Metallurgical processing of converter slag

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ABSTRACT

Converter slurries at modern metallurgical plants represent a significant part of metal-containing industrial waste with a high concentration of iron. Currently, there is a problem of their utilization and use as raw materials for metallurgy. The purpose of this work is to study the processes of briquetting and recovery of briquetted products, based on a mixture of converter slurries of gas purification and converter slags. When performing experimental studies on the preparation of sludge briquettes from a mixture of converter sludge of gas purification and converter slag, their metallization and reduction melting in laboratory conditions, the optimal composition of the components of the mixture of converter slag and gas purification sludge was determined by the percentage of iron, which is appropriate for use as a raw material for steel smelting. Experimental studies on the preparation of sludge-coal mixtures from dispersed metal-containing and carbon-containing industrial waste with stoichiometric coal consumption for the recovery of extracted metals have proved the possibility of obtaining sludge-coal briquettes, which are further subjected to metallization and reduction melting. Sequential processing of dispersed production waste, namely drying, metallization and reduction melting, allowed us to obtain at the final stage a metal sample that corresponds to high-quality steel in its composition. Based on the analysis of the results of experimental studies, the technology of reducing melting of metal-containing waste has been developed. As a result of the implementation of the technology, high-quality steels and alloys can be obtained without carburizing the metal, bypassing the production stages of cast iron and high-carbon alloys. The content of harmful impurities of sulfur and phosphorus meets the technical requirements of high-quality steel. The proposed technology for processing slag and sludge from oxygen-converter production will reduce the volume of accumulated production waste.

Keywords: converter slag, converter sludge, coal sludge, briquette, recovery, smelting.

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Introduction

At the traditional two-stage metallurgical complex "Blast Furnace - Oxygen Converter", converter slag [1, 2] is formed during the cast iron conversion into the steel. Oxidative melting of molten iron with a high carbon content of 4,2-4,5% is associated with the need to reduce the concentration of carbon in the metal to 0,2-0,5%, corresponding to the composition of the structural metal - steel. Removing carbon from cast iron composition is carried out by purging its melt by technical oxygen in converter, where by the reaction (1)

$$2C + 1,5O_2 = CO + CO_2, \qquad (1)$$
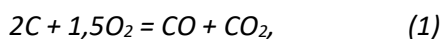
the carbon dissolved in cast iron in gaseous form $CO + CO_2$ is removed from melt into atmosphere.

It should be noted, that while purging a bath of cast iron by oxygen, not only carbon is oxidized. Allso a significant part of iron and other impurity alloying metals are oxidized too. They form a slag melt, which is a converter slag, in the form of oxides with the injected flux form. Its value is currently estimated only in the case when it contains an oxide of such a valuable metal as vanadium [3]. Vanadium-containing converter slags are by-products and further used to recover vanadium. The remaining converter slags containing iron oxides, partially manganese and chromium oxides are usually thrown into dumps. Today, huge masses of waste converter slags have been accumulated in dumps, the processing of which is an urgent problem. Processing of metal-containing industrial wastes, including converter slags, should be evaluated by the criterion of economic feasibility of useful metals' extraction from the slag (waste) mass unit, dependent on its chemical composition [2, 3].

The produced converter slag during conventional iron ore raw materials smelting at the Domna-Converter two-stage complex contains up to 25% iron, 4-5% manganese and a small amount of chromium. Furthermore, converter slag is characterized by high basicity on the $(CaO)/(SiO_2)$ relation at the level of 1,5÷2,0. As you can see by the iron and manganese content, the metal yield from the slag mass unit will be $e_m = 0,28$ kg/kg. This means that 3,57 tons of slag, i.e., empty rock, must be melted to smelt 1 ton of metal. Therefore, the inexpediency of direct metallurgical processing of converter slag is obvious. It is necessary to note that the possibility of its processing is to use as a component of a concentrate-based burden or metal-containing industrial waste with a high metal content. It should be noted that in parallel there exists and accumulates still a converter sludge with a high iron content. This symbiosis allows you to organize the production of a mixture with an average iron content of 50-55%, which corresponds to the expediency of its metallurgical processing.

Exhaust converter gases carry a lot of dust – about 10-30 g/m^3, in some cases this amount ups to 60 g/m^3. As a result, upper oxygen purge converters are typically equipped with gas treatment devices. Wet methods are used to clean converter gases.

Wet gas purification serves as the main source of sludge formation. The sludge of converter gas purges usually refers to iron rich by content. Their composition by main components are: Fe_{total} - 40 – 65%; CaO - 1,5 – 12,9%; SiO_2 - 1,4 – 2,8%; Al_2O_3 - 0,1 – 0,3%; MgO - 0,3 – 1,5%; P - 0,04 – 0,15%; S_{total} - 0,16 – 0,25%; C_{total} - 0,9 – 3,2%; Zn - 0,2 – 1,5%.

The humidity of these sludges can reach 50-60%, and the iron content - 60%. 10-30 kg of sludge per 1 ton of smelted steel (1-3%) is formed during the wet gas purification [4, 5].

The dispersion of converter sludges is quite high, although in some cases large particles are contained in the sludge.

The density of converter sludges is 3,5-5,0 g/cm^3. The specific dust release depends on the blasting intensity, tuyere designs, the oxygen pressure and the granulometric composition of the bulk materials.

When cooling with scrap, the amount of dust is 1,3-1,7 times more than when cooling with ore. In addition, the specific yield of dust is affected by the carbon content of the metal: with its increase, the yield of dust increases. The concentration of dust at the time of bulk materials supply can increase by 5 to 6 times [6, 7].

Experimental part

As the initial metal-containing material, converter slag and sludge of the converter plant of "ArcelorMittal Temirtau" JSC and coal sludge of coal enrichment were used.

The highest concentration of iron (Fe_{total} = 68,04%) of the converter sludge was observed in small fractions less than 0,05-0,1 mm. The converter sludge's granulometric composition of the converter plant of ArcelorMittal Temirtau JSC is given in Table 1.

The converter slurry must be dewatered before use. For the experience, 20 L of aqueous suspension (6000 g of solid) sludge was taken and the total precipitation time was 6 hours 40 minutes, from where the precipitation rate was 50 g/min. Further, the resulting sludge precipitate was dried in a drying chamber at a temperature of 100°C.

Further, the experiment was repeated under the directed effect of the magnetic field, depending on the layer height of the water-slurry phase in the same proportions. The precipitation time was 5 minutes, it indicates the magnetic field effect on the iron particles and the acceleration of the precipitation process.

Table 1 - Granulometric composition of the converter sludge

№	Fraction	%	Fe$_{total}$	FeO	SiO$_2$	CaO	MgO	MnO	P	Al$_2$O$_3$	Cu	Zn	Pb	Na$_2$O	K$_2$O	ППС
1	+1	7,4	42,2	43,07	6,02	27,56	4,3	0,96	0,36	0,86	0,021	>0,5(1,21)	0,068	0,146	0,155	3,08
2	0,63–1	3,6	46,88	55,38	6,41	28,25	7,25	1,46	0,65	0,74	0,019	0,42	0,014	0,074	0,088	-7,02
3	0,4–0,63	3,8	56,03	68,7	6,54	22,44	6,31	1,26	0,56	0,59	0,022	0,23	0,007	0,058	0,048	-12,87
4	0,315–0,4	8,5	59,97	72,34	4,90	19,81	6,98	1,43	0,47	0,86	0,023	0,20	0,007	0,045	0,052	-13,57
5	0,16–0,315	6,9	67,65	83,28	3,24	12,96	4,01	0,8	0,4	0,4	0,028	0,20	0,008	0,026	0,034	-10,95
6	0,1–0,16	4,0	66,96	78,97	2,82	12,95	3,83	0,72	0,39	0,38	0,030	0,34	0,013	0,035	0,036	-8,86
7	0,05–0,1	1,9	68,04	78,7	2,67	10,11	3,91	0,6	0,31	0,87	0,067	0,44	0,020	0,079	0,056	-8,01
8	0–0,05	9,0	47,26	46,85	1,96	20,63	6,38	0,65	0,18	0,37	0,030	>0,5(1,07)	0,057	0,101	0,068	6,95

The wet sludge was dried in a 100°C oven. The drying time was 26 minutes. A further increase in drying time does not result in a decrease in the moisture content of the test sample. The main values of the dried converter sludge sample are given in Table 2.

**Table 2** – The main properties of converter sludge during dewatering process

Parameters	Converter sludge
Bulk density, kg/cm^3	3,75
Volume of water-sludge samples, ml	78
Weight of solid component in water-slurry suspension, gr	6000
The precipitation time under the gravity influence, min	5
Precipitation rate, gr/min	6,0
The precipitation time under the magnetic field influence, min	4
Precipitation rate, gr/min	7,5
Temperature in drying chamber, ºC	100
Drying time, min	26

The content of the main components in the converter sludge dry sample is shown in Table 3.

As shown in Table 3 from the results of the chemical analysis, the iron content of the converter sludge is comparable to the iron ore concentrate. In addition, the converter sludge contains 1,56% manganese oxide as a useful impurity metal, which significantly increases the value of the used sludge.

At the same time, converter slag and converter sludge contain harmful impurities like sulfur and phosphorus, the concentration of which significantly exceeds the permissible limits (0,02 – 0,03%). However, the P$_2$O$_5$ cannot exist in the slag in the free state, because it is not stable at high temperatures at which phosphorus is easily reduced by carbon, manganese and even iron (2).

$$(P_2O_5) + 5 [Fe] \leftrightarrow 2 [P] + 5 (FeO) \qquad (2)$$
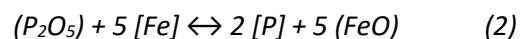
The strongest connection P$_2$O$_5$ gives CaO (3).

$$4 (CaO) + (P_2O_5) = (4 CaO \times P_2O_5) \qquad (3)$$

Hence the total phosphorus removal reaction can be represented (4):

$$2[P] + 5(FeO) + 4(CaO) = (4 CaO \times P_2O_5) \qquad (4)$$
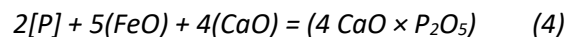
A compound Ca$_3$(PO$_4$)$_2$ may also be formed, and phosphorus starts reduction reaction at temperatures above 1200°C.

Sulfur is completely rigidly bound to calcium oxide (CaO) in the form of CaS, from which sulfur cannot be reduced during reducing melting (5).

$$(FeS) + (CaO) \leftrightarrow (FeO) + (CaS) \qquad (5)$$
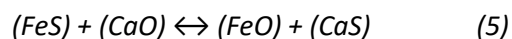
Therefore, in order to prevent the reduction and transition of phosphorus to the metal composition, it is necessary to provide a temperature-thermal mode of reducing melting of raw materials prepared from a mixture of converter slag and sludge [8, 9].

All used materials except converter slag are fine and dispersed. In order to ensure the dispersion of the burden, pieces of converter slag with a fraction of 40÷80, 40÷20, 20÷10, 10÷5 mm were crushed on a laboratory jaw crusher and then on a vibrator.

Table 3 - Chemical composition of burden components

Component name	Chemical composition, %									
	Fe_{total}	FeO	MnO	SiO_2	Al_2O_3	CaO	MgO	S	P	C
Converter sludge	63,7	15,0	1,28	6,66	0,53	15,2	6,9	0,68	0,06	1,8
Vessel slag	25	32,0	4,5	12,8	4,62	30,5	6,56	0,30	0,34	-
Coal sludge	-	-	-	16,07	7,69	1,35	0,35	0,5	-	65,0

As it shown on analysis of the data in Table 3, in the composition of the converter slag and the sludge, most of the slag-forming compounds SiO_2, Al_2O_3, CaO, MgO makes up 40% calcium oxide.

In order to increase the iron content of the resulting metal product, the converter sludge and the converter slag were enriched on a single cone magnetic separator at 0,5A.

In the non-magnetic part, 5% of the fraction was separated from the starting mass of 1035 g of converter slag, and in the converter sludge, the non-magnetic part also amounted to 5% of the starting mass of 730 g.

It should be noted that the type of reducing agent should be selected depending on the content of the reducing element in the metal-containing feedstock. For example, if the iron content of the sludge is about 20%, it is inappropriate to use coal flotation waste as a reducing agent, since the iron from the sludge will practically not be reduced, and the amount of slag which is formed during the smelting process will be very large.

In this case, the industrial waste used in the reduction is a mixture of converter slag and sludge having an iron content more than 50%.

Therefore, it was decided to use coal sludge formed during coal enrichment as a reducing agent.

The composition of coal sludge contains a large number of slag-forming oxides about 15%. In order to separate the coal particles (having a small specific gravity) from the slag-forming particles, the coal sludge was subjected to gravitational enrichment. Small and lightweight particles were blown into a special gas outlet, then entered the second container in the form of a dust collector. The caught dust mainly contains carbon particles.

Repeated chemical analysis of enriched metal-containing materials and coal sludge showed a significant increase in iron content. The chemical composition of the enriched components is shown in Table 4.

Studies were carried out with burden, in the ratio of converter sludge to converter slag 90:10, respectively. The chemical composition of the mixture of converter sludge and converter slag is shown in Table 5.

The solid carbon reduction process was carried out in a temperature range of 900-1200°C. The choice of these temperatures is explained, on the one hand, by the impossibility of melting the material, on the other hand, by the impossibility of reducing phosphorus.

As noted earlier, coal sludge is used as a reducing agent. For the complete reduction of metals, the stoichiometric flow rate of coal sludge was calculated per unit of iron-containing material according to the method of Tleugabulov S.M. [10, 11]. As a result of the calculations, the consumption of coal sludge per unit of iron-containing material in the amount of 0,300 kg/kg was obtained. Obtained mixture of converter slag with converter sludge and coal sludge results to chemical composition changes of mixture. The mass fraction of the iron- and coal-containing parts was calculated as follows: $y_p = 1,0: 1,300 = 0,76$ and $y_y = 0,300:1,300 = 0,23$. After mixing the ore-coal mixtures, sludge-coal briquettes were obtained on the laboratory mold. Liquid glass was used as the binder in the briquetting process.

The resulting briquettes were dried at 300-350°C in a muffle oven.

The drying temperature was selected according to the following principle. At a temperature of 350°C, solid carbon in the mixture of converter sludge and slag is practically not oxidized in the air atmosphere. Its reaction with metal oxides does not occur.

After drying, the briquettes acquired higher mechanical strength for processing in two stages: 1) reduction firing in a shaft furnace at 1000-1200°C; 2) smelting reduction in a melting furnace at a temperature of 1600°C.

For metallization, briquettes with size of 5×4cm and a mass of 125 g. entered into a chamber furnace SNOL-1,6.2.0.0,8/9-M1-U4 preheated to 500°C. During the experiment, the time and temperature of the start of gas evolution was fixed at 900°C, it indicated the onset of a direct reduction reaction.

Table 4 - Chemical composition of enriched components

Components	Chemical composition, %									
	Fe_{total}	FeO	MnO	SiO_2	Al_2O_3	CaO	MgO	S	P	C
Converter sludge	71,15	16,4	1,33	3,0	0,34	9,2	5,7	0,59	0,03	1,8
Vessel slag	32	33,4	4,7	9,2	4,43	30,5	5,3	0,21	0,31	-
Coal sludge	-	-	-	8,6	5,65	0,85	0,35	0,5	-	65,0

Table 5 - Chemical composition of converter sludge and converter slag mixture

Name	Fe_{total}	FeO	MnO	SiO_2	Al_2O_3	CaO	MgO	P	S
Ratio 90:10	62	16,5	1,5	3,2	0,6	10,2	5,2	0,05	0,51

Table 6 - Chemical composition of metalized product

Material	Chemical composition, %									
	Fe_{total}	FeO	Fe_{metal}	MnO	SiO_2	CaO	MgO	P	S	Al_2O_3
Metal briquette 1	80	3,2	78,4	1,2	12,2	9,8	4,7	0,07	0,69	4,5

Then, as the temperature increased to 1000°C, the CO and SO_2 gas were strongly released by the reaction (6).

$$5FeO + 4C = 5Fe_{metal} + 3CO + CO_2 \qquad (6)$$
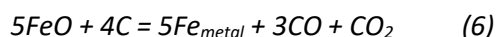
The experiments continued until gas release stoped. This meant almost complete or marginal reduction of iron at a temperature of 1000-1200°C.

Initial mass of the sample before the test: m1 = 126 g; mass of the sample after the test: m2 = 111 g; loss of mass: Δm = m1 - m2 = 14,9 g.

On the basis of experimental studies, the mode of the reduction process was established, during which a metallized product was obtained. The characteristics of the metallized product is shown in Table 6.

Sludge-coal briquettes weighing 250 g were placed in an alund crucible and installed in a "Tammana" smelting furnace. Immediately after turning on the furnace, in order to avoid secondary oxidation of the metallized product, the surface of the crucible was blown with argon for 10 seconds at 90A. The temperature inside the reaction crucible was controlled by tungsten/tungsten-rhenium VR5/20 thermocouple. When the temperature reached 1600°C, the briquettes completely converted to melt. The exposure in the furnace was 25 minutes. Further, argon was purged to cool the melt, then the melt crucible was removed and cooled in air. The results of the chemical analysis of the resulting metal casting sample are shown in Table 7.

Table 7 - Chemical composition of the obtained metal sample

Metal	Chemical composition, %				
	[Mn]	[C]	[Si]	[S]	[P]
	1,20	0,32	0,26	0,025	0,03

Conclusion

1. The optimal composition of the components of the converter slag and gas cleaning sludge mixture is established in terms of the percentage of iron, which is expedient for use as a raw material in steel smelting.

2. The metal yield was 38% and the slag yield 62% from the starting 250 g of sludge-coal briquettes.

3. According to the results of chemical analysis, the melted metal sample is natural alloy steel. The content of harmful impurities of sulfur and phosphorus meets the technical requirements of qualitative steel.

4. The proposed technology of processing slag and sludge of oxygen-converter production will reduce the volume of accumulated production wastes.

Conflict of interest. On behalf of all authors, the correspondent author states that there is no conflict of interest.

Конвертерлі қожды металлургиялық қайта өңдеу

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ТҮЙІНДЕМЕ

Қазіргі металлургия комбинаттарында конвертер шламдары темір мөлшері жоғары металға бай өнеркәсіптік қалдықтарға жатады. Қазіргі уақытта осы қалдықтарды пайдаға жарату және металлургияда шикізат ретінде пайдалану мәселесі туындап отыр. Бұл жұмыстың мақсаты газтазартудың конвертер шламдары мен конвертер қождарының қоспасы негізінде брикеттеу және брикеттелген өнімдерді тотықсыздандыру процестерін зерттеу болып табылады. Мақалада конвертерлі балқыту қалдықтарын, атап айтқанда конвертерлі қождың газ тазартудың конвертерлі шламымен қоспасын металлургиялық қайта өңдеуге тарту мүмкіндіктері қарастырылған. Осы негізде болат балқытуға арналған брикеттерді балқыту қалдықтарының қоспасынан шығару ұсынылады. Газтазартудың конвертерлік шламы мен конвертерлік қож қоспасынан шламкөмірлік брикеттерді дайындау, оларды металдандыру және зертханалық жағдайда тотықсыздандырып балқыту бойынша эксперименттік зерттеулерді орындау барысында болатты балқытуға шикізат ретінде пайдалануға лайықты болатын темірдің пайыздық құрамы бойынша конвертерлік қож бен газтазарту шлам қоспасы компоненттерінің қолайлы құрамы анықталды. Алынатын металдарды тотықсыздандыруға көмірдің стехиометриялық шығыны және металдан тұратын және құрамында көміртегі бар өнеркәсіптік қалдықтардан шламкөмірлік қоспаларды дайындау бойынша эксперименттік зерттеулер металдандыруға және тотықсыздандырып балқытуға жататын шламкөмірлік брикеттерді алуға мүмкін болатынын дәлелдеді. Өндірістің дисперсті қалдықтарын кезек кезегімен өңдеу, атап айтқанда кептіру, металдандыру және тотықсыздандырып балқыту, ең соңында сапалы болатқа сәйкес келетін металл үлгісін алуға мүмкіндік берді. Эксперименттік зерттеулердің нәтижелерін талдау негізінде құрамында металл бар қалдықтарды тотықсыздандырып балқыту технологиясы жасалды. Технологияны енгізу нәтижесінде шойын мен жоғары көміртекті қорытпалар өндірісінің кезеңдерін айналып өтіп, металды көміртектендірмей жоғары сапалы болаттар мен қорытпаларды алуға болады. Күкірт пен фосфордың зиянды қоспаларының құрамы жоғары сапалы болаттың техникалық талаптарына сәйкес келеді. Оттегі-конвертер өндірісінің қождары мен шламдарын өңдеудің ұсынылған технологиясы жинақталған өндіріс қалдықтарының көлемін азайтуға мүмкіндік береді.

Түйін сөздер: конвертерлі қож, конвертерлі шлам, көмір шламы, брикет, тотықсыздандыру, балқыту.

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Металлургическая переработка конвертерного шлака

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АННОТАЦИЯ

Конвертерные шламы на современных металлургических комбинатах представляют значительную часть металлосодержащих промышленных отходов с высокой концентрацией железа. В настоящее время существует проблема их утилизации и применения в качестве сырья для металлургии. Целью данной работы является исследование процессов брикетирования и восстановления брикетированных продуктов, на основе смеси конвертерных шламов газоочистки и конвертерных шлаков. При выполнении экспериментальных исследований по подготовке шламоугольных брикетов из смеси конвертерного шлама газоочистки и конвертерного шлака, их металлизации и восстановительной плавке в лабораторных условиях, установлен оптимальный состав компонентов смеси конвертерного шлака и шлама газоочистки по процентному содержанию железа, представляющему целесообразность для использования в качестве сырья при выплавке стали. Экспериментальные исследования по подготовке из дисперсных металлосодержащих и углеродсодержащих промышленных отходов шламоугольных смесей со стехиометрическим расходом угля на восстановление извлекаемых металлов, доказали возможность получения шламоугольных брикетов, которые далее подвергаются металлизации и восстановительной плавке. Последовательная обработка дисперсных отходов производства, а именно сушка, металлизация и восстановительная плавка позволила получить на завершающей стадии образец металла, который по своему составу соответствуют качественной стали. На основе анализа результатов экспериментальных исследований разработана технология восстановительной плавки металлосодержащих отходов. В результате реализации технологии могут быть получены качественные стали и сплавы без науглероживания металла, минуя стадии производства чугуна и высокоуглеродистых сплавов. Содержание вредных примесей серы и фосфора соответствует техническим требованиям качественной стали. Предлагаемая технология переработки шлака и шламов кислородно-конвертерного производства позволит сократить объемы накапливаемых отходов производства.

Ключевые слова: конвертерный шлак, конвертерный шлам, угольный шлам, брикет, восстановление, плавка.

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