute (3 M KOH), washing solutions. However, such technological decision is inexpedient, as the composition of the purified mother liquor (11 M) in the main component does not correspond to the initial solution (14 M) necessary for synthesis, i. e., the main principle of recycling is the identity of the compositions.

As it was mentioned above, recycling of KOH based on mother liquors after electrosynthesis has no problems. Only a careful selection of electrode materials (cast iron and steel with a minimum content of technological impurities of heavy metals and complete exclusion from the redistribution of alloy ferroalloys) is necessary. In these solutions, even after prolonged electrolysis, extraneous anions are contained in permissible amounts that do not have a significant effect on the technological parameters of electrosynthesis and the quality of the target product. Therefore, after the separation of K₂FeO₄ from the mother liquor, only the normalization of the latter over the OH⁻ anions is necessary.

So, it should be focused on the synthesis of K_2FeO_4 based on sodium hydroxide. During performing technological operations, it is much more difficult to work with NaOH solutions compared to KOH, especially in the area of low temperatures, which is due to higher viscosity values. Besides, after performing the main steps of the technological cycle (reactions 1 and 2 or 5 and 2), the mother liquor, without regard to the method of obtaining ferrates, is a hard-to-separate mixture of sodium and potassium hydroxides, which makes it practically unsuitable for recycling. And the absence of the latter leads to low competitiveness of this production technology. Therefore, despite the higher price of KOH, a preference for the production of potassium ferrate should still be given to the mother liquor based on potassium hydroxide, rather than NaOH.

It is obvious that the scope of NaOH should be limited to obtaining either ferrate-containing alkaline solutions or crystalline $BaFeO_4$, when for ferrates precipitation from the mother liquor barium hydroxide should be used, but not KOH:

$$Ba(OH)_2 + FeO_4^{2-} \rightarrow BaFeO_{4(s)} + 2OH^{-}.$$
 (7)

In this case, the production of the target product (barium ferrate) and the purification of the mother liquor from ex-

traneous anions are combined in one process, since $BaFeO_4$ resulting from the reaction (7) will include insignificant amounts of $BaMnO_4$, $BaCrO_4$ and $BaCO_3$.

7. Conclusions

1. The peculiarities of mother liquors recycling in various technologies of obtaining ferrates(VI) are considered. As a basic technological decision for precipitation of K_2FeO_4 and purification of spent alkaline solutions, it has been suggested to use two-stage salting out in concentrated KOH solutions (the first stage is at room temperature and the second one is at low temperatures).

2. It has been shown that the solubility of ballast salts with increasing alkali concentration and decreasing temperature reduces in the $\text{KNO}_3-\text{KCl}-\text{K}_2\text{SO}_4$ series.

3. It has been established that recycling of mother liquors is much more effective if alkaline solutions based on KOH are used for synthesis, and FeO(OH) is used as an iron precursor.

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