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STUDY OF COMPOSITION OF WASTE FROM METALLURGY PRODUCTION AIMED IN USE THEM AS ACTIVE PHASES OF CATALYSTS FOR HYDROCARBON RAW MATERIALS REFINING

Abstract. Wastes from metallurgical production contain a significant amount of valuable elements such as iron, chromium, manganese, titanium, aluminum. The introduction of small and dispersed fractions in the smelting furnace leads to disturbances in the technological regime. Therefore, these wastes are accumulated in various types of storages. To increase the efficiency of the use of resources, it is necessary to utilize these wastes. The main ways of utilization are agglomeration, as well as the addition in building materials as additives. It is proposed to combine these ways with a new approach to utilization. Based on the data on the composition, it is proposed to use waste from ferrous metallurgy in the Pavlodar region as active phases of catalysts for processing hydrocarbon feedstocks. The obtained results showed that the waste: wet-scrubbing sludge and dry gas scrubbing of the Aktyubinsk Ferroalloy Plant, mixed waste from the ash-slime storage of the Aksu ferroalloy plant, red mud and ferrous sand of the Pavlodar aluminum plant contain significant amounts of Fe, Cr, Mn, Al, Ca. This allows us to predict the possibility of creating catalysts using these types of waste. Thus, it is proposed to study a new approach to solving the problem of utilization of waste products of the metallurgical industry with the production of valuable products - catalysts for processing hydrocarbon raw materials.
Keywords: industrial waste, metallurgical production, utilization, secondary resources, catalysts.

Introduction. In present time metallurgy industry produces great amount of waste. Non-ferrous and ferrous metallurgy take up one of the leading positions in amount of emissions by data given in [1]. Their amounts in the total volume of emissions are correspondingly 22.8 and 15%. Significant part of metallurgy industry of Kazakhstan is focused in Pavlodar region a large industrial center. Thus, by the data given in [2], more than 4 million tons of ferrous metallurgy waste and more than 50 million tons of non-ferrous metallurgy waste are accumulated in Pavlodar by 2013. By experts opinion [3] production of one ton of ferrous metal leads to accumulation of 5-17 tons of waste. Dumps and slime storages are often considered as secondary deposit of valuable metallic components by volumes of their reserves. It is noted, that slags of ferrous metal production contain about 15% of metallic and 27% of oxide iron. In the red mud of aluminium production, which is also stored in storages in great amounts, the presence of iron oxides is 45-50% [4]. About half of total mass of iron ore is conversed into waste called “tailings”, which are quartz-ferruginous sand represented by particles of 0.14-0.63 mm in diameter. The wastes are denser in comparison with natural sand because it contains large amounts of iron compounds.

Significant collected amounts of metallurgy wastes require in-depth consideration of opportunities of utilization of them. The involvement of these wastes in industrial processes is one of the most important aspects of the effective use of mineral and energy resources, creation of waste-free and low-waste technologies and improvement of the environment in the area adjacent to the plant [5]. There are two main ways in this process - agglomeration of dispersed materials and return to the smelting process, as well as utilization by introducing as additives in various building materials. Each of these ways has advantages and disadvantages. So, in the work [5] it is pointed, that agglomeration allows the commercial use of dispersed and shredded waste. One of the simplest and most promising methods of agglomeration is briquetting using binding components. This technology makes it possible to obtain solid briquettes from almost any fine-grained waste [6]. Such briquettes are primarily used in the processes of smelting directly ferroalloys. Their use makes it possible to reduce the amount of accumulated waste, and also to improve the quality of the smelting of the product. Such technology was investigated in industrial conditions at the Aktobe ferroalloy plant in 2001. It is shown that the use of chrome ore briquettes led to a reduction in energy consumption in the production of a ton of product from 4196 kWh to 3742.58 kWh. The level of reduction of chromium reached 87.37-90.56% under conditions when 68.9% of briquettes were fed into the furnace. As a result, briquetting allows to reduce the energy and coke consumption, to increase the amount of reduced chromium and to reduce the amount of dust in gases discharged to the gas cleaning system [5].

On the other hand, a number of works show the use of waste from the metallurgical industry for the production of building materials [7-9]. Thus, in [10], the results of using calcium-containing waste of ferrous metallurgy as a diluent and flux and as a clay component, i.e. interlayer clay (shale oil waste), for reducing the drying time of brick raw materials are described. The use of these wastes in the production of ceramic bricks can reduce the consumption of natural raw materials, reduce the amount of industrial waste and reduce the burden on the environment. In addition, this leads to a reduction in the cost of the final product, provides an alternative raw material for the production of ceramic bricks, preserves irreplaceable resources and reduces energy consumption. The results of a study of the chemical and mineral composition, microstructure, and properties of the readily disintegrating aluminothermic slag of ferrochromium are shown in [11]. A fundamentally new high-efficiency technology for non-waste production of $Mg(Al,Cr)_2O_4$ spinel was developed. The resulting slag has binding properties. After the extraction of spinel, its lime-silicate-aluminate component can be used to produce more than ten forms of the product: cement, pigments, building materials and other materials.

At ferroalloy plants, ferromanganese, ferrosilicon, ferrochromium are produced. Industrial products determine the composition of waste. Thus, in slime of ferroalloys production, significant amounts of manganese and chromium are contained in addition to iron [12]. These elements are catalytically active and are included in the active phases of many catalysts [13-17]. Waste of aluminum production besides iron compounds also contains valuable components - aluminum and titanium. These metals are also actively involved in catalytic processes [18-21]. Thus, we see the potential for a third way of utilization of this type of mineral raw materials - by creating catalysts using these industrial wastes as an active phase. The proposed recycling path is the most cost-effective, as it offers the receipt of a new valuable product that is in demand on the market of catalysts for oil refining. In addition, any combination of the above approaches can be combined with a parallel use of the waste from the metallurgical industry to produce catalysts.

In order to determine the potential use of a material as an active phase, it is necessary to conduct a preliminary analysis of the elemental composition of the research object. In this paper, we present data on the elemental composition of waste metallurgical industry in the Pavlodar region (Kazakhstan), obtained by energy dispersive X-ray fluorescence spectroscopy.

Experimental. Investigation of the elemental composition of the obtained samples was performed by the method of energy dispersive X-ray fluorescence spectroscopy on the energy dispersive microanalysis system INCA Energy 450 mounted on a scanning electron microscope JSM 6610 LV ("JOEL", Japan). The device is equipped with the system of energy-dispersive (ED) microanalysis, a system of wave dispersion microanalysis, a system for analyzing the diffraction of backscattered electrons by a reflected electron detector, a secondary electron detector of Everhart-Thornley, a secondary electron detector for a low-vacuum regime and equipment for sample preparation. The spectra were obtained three times with the calculation of the average value.

Samples of wastes were used for the study: the wet gas scrubbing sludge of the Aktyubinsk Ferroalloy Plant (AFZ), the dust of the dry gas cleaning of the AFZ, the production waste from the ash sludge collector of the Aksu Ferroalloys Plant (ALF), red mud and ferruginous sand of the Pavlodar aluminum plant (PAZ).

Results and discussion. Figure 1 shows the standard spectrogram of the samples studied.

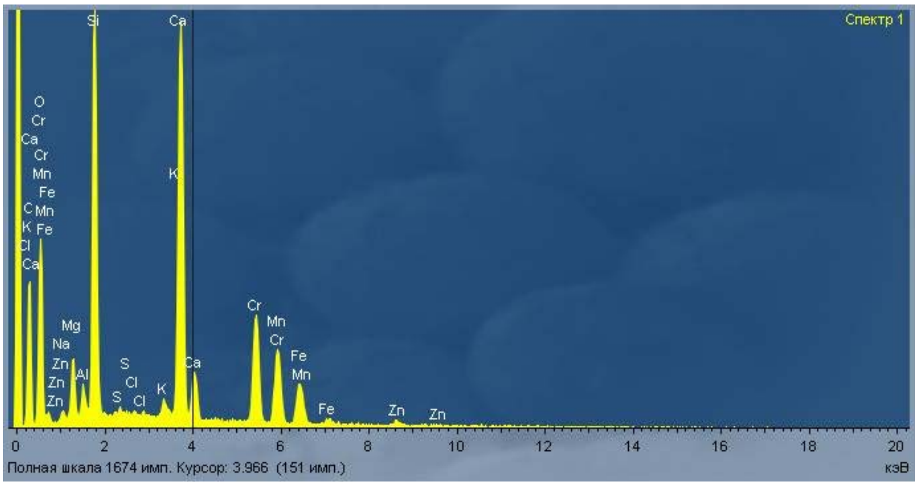

Figure 1 – Spectrogram of a sample of the waste produced by the AZF

Tables 1 and 2 show data on the elemental composition of waste samples of Pavlodar aluminum plant – ferrous sand and red mud. From the obtained data it is seen, that red mud in comparison with ferruginous sand has more homogeneous composition, since the scatter of the content of elements between sample spectra is small. Ferruginous sand, unlike red mud, also contains K and Mn in small amounts. The amount of iron in a sample of ferruginous sand is twice higher than in the composition of red mud. However, the scatter in its content between the spectra indicates a possible lower degree of dispersion of crystals containing Fe. At the same time, red mud contains a high amount of calcium (25-27%). This can affect the course of catalytic reactions, since it is known that in many cases calcium is added to modify the properties of the catalysts [22-24].

Table 1 – The elemental composition of the ferruginous sand sample of the Pavlodar aluminium plant

Spectrum	O	Na	Mg	Al	Si	S	K	Ca	Ti	Mn	Fe	Total
	wt.%											
spectrum 1	43,65	0,66	0,72	8,95	4,95	0,69	0,05	4,19	2,19	0,41	33,53	100
spectrum 2	44,5	0,7	0,11	9,12	18,18	0,26	6,31	1,7	0,6	0,1	18,42	100
spectrum 3	40	0,91	0,23	9,68	3,8	0,54	0,08	2,18	1,76	0,37	40,45	100
Average	42,72	0,76	0,35	9,25	8,98	0,5	2,14	2,69	1,52	0,29	30,8	100

Table 2 – The elemental composition of the red mud sample of the Pavlodar aluminum plant

Spectrum	O	Na	Mg	Al	Si	S	Cl	Ca	Ti	Fe	Total
	wt.%										
spectrum 1	44,77	1,91	0,16	2,45	7,99	0,55	0,09	25,7	1,97	14,39	100
spectrum 2	46,48	1,48	0,19	2,39	7,68	0,22	0,08	27,79	1,47	12,23	100
spectrum 3	45,69	1,6	0,18	2,47	7,99	0,24	0,05	27,05	1,57	13,16	100
Average	45,65	1,66	0,18	2,44	7,89	0,34	0,07	26,85	1,67	13,26	100

Tables 3 and 4 show data on the elemental composition of waste samples of the Aktyubinsk Ferroalloy Plant - dust of dry gas scrubbing and wet gas scrubbing sludge. Unlike previous samples, Ca is present in small amounts both in dust of dry gas scrubbing and in wet gas scrubbing sludge. However, the content of magnesium and chromium increases significantly. But, magnesium is mostly observed in the slime, and chrome in the dust of the gas cleaning system. The amount of iron in these samples is smaller, but the spread between the spectral indices is small, indicating a uniform distribution in the sample mass due to the rather high dispersion of the iron-containing components in these samples. In waste samples of the Aktobe ferroalloy plant, manganese is present in small quantities only in wet gas scrubbing sludge.

Table 3 – The elemental composition of dry dust sample of Aktobe ferroalloy plant

Spectrum	C	O	Mg	Al	Si	S	K	Ca	Ti	Cr	Fe	Ni	Total
	wt.%												
spectrum 1	16,20	30,13	14,84	2,64	6,65	0,17	0,04	0,65	0,19	22,43	5,94	0,12	100,00
spectrum 2	16,88	31,54	16,28	2,68	8,03	0,08	0,08	0,45	0,09	18,67	5,04	0,17	100,00
spectrum 3	15,05	31,36	16,31	2,80	7,95	0,09	0,02	0,77	0,14	19,80	5,59	0,14	100,00
Average	16,05	31,01	15,81	2,71	7,55	0,11	0,05	0,62	0,14	20,30	5,52	0,14	100,00

Table 4 – The elemental composition of a sample of wet gas scrubbing sludge of the Aktyubinsk Ferroalloy Plant

Spectrum	C	O	Mg	Al	Si	S	K	Ca	Cr	Mn	Fe	Zn	Total
	wt.%												
spectrum 1	15,55	31,84	29,35	1,32	7,15	1,22	0,08	0,29	6,95	0,30	5,73	0,22	100,00
spectrum 2	15,42	32,68	29,58	1,21	7,25	1,25	0,11	0,34	6,23	0,21	5,52	0,21	100,00
spectrum 3	14,62	31,94	29,90	1,09	7,35	1,21	0,08	0,34	7,05	0,28	5,85	0,27	100,00
Average	15,20	32,15	29,61	1,21	7,25	1,23	0,09	0,32	6,74	0,26	5,70	0,23	100,00

Table 5 shows the data on the elemental composition of the sample of mixed ash from the ash sludge storage of the Aksu ferroalloy plant. Table 6 shows the averaged data on the content of elements in 16 combined samples taken in different locations of the ash sludge storage of the Aksu ferroalloy plant. It is characteristically that titanium appears in these samples, although in small amounts. Chromium in these samples is also less, which may be due to differences in the output of the two plants products. On the other hand, in these wastes the manganese content is increased to 2%. The iron content here is the lowest and is 1 - 2%. The presence of zinc is becoming greater.

Table 5 – The results of elemental analysis of a sample of mixed waste taken from a local site of an ash sludge storage of the Aksu Ferroalloy Plant

Spectrum	O	Na	Mg	Al	Si	S	Cl	K	Ca	Ti	Cr	Mn	Fe	Zn	Total
	wt.%														
spectrum 1	43,34	0,41	6,67	1,70	36,94	0,31	0,14	1,30	0,81	0,06	2,76	2,04	1,70	1,82	100,00
spectrum 2	42,63	0,25	5,71	1,65	37,25	0,31	0,16	1,29	1,13	0,26	4,07	1,90	2,26	1,13	100,00
spectrum 3	41,29	0,68	3,11	5,37	36,64	0,30	0,14	1,42	0,91	0,19	4,43	2,16	2,04	1,31	100,00
Average	42,42	0,45	5,16	2,91	36,94	0,31	0,15	1,33	0,95	0,17	3,75	2,04	2,00	1,42	100,00

Table 6 – The average elemental composition of the ash sludge storage of the Aksu ferroalloy plant (averaged values for 16 combined samples taken in different locations of the ash sludge storage area)

Element	O	Na	Mg	Al	Si	S	Cl	K	Ca	Ti	Cr	Mn	Fe	Zn	Sn	Total
W, wt.%	46,18	0,54	5,01	1,23	34,16	0,40	0,26	1,41	4,44	0,01	1,73	2,18	1,01	1,38	0,06	100,00

Conclusion. Thus, based on the obtained data, it was established that all the studied samples contain catalytically active components in different amounts, which is determined by the production process. Therefore, they can be used as additives or active phases of catalysts with varying catalyst component ratios. When creating catalysts based on waste from the metallurgical industry, it is recommended to use clays or zeolites as a binder and a structure-forming component. The resulting catalysts must necessarily undergo a high-temperature treatment stage. Since the sources of origin of these components can be contaminated with organic impurities. In addition, the calcination step can lead to crystalline reorganization of the active phase filled with heterogeneous active elements. The received results allow considering dumps, tailing dumps and storages of metallurgical industries as secondary deposits of useful raw materials, suitable for obtaining active phases of non-specific industrial catalysts of hydrocarbon processing processes.

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МЕТАЛЛУРГИЯЛЫҚ ӨНДІРІС ҚАЛДЫҚТАРЫНЫҢ ҚҰРАМЫН ОЛАРДЫ КӨМІРСУТЕКТІ ШИКІЗАТ ӨҢДЕУ КАТАЛИЗАТОРЛАРЫНЫҢ АКТИВТІК ФАЗАСЫ РЕТІНДЕ ПАЙДАЛАНУ МАҚСАТЫНДА ЗЕРТТЕУ

Аннотация. Металлургиялық өндірістегі қалдықтар құрамында едәуір мөлшерде темір, хром, марганец, титан, алюминий тәрізді бағалы элементтер бар. Металлургиялық пешке кіші және дисперсті фракцияларды енгізу технологиялық режимнің бұзылуына әкеледі. Сондықтан, бұл қалдықтар әр түрлі сақтау жерлерінде жинақталады. Ресурстарды кәдеге жаратудың тиімділігін арттыру үшін осы қалдықтарды пайдалану қажет. Кәдеге жаратудың негізгі тәсілдері агломерация болып табылады, сондай-ақ құрылыс материалдарына қоспа ретінде енгізу. Бұл тәсілдерді қайта өңдеуге жаңа тәсілмен біріктіру ұсынылады. Құрамы бойынша мәліметтер негізінде көмірсутек шикізатын өңдеуге арналған катализаторлардың белсенді фазалары ретінде Павлодар облысындағы қара металлургия қалдықтарын пайдалану ұсынылады. Алынған нәтижелер Ақтөбе ферроқорытпа зауытынан газды ылғалды тазалау шламында және газды құрғақ тазалау шаңында, Ақсу ферроқорытпа зауытының күл-қоқыс жинау қондырғысынан аралас қалдықтардында, Павлодар алюминий зауытының қызыл шламы мен темір құмында айтарлықтай Fe, Cr, Mn, Al, Ca бар екендігін көрсетті. Бұл қалдықтардың осы түрлерін қолдану арқылы катализаторларды жасау мүмкіндігін болжауға мүмкіндік береді. Осылайша, көмірсутек шикізатын өңдеуге арналған катализаторлар, құнды өнімдер алу арқылы металлургия өнеркәсібінде қалдықтарды кәдеге жарату мәселесін шешудің жаңа әдісін зерделеу ұсынылады.

Түйін сөздер: өндірістік қалдықтар, металлургиялық өндіріс, қайта өңдеу, қайталама ресурстар, катализаторлар.

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ИССЛЕДОВАНИЕ СОСТАВА ОТХОДОВ МЕТАЛЛУРГИЧЕСКОГО ПРОИЗВОДСТВА С ЦЕЛЬЮ ИХ ИСПОЛЬЗОВАНИЯ В КАЧЕСТВЕ АКТИВНЫХ ФАЗ КАТАЛИЗАТОРОВ ПЕРЕРАБОТКИ УГЛЕВОДОРОДНОГО СЫРЬЯ

Аннотация. Отходы металлургического производства содержат значительное количество ценных элементов, таких как железо, хром, марганец, титан, алюминий. Введение мелких и дисперсных фракций в печи выплавки приводит к нарушениям технологического режима. Поэтому эти отходы накапливаются в хранилищах различного типа. Для повышения эффективности использования ресурсов необходимо утилизировать эти отходы. Основные пути утилизации это агломерация, а также введение в строительные материалы в качестве добавок. Предлагается эти пути комбинировать с новым подходом к утилизации. Основываясь на данных по составу предлагается использовать отходы черной металлургии Павлодарской области в качестве активных фаз катализаторов переработки углеводородного сырья. Полученные результаты показали, что отходы: шлам мокрой газоочистки и пыль сухой газоочистки Актюбинского ферросплавного завода, смешанные отходы с золошламонакопителя Аксуского завода ферросплавов, красный шлам и железистый песок Павлодарского алюминиевого завода содержат в значительных количествах Fe, Cr, Mn, Al, Ca. Это позволяет прогнозировать возможность создания катализаторов с использованием указанных видов отходов. Таким образом, предлагается исследование нового подхода к решению проблемы утилизации отходов металлургической промышленности с получением ценных продуктов – катализаторов переработки углеводородного сырья.

Ключевые слова: промышленные отходы, металлургическое производство, утилизация, вторичные ресурсы, катализаторы.

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