Pelletizing of ferruginous quartzites with red mud addition and heat treatment of obtained pellets in carbon monoxide atmosphere

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Use of concentrates with coarseness from 0.063 mm and lower as metallurgical raw material, is possible only after their corresponding enlargement, what is a complicated problem. This research describes comminution of ferruginous quartzites at first, and then sieving in the screen with coarseness < 0.063 mm. Red mud from Ural aluminium works was added as a binder. To increase pelletizing degree, the technique of red mud powdering on charge surface by compressed air was suggested. Charge humidity makes 6–9 % due to presence of high-dispersed clay of fine-porous particles with high moisture capacity in this charge. It was established that tensile strength during compression of green pellets with diameter 14–16 mm, which were obtained via this method, are 2 kg per a pellet. Aggregation of separate grains is noted already at the temperature 200–300 °C, what is explained by their partial surface oxidizing in hematite; at the same time, strength of pellets increases. It was also established that red mud addition during pelletizing of ferruginous quartzites is sufficient in the amount up to 5 %, while magnetite crystals show good aggregation with each other at the temperature 900 °C in reducing atmosphere of carbon monoxide, what leads to recrystallization of magnetite grains with forming of magnetite binding. The forming processes of slag, silica and quartzite are passing in the neutral media at the temperature 1000–1200 °C. It was found out that pellets with diameter 14–16 mm from ferruginous quartzites, which are subjected to roasting up to 1100 °C in reducing atmosphere, have hematite binding with relatively low strength. When increasing the temperature up to 1290 °C, pellets reach their tensile strength during compression up to 240–250 kg per a pellet. It was also found out that a sample at the temperature 1290 °C presents dense magnetite concentration in the central part of a pellet and melting of grains around their periphery provides strong structure; in this case qualitative composition of a pellet varies during phase transition of oxides from hematite to wustites.

Key words: fine ferruginous quartzites, granulometric composition, red mud, disk granulator, sintering, agglomeration, crushing strength.

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Introduction

Use of concentrates with coarseness from 0.063 mm and lower in metallurgical production requires their enlargement via pelletizing with adding of binding materials, what causes necessity of development of a special technology [1–3].

It is known that decrease of coarseness of charge granulometric composition leads to quick reducing of agglomeration efficiency due to dropping of gas penetration of a layer, which is the main condition of successful sintering. Agglomeration of fine concentrates also is accompanied by dramatic dropping of gas penetration, what leads to diminishing of equipment productivity and requires special sintering techniques [4–6].

The pelletizing method is based on ability of moisturized fine materials to form strong balls (pellets) during processing in rotating drums or cups. Granulation of concentrates is stipulated by appearance of capillary forces. Negative pressure, which braces particles in perpendicular direction to the contact surface with a pelletizing unit appears in the system of pores and capillaries, between pellet particles [7–9]. The essence of capillary pressure is presented in the **Fig. 1**. Thin capillary pores are striking by one end to moisture skull of a pelletizer, and water forced to penetrate into the space between pores (elongated S_1 and single-point S_2). When overrolling, water is in the same state (ΣX and ΣY), staying in the inter-particle space (see the hatched areas), with forming of dense fixed particles [10–12].

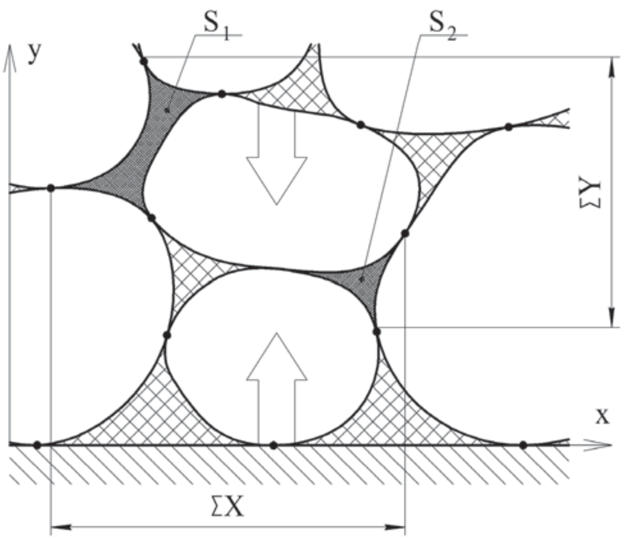


Fig. 1. The scheme of capillary tension presentation (S_1, S_2)

Finished granules after corresponding roasting present very strong and transportable product with the same size [13—15]. Such pellets are successfully used in blast furnaces and shaft metallization furnaces, providing more homogenous pas penetration of charge and high reducing ability of iron [16—18].

The aim of this work is substantiation of efficiency of red mud use as a binding additive during pelletizing; this additive supports reduction of iron component in fine concentrates during oxidizing roasting in the sintering process. The main task is increase of percentage of Fe extraction in the conditions of operation with complex ores. Red mud use as a binder helps also to solve the problem of active use of accumulated wastes in alumina production [19, 20].

Theoretical prerequisites

To provide primarily evaluation of the features of pellets behaviour in reducing atmosphere, the samples were subjected to thermodynamic analysis during phase transitions from hematite to magnetite. As soon as hematite is the main compound in pellets, reactions with hematite can be classified as direct reduction or transition state. The equations of direct phase transitions are presented in the works [21, 22] as a complex of reactions:

$$3Fe_2O_3(s) + CO(g) \rightarrow 2Fe_3O_4(s) + CO_2(g) \quad (1)$$

$$Fe_3O_4(s) + CO(g) \rightarrow 3FeO(s) + CO_2(g) \quad (2)$$

$$FeO(s) + CO(g) \rightarrow Fe(s) + CO_2(g) \quad (3)$$

The accompanying stage of Fe oxide reduction is the folowing:

$$Fe_3O_4 \cdot CaO(s) + 3C(s) \rightarrow 2Fe(s) + CaO(s) + 3CO(g)\uparrow \quad (4)$$

It explains presence of γ-Fe_2O_3 phase or traces of Fe_3O_4 and α-Fe in decomposition products of hematite Fe in the air. Such mechanism can be realized, e.g., for large sample thickness and heating speed, when oxygen can be late to supply to a reactant.

4 types of bonds, appearing during roasting of pellets with red mud addition, are revealed by the authors [23, 24] at different temperatures in the structure of pellets:

1. Moderate sintering of separate grains is noted already at the temperature 200—300 °C, what is explained by partial surface oxidizing of hematite Fe. Owing to high mobility of atoms during oxidizing, hematite crystals in the contact points form binding presented by a tie plates between single grains, what leads to apparent increase of a pellet strength just at the low temperatures (**Fig. 2*a***, expression 1).

2. At the temperature 900 °C, recrystallization of magnetite grains starts, it is accompanied by forming of a magnetite binder, similar to a hematite one (**Fig. 2*b***, expression 2).

3. At the temperature 1000—1200 °C, slag forming from FeO, silica and quartzite occurs in neutral or low-oxidizing atmosphere. Slag moisturized magnetite grains and hinders their further oxidizing, while during cooling of pellets it cemented single grains in concentrate with forming of reliable structure (**Fig. 2*c***, expression 3).

4. Melting of grains of ferruginous quartzites between themselves is observed at the temperature 1290 °C, when magnetite and hematite crystals, bonded with each other, are forming (**Fig. 2*d***, expression 4). It should be noted that Fe_2O_3 does not form slag with SiO_2 at the higher roasting temperature 1300 °C, and sintering takes place in a solid phase.

Then oxide form of hematite (α-β Fe_2O_3) transforms in low-magnetic maghemite (γ-Fe_2O_3) with varying the lattice structure, and then through the stages of magnetite (Fe_3O_4) and wustites ($Fe_{1-x}O$) till appearance of rare forming of pure iron.

Consequently, the roasting temperature varies within the range 200—1290 °C, depending on concentrate properties.

Achievements of the cited works conclude in reveal of the mechanism of frame structure forming due to step-by-step hematite reduction till obtaining pure iron. As a result of theoretical analysis of roasting of iron ore concentrate pellets, it was found out that particles sintered at the temperature 1290 °Cwith forming Fe frame structure having sufficient strength.

Materials and methods

The sample of ferruginous quartzites from Mikhailovskiy mining and concentrating works was used in this research. Red mud from Ural aluminium works (Kamensk-Uralskiy, Sverdlovsk region) was added as a binder. It was taken directly from a slime field. Chemical composition of ferruginous quartzites and red mud is presented in the **Table 1**.

Granulometric material composition was determined using laser analyzer of particles "Microsizer 201". The results of distribution of these particles are displayed in the **Table 2**.

Fig. 2. The scheme of presentations of different bond types between a pellet grains:
a – forming of the first aggregated states at the temperature 200—300 °C, with moisture loss;
b – magnetite recrystallization with forming of magnetite binder at the temperature 900 °C, with removal of gaseous reaction products from inter-grain pores;
c – forming of hematite binder at the temperature 1000—1200 °C;
d – melting of grains of ferruginous quartzites between themselves at the temperature 1290 °C

Table 1. Chemical composition of materials, % (mass.)

	FeO	Fe_2O_3	Al_2O_3	CaO	SiO_3	MgO	Na_2O	K_2O
Concentrate of ferruginous quartzites at Mikhailovskiy mining and concentrating works named after A. V. Varichev	13.46	52.11	9.65	3.89	17.57	2.86	0.34	0,12
Red mud from Ural aluminium works	6.67	42.14	23.62	15.20	5.12	2.49	3.35	1.41

Table 2. Mass part of particles for material with various coarseness, % (mass.)

Coarseness, mm	Ferruginous quartzites		Red mud from Ural aluminium works
	Before comminution	After comminution	
+ 1.000	3.76	–	8.31
+ 0.063	70.84	27.44	56.32
– 0.063	25.40	72.56	35.37

Conducted researches

Obtaining of a pellet includes adding of red mud in the amount 1–15 % (mass.) in composition of ferruginous quartzites (which are comminuted to coarseness < 0.063 mm) during pelletizing process with consequent roasting (**Fig. 3**).

Red mud of the fraction -63 µm was added to ferruginous quartzite, which was sieved to the same fraction. Mixing process was carried out in a shaker mounted in a driven rolling table AZ-VTR.36 during 15 min with speed 25 min^{-1}. A specimen was used for pelletizing and placed in a pelletizer cup with diameter 300 mm and rotation speed 35 min^{-1}. A pelletizer was designed and assembled according to the author's drawings for laboratorial works. Each pelletizing work used initial specimen of mix of ferruginous quartzites with mass 100 g. water was added by sprinkling on the surface of comminuted ore during pelletizing.

When added water exceeds 9 %, forming of a pellet with moisture over-saturation was observed just immediately. Pellet growth was observed during 20 min until obtaining the form close to ellipsoid, with cross section size 35 mm and length 60 mm. Then pelletizing process was hindered and obtained cluster, which absorbed whole material and moisture reminder, rolled over pelletizer cup sides. When moisture content decreased to 6–9 g of water per 100 g of comminuted ore, the period of pellets forming was 30–45 min. the first forming of pellets agglomerates was observed after 7–10 min since the start of pelletizing, then they increased to 14–16 mm.

Influence of red mud additive in composition of ferruginous quartzites was examined in the range 1–20 % (mass.);

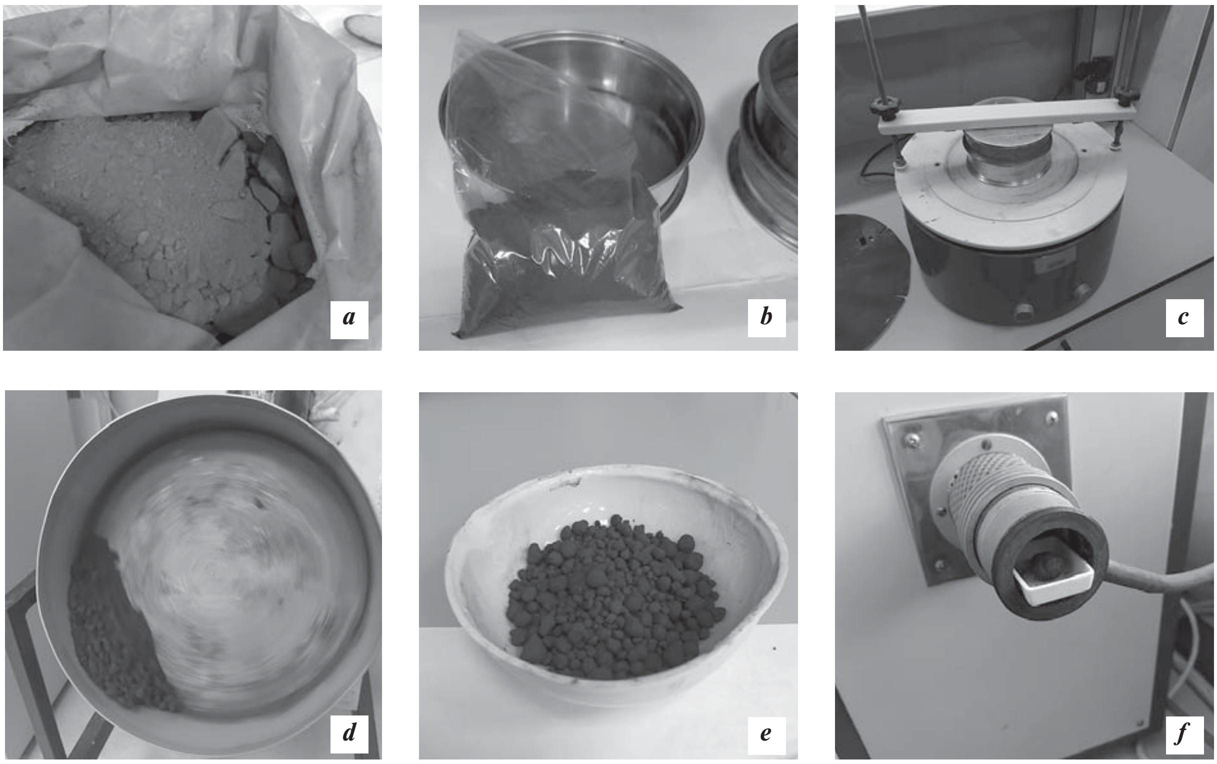


Fig. 3. The scheme of pellets obtaining from ferruginous quartzites in laboratorial conditions:
a – ferruginous quartzite; *b* – red mud; *c* – material classification to -0.063 mm; *d* – forming of pellets in a plate pelletizer; *e* – finished green pellets; *f* – roasting of pellets

in this case red mud addition was conducted via powdering. Optimal red mud amount, which was introduced in pellet composition, was determined experimentally after sintering and destruction via investigation for crushing strength in a hydraulic press. So, when adding of red mud more than 5 % (mass.), there was no visible variations; at the same time, Fe content did not increase, while lowering of cold crushing strength of pellets was noted after introduction of 10, 15 and 20 %. It was found out experimentally, that optimal amount of red mud additive makes 3–5 % (mass.).

The specimen of formed pellets with weight 100 g was placed in the ceramic heat-resistant glass and then charged in the muffle furnace, where the samples were subjected to roasting in reducing atmosphere of carbon monoxide according to the technique suggested by the authors [25–27]. The roasting process was carried out at the selected temperature conditions, separately for each testing. The samples were left in the furnace during 12 hours for cooling to the room temperature. After roasting, a glass with pellets was taken out of the furnace for classification of valid samples for consequent analysis. There were 100 valid samples with size to 16 mm with mostly round form. Roasting of valid samples was conducted together with other samples for simulation of sintering process in industrial conditions [28–30].

The presented samples of cooled pellets were subjected to analysis for cold crushing strength by the manual hydraulic press (Matest C094). Initial phase composition of materials was determined via the method of X-ray diffraction (XRD) using the analyzer Bruker AXS D8 ADVANCE (Germany) with Cu. Porosity and structure of samples were examined by the scanning optical microscope JSM-6460LV of the company Jeol «Oxford».

To determine phase composition, pellets were destructed, comminuted and sieved to the fraction 400 µm. Then a pellet with diameter 15 mm and height 4 mm was formed for phase analysis. Microstructure was analyzed after preliminary preparation of ground samples in the laboratorial metallographic grinding machine MP 2B at the speed 300 min^{-1}, while consequent polishing was carried out at the speed 150 min^{-1} according the technique.

Results and discussion

To determine microstructure of a sintered pellet, a sample with ground surface is prepared and placed in an optical microscope. The boundaries of hematite grains and reduced austenite are seen clearly. The sample obtained at the temperature 1290 °C demonstrates dense concentration of magnetite in the central part of a pellet (**Fig. 4*a***).

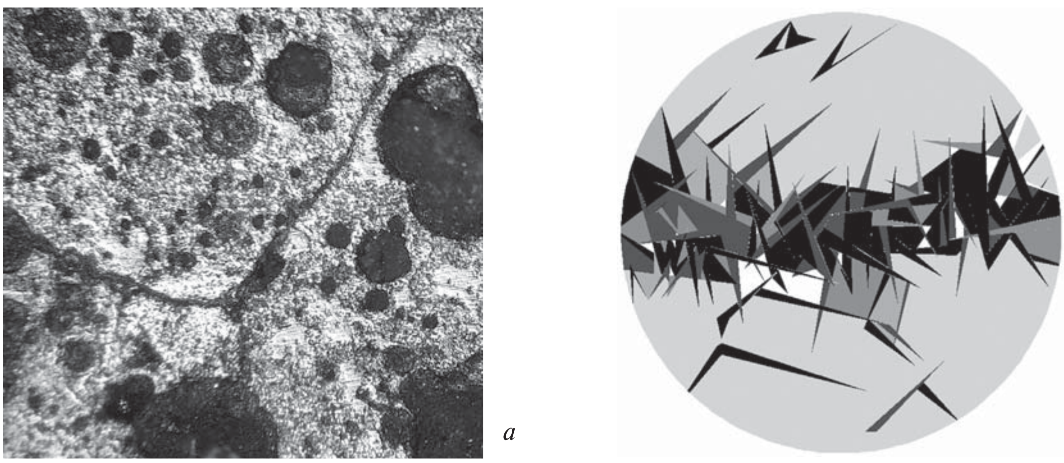
Fig. 4*b* illustrates the process of Fe reduction in fine ferruginous quartzites. Magnetite crystals display good aggregation with each other during 2 hour heating at the temperature 900 °C, what is accompanied by removal of gaseous products of interaction reactions among the components; it is considered as a cause of hematite binder forming in the contact plane and starting of reduction process.

The results of phase analysis of pellets from ferruginous quartzites with red mud addition are presented in the **Table 3**.

Table 3. General results of the research

Parameter	Concentrate	Comminuted green pellet	Comminuted roasted pellet
W, % (mass.)	10.08	9.92	4.15
Femag., % (mass.)	65.85	65.71	67.32
Binder additive, % (mass.)		0.57	
Carbon monoxide consumption, m^3/s	–	–	1.5
Compression strength in raw form, kg/pel.	–	1.43	245.84
Flowability to 0.5 mm, %	–	98.27	13.59
Porosity, %	–	21.87	33.04

Investigations of the effect of various temperature procedures on iron oxide reduction in a pellet composition were conducted. Reduction occurred during phase transition of oxides, from hematite transformation (α→β Fe_2O_3), passing the stages of magnetic maghemite (γ Fe_2O_3), magnetite (Fe_3O_4) until forming of wustites ($Fe_{x-1}O$). So, magnetite



a *b*

Fig. 4. Forming of sintered pellet structure:
a – after heating in inert atmosphere at the temperature 1000° C during 2 hours (magnification x325);
b – the scheme for presentation of hematite binder (dark areas) between two crystals of magnetite (light area)

peak in the phase diagram coincides with maghemite peak, which is also presented in a pellet structure.

Strength of hematite pellets with coarseness < 0.063 mm starts to increase quickly during pelletizing just at the temperatures above 900 °C [31–33]. Above-mentioned prevailing magnetite peak in the phase diagram is confirmed by the author's researches.

The amount of red mud additive has positive effect on binder quality during pelletizing. Sufficient amount of red mud additive is 5 %, the larger amount decreases Fe concentration, but does not influence anyhow on pellet mechanical properties.

Hematite-based pellets of ferruginous quartzites displayed rather difficult sintering in comparison with usual iron ore magnetite-based concentrate, which has sintered structure already at the temperature 1100 °C; at the same time, oxidizing atmosphere does not provide essential effect on strength properties of a sample [34, 35]. So, when the temperature reached 1290 °C in the conditions of reducing atmosphere, tensile crushing strength of pellet samples reached 240–250 kg per a pellet.

Practical value of this research concludes in improvement of pellets quality when using red mud as a binder, with consequent sintering in carbon monoxide atmosphere; it rises technical and economical parameters of production at Mikhailovskiy mining and concentrating works named after A. V. Varichev.

Putting into practice potential of this technology was determined via mathematical simulation on the base of experimental data. The results of evaluation of the technical and economical effect during roasting of pellets containing red mud as a binder in the carbon monoxide media are presented in the **Table 4**.

Possible ways of further research in this direction can be marked in development of pellets production on the base of other ores, e.g. at "Karelskiy okatysh" enterprise. Perspectives of manufacturing commercial products with large content of industrial wastes (slimes, slags) without raw material quality deterioration can be considered. Search of the new suggestions for solving the problem of additional extraction from gangue, which can contain significant amount of non-magnetic iron, seems to be prospective.

It seems interesting to use directions of pellets metallization without the stage of oxidizing roasting. However, introduction of positive influencing additives in semiproduct composition at the forming stage can lead to obtain of low quality raw material as a result of consequent processing; e.g. alkali compounds, which are presented in semiproduct composition, deteriorate quality parameters during melting process.

Additionally, the problem of lowering of CO emission parameters is also actual, but not so important, though it finalizes in subsequent imposition of penalties.

To provide financial evaluation of each project, it is necessary to construct the units of pilot-industrial equipment with maximal approximation to industrial operating conditions for manufacture of finished semiproduct without quality deterioration.

Conclusion

Influence of red mud additive on pelletizing process of ferruginous quartzites is examined.

1. Red mud additive as a binder in fine ferruginous quartzites is sufficient in the amount up to 5 %.
2. Water addition should not exceed 9 % in pellet composition on the base of ferruginous quartzites.
3. Magnetite crystals show good aggregation with each other at the temperature 900 °C, forming hematite binder in the contact plane.
4. Strength of hematite-based pellets during roasting at the temperature up to 1100 °C in reducing atmosphere of carbon monoxide rises slowly, but later increases quickly.
5. It was established that crushing strength reaches 240–250 kg per a pellet when the temperature increases up to 1290 °C.
6. It was found out that qualitative composition of pellets varies during phase transition of oxides from hematite to elementary Fe.
7. It was established that a sample forms dense magnetite concentration in the central part of a pellet at the temperature 1290 °C, while melting of grains along their periphery provides firm structure.
8. Necessity of solving several problems in this direction is revealed; the following such problems can be mentioned here: manufacture of semiproducts from other ores, complex evaluation with taking melting into account, use of gangue and slags, metallization of pellets without oxidizing roasting, decrease of CO emission parameters, with resuming financial assessment of each project.

Table 4. Technical and economical effect during roasting of pellets containing red mud as a binder in the carbon monoxide media

Parameters	Roasting in the air media	Roasting in the carbon monoxide media	Effect, %
Electric energy consumption, kWt·h/t of pellets	33.27	32.23	3.23
Fuel consumption, m^3/t	13.75	12.47	10.26
Expected productivity, t/h	500	510	1.96
Roasting temperature, °C	1250	1200	4.17
Compression strength, kg/pel.	220	230	4.35
Drum sample (> 5 mm), %	92	95	3.16
Drum sample (< 0.5 mm), %	3.04	3.05	0.33
Porosity of pellets, %	23.14	23.26	0.52

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