MINING SCIENCE AND TECHNOLOGY (RUSSIA)
ГОРНЫЕ НАУКИ И ТЕХНОЛОГИИ
2024;9(1):21–29

eISSN 2500-0632
https://mst.misis.ru/

BENEFICIATION AND PROCESSING OF NATURAL AND TECHNOGENIC RAW MATERIALS

Research paper

https://doi.org/10.17073/2500-0632-2023-12-196
UDC 622.765.4

Electrochemical action on the flotation beneficiation of ordinary iron ore concentrate

Kh.K. Rakhimov[1], **E.L. Chanturiya**[1, 2], **D.V. Shekhirev**[1]
[1] *University of Science and Technology MISIS, Moscow, Russian Federation*
[2] *N.V. Melnikov Institute for the Problems of Integrated Subsoil Development, Russian Academy of Sciences (IPKON RAS), Moscow, Russian Federation*
✉ *elenachan@mail.ru*

Abstract

One of the main challenges in processing fresh ferruginous quartzites is to obtain high-quality iron ore concentrates containing more than 70% total iron and less than 1.8% silica to produce DR pellets and hot Briquetted Iron (HBI). Currently, it is widely recognized that the most effective methods to achieve high-quality iron ore concentrates is through reverse flotation using cationic amine collectors in an alkaline medium. However, due to the very fine impregnation of magnetite in quartz, the insufficiently complete release of magnetite even with fine grinding, and the proximity of the flotation (surface) behavior of the separated minerals, high-quality concentrates are not always achievable in the flotation process. Consequently, exploring methods to enhance the efficiency of flotation separation of minerals and improve concentrate quality remains a pertinent issue. Historical studies have shown that electrochemical treatment can adjust the properties of reagents, enhance their effect on specific minerals, and thus control the flotation process. The efficiency of quartz and other silicates flotation by amines significantly depends on the ratio of ionic and molecular forms of the reagent in aqueous solutions of the collector and in the flotation pulp. Altering this ratio can impact the outcomes of reverse cationic flotation of iron ores. It is feasible to change the ratio of the amine forms through electrochemical oxidation or reduction of the reagent solution. Moreover, the electrochemical treatment facilitates the dispersion of the amine in the aqueous medium and its physical adsorption on minerals. Therefore, electrochemical pretreatment of amines can be considered a promising method for intensifying the reverse flotation of iron ore. This paper presents research results aimed at improving the quality of the oversize of the fine screening of ordinary magnetite concentrate from Mikhailovsky GOK, named after A.V. Varichev, through the use of electrochemically treated solutions of cationic amine class collectors in the process of reverse cationic flotation. The research findings confirmed the feasibility of using preliminary diaphragmless electrochemical treatment of reagents Tomamine RA-14 and Lilaflot 811M (esters of monoamine of different composition) for the targeted modification of their properties and for increasing the efficiency of reverse flotation. Consequently, the silica content in the flotation cell product decreased from 1.66–1.7% to 1.51–1.56, with the grade of total iron exceeding 70%.

Keywords

fresh ferruginous quartzites, magnetite concentrate, reverse cationic flotation, amines, electrochemical treatment

For citation

Rakhimov Kh.K., Chanturiya E.L., Shekhirev D.V. Electrochemical action on the flotation beneficiation of ordinary iron ore concentrate. *Mining Science and Technology* (*Russia*). 2024;9(1):21–29. https://doi.org/10.17073/2500-0632-2023-12-196

ОБОГАЩЕНИЕ, ПЕРЕРАБОТКА МИНЕРАЛЬНОГО И ТЕХНОГЕННОГО СЫРЬЯ

Научная статья

Использование электрохимических воздействий в процессе флотационного дообогащения рядового железорудного концентрата

Х.К. Рахимов[1], **Е.Л. Чантурия**[1, 2], **Д.В. Шехирев**[1]
[1] *Университет науки и технологий МИСИС, г. Москва, Российская Федерация*
[2] *Институт проблем комплексного освоения недр им. академика Н.В. Мельникова Российской академии наук (ИПКОН РАН), г. Москва, Российская Федерация*
✉ *elenachan@mail.ru*

Аннотация

Одной из основных задач при переработке неокисленных железистых кварцитов является получение высококачественных железорудных концентратов, содержащих более 70 % железа общего и менее 1,8 % кремнезема для получения DR-окатышей и горячебрикетированного железа.

В настоящее время общепризнано, что наиболее эффективным способом получения высококачественных железорудных концентратов является обратная флотация катионными собирателями аминами в щелочной среде, однако из-за тончайшей вкрапленности магнетита в кварц, недостаточно полного раскрытия магнетита даже при тонком измельчении, а также из-за близости флотационных (поверхностных) свойств разделяемых минералов даже в процессе флотации не всегда возможно выделить высококачественные концентраты. В этой связи остается актуальным поиск способов повышения эффективности флотационного разделения минералов и повышения качества концентрата.

Ранее проведенными исследованиями показано, что с помощью электрохимической обработки можно регулировать свойства реагентов, усиливать их воздействие на определенные минералы и таким образом управлять процессом флотации. Поскольку эффективность флотации кварца и других силикатов аминами в существенной мере зависит от соотношения ионной и молекулярных форм реагента в водных растворах собирателя и во флотационной пульпе, изменение этого соотношения может влиять на результаты обратной катионной флотации железных руд. Изменение соотношения форм амина возможно при электрохимическом окислении или восстановлении раствора реагента. Кроме того, электрохимическая обработка способствует диспергации амина в водной среде и его физической адсорбции на минералах. Соответственно, предварительная электрохимическая обработка аминов может рассматриваться как одно из перспективных направлений интенсификации обратной флотации железных руд.

В статье представлены результаты поисковых исследований по улучшению качества концентрата, полученного флотацией мелкого грохочения рядового магнетитового концентрата Михайловского ГОКа им. А.В. Варичева за счет использования в процессе обратной катионной флотации электрохимически обработанных растворов катионных собирателей класса аминов.

Результаты поисковых исследований подтвердили возможность применения предварительной бездиафрагменной электрохимической обработки реагентов Tomamine PA-14 и Lilaflot 811M (эфиров моноамина различного состава) для направленного модифицирования их свойств и повышения эффективности обратной флотации надрешетного продукта: содержание кремнезема в камерном продукте снизилось с 1,66–1,7 % до 1,51–1,56 при содержании железа общего более 70 %.

Ключевые слова

неокисленные железистые кварциты, магнетитовый концентрат, обратная катионная флотация, амины, электрохимическая обработка

Для цитирования

Rakhimov Kh.K., Chanturiya E.L., Shekhirev D.V. Electrochemical action on the flotation beneficiation of ordinary iron ore concentrate. *Mining Science and Technology* (*Russia*). 2024;9(1):21–29. https://doi.org/10.17073/2500-0632-2023-12-196

Introduction

One of the primary challenges in the processing of fresh ferruginous quartzites is obtaining high-quality iron ore concentrates with over 70% total iron and less than 1.8% silica for the production of DR pellets and direct reduction iron. Recent studies [1, 2] explore the potential of enhancing the efficiency of ferruginous quartzite processing, including improving the quality of ordinary magnetite concentrate through fine screening. The unique texture and structure of these ores, characterized by the finest unreleased impregnation of quartz in magnetite and vice versa, along with complex intergrowths of magnetite, quartz, and other silicates, make it challenging to achieve the desired quality of magnetic separation concentrates, even with fine grinding. Therefore, fine screening of ordinary magnetite concentrates separated by magnetic separation, additional grinding of the oversize, and further finishing by flotation are required to improve the magnetite concentrate quality. Studies [3, 4], analyzing the material composition of the fine screening oversize from ordinary magnetite concentrate of magnetic separation, reveal that a significant part of all mineral phases in the initial oversize are intergrown with each other. Grinding the material to a grain size of 80% –30 microns leads to a more complete release of lean magnetite intergrowths, increasing the proportion of magnetite in the form of rich intergrowths while maintaining the proportion of magnetite as released grains. Moreover, inclusions of quartz and celadonite in magnetite, indiscernible as separate mineral phases and identified only through average elemental composition, remain unreleased even with fine grinding, leading to a decreased quality of the magnetite concentrate. Study [3] presents theoretical performance predictions, while studies [5, 6] provide experimental results of reverse cationic flotation of the oversize.

It is now widely acknowledged that reverse flotation using cationic amine collectors in an alkaline medium is the most effective method for iron ores and concentrate flotation [7–9]. Study [7] offers a comprehensive review of collectors, depressors, and medium regulators globally utilized in iron ore flotation. Publications [8, 9] detail the characteristics of amine cationic collectors, explore various amines, and present the results of their application in iron ore flotation based on pulp pH and quartz grain size distribution. The superiority of cationic reverse flotation over anionic flotation of iron ores has been established through comparison [10], and the study on the role of starch consumption as a depressor of iron minerals in reverse flotation by amines in an alkaline medium showed the potential to increase iron recovery into the cell product [11]. Reviews [12, 13] also highlight the efficiency of reverse cationic flotation of iron ores by amines in an alkaline medium. However, achieving high-quality concentrates through reverse flotation with amines is sometimes hindered by the insufficient release of magnetite even with fine grinding, the presence of magnetite intergrowths with quartz and other silicates, and the proximity of flotation (surface) behavior of the separated minerals. Attempts to enhance the selectivity of silicates and Ca – containing mineral flotation to separate magnetite using a mixture of collectors, however have been explored in [14, 15], but finding ways to improve mineral separation efficiency and magnetite concentrate quality remains a crucial pursuit.

Historical studies have shown that electrochemical treatment can modify the properties of reagents, amplify their effect on certain minerals, and thereby control the flotation process [16, 17].

The reagent's form in aqueous solution significantly influences the structure and composition of the mineral particles' surface hydrophobic layer, affecting their flotation behavior. The aqueous medium may contain ionic, molecular, polymeric, and micellar forms of amine in ratios that depend on reagent concentrations and medium conditions [18]. Since the flotation efficiency of quartz and other silicates by amines largely relies on the ratio of ionic and molecular forms of the reagent in collector solutions and the flotation pulp, changing this ratio can impact the results of reverse cationic flotation of iron ores [18]. Modifying the amine forms ratio is possible through electrochemical oxidation or reduction of the reagent in an aqueous solution. Furthermore, electrochemical treatment aids in the dispersion of the amine in the aqueous medium and its physical adsorption on minerals.

Given the foregoing, and based on literature data, the potential for intensifying the reverse flotation of iron ores with amines after their preliminary electrochemical treatment is considered promising.

This paper presents research findings on improving the quality of the oversize of fine screening of ordinary magnetite concentrate through the use of electrochemically treated solutions of cationic amine class collectors in the process of reverse cationic flotation i.e., the application of additional flotation beneficiation of the oversize.

Research Subjects and Methods

The research focused on the oversize from fine screening of the ordinary magnetite concentrate from the A.V. Varichev Mikhailovsky GOK (sample ND). The mineral and chemical compositions of this sample are detailed in Tables 1 and 2, respectively.

For the flotation beneficiation process, cationic reagents produced on an industrial scale by Nouryon Surface Chemistry AB Akzo Nobel and Clariant, TOMAH PRODUCTS ING, were used as collectors (Table 3).

Table 1

Chemical composition of ND sample, %

ND sample	Fe_{total}	SiO_2
ND, according to MGOK data	61.7	12.6
ND, according to X-ray fluorescence analysis data, direct analysis	62.22	11.78
ND, according to X-ray fluorescence analysis data, estimated, weighted average	61.32	11.80

Table 2

Mineral composition of the oversize by mineral group (according to MLA data)

Mineral group	Minerals included in the group	Mineral group share, %
Magnetite	“Pure” magnetite, magnetite with fine quartz inclusions, magnetite with fine quartz and celadonite inclusions	84.77
Hematite	Hematite, goethite	3.82
Carbonates:	Ankerite, siderite, calcite	0.89
Quartz	Quartz, quartz with fine inclusions of magnetite	7.73
Aegirine	Aegirine	0.71
Celadonite	Celadonite	1.73
Aluminosilicates	Minerals of aluminosilicate group	0.07
Other	Other	0.20
Iron scrap	Iron scrap	0.08
Total	Sum of minerals	100.00

The electrochemical treatment (ECT) of a 1% aqueous solution (using recycled water from MGOK) of the reagents Lilaflot 811 M (mixture of monoamine esters (alkoxypropylamines) alkene $C_{13}H_{29}NO$ (isomer) (50–70%) + alkane $C_{15}H_{33}NO_3$ (30–50%), degree of neutralization of 20–40%) and Tomamine PA-14 (monoamine ester $R–O–CH_2CH_2CH_2–NH_2$; [3-(isodecyloxy) propylamine-1] 95%, degree of neutralization of 30% + iso-alcohols $C_9–C_{11}$ 5%; iso-C10 saturated) was carried out in laboratory setting. The ECT was performed using direct electric current in a diaphragmless glass electrochemical cell of 150 ml capacity (Fig. 1). For electrodes, insoluble titanium drop-forged mesh coated with ruthenium oxide coating was employed, maintaining a working areas ratio of 1 : 2 between the anode and cathode. The reagent solution's treatment involved applying electric current concentrations of 0.28, 0.4, and 0.6 Ah/L in both "cathode" and "anode" modes, where the electrode with the larger area functioned as the cathode in cathode mode and as the anode in anode mode. The electric current was supplied to the electrodes through a rectifier, and the solution was agitated using a magnetic stirrer throughout the treatment process.

The electrochemical treatment (ECT) of the reagent solution lasted for 10 min. Before and after the treatment, the pH and Eh of the reagent solution were measured.

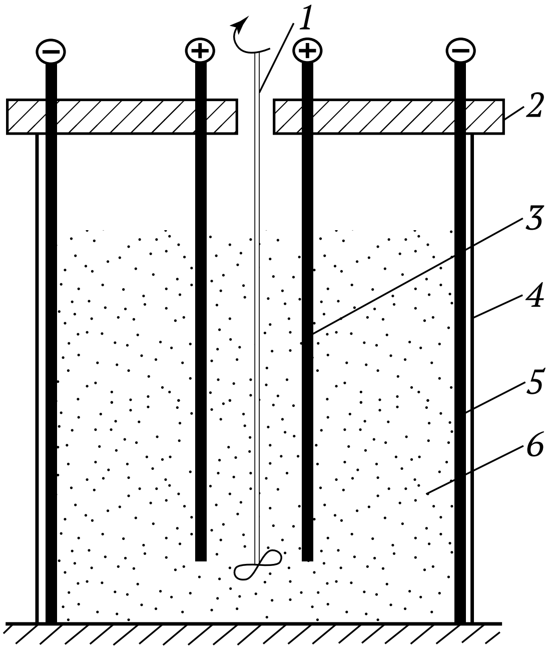

Fig. 1. Apparatus for diaphragmless treatment of a reagent solution:
1 – mechanical stirrer; *2* – removable cover made of non-conductive material with an opening for the mechanical stirrer; *3* – cylindrical anode (insoluble titanium drop-forged mesh with ruthenium oxide coating); *4* – beaker made of non-conductive material (glass); *5* – cylindrical cathode (insoluble titanium drop-forged mesh with ruthenium oxide coating); *6* – reagent solution

Table 3

Conditions for electrochemical treatment of recycled water and reagent solutions

Treatment conditions	Current concentration, Ah/L	Treatment time, min	Initial pH	Initial Eh	Final pH	Final Eh
Recycled water						
Anode	0.28	10	7.784	+176.1	8.77	−243.2
Anode	0.4	10	7.784	+176.1	8.11	−72.2
Anode	0.6	10	7.784	+176.1	7.926	+142
Cathode	0.28	10	7.784	+176.1	8.34	+86.7
Cathode	0.4	10	7.784	+176.1	7.87	+134.8
Cathode	0.6	10	7.784	+176.1	8.06	−276
Tomamine						
Anode	0.28	10	9.18	+60.5	9.06	+112
Anode	0.4	10	9.18	+60.5	9.152	+125
Anode	0.6	10	9.18	+60.5	9.23	+148.6
Cathode	0.28	10	9.18	+60.5	9.1	+32.3
Cathode	0.4	10	9.18	+60.5	9.33	+40.4
Cathode	0.6	10	9.18	+ 60.5	9.24	−302.3
Lilaflot 811M						
Cathode	0.28	10	7.91	+84	9.195	−360
Cathode	0.4	10	7.91	+84	9.0	−308.6
Cathode	0.6	10	7.91	+84	8.8	−330

Reverse cationic flotation was conducted on the ground oversize (a 294 g subsample) of fine screening from the ordinary magnetite concentrate using the Tomamine PA-14 reagent and recycled water from Mikhailovsky GOK. The process followed a flow sheet that included preliminary magnetic consolidation of the oversize, rougher reverse flotation of the magnetic fraction, and recleaner reverse flotation of the cell product [3]. Similarly, the Lilaflot 811M reagent was used according to an analogous flow sheet but without preliminary magnetic consolidation. In both cases, the grinding time was set at15 min. The collector's consumption rate was 240 g/t for the rougher flotation and 100 g/t for the cleaner flotation. Dextrin, used as a magnetite depressor, was added during the rougher flotation at a rate of 600 g/t. The pH level was maintained at 10–10.5 using caustic soda.

Research Findings and Discussion

The conditions for electrochemical treatment and the results from the flotation experiments are presented in Tables 3–5 and Figs. 2 and 3.

Table 4

Results of reverse cationic flotation with preliminary magnetic consolidation using electrochemically treated Tomamine reagent

Treatment conditions	Current concentration, Ah/L	Product	Yield, %	Percentage		Recovery, %	
				Fe	SiO_2	Fe	SiO_2
No treatment	–	n/m	6.3	16.32	67.13	1.65	35.42
		Flotation froth (product) 1	29.2	53.71	21.93	25.29	53.87
		Flotation froth (product) 2	7.4	67.47	4.07	8.02	2.52
		Cell flotation product	57.2	70.39	1.70	65.04	8.19
		Initial	100.0	61.9285	11.870	100.00	100.00
Anode	0.28	n/m	4.5	15.85	67.82	1.17	24.81
		Flotation froth (product) 1	33,6	51.9	23.88	28.36	64.83
		Flotation froth (product) 2	8.2	66.29	4.49	8.81	2.96
		Cell flotation product	53.7	**70.51**	**1.7**	61.67	7.39
		Initial	100.0	61.445	12.365	100.00	100.00
Anode	0.4	n/m	5.8	15.56	67.7	1.47	31.62
		Flotation froth (product) 1	31.5	52.51	23.16	27.04	58.92
		Flotation froth (product) 2	7.4	66.71	4.17	8.05	2.49
		Cell flotation product	55.4	**70.06**	**1.56**	**63.43**	6.98
		Initial	100.0	61.137	12.377	100.00	100.00
Anode	0.6	n/m	5.6	15.8	67.71	1.45	30.39
		Flotation froth (product) 1	32.1	52.44	23.23	27.55	59.58
		Flotation froth (product) 2	7.7	66.81	4.45	8.45	2.75
		Cell flotation product	54.6	**69.99**	**1.67**	**62.55**	7.29
		Initial	100.0	61.071	12.510	100.00	100.00
Cathode	0.28	n/m	5.4	15.06	68.73	1.33	29.54
		Flotation froth (product) 1	34.3	53.14	22.06	29.99	60.48
		Flotation froth (product) 2	6.3	66.25	4.73	6.90	2.39
		Cell flotation product	54.0	**69.62**	**1.76**	61.78	7.59
		Initial	100.0	60.815	12.518	100.00	100.00
Cathode	0.4	n/m	6.1	19.47	63.02	1.96	31.36
		Flotation froth (product) 1	31.3	52.18	22.99	26.74	58.34
		Flotation froth (product) 2	6.7	66.19	4.4	7.32	2.41
		Cell flotation product	55.9	**69.9**	**1.74**	**63.99**	7.89
		Initial	100.0	61.018	12.321	100.00	100.00
Cathode	0.6	n/m	5.4	16.29	66.48	1.44	29.71
		Flotation froth (product) 1	33.2	53.01	22.27	28.78	60.95
		Flotation froth (product) 2	8.1	66.8	3.83	8.81	2.55
		Cell flotation product	53.3	**70.1**	**1.55**	**60.97**	6.80
		Initial	100.0	61.232	12.146	100.00	100.00

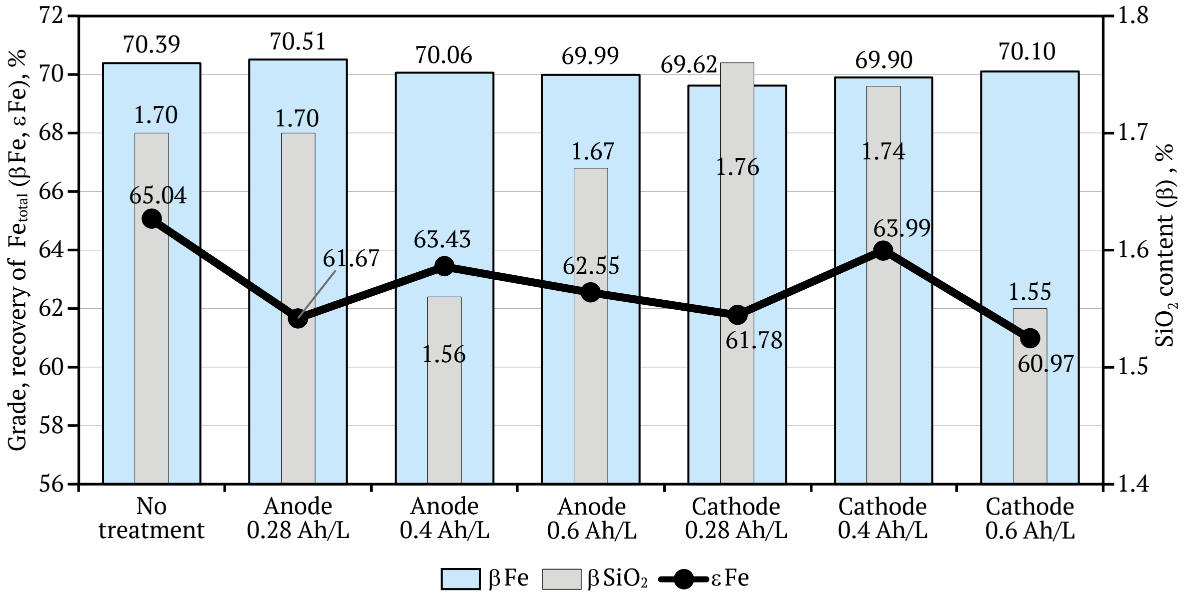

Fig. 2. Results of reverse cationic flotation with preliminary magnetic consolidation using electrochemically treated Tomamine reagent

Table 5

Results of reverse cationic flotation without preliminary magnetic consolidation using electrochemically treated Lilaflot 811 M amine reagent

Treatment conditions	Current concentration, Ah/L	Product	Yield, %	Percentage, %		Recovery, %	
				Fe	SiO_2	Fe	SiO_2
No treatment	–	Flotation froth (product) 1	41.28	50.24	25.27	33.61	82.79
		Flotation froth (product) 2	9.26	67.92	14.57	10.20	10.71
		Cell flotation product	49.46	**70.10**	**1.66**	**56.19**	6.50
		Initial	100.00	61.70	12.60	100.00	100.00
Cathode	**0.28**	Flotation froth (product) 1	40.98	49.55	26.99	32.91	87.79
		Flotation froth (product) 2	8.42	68.08	9.19	9.29	6.14
		Cell flotation product	50.59	**70.48**	**1.51**	**57.79**	6.06
		Initial	100.00	61.70	12.60	100	100.00
Cathode	**0.4**	Flotation froth (product) 1	38.54	47.15	29.50	29.45	90.23
		Flotation froth (product) 2	7.10	68.30	5.58	7.86	3.15
		Cell flotation product	54.36	**71.15**	**1.54**	**62.69**	6.63
		Initial	100.00	61.70	12.60	100.00	100.00
Cathode	**0.6**	Flotation froth (product) 1	31.44	45.10	34.55	22.98	86.21
		Flotation froth (product) 2	5.64	65.36	9.62	5.97	4.30
		Cell flotation product	62.92	**69.67**	**1.90**	**71.05**	9.49
		Initial	100.00	61.70	12.60	100.00	100.00

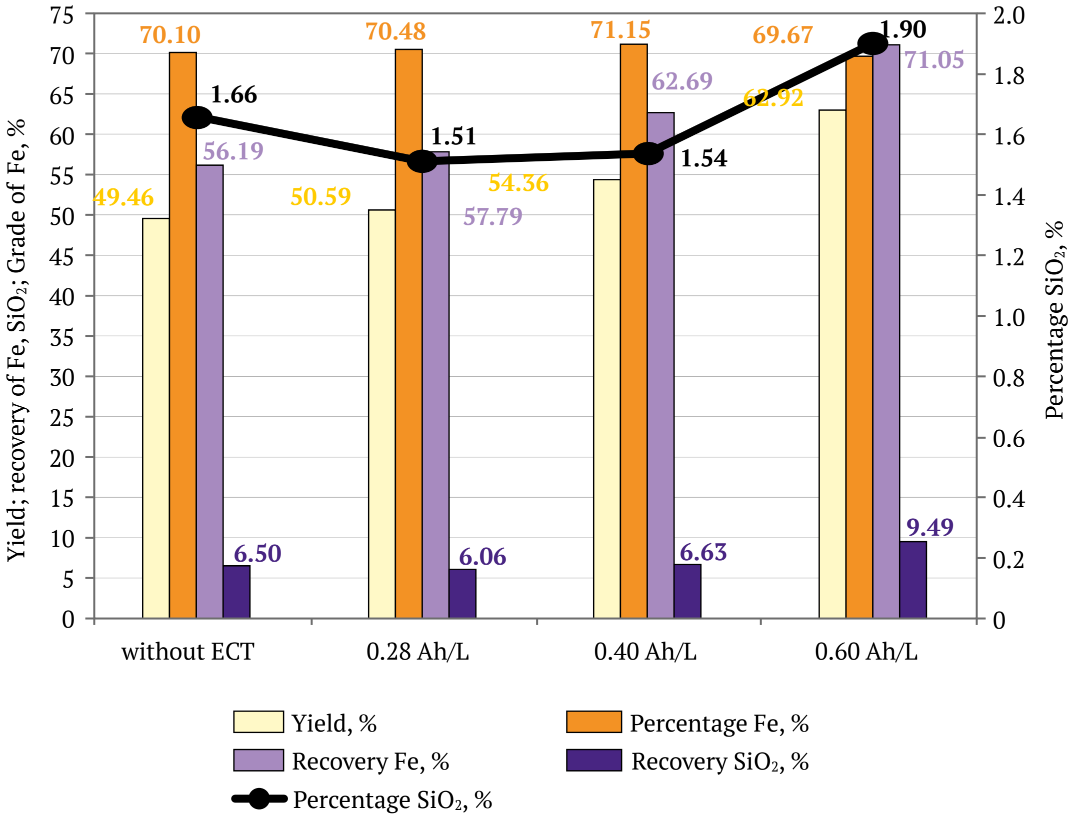

Fig. 3. Results of reverse cationic flotation without preliminary magnetic consolidation using electrochemically treated Lilaflot 811 M amine reagent

The analysis of the obtained results indicates that the anode mode of diaphragmless electrochemical treatment using Tomamine PA-14 reagent at a current concentration of 0.4 Ah/l had a noticeable effect. It allowed for a reduction in silica content from 1.7 to 1.56% while maintaining the required quality of total iron grade at 70.06 %, albeit with a slight decrease in Fe recovery from 65.04% to 63.43% compared to the reference test without ECT. Somolarly, the cathode mode at 0.6 Ah/L yielded a high-quality concentrate containing 70.1 % total iron with a recovery of 60.97%, versus 65.04% total iron in the test without ECT, and a silica content of 1.55 % (refer to Table 4, Fig. 2).

Therefore, the electrochemical treatment of the Tomamine PA-14 reagent positively influenced the concentrate's quality in terms of silica content, significantly reducing its while maintaining the iron grade at over 70%.

The electrochemical diaphragmless treatment of the Lilaflot 811 M reagent yielded positively results in the cathode mode. Without ECT, the flotation product contained 70.10% Fetotal and 1.66% SiO_2, with recoveries of 56.19 and 6.50%, respectively. Treating the reagent at a current concentration of 0.28 Ah/l increased the total iron grade in the concentrate to 70.48% and reduced the silica content to 1.51%, with recoveries of 57.79% for iron and 6.06% for silica, respectively. Treating the reagent at a current concentration of 0.4 Ah/L improved performance, resulting in a high-quality concentrate with a total iron grade of 71.15% and a silica content of 1.54%, with recoveries of 62.69% for total iron and 6.63% SiO_2 for silica (refer to Table 5, Fig. 3). Further increasing the current concentration to 0.6 Ah/L compromised the process's selectivity, decreasing the quality of the iron ore concentrate produced while increasing the recovery of total iron into the concentrate.

The electrooxidation and electroreduction reactions of amine are highly complex and, despite numerous studies, the mechanisms of these reactions remain unclear. At the anode, typically, the removal of an electron from the electron pair of a nitrogen atom usually occurs, initiating of sequence of complex transformations. Meanwhile, at the cathode, the reagent gains an additional electron. It is important to consider that, in the case of amines used as flotation reagents, they are often complex mixtures composed of monoamines, mono- and diamine esters, and others, further complicating the determination of the electrooxidation or electroreduction mechanisms.

Conclusions

The research findings suggest that preliminary diaphragmless electrochemical treatment of reagents such as Tomamine RA-14 and Lilaflot 811M (esters of monoamine with varying composition) could be used to purposefully modify their properties and enhance the efficiency of reverse flotation of the oversize. This could lead to a decrease in silica content in the cell flotation product and an increase in the grade and recovery of total iron in the flotation concentrate.

It should be noted that since all cationic amine collectors (including monoamines, diamines, and their esters) have diverse compositions and are often initially mixtures, varying effects of electrochemical treatment can be anticipated for each reagent. Consequently, it is necessary to test each collector under different ECT conditions on a case-by-case basis.

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Information about the authors

Khusrav K. Rakhimov – PhD-Student of the Department of Enrichment and Processing of Mineral Resources and Technogenic Raw Materials, University of Science and Technology MISIS, Moscow, Russian Federation; ORCID 0009-0007-8644-7055; e-mail rakhimov.khusrav@mail.ru

Elena L. Chanturiya – Dr. Sci. (Eng.), Professor, Professor of the Department of Enrichment and Processing of Mineral Resources and Technogenic Raw Materials, University of Science and Technology MISIS, Moscow, Russian Federation; Research Institute of Comprehensive Exploitation of Mineral Resources of the Russian Academy of Sciences, Moscow, Russian Federation; ORCID 0000-0002-5757-4799, Scopus ID 57196009376, ResearcherID J-4214-2014, AuthorID 67982; e-mail elenachan@mail.ru

Dmitry V. Shekhirev – Cand. Sci. (Eng.), Senior Researcher, Associate Professor of the Department of Enrichment and Processing of Minerals and Technogenic Raw Materials, University of Science and Technology MISIS, Moscow, Russian Federation; ORCID 0000-0002-9668-9716, Scopus ID 57170910700, AuthorID 589402; e-mail shekhirev@list.ru

Received 27.12.2023
Revised 19.01.2024
Accepted 20.01.2024