On the problem of processing of manganese wastes from ore beneficiation: preparation of a single-component charge for ferromanganese melting

N. K. Dosmukhamedov, *Cand. Eng., Prof.* [1], *e-mail: nurdos@bk.ru;*
E. E. Zholdasbay*, Ph. D.* [1];
E. B. Tazhiev*, Ph. D., Senior lecturer* [1], *e-mail: eleusiz_t1990@mail.ru (Corresponding author);*
A. A. Argyn*, Ph. D.* [1]

[1] *Satbaev University (Almaty, Kazakhstan)*

Large volumes of wastes from beneficiation of manganese ores, dispersed iron and carbon-containing sludges are accumulated at large concentrating and metallurgical plants. Millions of tons of manganese wastes have been accumulated at the Dzhezdinsky mining and concentrating plant. Processing of small and dispersed metal-containing industrial wastes cannot be carried out via conventional technology. Development and implementation of the new technology for the processing of these wastes into valuable metal alloys is a very urgent problem. The highly efficient technology for the preparation and assembling of charge based on manganese wastes from beneficiation of manganese ores at the Dzhezdinsky mining and concentrating plant has been developed. The device for air-gravitation beneficiation of manganese wastes has been developed, which makes it possible to obtain a manganese-containing product with a high manganese content (up to 28 %). The method of stage-by-stage preparation of complex fine mixtures (with fraction less than 1 mm), consisting of manganese wastes, mill scale and charcoal, is shown, with substantiation for each operation with determination of the optimal parameters (temperature, consumption of components, etc.) is displayed. The composition of the final single-component charge obtained in the form of granulated pellets is given. Comprehensive analytical studies of intermediate and final products were performed using atomic emission spectrometry with inductively coupled plasma and X-ray phase analysis. It ensured step-by-step quality control of the products obtained during preparation of the single-component charge. The results of the research will be used for the preparation and efficient processing of fine manganese wastes from the Dzhezdinsky mining and concentrating plant in Kazakhstan for production of high-quality alloys that are demanded on the domestic and world markets.

Key words: manganese wastes, air gravitation beneficiation, mill scale, charcoal, carbon, reduction, mixture, single-component charge, granulated pellets.
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Introduction

The conventional manufacturing technology of ferroalloys is based on use od lump manganese and chromite ores with additives of metallurgical coal and fluxes; it is characterized by necessity of solving of several problems. In particular, the problems of saving of primary raw materials, forming and processing of wastes, reducing the cost of products owing to cutting the consumption of electric power and coke seems to be rather actual.

At present time, production of iron, steel and ferroalloys is accompanied with large emission of greenhouse gases [1–3]. To decrease these emissions, it seems evident to provide putting into practice innovative technologies and use of alternative fuel, which is depleted by CO_2. Use of biomass, biocarbon of hydrogen can be considered as the possible ways to solve this problem. Replacement of fossil carbon-bearing fuels by charcoal, for example, can lead to significant decrease of specific CO_2 emission in the atmosphere [4, 5].

Searching the methods for processing of fine-dispersed wastes after beneficiation of manganese ores is a separate problem. Accumulated volumes of these wastes determine the critical level of their influence on underground waters, aqueous flora and the environment [6–8].

Fine-dispersed wastes, which are forming during preparation of raw materials. Contain increased Fe and Mn contents comparable with their content in conditional raw materials. Large-scale use of such wastes in conventional technology is practically impossible and restricted by technical conditions of their application. At present time, accumulated volumes of the above-mentioned wastes achieved such amount, that they can be rather considered as secondary raw resources for a large metallurgical works.

At present time there are about 15 mln tons of fine wastes from beneficiation of chromite and manganese ores accumulated in the territory of Donskoy and Dzhezdinsky mining and concentrating works [9]. They continue to be accumulated in geometrical progression due to absence of a rational technology for their processing. It finalizes already in a critical ecological problem.

The new theoretical suggestions were published during last decade [10–12], which can be used as a base for development of a new technology for processing of fine-dispersed Mn- and Cr-containing wastes. Organization of ferroalloys production via use of solid phase reactions for reduction of metal oxides by carbon is recognized as the most important component of this technology. According to the conclusions of the authors of the researches [12, 13], Fe, Mn and Cr oxides in dispersed state acquire higher interaction activity with solid carbon at low temperatures in comparison with a liquid phase system, which is used in conventional technology. Development of this technology is focused on dissociation of metal oxides, which passes identically to their forming from separate elements, not depending on external conditions. The new approaches to technology of steel and ferroalloys production are based on the common technique for calculation of initial single-component charge with varying composition. Use of charcoal, which is produced via pyrolysis from biological wastes (starting from weed plants to agricultural wastes, such as straw, cotton stems etc.) instead of stone coal as a reducing agent provides essential rise of ecological efficiency of this technology.

The technical solutions which are suggested by the authors present an extraordinary practical interest from the point of view of involvement in processing of fine-dispersed Mn- and Cr-containing wastes from beneficiation of corresponding ores, as well as other kinds of multi-component raw materials in order to extract valuable metals from them.

The aim of this research is development of the technique for preparation of granulated pellets from a complex system (single-component charge), composed on the base of manganese wastes. To achieve this aim, the following problems were solved step-by-step: the experimental air-gravitation unit for beneficiation of manganese wastes was developed; the new technique for common calculation of a single-component charge and preparation of granulated pellets was displayed on the base of dosing of mass relationships in such components of this unit as manganese wastes, rolling scale and solid carbon. Realization of such mechanism and technological procedure of preparation of granulated pellets allows obtaining high-quality ferromanganese from industrial wastes during their consequent processing.

The research methods

Manganese wastes of Dzhezdinsky mining and concentrating works as well as rolling scale from Arcelor Mittal steel production, charcoal obtained via pyrolysis of biological wastes (starting from weed plants to agricultural wastes) were used as initial materials for manufacture of a single-component charge.

Charcoal consumption per unit of charge was calculated on the base of stoichiometry of reaction for reduction of Fe and Mn oxides by carbon. Introduction of carbon stoichiometric consumption without excess in charge composition is considered as a determining factor in the process of organization of such experiments.

This work suggested the method of charge preparation including presence of Fe and Mn oxides as well as solid carbon in a one particle in dispersed state. In general, the obtained coal-containing granulated pellets present a single-component charge. Consequent processing of prepared single-component charge in a reduction furnace provides complete Fe and Mn reduction by solid carbon and obtaining of high-quality ferromanganese.

Elemental compositions of the initial components of charge and obtained products was determined using mass spectrometer Agilent 7700 Series ICP-MS (USA) with inductively coupled plasma and chemical analysis.

X-ray phase analysis of samples was carried out using the sensor D8 Advance (Bruker AXS GmbH), α-Cu, with voltage in X-ray pipe 40/40. Data processing from obtained diffraction patterns and calculation of interplane spacing was conducted using EVA software. Analysis of samples and search of phases was implemented using Search/match program and ASTM cards data base. The error of semi-quantitative analysis is ± 5 %.

Experimental equipment and conducted researches

To conduct experiments on beneficiation of manganese wastes, the pilot unit for air-gravitation beneficiation was developed, its general scheme is presented in the **Fig. 1**.

The unit consists of metallic cylinder shell *1*, its lower part through mesh partition *2* is connected with air blowing chamber *3*, which is equipped by branch pipe *4* for injection of air flow. The upper part of the shell is followed by dome *5* presented by truncated cone, which is connected from the top with gas flowing system *6*. The feeding unit is mounted in the surrounding gas offtake pipe, along the shell axis. This unit contains cylinder branch pipe *7*, sliding gate valve *8* and feed hopper *9*. This gas offtake pipe is directed below and enters the dust-collecting unit *10*, which is connected in its upper cone part with the gas offtake pipe of processed air *11*. The branch pipe *12* for discharge of collected dust is mounted under the dust-collecting unit.

Product obtained after air-gravitation beneficiation was used for preparation of single-component charge.

The essence of conducted experiments was as follows. Initial material was previously crushed in the jaw crusher reaching fraction less than 2.0 mm and then was charges by portions in the air-gravitation beneficiation unit through the feed hopper *9*. Then the sliding gate valve *8* was opened and loaded material portion was passed down. Material was accumulated on the mesh partition *2*. When the shell was filled up to middle of its height, loading was stopped via closing of the sliding gate valve *8*. Then compressor was switched on and compressed air was introduced in the air blowing chamber *3* through the branch pipe *4*. Air flow filtration was carried out through the material layer above the mesh *2*. Technological procedures and optimal parameters of the unit (such as air consumption, hovering velocity of particles, time of particle presence in the working space) were determined experimentally.

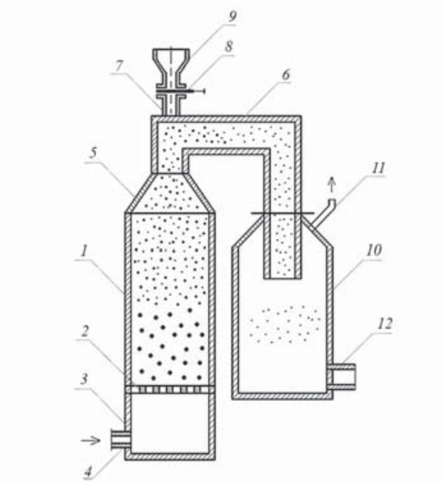

Fig. 1. Pilot unit for air-gravitation beneficiation of manganese wastes:
1 – cylinder shell; *2* – mesh partition; *3* – air blowing chamber; *4* – branch pipe; *5* – truncated cone dome; *6* – gas flowing system; *7* – cylinder branch pipe; *8* – sliding gate valve; *9* – feed hopper; *10* – dust-collecting unit; *11* – gas offtake pipe; *12* – branch pipe for dust discharge

In order to provide efficient physical-chemical interaction between charge components (beneficiated manganese product, rolling scale, charcoal), each of them was subjected to preliminary crushing reaching fraction 1.0 mm in the electric vibration crusher, which is presented in the **Fig. 2**.

Fig. 2. General view the electric vibration crusher

Granulated pellets were prepared from single-component charge using special laboratorial equipment (**Fig. 3**).

Granulation was carried out in the plate granulator (see Fig. 3, *a*). Molasses solution was used as a binding component. Granulation process was conducted as follows. Molasses solution with water (3 % of molasses for a mix of wet (6 %) material) was introduced on dry mass. Obtained and graded granulated material of 8.0–20.0 fraction was subjected to drying at 400 °C in a drying durance (see Fig. 3, *b*). Solid carbon in composition of the complex mix in the air atmosphere is not oxidized practically and does not interact with Fe and Mn oxides at preset temperature. Pellets which were obtained after during (see Fig. 3, *c*) are characterized by high mechanical strength properties; it is very important for their transportation, loading and consequent reducing roasting. Chemical composition of obtained pellets was identical with chemical composition of the single-component charge.

Results and discussion

Elemental and phase composition of manganese-containing wastes from Dzhezdinsky mining and concentrating works are shown in the **Tables 1** and **2**.

It can be seen from the above-presented results, that manganese-containing wastes are characterized by low content of Fe and Mn oxides – 6.0–7.5 % and 15.0–20 %, respectively.

In order to provide beneficiation of the initial material, the experiments with use of the air-gravitation unit were carried out in this research (see Fig. 1). Technological parameters of the unit, providing obtaining of product with its beneficiation by Mn and Fe, were determined in the following way. The dense layer of material was processed until its boiling state, i.e. to the first critical velocity, via adjusting of air flow. The task was to provide fine adjustment of air flow velocity to the second critical velocity, in relation to fine particles with specific volumetric mass to $\gamma_{vol} = 2.2–2.5$ g/cm^3. SiO_2 and Al_2O_3, which are contained in the mass pf prepared fine wastes, can be related to such particles. Hovering velocity, i.e. removal velocity of particles with preset specific mass, was determined via the formula:

a *b* *c*

Fig. 3. Laboratorial equipment: *a* – plate granulator; *b* – drying furnace SNOL; *c* – pellets

Table 1. **Elemental analysis of manganese wastes**

Elements	Content of components, % (mass.)	Elements	Content of components, % (mass.)
O	44.64	K	0.258
Mn	16.32	Ca	16.631
Fe	4.68	Ti	0.167
Na	0.28	As	0.04
Mg	0.831	Cu	0.012
Al	1.38	Zn	0.072
Si	7.04	Pb	0.244
P	0.022	Sr	0.133
Cl	0.038	Ba	6.521
S	0.682	Pb	0.002

Table 2. **Results of semi-quantitative analysis of waste**

Minerals	Formula	Content, %
Calcite	$Ca(CO_3)$	41.2
Quartz, syn	SiO_2	13.0
Braunite-1Q, syn	$Mn_7O_8(SiO_4)$	10.1
Bixbyite, ferrian	$FeMnO_3$	9.9
Baryte	$BaSO_4$	6.6
Pigeonite	$Mg_{0.69}Fe_{0.23}Ca_{0.08}SiO_3$	6.0
Dickite-2M1	$Al_2Si_2O_5(OH)_4$	5.6
Hematite, syn	$Fe_{1.957}O_3$	4.6
Iron Manganese	Fe_3Mn_7	3.0

$$\omega_h = \sqrt{\frac{g \cdot d_r}{\psi \cdot \gamma_{\text{vol}}}}, \text{ m/s} \qquad (1)$$

where d_r — diameter of particles, m;
ψ — coefficient of material layer resistance;
γ_{vol} — volumetric mass of particles, kg/m³.

Substituting the corresponding physical parameters of fine-dispersed in the equation (1), the values of hovering velocity of particles were determined.

The above-mentioned values of hovering velocity of particles were set via adjusting of consumption of compressed air, which was introduced in the unit via the branch pipe 4. Air flow velocity on free cross section of a shell was determined from the following relationship:

$$w_{\text{af}} = \frac{4 \cdot V_2}{60 \pi D^2}, \text{ m/s} \qquad (2)$$

where w_{af} — compressed air consumption, m³/min;
D — diameter of cylinder shell, m;
60 — converting of 1 min in s.

Half of the shell operating space was filled by dense layer of bulky material. Porosity (ε) — is a free space between particles in the dense layer, it was equal to 0.3. When the dense layer reaches boiling state, porosity value increases up to the values 0.5–0.6. Based on these data, compressed air consumption in dependence on hovering velocity of particles was determined by the following equation:

$$V_h = 15 \cdot \pi D^2 \cdot \varepsilon \cdot w_h, \text{ m}^3\text{/min} \qquad (3)$$

where ε — porosity in the boiling layer;
w_h — hovering velocity of particles.

Substituting the value of hovering velocity from the equation (1) to the equation (3), we adjusted air flow consumption with the equation:

$$V_h = 15 \cdot \pi D^2 \cdot \varepsilon \cdot \sqrt{\frac{4 \cdot V_h}{60 \pi D^2}}, \text{ m}^3\text{/min.} \qquad (4)$$

When establishing the calculated consumption of compressed air through the mesh partition, we observed carry-over of fine particles. The procedure of hovering of particles in the layer was held during 15–20 min. In this way 4 experiments were conducted. Mass of loaded fine wastes made 1 kg. Residual beneficiated products, with their mass varying within the range 0.78–0.80 kg, were obtained after gravitation processing in a boiling layer. Chemical composition of the obtained samples is presented in the **Table 3**.

Slight deviations in content of the components (+/- 0.8 % abs.) in the obtained samples 1–4 (see Table 3) show on the stable operating procedure of the unit at the preset parameters (air consumption, hovering velocity of particles in the boiling layer etc.) and obtaining of beneficiated product with stable composition. The obtained pilot samples of manganese wastes with increased Mn content were used for preparation of a complex Fe-Mn-C-containing single-component charge.

It can be seen from the Table 3, that Fe content in the obtained samples is low (10–11 %), what is insufficient for preparation of the final single-component charge. To prepare

Table 3. **Chemical composition of the samples of manganese wastes after gravitation beneficiation**

Obtained products	Chemical composition, % (mass.)							
	Fe_{tot}	Mn	SiO_2	Al_2O_3	CaO	MgO	S	P
Sample 1	9.82	27.90	34.30	4.33	1.82	0.62	0.22	0.82
Sample 2	10.11	28.12	33.12	4.26	1.81	0.58	0.21	0.81
Sample 3	10.56	28.53	32.20	4.21	1.80	0.56	0.20	0.79
Sample 4	10.62	28.65	31.40	4.20	1.76	0.55	0.20	0.80

Table 4. **Average-weighted chemical composition of the mix**

Charge name	Chemical composition, % (mass.)							
	Fe_{tot}	Mn	SiO_2	Al_2O_3	CaO	MgO	P	S
Scale/Mn wastes 0.1/0.9	15.85	25.56	29.02	3.78	1.60	0.49	0.19	0.77

the charge with optimal composition, rolling scale containing up to 67 % of Fe was introduced additionally. The mix was prepared, consisting of rolling scale and manganese wastes, with their mass parts relation 0.1/0/9. The average-weighted chemical composition of the prepared mix is presented in the **Table 4**.

Coal was introduced in charge composition as a reducing agent. The choice was made based on the following arguments. Reduction of manganese oxides to metallic state using hot reducing gas (HRG) is not achieved via conventional processes, because dissociation elasticity of MnO and Cr_2O_3 is lower by many times in comparison with dissociation degree of CO_2 and H_2O gases. As soon as temperature rises, dissociation of CO_2 and H_2O gases occurs earlier than for MnO oxide. At the same time, a gas phase becomes not reducing atmosphere, but on the contrary oxidizing one relating to manganese oxides. That's why HRG has not sufficient reducing potential relating to manganese oxides, to subject them to metallization. In this connection, choice of coal as the main reducing agent, having high reducing potential, seems to be rather correct.

Wide use of coal in solid phase systems was hindered for a long time by theoretical regulations about adsorption-autocatalyst mechanism of reducing processes, which consider interaction reactions between solid carbon and solid metal oxides via so-called contact-diffusive mechanism. As a result of such consideration, reducing and smelting processes in manganese production are practically organized in a liquid phase system. In ore smelting furnaces, molten oxide ore flows around the layers of metallurgical coke at high temperature (1500–2000 °C). In this case reduction of oxides by solid carbon occurs due to development of reacting surface. Opinion on interaction between solid carbon and metal oxides only via contact-diffusive mechanism was caused by underestimation of structural and physical-chemical transformations in solid carbon particles during system heating. It was established that system heating is accompanied by splitting of crystal lattice in solid carbon particles both in plane direction and in interplane distances with intensive flow of electrons (electronic emission). The flow of electrons covers not only a point, but the whole surface of solid particles of oxides and provides negative charging of gasified oxygen in oxides, which are also been in excited state during heating. It leads to starting of non-reversible chemical reactions. This concept is tightly connected with the fundamental mechanism of all mass exchange processes — the donor-acceptor mechanism. HRG molecules of CO and H_2 are considered as a weak electronic donor, thereby they have restricted reducing potential. Solid carbon has high electronic potential and is characterized as a strong electronic donor, so it has high reducing potential.

Based on the composition of obtained mix, we took into account that Fe and Mn oxides are reducing by solid carbon. The required stoichiometric amount of solid carbon was calculated in accordance with reaction of direct metal reduction from their oxides; then this amount was added in the prepared mix. In our case, carbon consumption (GC) for reduction of Fe and Mn oxides made 0.2068 kg, what is equivalent to 0.211 kg of charcoal per 1 kg of charge.

Thus, finally prepared complex single-component charge presented the mix of Fe-, Mn- and C-containing charge materials.

Chemical composition of initial components, which were used for charge preparation are presented in the **Table 5**.

The obtained dispersed complex mix contained all required chemical components for realization of reducing processes. The average-weighted chemical composition of the prepared complex mix is presented in the **Table 6**.

At the next stage it was necessary to solve the problem of choosing the method for supply of heat amount, which is sufficient to provide the system heating and intensive passing of reducing reactions for Fe and Mn oxides. Direct reduction

Table 5. **Chemical composition of initial charge components**

Material	Chemical composition, % (mass.)									
	Fe	FeO	Mn	SiO_2	Al_2O_3	CaO	MgO	S	P	C
Manganese wastes	11.21	—	28.35	32.25	4.20	1.78	0.55	0.8	0.21	—
Rolling scale	66.67	32.21	0.44	—	—	—	—	0.01	0.50	—
Charcoal	—	—	—	1.02	0.26	—	—	—	0.37	98.0

Table 6. **Average-weighted chemical composition of the complex mix**

Elements	Fe	Mn	SiO_2	Al_2O_3	CaO	MgO	S	P	C
Content, % (mass.)	11.94	18.67	20.46	4.21	17.10	0.73	0.58	0.02	11.40

by solid carbon is accompanied by endothermic heat effect. To provide initiation of direct reduction reaction of oxides, it is required to heat the system up to the temperature 700—1200 °C. Heat supply to a dispersed complex system leads at first to intensive emission of CO and CO_2 gases as reaction products and exhausting of essential part of fine-dispersed particles together with gas flow. Choosing the thermal energy source is also very important. In this case, it is expedient to base on a fuel source of thermal energy, which is cheaper than heat extracted from electric power by 4 times. The conventional technology worldwide is based on processing of manganese ores in electric thermal furnaces.

Supply of heat energy from a fuel power source to a complex system is realized by fuel burning, where high temperature gas flow is a heat carrier. To provide heat exchange between gas flow and complex carbon-containing system, granulated pellets are prepared from a single-component charge. These pellets are used for conduction of the following researches in the field of their low-temperature roasting and reduction smelting, in order to obtain high-quality ferromanganese.

Conclusion

This work describes the technology for processing of fine wastes of manganese ores, which applies the principle of single-component charge preparation on the base of beneficiated manganese-containing product, using coal as carbon-containing reducing agent.

Based on this position, three techniques were developed: the technique for preparation of separate charge components (manganese-containing wastes, carbon-containing reducing agent, rolling scale) via their crushing and mixing; the united calculating technique for preparation of the complex system (single-component charge) via dosed selection of charge components; the technique for preparation of granulated pellets, which are ready for solid phase reduction of metals from their oxides by solid carbon.

On the contrary to the conventional technique for metals reduction from agglomerated raw materials, produced via sintering using HRG technology, this research presents the technique for preparation of granulated pellets. It is based not on agglomeration, but opposite on crushing of manganese-containing wastes and added separate charge components, such as solid carbon-containing reducing agent and rolling scale.

Experimental part of the research presents construction of the new air-gravitation unit and the technique for beneficiation of manganese wastes from Dzhezdinsky mining and concentrating works. As a result of conducted technological experiments, a beneficiated product was obtained and manganese content in this product was increased from 19 to 28.35 %. Based on this new technique, a single-component charge containing 24.38 % of manganese and 16.72 % of carbon was prepared. Relation of manganese wastes to rolling scale makes 0.9/0.1.

Granulated pellets, which are identical to a single-component charge by manganese and carbon content, were prepared. They will be used in consequent low-temperature roasting and reduction smelting, in order to obtain high-quality ferromanganese.

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