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Д.В. Сокольский атындағы «Жанармай,
катализ және электрохимия институты» АҚ

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ИЗВЕСТИЯ

НАЦИОНАЛЬНОЙ АКАДЕМИИ НАУК
РЕСПУБЛИКИ КАЗАХСТАН
АО «Институт топлива, катализа и
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Қазақстан Республикасы Ұлттық ғылым академиясы "ҚР ҰҒА Хабарлары. Химия және технология сериясы" ғылыми журналының Web of Science-тің жаңаланған нұсқасы Emerging Sources Citation Index-те индекстелуге қабылданғанын хабарлайды. Бұл индекстелу барысында Clarivate Analytics компаниясы журналды одан әрі the Science Citation Index Expanded, the Social Sciences Citation Index және the Arts & Humanities Citation Index-ке қабылдау мәселесін қарастыруда. Webof Science зерттеушілер, авторлар, баспашылар мен мекемелерге контент тереңдігі мен сапасын ұсынады. ҚР ҰҒА Хабарлары. Химия және технология сериясы Emerging Sources Citation Index-ке енуі біздің қоғамдастық үшін ең өзекті және беделді химиялық ғылымдар бойынша контентке адалдығымызды білдіреді.

НАН РК сообщает, что научный журнал «Известия НАН РК. Серия химии и технологий» был принят для индексирования в Emerging Sources Citation Index, обновленной версии Web of Science. Содержание в этом индексировании находится в стадии рассмотрения компанией Clarivate Analytics для дальнейшего принятия журнала в the Science Citation Index Expanded, the Social Sciences Citation Index и the Arts & Humanities Citation Index. Web of Science предлагает качество и глубину контента для исследователей, авторов, издателей и учреждений. Включение Известия НАН РК в Emerging Sources Citation Index демонстрирует нашу приверженность к наиболее актуальному и влиятельному контенту по химическим наукам для нашего сообщества.

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ELEMENTAL ANALYSIS OF LOCAL AND FOREIGN RAW MATERIALS FOR THE PRODUCTION OF TRIMANGANESE TETRAOXIDE PIGMENT

Abstract. In the course of continuation of scientific research in the development of Kazakhstani technology for the production of trimanganese tetraoxide pigment, there have been proposed new methods of analysis which make it possible to investigate new nanostructural materials sourced from local deposits. The results obtained are of practical importance and serve as the foundation for developing new technologically advanced domestic production of a competitive product that is in demand both on the national and international markets. The available methods are not able to highlight the particularity and diversity of Kazakhstani manganese ores due to the uneven distribution of manganese minerals and host (waste) rock, the quantitative ratio of rock-forming components and their variation instability. Methods of energy-dispersive analysis of manganese ores from Kazakhstani Zhaksylyk and Bogach deposits have been put forward, electronic micrographs, spectrograms of samples, diagrams of normalized distribution of elements on the surface of samples have been obtained and a comparative analysis with raw analogue samples of some foreign manufacturers has been performed.

Key words: hausmannite, pigment, manganese ore, nanopowders, electronic microscopy, energy-dispersive analysis.

Introduction. The main direction of The Strategic Development Plan of the Republic of Kazakhstan is creation of an export-oriented [1] economy with high added value. The export orientation results from the need to expand target markets for the growing volume of goods and services and to integrate into global value chains. Export-oriented industrialization will give the opportunity to bring new Kazakhstani goods to foreign markets, including high-tech processed goods.

These include trimanganese tetraoxide used for volumetric coloring of ceramic products made of white and red burnt clay in the production of light brown, yellow-brown, brown, dark brown bricks and ceramic tiles, as a combined pigment for painting, both in bulk and as a surface decorative finish [2-4]. The distinction of high quality manganese pigments is high manganese content, uniformity, low dusting and stable quality as well as the ability to form stable suspensions [5]. The outlined technological qualities of the proposed product determine a high demand for this pigment having a very strong effect on the average market value of the product.

For manufacturing the trimanganese tetraoxide product, it is planned to use raw materials mined in Zhaksylyk and Bogach deposits (Kazakhstan) [6-9], which will have a positive effect on the domestic economy of the country, and this production is also included in the list of approved state programs for industrial and innovative development and import substitution "Made in Kazakhstan". The development of national technology is necessary so that it is fully suitable for the use of raw materials mined in the territory of the Republic of Kazakhstan [10]. In addition, the developed technology will allow creating a scientific and technical groundwork for the production of finished products that meet the demand not only in Kazakhstan, but abroad as well.

In the present work there has been performed physicochemical analysis of samples of raw materials from manganese ore deposits Zhaksylyk and Bogach; electronic micrographs, spectrograms of the samples, diagrams of normalized distribution of elements on the surface of samples have been obtained and a comparative analysis with raw samples produced in Georgia and India has been carried out.

Materials and methods. For conducting the research, there were prepared 4 samples of raw materials for the production of trimanganese tetraoxide: 1) manganese ore of the Bogach deposit (Kazakhstan); 2) manganese ore of the Zhaksylyk deposit (Kazakhstan); 3) manganese ore produced in Georgia; 4) manganese ore produced by Raigarh Ridmakatra Ind., India.

Samples of manganese ore from the Bogach and Zhaksylyk deposits were presented in the form of broken fragments of rock (lumpy form). The initial crushing to granules of 1-5 mm was carried out on a jaw crusher “DSH 100x200”. Further grinding was performed for 8 hours on a laboratory ball mill “MSHL-1”.

The study by electronic microscopy was conducted at the Engineering Laboratory of the Eurasian National University named after L.N.Gumilyov on a Hitachi TM3030 scanning electronic microscope with a Bruker XFlash MIN SVE microanalysis system at an average voltage of 15kV. Elemental analysis was carried out by the method of energy-dispersive analysis by taking at least 7 spectra from each sample to obtain an average normalized value. The data on the content of elements are given in weight percent.

Results. Figure 1 shows micrographs of the investigated samples No.1-4 at resolution x1000. The analysis of micrographs reveals that dispersive capacity of samples 1, 2 and 4 is similar to each other and ranges from one to several tens of microns. At the same time, the dispersive capacity of sample of raw materials produced in Georgia (sample 3) is higher and larger aggregates reach up to 10 microns. It is obvious that in this sample enlarged aggregates arise due to the agglomeration of smaller particles.

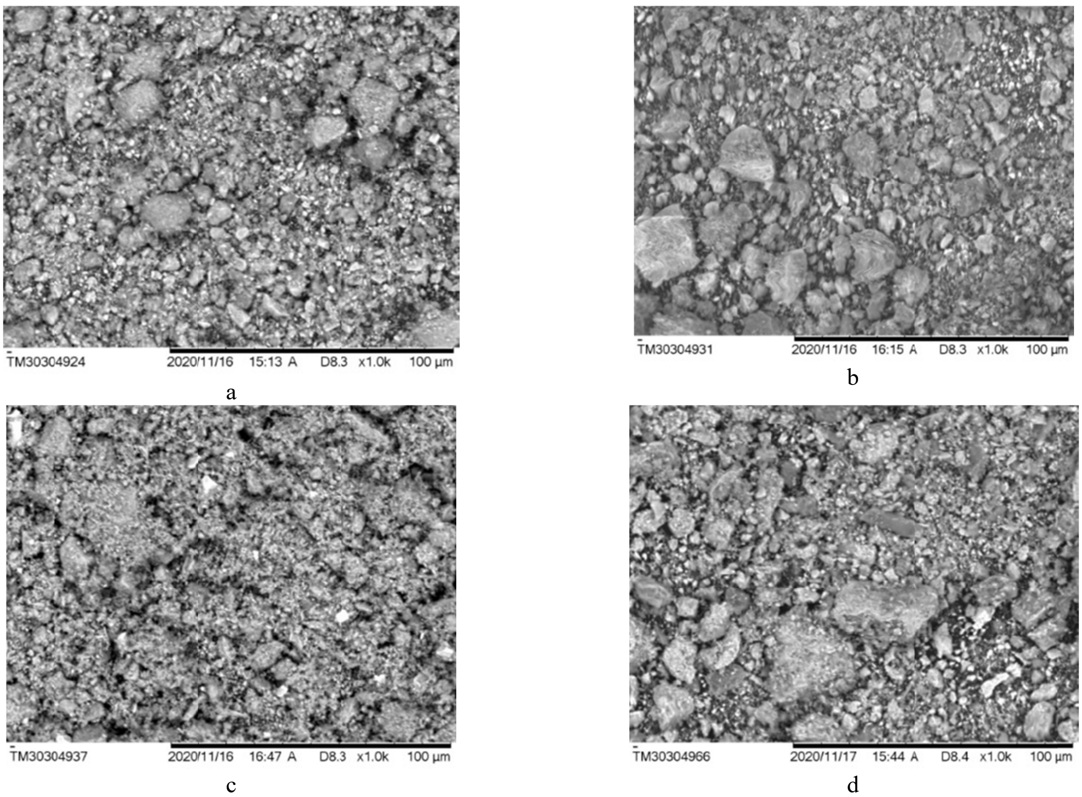

Figure 1 - Electronic micrographs (x1000) of the samples: a) manganese ore from the Bogach deposit; b) manganese ore from the Zhaksylyk deposit; c) manganese ore from Georgia; d) manganese ore from India

The microanalysis system made it possible to analyze the elemental composition of the samples by the energy-dispersive analysis method.

Tables 1-4 present the findings of the elemental analysis of the investigated samples.

Table 1 – Findings of the elemental analysis of manganese ore samples from the Bogach deposit

Point	Content of elements wt.%											Total
	C	O	Mn	Ca	Si	Na	Al	Mg	K	Fe	Cu	
1	36,2	40,04	17,75	2,13	1,26	0,67	0,69	0,52	0,73	0	0	100
2	10,75	33,55	40,56	4,78	6,53	0	0,98	0,99	1,87	0	0	100
3	5,54	32,55	47,21	7,18	3,26	0,28	0,95	0,98	2,05	0	0	100
4	7,57	36,48	36,34	6,54	6,56	1,71	2,38	0,9	1,53	0	0	100
5	3,5	25,36	51,63	11,28	4,78	0,09	1,22	0,35	1,79	0	0	100
6	5,55	29,03	50,58	9,41	2,01	0,79	0,94	0,18	1,51	0	0	100
7	3,11	16,76	68,48	4,13	1,39	0,16	1,46	0,48	4,03	0	0	100
8	13,21	42,22	33,56	5,47	2,27	0,43	0,44	1,09	1,31	0	0	100
Average	10,68	32	43,26	6,36	3,51	0,52	1,13	0,69	1,85	0	0	100

Table 2 – Findings of the elemental analysis of manganese ore samples from the Zhaksylyk deposit

Point	Content of elements wt.%											Total
	C	O	Mn	Ca	Si	Na	Al	Mg	K	Fe	Cu	
1	42,38	41,72	7,58	0	4,92	0	1,23	0	0,32	1,85	0	100
2	22,86	47,48	7,99	0	16,93	0	1,52	0	0,39	2,84	0	100
3	3,87	5,14	58,14	0	8,85	0	8,79	0	3,48	11,73	0	100
4	40,37	37,11	11,39	0	5,55	0	1,74	0	0,59	3,25	0	100
5	36,4	45,09	7,94	0	6,46	0	1,54	0	0,29	2,28	0	100
6	18,5	41,75	22,32	0	9,41	0	3,29	0	1,03	3,7	0	100
7	2,02	6,38	37,33	0	21,49	0	17,8	0	2,35	12,63	0	100
Среднее	23,77	32,1	21,81	0	10,52	0	5,13	0	1,21	5,47	0	100

Table 3 – Findings of the elemental analysis of manganese ore samples from Georgia

Point	Content of elements wt.%											Total
	C	O	Mn	Ca	Si	Na	Al	Mg	K	Fe	Cu	
1	2,82	29,52	65,22	0	1,28	0	1,16	0	0	0	0	100
2	2,76	34,9	58,25	0	2,61	0	1,49	0	0	0	0	100
3	2,35	19,33	75,17	0	1,13	0	2,02	0	0	0	0	100
4	2,1	29,43	64,56	0	1,79	0	2,12	0	0	0	0	100
5	2,62	26,41	64,96	0	3,12	0	2,89	0	0	0	0	100
6	2,2	31,74	49,06	0	15,71	0	1,28	0	0	0	0	100
7	3,74	35,36	58,35	0	1,61	0	0,94	0	0	0	0	100
8	2,97	36,94	20,06	0	39,57	0	0,45	0	0	0	0	100
9	2,81	28,37	66,2	0	1,39	0	1,23	0	0	0	0	100
Среднее	2,71	30,22	57,98	0	7,58	0	1,51	0	0	0	0	100

Table 4 – Findings of the elemental analysis of manganese ore samples from India

Point	Content of elements wt.%											Total
	C	O	Mn	Ca	Si	Na	Al	Mg	K	Fe	Cu	
1	3,63	48,48	16,08	0	13,1	2,73	12,4	0	0,56	1,93	1,09	100
2	0,82	10,35	60,67	0	4,69	0,46	9,63	0	1,46	7,66	4,26	100
3	3,93	45,09	36,55	0	2,92	0,63	4,62	0	0,67	4,01	1,58	100
4	0	14,64	32,93	0	9,56	0,41	10,53	0	0,65	27,17	4,12	100
5	10,35	43,47	28,73	0	5,5	0,6	6,44	0	0,64	2,37	1,89	100
6	1,94	25,95	40,3	0	21,25	0,24	4,18	0	0,67	3,91	1,55	100
7	3,46	32,9	35,38	0	10,62	0,54	7,12	0	0,65	6,33	2,99	100
Среднее	3,45	31,55	35,81	0	9,66	0,8	7,85	0	0,76	7,63	2,5	100

According to the elemental analysis data, it is seen that in terms of manganese content, the ore of the Bogach deposit ranks second after raw materials of Georgian origin and contains about 43,26% of manganese. At the same, the iron content in the same ore is not significant. The best results regarding the manganese content were revealed in the ore from Georgia, where the manganese content reached 57.95%. The lowest manganese content in the ore of the Zhaksylyk deposit is 21.81% which confirms the data of the technical literature that manganese ores in Kazakhstan are rather poor.

Figure 2 presents typical diagrams of the concentration distribution of atoms of elements in the investigated samples.

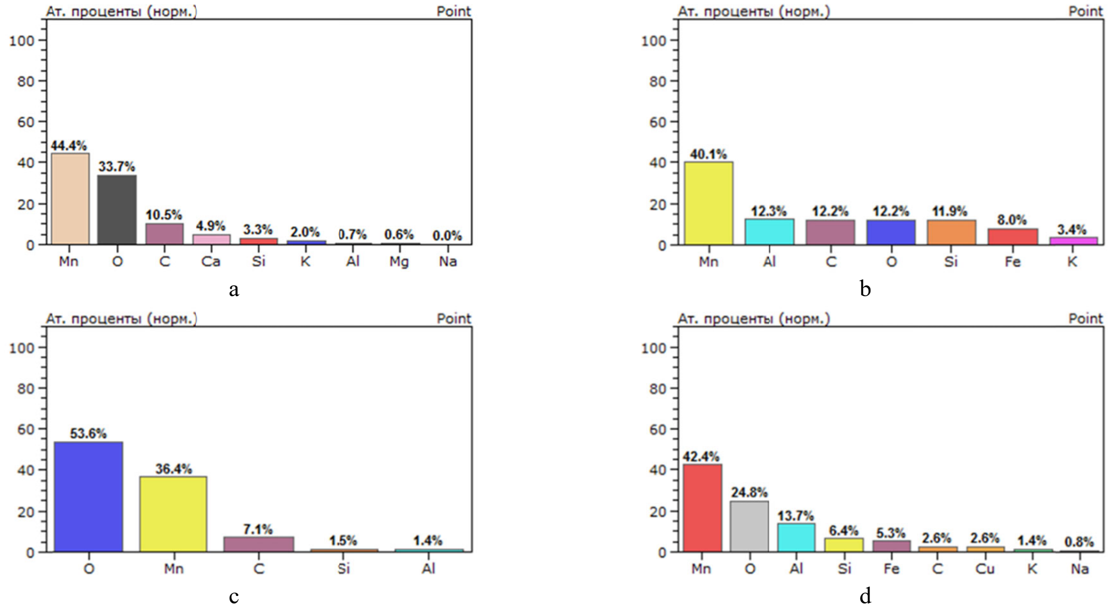

Figure 2 - Normalized distribution of the content of atoms of elements (at. %) in the samples: a) manganese ore from the Bogach deposit; b) manganese ore from the Zhaksylyk deposit; c) manganese ore from Georgia; d) manganese ore from India

Figure 3 present the micrographs with mapping of the distribution of elements on the focused surface of the sample. In all samples there are inclusions of dense crystallites containing silicon. These formations may belong to waste rock particles.

In all micrographs, these formations look like dense monolithic particles with a size of 20-30 microns. In all samples manganese gives a more a less dense background, which indicates a sufficiently high or average content of it in the composition of the raw ore.

The analysis of the obtained micrographs of the samples under study revealed that the dispersive capacity of ore samples from the Bogach deposit (sample 1), Zhaksylyk deposit (sample 2) and the ones produced in India (sample 4) is similar to each other and ranges from one to several tens of microns. At the same time, the dispersive capacity of sample of raw materials produced in Georgia (sample 3) is higher, the enlarged aggregates reach sizes up to 10 microns due to the agglomeration of smaller particles.

Micrographs with mapping of the distribution of elements on the focused surface of the sample provide the opportunity to study highly relief surfaces and obtain information not only about the state of the surface layer but also allows adjusting the image of sub-layers.

Thus, in all investigated samples 1-4 there are inclusions of dense crystallites containing silicon as the main rock-forming component representing dense monolithic conglomerates with a size of 20-30 microns. It should be highlighted that in all samples manganese gives a more or less dense background which corresponds to a high or average content of it in the composition of the raw ore. In particular cases it depends on the source of origin, sample preparation, degree of grinding, moisture content, etc.

On the basis of the obtained elemental analysis data, it was found that in terms of manganese content, the ore of the Bogach deposit ranks second after the raw materials of Georgian origin (Mn 57.98 wt.%) and contains about 43.06 wt.% while the iron content in the same ore is reduced to zero. The lowest content of manganese mineral is found in the poor oxidized ore of the Zhaksylyk deposit which comprises about 22.00 wt. % due to the presence and influence of silicates, alumina, lime and magnesia as well as due to its mineralogical features, it is represented by oxide forms of manganese and iron (5.47 wt.%) and as a result has a dark brown color. Regarding the sample of manganese ore of Indian origin, it should be noted that the maximum manganese content at a single point reaches about 60.00 wt. %. However, when the indicator is averaged over seven repetitions, its amount is almost halved and comprises 35.81 wt. % due to the influence of the host (waste) Rock and oxide compounds. In addition, there was found a significant, in comparison with the rest of the samples, iron content up to 7.63 wt. %.

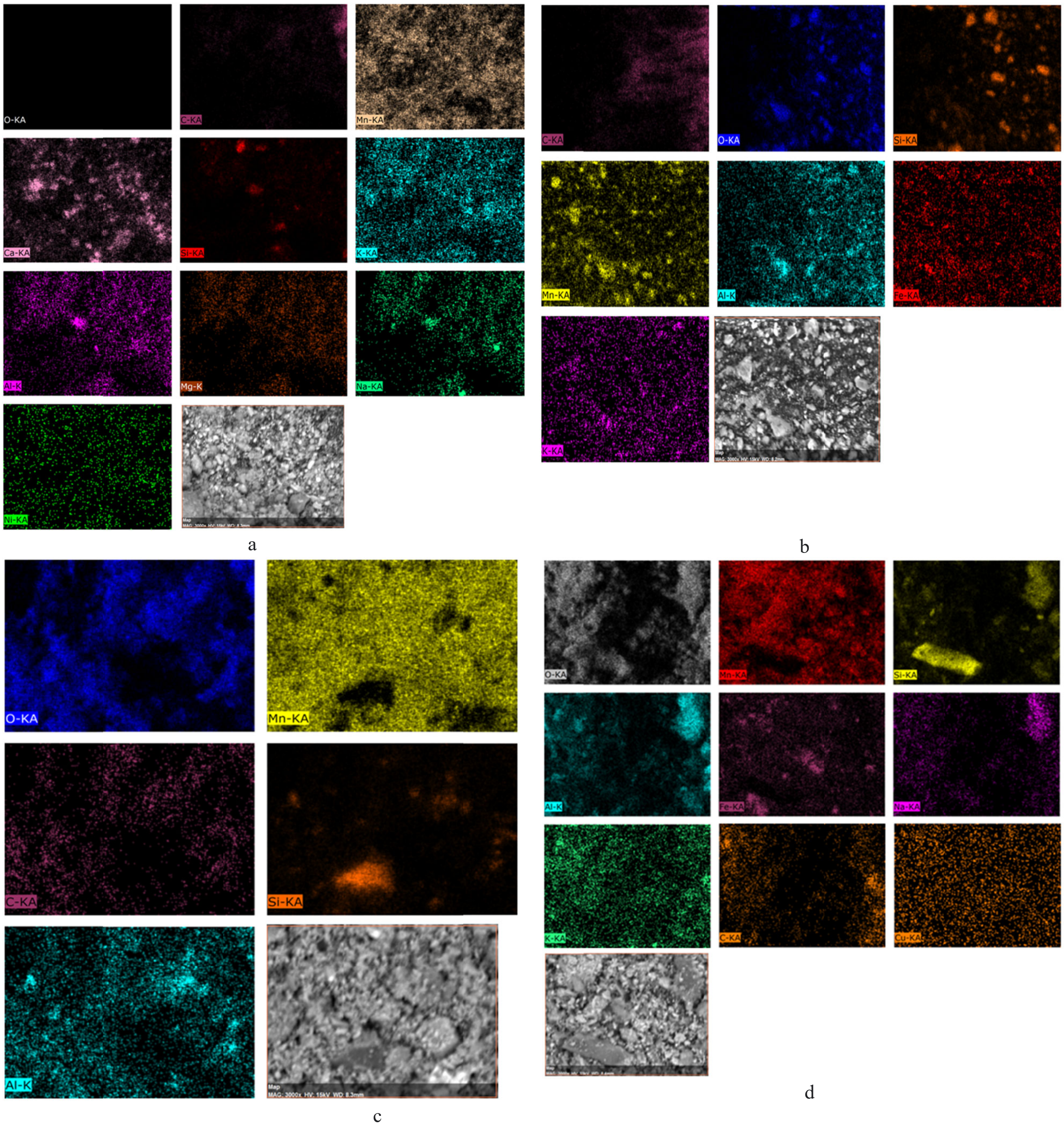

Figure 3 - Micrographs with mapping of the distribution of elements on the focused surface of the samples:
a) manganese ore from the Bogach deposit; b) manganese ore from the Zhaksylyk deposit;
c) manganese ore from Georgia; d) manganese ore from India

Conclusion. The findings obtained do not contradict and are in good agreement with the literature data regarding the fact that Kazakhstani raw materials are classified as medium and low in terms of the manganese mineral content.

Taking into account the quantitative characteristics of the content of manganese, iron, silicon, aluminum, oxygen as well as other trace elements, obtained by the energy dispersive method, in the light of the investigated samples, it should be highlighted that the given study considered mineral raw materials which have virtually uncontrollable average elemental composition. A different picture may appear in the study of technological samples, concentrates or finished products where the influence of the host rock is minimized.

The identity and homogeneity of the morphology of the surface and sub-layers of the samples, their nanoscale characteristics during electronic scanning provide a basis for differentiating the samples according to their component composition, depending on the source of origin.

Based on the totality of the obtained data and results of the analysis, it can be concluded that samples of Kazakhstani raw materials are promising materials and can be used in various processes of technological processing of manganese raw materials, in particular, they provide unlimited prospects for the further synthesis of powder pigments based on trimanganese tetraoxide.

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ТРИМАРГАНЕЦ ТЕТРАКСИД БОЯҒЫШЫ ӨНДІРІСІ ҮШІН ЖЕРГІЛІКТІ ЖӘНЕ ШЕТЕЛДІК ШИКІЗАТТЫ ЭЛЕМЕНТТІК ТАЛДАУ

Аннотация. Тримарганец бояу өндірудің Қазақстандық технологиясын әзірлеу саласындағы ғылыми-зерттеу ізденістерін жалғастыру шеңберінде тетраоксид отандық кен орындарының көзі болып табылатын жаңа наноқұрылымдық материалдарды зерттеуге мүмкіндік беретін талдаудың жаңа әдістері ұсынылды. Алынған нәтижелер қолданбалы мәнге ие, сонымен қатар ішкі және сыртқы нарықта сұранысқа сай бәсекеге қабілетті өнімнің жаңа технологиялық отандық өндірісін құруға себеп болады. Қолда бар әдістемелер марганец минералдарының және сыйымды (бос) жыныстардың біркелкі бөлінбеуіне, жыныс құраушы компоненттердің сандық қатынасына және олардың вариациялық өзгергіштігіне байланысты Қазақстандық марганец кендерінің ерекшелігі мен алуан түрлілігін қамтуға қабілетсіз. Қазақстандық Жақсылық және Богач кен орындарының марганец кендерін энергодисперсиялық талдау әдістері ұсынылды, электрондық микрофотографиялар, үлгілер спектрограммалары, үлгілердегі элементтер атомдарының құрамын нормаланған бөлу диаграммалары, үлгілердің бетінде элементтердің таралуын картаға түсіру микрофотографиялары алынды, сондай-ақ кейбір шетелдік өндірушілердің ұқсас шикізат үлгілерімен салыстырмалы талдау орындалды.

Түйін сөздер: Хаусманнит, пигмент, марганец кені, наноұнтақтар, электрондық микроскопия, энергия дисперсиясын талдау.

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ЭЛЕМЕНТНЫЙ АНАЛИЗ МЕСТНОГО И ЗАРУБЕЖНОГО СЫРЬЯ ДЛЯ ПРОИЗВОДСТВА КРАСИТЕЛЯ ТРИМАРГАНЦА ТЕТРАОКСИД

Аннотация. В рамках продолжения научно-исследовательских изысканий в области разработки казахстанской технологии производства красителя тримарганца тетраоксид предложены новые методы анализа, позволяющие исследовать новые наноструктурные материалы, источником которых являются отечественные месторождения. Полученные результаты имеют прикладное значение и служат фундаментом для построения нового технологичного отечественного производства конкурентноспособного продукта, востребованного на внутреннем и внешнем рынке. Имеющиеся методики не способны охватить специфику и разнообразие казахстанских марганцевых руд, обусловленную неравномерностью распределения марганцевых минералов и вмещающей (пустой) породы, количественным соотношением породообразующих компонентов и их вариационной изменчивостью. Предложены методы энергодисперсионного анализа марганцевых руд казахстанских месторождений Жаксылык и Богач, получены электронные микрофотографии, спектрограммы образцов, диаграммы нормализованного распределения содержания атомов элементов в образцах, микрофотографии картирования распределения элементов на поверхности образцов, а также выполнен сравнительный анализ с сырьевыми образцами-аналогами некоторых зарубежных производителей.

Ключевые слова: хаусманнит, пигмент, марганцевая руда, нанопорошки, электронная микроскопия, энергодисперсионный анализ.

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