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PRODUCTION OF REFINING ALUMINA-CONTAINING FLUXES BY SINTERING FROM TECHNOGENIC RAW MATERIALS

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Abstract. Modern Russian steelmaking plants use predominantly alumina-containing materials for liquefying lime in a ladle-furnace unit, which replaced fluorspar. Alumina-containing materials currently available on the market cannot be used directly in steelmaking without preliminary preparation (refining, heat treatment or briquetting), or are simply unsuitable for ladle processing of steel. This work describes laboratory studies on the production of refining alumina-containing fluxes by sintering in units such as machines for pellets firing or producing agglomerate (in the temperature range of 1200 – 1500 °C) from clean metallurgical waste (fine dust from the production of alumina and burnt lime), meeting the requirements of steelmaking plants by chemical composition and mechanical properties. A comparison was made of sintering technological schemes with the introduction of hydrated lime and a mixture of hydrated lime and calcium carbonate in a 1:1 ratio as a source of CaO. We determined that the maximum permissible CaO content in sintered briquettes when using a mixture of hydrated lime and calcium carbonate in the charge, which does not lead to hydration destruction in air, is in the range of 2.3 – 3.6 %, depending on the holding temperature. The maximum permissible content of Al_2O_3 in sintered briquettes when using hydrated lime in the charge, which does not lead to hydration destruction in air, is in the range of 9.5 – 31.7 %, depending on the holding temperature. In existing fuel units it is possible to obtain fluxes by sintering only when using hydrated lime as a source of CaO, because adding calcium carbonate to the charge (9 – 22 %) requires an increase in holding temperature (above 1500 °C) or holding time (more than 25 min).

Keywords: ladle-furnace, alumina dust, refining fluxes, alumina-containing materials, calcium aluminates, sintering, hydration destruction

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ПОЛУЧЕНИЕ РАФИНИРОВОЧНЫХ ГЛИНОЗЕМСОДЕРЖАЩИХ ФЛЮСОВ МЕТОДОМ СПЕКАНИЯ ИЗ ТЕХНОГЕННОГО СЫРЬЯ

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Аннотация. Современные сталеплавильные предприятия России для разжижения извести на агрегате ковш-печь применяют преимущественно глиноземсодержащие материалы, которые пришли взамен плавиковому шпату. Доступные сейчас на рынке глиноземсодержащие материалы не могут быть использованы напрямую в сталеплавильном производстве без предварительной подготовки (рафинирования, термообработки или брикетирования), либо просто непригодны для ковшевой обработки стали. В данной работе описаны лабораторные исследования по получению рафинировочных глиноземсодержащих флюсов методом спекания в агрегатах по типу машин для обжига окатышей или производства агломерата (в температурном интервале 1200 – 1500 °C) из чистых отходов металлургического производства (мелкодисперсная пыль производства глинозема и обожженной извести), отвечающих требованиям сталеплавильных предприятий по химическому составу и механическим свойствам. Проведено сравнение технологических схем спекания с введением в качестве источника CaO гидратированной извести и смеси гидратированной извести и карбоната кальция в соотношении 1:1. Предельно допустимое содержание CaO в спеченных брикетах при использовании в шихте смеси гидратированной извести и карбоната кальция, не приводящее к гидратационному разрушению на воздухе, находится в диапазоне 2,3 – 3,6 % в зависимости от температуры выдержки. Предельно допустимое содержание Al_2O_3 в спеченных брикетах при использовании в шихте гидратированной извести, не приводящее к гидратационному разрушению на воздухе, находится в диапазоне 9,5 – 31,7 % в зависимости от температуры выдержки. В существующих топливных агрегатах возможно получить флюсы методом спекания только при использовании в качестве источника CaO гидратированной извести, так как добавление карбоната кальция в шихту (9 – 22 %) требует увеличения температуры выдержки (выше 1500 °C) или ее продолжительности (более 25 мин).

Ключевые слова: агрегат ковш-печь, глиноземная пыль, рафинировочные флюсы, глиноземсодержащие материалы, алюминаты кальция, спекание, гидратационное разрушение

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INTRODUCTION

The fluxes and slag-forming materials used in metallurgy exert significant influence on production technology, as well as the chemical composition and quality of smelted steel. The quantity of detrimental impurities (such as sulfur, phosphorus, and gases like oxygen, hydrogen, and nitrogen) is contingent upon the type of additives employed, which constitutes a fundamental determinant in achieving high-quality steel.

In the ladle-furnace unit (LFU) utilized for steel processing, lime serves as the primary slag-forming material, boasting a melting point exceeding 2500 °C. To lower the slag's melting point during extraction, flux is introduced alongside lime to induce liquefaction. In the past, fluorspar was widely employed for slag liquefaction in LFUs; however, its usage has recently been minimized or entirely phased out due to various adverse factors including its short-term efficacy, diminishment of lining resistance in the slag belt zone, and environmental concerns [1 – 3].

Alumina-containing fluxes, surpassing fluorspar in several aspects, emerge as a promising alternative to fluorine-based materials. These alumina-containing materials can be employed either independently or in conjunction with fluorspar, even for steel grades designated as "aluminum-free" [4]. Notably, aluminothermal slags derived from ferrovanadium and aluminothermal chromium production have gained significant traction as alumina-containing fluxes. However, a major drawback lies in their market scarcity due to the limited production of ferroalloys via aluminothermal reduction. Additionally, other available alumina-containing materials often require preliminary treatment (such as refining, heat treatment, or briquetting) before they can be utilized in steelmaking, or they may prove unsuitable for the ladle processing of steel [5 – 7].

When procuring fluxes, steelmaking plants impose criteria concerning both chemical composition and mechanical properties. Fluxes are expected to be supplied in the form of lumps or briquettes, with overall dimensions ranging from 10 to 50 mm. The fine fraction (0 – 5 mm) should not exceed 10 % of the total mass, while moisture content is to be kept below 1 % in summer and up to 6.5 % in winter. Consumers may also specify requirements regarding the strength properties of briquettes/lumps. A summarized outline of the chemical composition requirements for alumina-containing fluxes is presented in Table 1.

The required quantity of alumina-containing flux for processing in the LFU is determined through the balance equation of aluminum utilization in ladle processing:

$$\mathrm{Al_{sec}} = \mathrm{Al_{res}} + \mathrm{Al_{deox}} + \mathrm{Al_{air}} + \mathrm{Al_{sl}} + \mathrm{Al_{evap}},$$

where $\mathrm{Al_{res}}$ is residual aluminum, $\mathrm{Al_{deox}}$ is deoxidation aluminum, $\mathrm{Al_{air}}$ is air oxygen-oxidized aluminum, $\mathrm{Al_{sl}}$ is furnace slag-oxidized aluminum, and $\mathrm{Al_{evap}}$ is evaporated aluminum.

A portion of Al_2O_3 needed for lime liquefaction is generated through the interaction of aluminum with dissolved oxygen in steel, while another portion results from combustion on the slag surface and subsequent deoxidation. The remaining portion is introduced in the form of flux to facilitate free-running slag. Despite its technological efficiency, aluminum is deemed economically impractical due to its high cost as a source of Al_2O_3. According to the balance equation, it's estimated that aluminum consumption is distributed as follows: $\mathrm{Al_{res}}$ – 15 %; $\mathrm{Al_{deox}}$ – 18 %; $\mathrm{Al_{air}}$ – 38 %; $\mathrm{Al_{sl}}$ – 28 %; $\mathrm{Al_{evap}}$ – 1 %.

Given the current industrial environmental constraints and stringent steel quality requirements, developing alumina-containing flux production technology in an environmentally sustainable and cost-effective manner emerges as an urgent challenge.

MATERIALS AND METHODS

An essential consideration in flux production via sintering is the meticulous selection of charge materials. Firstly, these materials must not introduce harmful impurities that resist removal during heat treatment and could potentially contaminate the processed steel. Secondly, they should be relatively amenable to briquetting, as sintering technology involves the heat treatment of lump material. Thirdly, availability on the market is crucial.

The recycling of metallurgical waste to yield marketable products has gained significant traction in recent times. Among such wastes is the dust collected from the filters of roasting furnaces. This paper focuses on the processing of dust originating from alumina calcination and limestone roasting furnaces.

Alumina calcination involves the dehydration of aluminum hydroxide at elevated temperatures. Aluminothermal slag, utilized in rotary tube furnaces or fluidized-bed furnaces at temperatures reaching up to 1200 °C, represents the final stage in the Al_2O_3 production process chain. Inevitably, during the calcination processes in

Table 1

Consolidated requirements for the chemical composition of alumina-containing fluxes imposed by metallurgical enterprises in Russia

Таблица 1. **Сводные требования к химическому составу глиноземсодержащих флюсов, предъявляемые металлургическими предприятиями России**

Element	Content in steelmaking flux, %		Features of use in the LFU
	min	max	
min, %			
Al_2O_3	50	80	Reduces the melting point of CaO
max, %			
CaO	20	30	Forms a low-melt eutectic with Al_2O_3
MgO	8	20	
Fe_2O_3	2	6	Worsens the desulfiding process Limitation on the use of corundum production slags and alumina-containing ores without refining
MnO	1.5	2.0	
SiO_2	3	15	
$P_{tot} + S_{tot}$	0.02	0.30	Transfer to metal and require additional refining
Cr_2O_3	2	10	Form carbides, worsen further processing Restriction on the use of aluminothermal slags from ferrotitanium and chromium metal production
TiO_2	1	5	
$V_2O_5 + Nb_2O_5$	1	1	Recovered and converted to metal Restriction on the use of aluminothermal slags from ferrovanadium and ferroniobium production
$Na_2O + K_2O$	1	8	Restriction on the use of slag from secondary aluminum production

various units, nanoscale dust is generated. Research cited in [8] indicates that the size of dust nanoparticles falls within the range of 50 – 300 nm. Approximately 14 % of the fine alumina dust is carried away from the furnace by flue gas during calcination, which is then directed to multicyclones and electric filters [9]. However, this dust, containing nanoparticles, is unsuitable for use in the classical technology of electrolytic decomposition of Al_2O_3 due to its hygroscopic nature, leading to excessive hydrogen content in aluminum metal. Nonetheless, this material finds applicability in the iron industry as a source of Al_2O_3 in steelmaking fluxes.

When selecting a source of CaO for flux production via sintering, one should consider a chain of chemical transformations: $CaCO_3 \rightarrow CaO \rightarrow Ca(OH)_2 \rightarrow CaCO_3$.

Fine-dispersed carbonate rocks, burnt, or slaked lime can serve as a source of CaO, each carrying its own set of advantages and disadvantages. Limestone ($CaCO_3$) necessitates no preliminary preparation before briquetting. However, during sintering, its decomposition into CaO and CO_2 absorbs heat (178 kJ/mol). Burnt lime (CaO), on the other hand, doesn't undergo significant mass loss during sintering due to the absence of crystal hydrate moisture. Yet, its slaking during briquetting releases heat (65 kJ/mol), which is not conducive to the process. Slaked lime ($Ca(OH)_2$) offers several advantages over the aforementioned materials:

– it requires no preliminary preparation and can hydrate in air during storage;

– it facilitates convenient briquetting as it doesn't generate heat when interacting with water;

– heat absorption during its decomposition into CaO and H_2O (65 kJ/mol) during sintering is almost 3 times less than the heat absorbed during the decomposition of $CaCO_3$.

In industrial settings, burnt lime is typically obtained by calcinating carbonate rocks in shaft or rotary furnaces at temperatures ranging from 1000 to 1250 °C [10]. Analogous to alumina calcination, this process generates micro-sized dust particles (6 – 60 μm), which are typically collected in bag filters or electric filters [11]. While this dust shares a similar composition with the burnt material, its smaller particle size renders it more prone to rapid hydration in the air during storage. Nonetheless, this fine dust can also serve as raw material for the production of sintered alumina-lime fluxes.

An integral aspect of working with dispersed materials involves their preparation for heat treatment. In this study, cold briquetting was employed. Drawing from successful experiments involving porous alumina-containing materials [12], a binder based on polyacrylamide was utilized. This binder exhibits low consumption (0.6 % of the mass of the briquetted raw material) and is completely removed at sintering temperatures.

The production technology of alumina-lime fluxes, designed to withstand hydration and subsequent destruction during sintering, from pure components encompasses the following operations:

– production of briquettes from pure components;

– heating the material to the holding temperature;

– maintaining a constant temperature during holding;

– cooling in ambient air.

In laboratory settings, sintering was conducted in a resistance furnace equipped with a graphite heater. The technologies employed in ore pellet roasting and agglomerate production served as the foundation for flux production. The temperature range for laboratory experiments was selected in accordance with the existing process characteristics of fuel units. Literature data indicates that the maximum roasting temperature for iron ore pellets is around 1400 °C [13 – 17], while for chromite pellets, it ranges from 1400 to 1500 °C [18 – 22]. Hence, the temperature holding interval ranged from 1200 to 1500 °C. During laboratory experiments, the average heating rate was maintained at 20 °C, and the duration of holding at a constant temperature ranged from 15 to 25 min. Following heat treatment, the sintered briquettes were cooled in ambient air, after which their mass and geometric parameters were measured. Subsequently, the briquettes were stored in air, at a temperature of 21 °C and a relative humidity of 50 %, to monitor any changes in mass. Weighing and recording the mass change of the briquettes were conducted once every seven days until the mass stabilized.

Two series of experiments were conducted utilizing different sources of CaO: a mixture of hydrated lime and calcium carbonate in a 1:1 ratio (series *1*) and hydrated lime along (series *2*). The proportion of Al_2O_3 in the briquettes before sintering varied from 50 to 80 % in both cases, with increment of 5 %. The chemical composition of the raw materials is provided in Table 2.

RESULTS AND DISCUSSION

After the briquettes had cooled in air, an external evaluation of their post-sintering state was conducted. Based on their appearance, the heat-treated briquettes were categorized into four conventional groups (Fig. 1).

The STATISTICA software package was employed to process the results and identify factors influencing the hydration destruction of sintered briquettes. The dependent variable was the percentage of mass gain during storage in air, while the independent variables included the temperature of sintering (°C), sintering time (min), composition of initial briquettes (%), change in density and volume of briquettes during sintering (%), and

Table 2

Chemical composition of raw materials

Таблица 2. **Химический состав исходных материалов**

Material	Al_2O_3 dust			CaO dust				
Element	Al_2O_3	Fe_2O_3	$Na_2O + K_2O$	CaO	MgO	SiO_2	P	S
Content, %	99.53	0.01	0.45	98.76	0.50	0.70	0.01	0.03

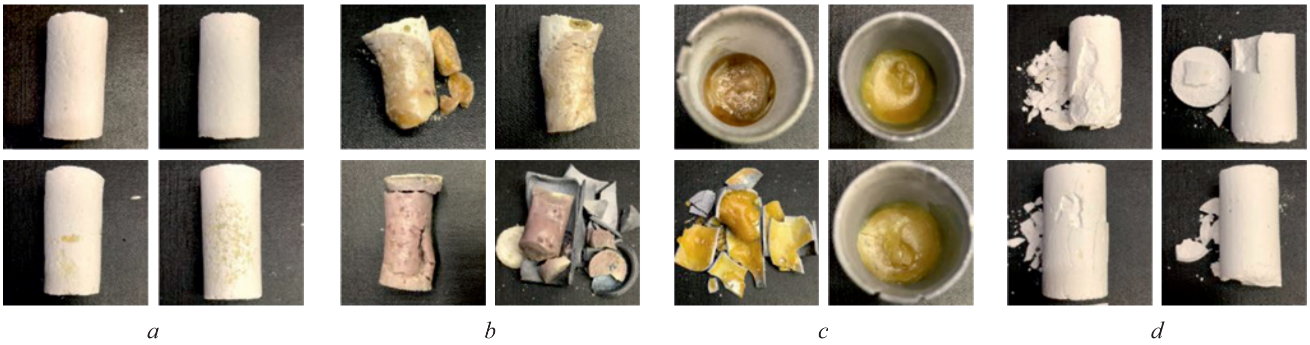
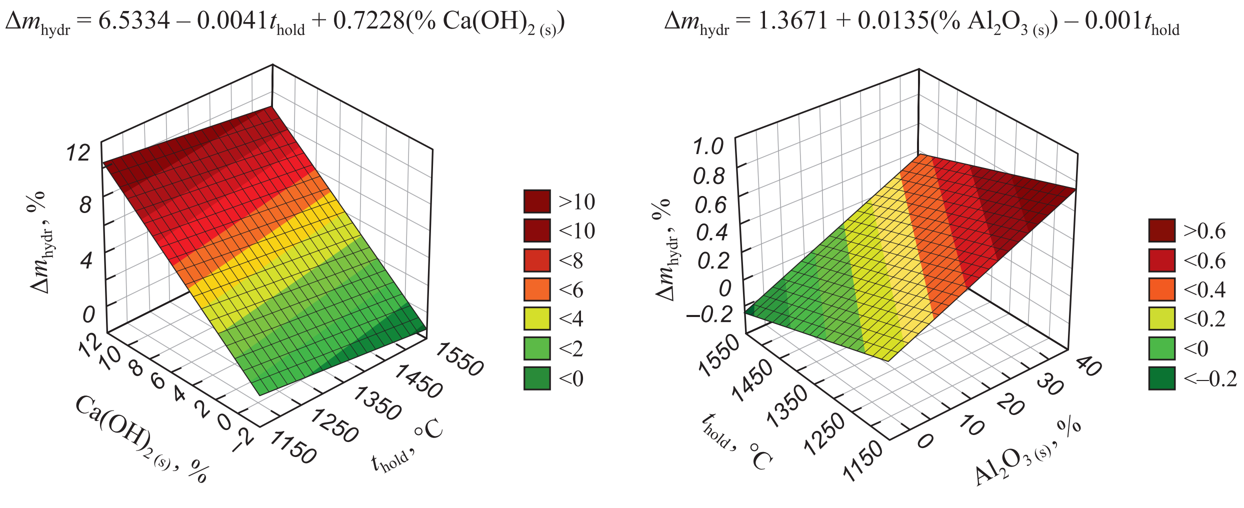


Fig. 1. Appearance of heat-treated briquettes:
a – sintered; *b* – semi-melted; *c* – melted; *d* – collapsed/cracked

Рис. 1. Внешний вид термообработанных брикетов:
a – спеченные; *b* – оплавленные; *c* – расплавившиеся; *d* – разрушившиеся/треснувшие



a *b*

Fig. 2. Influence of holding temperature, $Ca(OH)_2$ and Al_2O_3 content in sintered materials on change in mass:
a – when using a mixture of $CaCO_3$ and $Ca(OH)_2$; *b* – when using $Ca(OH)_2$

Рис. 2. Влияние температуры выдержи, содержания $Ca(OH)_2$ и Al_2O_3 в спеченных материалах на изменение массы:
а – при использовании смеси $CaCO_3$ и $Ca(OH)_2$; *b* – при использовании $Ca(OH)_2$

phase composition of sintered briquettes after holding in air (%). Fig. 2 illustrates the primary factors influencing the hydration destruction of briquettes for the two series of experiments.

In addition to quantitative evaluation, qualitative assessment of the visual state of briquettes during air storage was conducted. For experiments in series *1*, the initial signs of hydration degradation, accompanied by mass gain, were observed as early as the 7th day of air storage, with the mass gain concluding on the 56th day of observation. For the experiments in series 2, the first indicators of hydration degradation were observed only on the 28th day of air storage, with the mass gain ceasing on the 100th day of observation. The results of *X*-ray structural analysis of sintered briquettes that remained intact during air storage are presented in Table 3.

The results of the comprehensive evaluation by regression equations, determining the maximum permissible content of free CaO and Al_2O_3 in sintered briquettes for two laboratory series, are presented in Table 4.

It has been established that in series *1*, where the process of calcium aluminate formation coincides with the decomposition of calcium carbonate and calcium hydroxide, it is necessary to elevate the temperature

Table 3

X-ray structural analysis of sintered briquettes not collapsed during storage in air

Таблица 3. **Рентгеноструктурный анализ спеченных брикетов, не разрушившихся при хранении на воздухе**

Series number	Share of Al_2O_3 in briquette before sintering, %	Holding temperature, °C	Holding time, min	Al_2O_3, %	$CaO{\cdot}Al_2O_3$, %	$12CaO{\cdot}7Al_2O_3$, %	$CaO{\cdot}2Al_2O_3$, %	$3CaO{\cdot}Al_2O_3$, %	$CaO{\cdot}6Al_2O_3$, %
1	50	1500	15	0	9.10	90.90	0	0	0
1	75	1500	15	22.90	25.10	0	38.20	0	13.80
2	50	1200	15	0	0	78.30	0	21.70	0
2	50	1200	25	0	0	65.90	0	34.10	0
2	70	1400	20	14.00	53.40	15,30	16.10	1.20	0
2	55	1400	20	4.40	3.40	92.30	0	0	0
2	50	1400	20	0	2.50	97.50	0	0	0
2	50	1500	25	0	0	86.30	0	13.70	0
2	80	1500	25	26.90	25.90	0	40.60	6.50	0

Table 4

Ultimate content of CaO and Al_2O_3 in sintered briquettes

Таблица 4. **Предельное содержание CaO и Al_2O_3 в спеченных брикетах**

Series number	Limit content of elements in briquette, %	Holding temperature, °C						
		1200	1250	1300	1350	1400	1450	1500
1	CaO ($Ca(OH)_2$)	2.3 (3.0)	2.5 (3.3)	2.6 (3.5)	2.9 (3.8)	3.1 (4.1)	3.6 (4.7)	3.6 (4.7)
2	Al_2O_3	9.5	13.2	16.9	20.6	24.3	28.0	31.7

(>1500 °C) and/or extend the holding time (>25 min) to ensure the completion of all structural transformations. Conversely, series *2* demonstrates that hydration-resistant materials can be produced at relatively low temperatures (starting from 1200 °C) within the specified time interval (15 – 25 min).

Conclusions

The maximum allowable content of CaO in sintered briquettes, which prevents the destruction of the sintered material during air storage (in the case of using a mixture of hydrated lime and calcium carbonate as a source of CaO), falls within the range of 2.3 to 3.6 %, depending on the holding temperature during sintering, corresponding to a mass gain of 3.8 %.

The maximum allowable content of Al_2O_3 in sintered briquettes, which avoids the destruction of the sintered material during air storage (in the case of using hydrated lime as a source of CaO), ranges from 9.5 to 31.7 %, depending on the holding temperature during sintering, corresponding to a mass gain of 0.3 %.

Given the existing fuel units, fluxes can only be produced when hydrated lime (series *2*) is utilized as a source of CaO. This is because when a mixture of hydrated lime combined with calcium carbonate (series *1*) is used as a source of CaO, a holding temperature exceeding 1500 °C is required, which is unattainable with the existing fuel units, or by extending the holding time to over 25 min.

The most favorable source of CaO for flux production via the sintering method is the hydrated dust from limestone roasting furnaces. This is attributed to the fact that during sintering, its heat absorption is three times lower than that of limestone.

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В. В. Аксенова – планирование и проведение экспериментов, определение химического состава исходных материалов методом волнодисперсионной рентгенофлуоресцентной спектрометрии, обработка полученных экспериментальных данных, подготовка текста статьи.

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Contribution of the Authors

V. V. Aksenova – planning and conducting experiments, determination of chemical composition of the starting materials by wave-dispersive X-ray fluorescence spectrometry, processing the experimental data obtained, writing the text.

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