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RESULTS OF PHYSICAL MODELING OF REAGENT OXIDATION OF OILED SCALE OIL UNDER BLAST FURNACE SMELTING CONDITIONS

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Abstract. Using physical modeling methods, the authors studied the processes of hydrocarbon oxidation of oiled mill scale oil, as applied to its loading into a blast furnace inside metal containers measuring 85×83.4 mm, when an oxidizing reagent is included in the composition of the loaded material. The oil's hydrocarbon oxidizer was ammonium nitrate (NH_4NO_3), which undergoes thermal decomposition with the release of oxygen at a temperature of 210 °C corresponding to a temperature range of 200 – 400 °C with the highest intensity of oil vapor release from oiled scale and allows the oxidizer to actively interact with oil vapor. The proportion of the oxidizer changed in the range from 15 to 40 % of the oiled scale mass. With a saltpeter content of 40 % of the loaded oiled scale mass, the degree of oil decomposition in the blast furnace reaches 90 %. Chemical composition of the liquid products in oxidized and non-oxidized oil samples was determined by gas chromatography, and based on the data obtained, the group chemical composition of the samples was evaluated. In the group chemical composition of the products of hydrocarbon oxidation with ammonium nitrate, an increase in the content of oxygen compounds was revealed, compared with the remaining oil in the experiment without using a reagent, from 10.58 to 20.54 %, while the content of saturated hydrocarbons decreased from 35.44 to 29.64 % and the content of unsaturated hydrocarbons from 18.09 to 16.20 %. Since all saltpeters have a high solubility in water, and their aqueous solutions are explosion- and fire-proof, it is recommended to load an oxidizing reagent into a tank with oiled scale of current production, which has a high (from 10 to 30 %) humidity and a liquid consistency.

Keywords: recycling of industrial waste, oiled scale, incomplete oxidation of hydrocarbons, chromatographic analysis, chemical composition of oil, blast furnace

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РЕЗУЛЬТАТЫ ФИЗИЧЕСКОГО МОДЕЛИРОВАНИЯ РЕАГЕНТНОГО ОКИСЛЕНИЯ МАСЛА ЗАМАСЛЕННОЙ ОКАЛИНЫ В УСЛОВИЯХ ДОМЕННОЙ ПЛАВКИ

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Аннотация. Методами физического моделирования авторы изучали процессы окисления углеводородов масла замасленной прокатной окалины применительно к загрузке ее в доменную печь внутри металлических контейнеров размерами 85×83,4 мм при включении в состав загружаемого материала реагента-окислителя. В качестве окислителя углеводородов масла выступала аммиачная селитра (NH_4NO_3), которая подвергается термическому разложению с выделением кислорода при температуре 210 °С, что соответствует температурному интервалу 200 – 400 °С с наибольшей интенсивностью выделения паров масла из замасленной окалины и позволяет окисли-

телю активно взаимодействовать с парами масла. Долю окислителя изменяли в интервале от 15 до 40 % от массы замасленной окалины. При содержании селитры 40 % от массы загружаемой замасленной окалины степень разложения масла в доменной печи достигает 90 %. Методом газовой хроматографии определен химический состав жидких продуктов в окисленной и неокисленной пробах масла, и на основе полученных данных проведена оценка группового химического состава проб. В групповом химическом составе продуктов окисления углеводородов аммиачной селитрой выявлено увеличение содержания кислородных соединений, по сравнению с остатком масла в эксперименте без использования реагента, с 10,58 до 20,54 % при уменьшении содержания предельных углеводородов с 35,44 до 29,64 % и содержания непредельных углеводородов с 18,09 до 16,20 %. Поскольку все селитры имеют высокую растворимость в воде, а их водные растворы являются взрыво- и пожаробезопасными, рекомендуется вводить реагент-окислитель в резервуар с замасленной окалиной текущего производства, имеющей высокую (от 10 до 30 %) влажность и жидкую консистенцию.

Ключевые слова: утилизация техногенных отходов, замасленная окалина, неполное окисление углеводородов, хроматографический анализ, химический состав масла, доменная печь

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INTRODUCTION

The recycling of iron-bearing fine waste generated at metallurgical enterprises, including dust, sludge, and mill scale, is mainly carried out by using these materials as components of the sinter charge [1; 2]. Particular difficulties arise when oiled mill scale is recycled by this route [3]. The accumulation of oiled scale waste in sludge storage facilities creates environmental risks and leads to the loss of a valuable secondary resource with a high iron content [4].

When mill scale comes into contact with oil-containing water, a highly stable mixture is formed: scale + oil + water [5]. Under the influence of the surface adsorption energy of scale particles and the adhesive properties of oil, the oil is firmly retained on the scale surface [6]. As a result, a heterogeneous metallurgical waste consisting of scale, lubricants, and water is generated. This material, with particle sizes from 0 to 10 mm, is referred to oiled scale [7]. The iron content of oiled scale reaches 70 – 80 %, which makes this type of waste a valuable secondary resource [8].

Known methods for recycling oiled mill scale involve preliminary oil removal by chemical [9; 10] or thermal treatment [11; 12], followed by sintering [13 – 15] or briquetting [16]. The limitations of the existing recycling methods lead to a low recovery rate of this technogenic resource and its accumulation in sludge storage facilities.

In this context, the development of a method for recycling oiled scale by direct charging into a blast furnace without preliminary oil removal appears relevant [17; 18]. According to patent [18], oiled scale can be charged into a blast furnace in metal packaging, namely containers that melt at temperatures of at least 1500 °C. However, according to the calculations reported in [19], when lumped oiled mill scale is charged into a 1370 m³ blast furnace, the temperature of the container with oiled

scale at a level of 2 – 5 m below the stockline reaches 230 – 600 °C. This range corresponds to the onset and completion of oil evaporation from oiled scale. The small amount of burden materials located above the level at which oil evaporation begins cannot ensure complete decomposition of oil vapors as they pass through the burden layer. Laboratory experiments [20] showed that more than 25 % of the oil passes into blast furnace dust and sludge from vacuum filtration units (VFUs). This may complicate the operation of the furnace gas-cleaning equipment [21]. One measure aimed at increasing the degree of decomposition of oil contained in oiled mill scale is the use of oxidizing reagents. Therefore, the purpose of this study was to investigate, under laboratory conditions, the oxidation of oil contained in oiled scale by an oxidizing reagent in order to improve the recycling efficiency of this material.

MATERIALS AND METHODS

In this study, a steel reactor was used to reproduce the processes occurring in a metal container during charging into a blast furnace (Fig. 1). The reactor volume was 380 cm³, which allowed 500 g of oiled scale to be charged in the experiment without an oxidizing reagent. After the material had been charged, the reactor was sealed with a lid fitted with an asbestos gasket and a vapor–gas phase outlet. It was then heated in an electric muffle furnace to 700 °C at a rate of 10 °C/min. Water and oil vapors were captured and condensed using a 400-mm-long glass tube condenser cooled with running water.

Oil residues carried with the vapor phase through the glass condenser during the period of most intensive oxidation were additionally deposited in a 2.5-m-long copper coil condenser immersed in a cold-water tank. The experiment involving oil distillation from the reactor without additional oxidizing reagents was taken as the basis experiment. In the experiments involving oxida-



Fig. 1. Laboratory reactor for studying the oxidation processes of oiled rolled scale oil

Рис. 1. Лабораторный реактор для исследования процессов окисления масла замасленной прокатной окалины

tion of oil hydrocarbons by an external oxidizing reagent, ammonium nitrate (NH_4NO_3) was used. At 210 °C, it undergoes thermal decomposition with oxygen release. This temperature falls within the range of 200 – 400 °C, which corresponds to the most intensive release of oil vapors from oiled scale [19]. This enables oxygen, acting as the oxidizing reagent, to actively interact with oil vapors. The proportion of the oxidizing reagent was varied from 15 to 40 % of the mass of oiled scale.

Gas–liquid chromatography was used for qualitative and quantitative analysis of the composition of substances in the obtained samples that were capable of entering the vapor–gas phase and subsequently condensing in the distillate. The analyses were performed using a Chromatec Crystal 5000 gas chromatography system (Fig. 2), and data processing was carried out with Chromatec Analyst 3.0 software.

A 1- μL oil sample was collected with a microsyringe and introduced into the chromatograph through an evaporator heated to 320 °C. The analysis was performed on a ZB-5ms capillary column (5 % phenyl – 95 % dimethylpolysiloxane) with the following parameters: length, 30 m; inner diameter, 0.32 mm; film thickness, 0.25 μm .



Fig. 2. Gas chromatography system “Chromatec Crystal 5000”

Рис. 2. Газохроматографический комплекс «Хроматэк Кристалл 5000»

High-purity argon, grade 99.993 %, was used as the carrier gas in accordance with GOST 10157 – 79. The analysis was conducted in the linear column heating mode to 320 °C at a rate of 6 °C/min. A flame ionization detector was used to record the elution of the mixture components.

For qualitative analysis, linear retention indices of the components in the calibrated mixture of *n*-alkanes were calculated using the following formula [22]:

$$RI_{i, \text{lin}} = 100z + 100 \left(\frac{t_i - t_z}{t_{z+1} - t_z} \right),$$

where $RI_{i, \text{lin}}$ is the linear retention index of the *i*-th component of the mixture; *z* is the number of carbon atoms in the *n*-alkane eluted before the component being determined; t_i is the retention time of the component being determined; t_z is the retention time of the *n*-alkane with *z* carbon atoms; t_{z+1} is the retention time of the *n*-alkane with *z* + 1 carbon atoms, s.

For qualitative identification of individual compounds, the calculated indices were compared with tabulated retention indices for organic compounds from the NIST GC Method and Retention Index Database.

Component contents were quantified using the normalization method.

RESULTS AND DISCUSSION

Oil decomposition degree

The results of the experimental determination of the degree of oil decomposition, calculated from the amount of collected distillate, are presented below:

Ammonium nitrate content relative to the mass of oiled scale, wt. %	Decomposition degree, %
15	5
20	10
30	60
40	75

When ammonium nitrate was added at 15 wt. % of the oiled scale mass, the exothermic effect of the oxidation reaction was insignificant, and the degree of oil decomposition was only 5 %. Starting from a content of 20 wt. %, intense heat release was observed. At an ammonium nitrate content of 40 wt. % relative to the mass of oiled scale, the reaction became distinctly explosive, making quantitative collection of the distillate impossible.

Chemical composition of condensed oil products

Examples of chromatograms of oil samples obtained in the basic experiment and in the experiment with ammo-

nium nitrate added at 40 wt. % relative to the mass of oiled mill scale are shown in Fig. 3. The results of qualitative and quantitative analysis of the sample whose chromatogram is shown in Fig. 3, *a* are presented in Table 1.

As shown in Table 1 and Fig. 3, *a*, the characteristic “comb-like” pattern corresponds to regularly spaced peaks of linear alkanes, which exhibit the highest intensities on the chromatogram.

During oxidation of the oil with the external oxidizing reagent, ammonium nitrate, the number of individual compounds identified in the sample increased from 81 to 115. This increase was mainly associated with the formation of relatively light oxidation and oxidative decomposition products, which are recorded in the left-hand part of the chromatogram in Fig. 3, *b*. These compounds included acetone, propanal, propan-1-ol, butan-2-one, formic acid esters, and other oxygen-containing organic compounds. In total, 151 individual chemical compounds were identified in the analyzed oil samples by chromatographic analysis. Based on the identification of individual compounds, the group composition of the oils was evaluated. These results are presented in Table 2.

Analysis of the data in Table 2 shows that the oil recovered in the basic experiment was dominated by saturated,

unsaturated, and aromatic hydrocarbons of aliphatic and alicyclic structure, whereas the content of oxygen-containing compounds with various functional groups was relatively low. The formation of these oxygen-containing compounds can be explained by partial oxidation of oil hydrocarbons during rolling, when the oil comes into contact with the hot metal surface, as well as during subsequent storage of oiled scale in open-air dumps.

The use of ammonium nitrate reduced the total peak area of saturated hydrocarbons from 35.44 to 29.64 % (by 16.37 rel. %) and that of unsaturated hydrocarbons from 18.09 to 16.20 % (by 10.45 rel. %), whereas the total content of oxygen-containing compounds increased from 10.58 to 20.54 % (by 94.14 rel. %). The alcohol content increased from 4.28 to 5.65 % (by 32 rel. %). Overall, the use of ammonium nitrate as an oxidizing reagent resulted in a more complex and more highly oxidized sample composition, with an increased content of oxygen-containing compounds, mainly more deeply oxidized products such as carboxylic acids and esters.

Because nitrates are highly soluble in water and their aqueous solutions are fire- and explosion-safe, the oxidizing reagent can be introduced into the storage tank containing oiled scale from the current production stream,

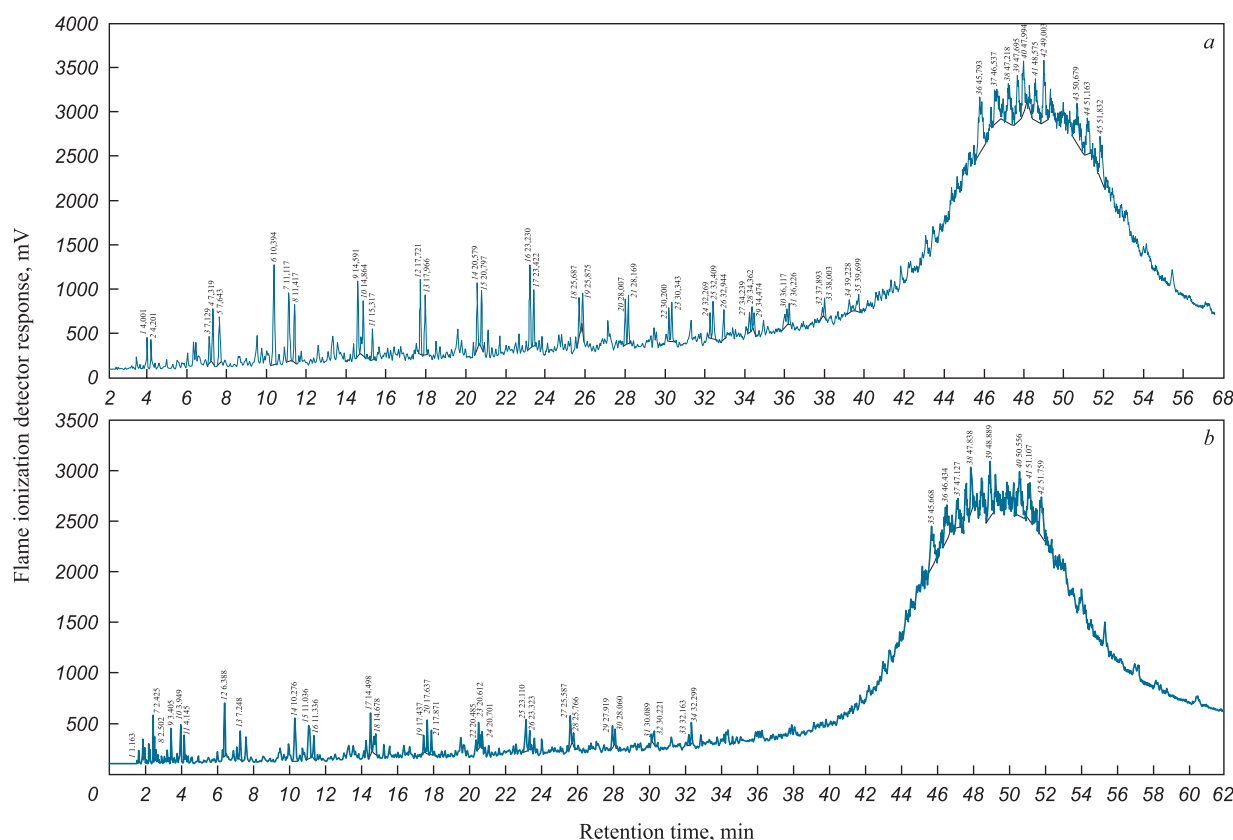


Fig. 3. Chromatograms of oils during the experiment without reagent addition (*a*) and when using ammonium nitrate as an oxidizing reagent (*b*)

Рис. 3. Хроматограммы масел при проведении эксперимента без добавления реагента (*a*) и при использовании в качестве реагента-окислителя аммиачной селитры (*b*)

Table 1. Chemical composition of oiled scale oil (in the absence of an oxidizing reagent; basic experiment)

Таблица 1. Химический состав масла замасленной окалины (в отсутствие реагента-окислителя; базовый эксперимент)

No.	Oil component	RI*	% (wt.)	No.	Oil component	RI	% (wt.)
1	1-heptene	680	0.44	42	tetradecane	1396	1.99
2	n-heptane	692	0.43	43	octylbenzene	1450	1.50
3	3-methylbutan-1-ol	719	0.23	44	1-pentadecene	1488	1.22
4	ethylcyclopentane	750	0.36	45	pentadecane	1495	1.29
5	5-methylhept-1-ene	758	0.53	46	5-methylpentadecane	1546	1.12
6	toluene	762	0.48	47	2,6,10-trimethyltetradecane	1553	0.65
7	4-methylheptane	765	0.58	48	dodecanoic acid	1559	0.60
8	1,4-dimethylcyclohexane	782	0.59	49	tridecan-2-one	1581	0.49
9	1-octene	787	1.22	50	tridecan-1-ol	1588	0.93
10	n-octane	797	1.29	51	hexadecane	1595	0.98
11	1,2,4-trimethylcyclohexane	847	1.11	52	benzophenone	1640	0.81
12	6-methyloct-1-ene	853	0.52	53	tridecanoic acid	1665	0.59
13	3-methyloctane	870	2.57	54	pentadecan-2-one	1682	0.52
14	4-methylpentan-1-ol	876	0.28	55	1-heptadecene	1688	0.73
15	2,2,4-trimethylheptane	883	0.52	56	heptadecane	1695	1.45
16	1-nonene	889	1.96	57	8-heptadecene	1722	0.77
17	nonane	897	1.24	58	2,6,10-trimethylhexadecane	1779	0.44
18	propylcyclohexane	931	0.68	59	hexadecan-2-one	1788	0.61
19	4-ethyloctane	951	0.98	60	1-octadecene	1794	0.63
20	5-methylnonane	959	0.65	61	octadecane	1800	0.49
21	4-decene	983	0.41	62	3-ethylbenzophenone	1826	0.48
22	1-decene	989	1.65	63	4,8,12-trimethylheptadecane	1837	0.31
23	decane	997	1.79	64	cyclohexadecane	1882	0.56
24	1-methylpropylbenzene	1011	0.60	65	1-nonadecene	1888	0.39
25	1-undecene	1089	1.66	66	nonadecane	1894	0.52
26	undecane	1096	1.42	67	1-eicosene	1988	0.53
27	3-rhexylcyclopentene	1115	0.41	68	eicosane	1994	0.54
28	pentylcyclohexane	1122	0.32	69	6-methyleicosane	2066	0.50
29	5-ethyldecane	1154	0.85	70	heneicosane	2094	0.69
30	nonan-1-ol	1161	0.42	71	4-ethyleicosane	2148	0.53
31	1-dodecene	1189	1.39	72	terphenyl	2158	0.55
32	dodecane	1196	1.20	73	2-methylheneicosane	2169	0.82
33	2,5-dimethylundecane	1209	0.64	74	docosane	2195	0.49
34	undecan-6-one	1231	0.65	75	eicosan-2-one	2206	0.45
35	2-methyldodecane	1267	0.41	76	2,6,10,14,19-pentamethyleicosane	2226	0.73
36	1-tridecene	1288	2.03	77	eicosan-1-ol	2252	0.68
37	tridecane	1296	1.32	78	heneicosan-2-ol	2307	1.73
38	2-tridecene	1312	0.41	79	methyl eicosanoate	2331	1.09
39	biphenyl	1346	0.47	80	tetracosane	2394	2.07
40	4-cyclohexyldecane	1352	0.34	81	benzo[a]pyrene	n.d.	0.001
41	1-tetradecene	1388	1.20				

* RI – linear retention index.

Table 2. Group chemical composition of the studied oils

Таблица 2. Групповой химический состав исследуемых масел

Class	Content, %	
	basic experiment	with addition of oxidizing reagent
Hydrocarbons:		
saturated	35.44	29.64
unsaturated	18.09	16.20
aromatic	3.61	3.63
Alcohols	4.28	5.65
Aldehydes	–	0.34
Ketones	4.01	4.49
Carboxylic acids	1.19	3.37
Esters	1.09	6.68
Unidentified compounds (content of each component less than 0.5 wt. %)	32.29	30.00
Total	100.00	100.00

which has a high moisture content (10 – 30 %) and a slurry-like consistency. Under these conditions, only minimal mixing is required to ensure uniform nitrate distribution. The resulting oiled scale–nitrate slurry can be stored in steel or polypropylene tanks, from which individual metal containers are subsequently filled for charging into the blast furnace.

These findings support a method for recycling oiled scale in metal containers using ammonium nitrate at 30 – 40 wt. % of the oiled scale charge, which ensures 60 – 75 % oil decomposition. Considering that up to 75 % of oil vapors decompose while passing through the burden layer above the level where oil evaporation begins [20], the overall degree of oil decomposition and oxidation reaches 90 %.

CONCLUSIONS

Under laboratory conditions, the efficiency of oxidation of oil contained in oiled mill scale was investigated using ammonium nitrate as the oxidizing reagent. An ammonium nitrate content of 40 wt. % of the charged oiled mill scale provided the highest degree of oil decomposition, reaching 75 %. During oil oxidation with ammonium nitrate, the residual unburned oil showed an increase in the content of oxygen-containing compounds from 10.58 to 20.54 %, while the total peak area of saturated hydrocarbons decreased from 35.44 to 29.64 % and that of unsaturated hydrocarbons from 18.09 to 16.20 %, compared with the residual oil obtained in the basic experiment.

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