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OLD IRON-BEARING WASTE TREATMENT TECHNOLOGY*

The article shows a possible way to solve the problem of alternative technology for treatment of solid waste and tailings in terms of the Kamysh-Burun Iron Ore Industrial Plant in the Republic of Crimea. Until 1993 the Plant operated the Kamysh-Burun and Eltigen deposits of ore considered as a rebellious high-phosphorus material. Accordingly, solid waste and tailings are also high-phosphorus and high-sulfuric.

To date the Kamysh-Burun Plant is closed. The former production infrastructure is put out of operation, the territory is polluted by toxic waste and represents an unauthorized disposal site in point of fact. The total area operated by the Kamysh-Burun Plant and taken out of service makes 8.3 km^2 (without open pit mines).

Experts of the Mining College, NUST MISIS, have developed the treatment technology for iron-bearing mining waste. This technology, with addition of charge-adjusting rich high-phosphorus iron-bearing waste material, allows production of technogenic material with iron content higher than 40%, which is a criterion of economically efficient metallurgical Romelt processing and detoxification of waste suitable for industrial use.

The target waste is represented by the Kamysh-Burun dump (average iron content 52%) with uncertain reserves and by the Upper Churbash tailings pond (average iron content 30.85–44.3%) with estimated reserves of 24.2 Mt.

The top layer of the tailings pond, due to natural atmospheric metamorphism, has low content of iron, phosphorus and sulfur. The bottom layers preserve much phosphorus. All layers of the tailings pond and dump are processible with magnetic separation, and iron content of concentrate, depending on depth of waste layer occurrence, has a wide range from 10 to 64%. Concentrates produced from the low-iron top layer material of the tailings pond contain no toxic admixtures of sulfur or phosphorus.

Mixing of the magnetic separation concentrates produced from materials sampled at different industrial infrastructure sites makes it possible to reduce sulfur and phosphorus content of the composite concentrate nearly 2 times.

The processing rejects contain 30–40% of iron. Based on the analysis of properties of these rejects, it is concluded that this material is a man-made iron-oxide pigment. Thus, the alternative treatment technology for the Kamysh-Burun waste enables production of:

—composite concentrate suitable for metallurgical treatment by the Romelt process for making cast iron and slug for structural application;

—rejects being a man-made iron-oxide pigment having similar properties with natural yellow earth and saturnine red.

Key words: *oolite ore, tailings, mineral composition, technological study, magnetic separation, ultrasonic treatment, iron-bearing product, pig iron, iron-oxide pigment*

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Introduction

It is needless to say that the problem of sound management and processing of accumulated mining and metallurgical industry waste is highly urgent. Being global sources of environmental pollution, mining and metallurgical waste are at the same time promising huge technogenic reserves of many valuable components. The identified problem is of special concern at the present time as natural reserves are being inexorably depleted. The tendency of mineral wealth decrement and iron ore deterioration actualizes treatment of mining waste with a view to using the resultant iron-bearing product in the iron and steel industry [1–6]. Sometimes recycling of waste can be profitable owing to integrated processing with manufacturing extra marketable by-products, for instance, for the needs of construction industry [7–9].

Currently mining and processing plants treating one or a few mineral deposits use classical magnetic processes with magnetite recovery to concentrate. Nonmagnetic tailings, containing hematite, hydrogoethite and other natural and technogenic iron pockets, often hold much more iron in terms of element than ore fed to a processing factory.

Tailings contain unrecovered magnetic iron that is advisable to extract using other, innovative methods. Nonmagnetic iron in tailings is extremely difficult. Production of concentrate with the iron content not less than 60% to be suitable for metallurgical processing overwhelmingly fails.

One of examples of the former USSR iron industry waste is solid waste dumps and tailings ponds at the Kamysh-Burun Iron Ore Industrial Plant (at Kerch town, now the Republic of Crimea) closed in 1993. For more than 50 years of operation, the Plant has accumulated above 25 Mt of waste after mining and processing of bog iron ore in the Kerch Iron Ore Field (Kamysh-Burun, Eltigen-Ortel and other deposits). The average thickness of pile layer is 9.0 m. The chemical composition of tailings is: Fe — 30.85–44.3%; FeO — 0.05%; Ca — 0.3–0.5%; Mg — 1.0%; Al — 0.3–0.5%; As — 0.03–0.001%; Mn — 1.0%; Ti — 0.5–1.0%; SiO_2 — 7.19–19.0%; Al_2O_3 — 0.13%; TiO_2 — 0.244%; V_2O_5 — 0.073%; Na_2O+K_2O — 3.10%; CaO — 1.83%, MgO — 1.38%.

At the Kamysh-Burun deposit, commercial-value tobacco ore and limonite were extracted using the open pit mining method. The ore featured increased contents of moisture (20–30% absorbed moisture and 10% hydrate moisture) as well as manganese (0.8–4.3%), phosphorus (0.6–1.1%) and arsenic (0.7–1.3%), and insignificant content of vanadium. Iron con-

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tent was approximately 30–40% (basic ore mineral was hydrogoethite). Such ore was ranked as rebellious, especially tobacco ore which made 60% of all reserves.

For enrichment brown-iron at the Kamysh-Burun processing plant used wash-gravitational technology in 1932 till 1993. The feature of the Kerch ore was a considerable content of iron-chlorite ore (cement), together with hydrogoethite, with the iron content of 28–35%. There was insignificant quantity of free minerals (quartz, calcite, etc.) in the ore. For this reason, the task of the Kerch ore benification was to separate hydrohematite oolite into concentrate and to remove lean chloritic oolite and chlorite cement to tailings.

Easy brown ore was treated by wash-gravitation technology; difficult (tobacco) ore making the major reserves of the Kerch Basin was subjected to gravity settling.

The wash-gravity and gravity settling concentrates were agglomerated. Fluxed agglomerate was mostly sent to the Azov-Steel plant (Mariupol, Ukraine). Later on, treatment of agglomerate was assumed inexpedient due to high content of phosphorus. The Kamysh-Burun Iron Ore Industrial Plant carried out ore mining and processing until 1993. Currently, the production capacities are out of service. Waste treatment is only possible with new technologies of processing and concentrate production at the minimal permissible mass fraction of toxic components, first of all, arsenic and phosphorus.

With this end in view, it is required to study occurrence forms of valuable components (first of all, iron) and toxic contaminants in waste, estimate their process properties and to identify the most efficient extraction methods [10].

With due regard to material constitution of waste, a high-quality iron product is hardly expected. Scientific research and pilot tests of flotation or agglomeration in reducing environment, etc. are currently undertaken [11–15]. All methods have right to exist but the cost of the concentrate is essentially higher than the cost of magnetic concentrate of traditional processing techniques. Efficient metallurgical processing of low-grade iron concentrate is possible through application of direct smelting technologies, for instance, the Romelt process that enables pig iron making from low-grade iron ore. Slugs can be used in road building, and electric energy can be used for the self-supply of the metallurgical plant [16, 17].

Results and discussion

Two samples of waste were tested: from the Upper Churbash tailings storage (UC) and from the Kamush-Burun Iron Ore Plant dump (KB). Mineral composition of the samples was analyzed using optical and precision microscopy with microprobe analysis of manufactured polished briquettes.

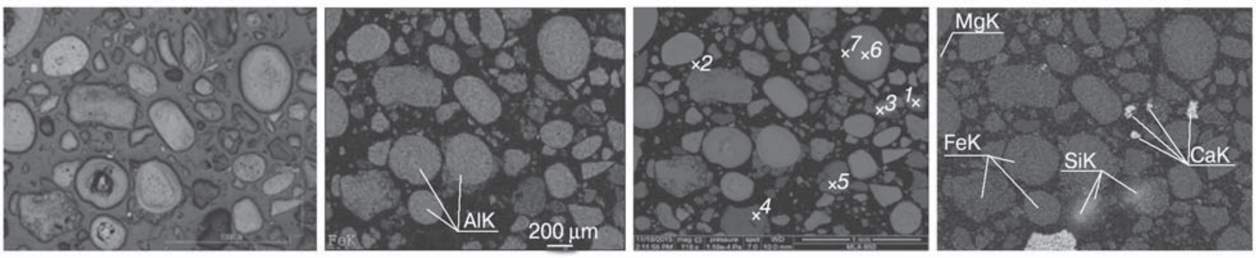
Table 1. Mineral composition of test samples

Component	Content, %	
	UC sample	KB sample
Hydrogoethite, hematite	0.11	34.06
Superparamagnetic hydrogoethite	6.93	31.02
Leptochlorite	17.99	10.47
Manganese hydroxide	Traces	1.62
Rare earth phosphate, apatite	0.40	4.40
Feldspar (potash feldspar, albite, plagioclase)	19.04	4.90
Mica	2.60	3.34
Carbonate (calcite, dolomite)	10.86	2.02
Clayey minerals	24.20	0.16
Quartz	17.87	7.71
Zircon	Traces	Traces
Pyrite	–	0.30
Total	100.00	100.00

The UC sample is a yellow-grey granular material composed of sand-size finely dispersed particles of micaseous clay material and hydrogoethite. Fragments 1–3 mm in size are represented by quartz, feldspar, carbonate, goethite and microscopic size aggregates with silica–hydrogoethite–clayey–micaseous cement.

The KB sample is a dark-brown and reddish-brown material composed of grains and fines. Grains are represented by separate oolites of goethite-hydrogoethite composition, fragments of quarts, feldspar and ancient shells (carbonate) as well as aggregates of these materials and oolites cemented by a leptochlorite-hydrogoethite substrate. Essential quantity of phosphorus-bearing phases represented by rare earth phosphate and apatite is observed (**Table 1**). The microprobe analysis also reveals phosphorus in hydrogoethite.

The main metallic mineral is hydrogoethite present in two forms: oolites and finely dispersed nodules. Oolites are typical for the KB sample, finely dispersed aggregates occur in a great measure in both samples. Ball-like and ellipsoid-shaped oolites have sizes from 0.1 to 1.0 mm, predominantly 0.2–04 mm. Microscopically, they have concentrically zonal structure and have quartz, clayey-chlorite and, seldom, feldspar fragments in the center (**Fig. 1**). Fine-dispersed hydrogeothite traced by the Mossbauer spectroscopy is present in aggregates with hydromica, clayey minerals and leptochlorite.



a *b* *c* *d*

Fig. 1. Mineral oolites and fragments in the KB sample:
***a* — in reflected light; *b* — subimages of in characteristic radiation of iron; *c* — back scattered electrons; *d* — composite display in characteristic rays. Figures in *c* mark the spectrum taking points**

Oolites and pseudo-oolites are cemented by leptochlorite–hydromica–hydrogoethite–clayey material. The mineral elements in the cement form finely dispersed aggregates where individual minerals are nonvisualizable. Aside from ball-like and ellipsoid-shaped nodules of iron hydroxide, there are angular loose fragments of fine-dispersed aggregates of iron hydroxide and clayey material with numerous small and microscopic nodules of quartz, chlorite and feldspar, as well as aggregates of oolites and pseudo-oolites in cemented matrix of different composition.

The UC samples consist of aggregates having uniform structure and composition, and are composed of finely dispersed loose micaseous–clayey cement binding differently grained particles of rock-forming minerals (mostly quartz and feldspar) and, more seldom, fragments and small oolites of hydrogoethite (**Fig. 2**). The composition uniformity is best of all displayed by the electron microscopy. Almost all aggregates are dark-shaded, highly porous and microjointy, which is indirectly reflective of their considerable content of hydroxide-bearing mineral phases.

By the microprobe analysis data, the dominating component in the composition of hydrogoethite is iron: its content varies from 53.04 to 62.95% in oolites and fine-crystalline grains and from 33.22 to 48.77% in finely dispersed aggregates. Constant admixtures in hydrogoethite are phosphorus the content of which exceeds 1% in most grains and calcite at the content less than 1%.

The chemistry of the cement in the UC and KB samples is different: the UC sample cement features higher contents of silica, aluminium, magnesium and potassium oxides from the compositions of lipochlorite, hydromica and clayey minerals, while the KB sample cement has higher content of iron oxide as a consequence of higher content of fine-dispersed hydrogoethite.

The grain-size analysis shows more uniform distribution of different size fractions in the UC sample as against the KB sample. The maximum mass fraction falls at the size of 0.4+0.071 m in both samples, which makes 31% of total material in the UC sample and approximately 55% of total material in the KB sample. The UC sample has high content of fractions larger than 0.8 mm — round 40%.

In the UC sample, the iron content increases from 6.4 to 10.2% as the grain size grows, reaches the maximum at the grains size of 3.7 mm and the drops. The highest iron recovery observed in the size range –0.4+0.071 mm with the maximum value at the size of 0.3 mm drops in the size range 1+0.5 mm with the minimum at 0.65 mm.

Table 2. Chemical composition of the original UC and KB samples

Size grades	US sample		KB sample	
	Yield, %	Fe content, %	Yield, %	Fe content, %
+5.00	9.31	7.55	2.63	54.60
–5+2.5	9.84	10.20	5.80	51.00
–2.5+1.6	7.87	8.11	0.94	56.00
–1.6+0.8	11.53	8.42	8.84	58.50
–0.8+0.63	3.56	7.66	3.07	60.80
–0.63+0.56	3.46	7.51	3.77	58.70
–0.56+0.4	6.60	7.41	13.04	61.10
–0.4+0.071	31.32	7.46	54.51	52.60
–0.071	16.51	6.39	7.40	51.10
Total	100.00	8.06	100.00	54.59

The recovery of iron with the particles larger than 1 mm insignificantly fluctuates in the range 8–14%. The pay size grade in the UC sample is 0.4 + 0.1 mm (Table 1).

In the KB sample, iron size distribution is different. Up to the size of –0.4 mm, the content of iron is the least while the recovery is the highest and reaches 55%. Under a jump in the iron content from 50 to 61% in the size grade –0.8+0.4 mm, the recovery of iron drops nearly to zero and then, with an increase in size, remains low and falls short of 10% in some size grades. The yield and recovery indexes totally coincide (**Table 2**).

Most probably, iron in waste of the KB sample is in bound state in large size grades which are rebellious. The same dependence is observed in the UC sample though it is not so pronounced. Consequently, recoverable iron is expected in the size grade –0.4 mm.

The processability tests of waste sampled in the top and bottom layers of tailings pond and in dumps were carried out in flotation, magnetic separation, roasting–magnetic separation and ultrasonic treatment. The outcome of the experimentation is described below [10, 17].

Magnetic fractioning on a tube magnetic analyzer displays high magnetic properties of the KB sample. If necessary, iron content of the concentrate can be raised to 65% by magnetic separation in weak field. At the same time, the magnetic analysis of the UC sample yields insufficient result, which implies that even strong-field magnetic separation, alone, fails to produce satisfactory technological parameters. In this case, the latter can be obtained by applying roasting to convert iron minerals to a more magnetic phase with the further high-gradient magnetic separation of roasting products.

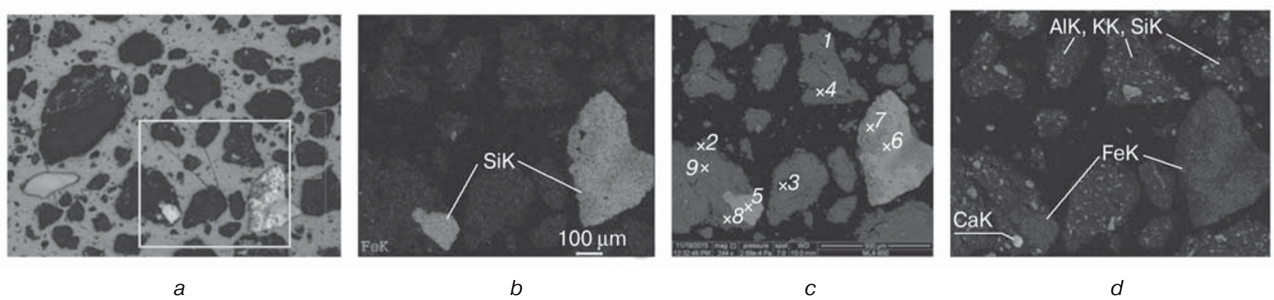
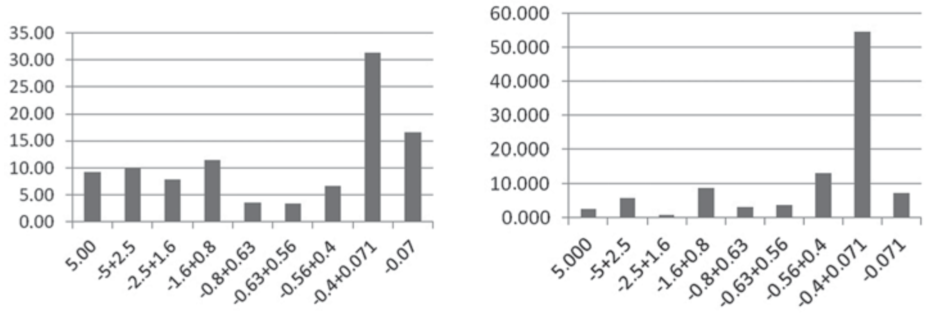


Fig. 2. Mineral aggregates in the UC sample:
***a* — in reflected light; *b* — subimages of in characteristic radiation of iron; *c* — back scattered electrons; *d* — composite display in characteristic rays. Figures in *c* mark the spectrum taking points**



a *b*

Fig. 3. Grain-size analysis results:
***a* — UC sample; *b* — KB sample**

Blending of materials sampled in the top and bottom layers of the Upper Churbash tailings pond allows obtaining magnetic concentrate with the yield of 16.2% with Fe_2O_3 content of 40.9% at the recovery of 16.4%. The Mossbauer spectroscopy shows that the materials from top and bottom layers of the tailings pond and the KB dump contain predominantly superparamagnetic iron phase and insignificant ferrous silicates and micron- and submicron-size hydrogoethite.

The top layer material to 87% is composed of superparamagnetic hydrogoethite with the total iron content of 8.54%; the bottom layer material contains superparamagnetic hydrogoethite (round 45%), finely dispersed hydrogoethite (Fe^{+3}) and ferrous silicate.

The doublets in the spectra of the samples, which are characterized as superparamagnetic hydrogoethite, make it possible to relate the material with ferrous coloring agents analogous to iron-oxide pigmentary ore from the South Ural deposits as a yellow or brown color raw material. The electron microscopy analysis of these ores displays prismatic, tabular, flaky and needle-like iron hydroxides, which typical for both synthetic and natural iron oxide pigments.

The sextet spectrum is recorded for the dump sample and is responsible for the low-ordered magnetic phase FeO(OH). Spectra of the doublets in both samples can be related either with the paramagnetic phase of nanodispersed hydrogoethite or with Fe^{+2} ions in the structure of silicates (**Fig. 3**).

Despite the nano- and submicron structure of iron-bearing waste, the magnetic fraction was successfully extracted by high-level poly-gradient separation and in magnetic chute, which supposes the use of sufficiently simple equipment later on.

The magnetic concentration rejects contain mostly silicates and aluminium silicates with nonmagnetic admixtures. Their iron content is less than 40%. It is impossible to state that the magnetic component is totally removed from them as it is rather difficult to dissociate magnetic and nonmagnetic species in such material.

The upper layer in the Upper Churbash tailings pond is characterized by the low content of phosphorus. The magnetic treatment of this sample (multi-stage high-intensity poly-gradient magnetic separation) increases the iron content to 20–21%, which enables using this concentrate as additive to melting batch together with the concentrate of processing of the bottom layer material from the tailings pond.

The magnetic treatment of the bottom layer material from the Upper Churbash tailings pond produces concentrates with the iron content to 58% and phosphorus content of 2%. It is possible to reduce phosphorus content by blending the concentrates obtained by processing the upper and bottom layer materials sampled in the tailings pond. The blend at a ratio of 1 : 1 allows production of the concentrate with the iron content of 40% and phosphorus content of 1%. Sulfur is not detected in the tailings pond sample. Thus, old tailings can be subjected to the Romelt process treatment with the preliminary magnetic separation and discharge of rejects with the permissible iron content of less than 5%.

Treatment of iron-bearing material sampled from the Kamysh-Burun Iron Ore Plant waste dump by traditional magnetic separation is inexpedient. Recovery of size –0.4 mm at the yield of 63% allows a product with the iron content of 58% and phosphorus content of 2%, which is 25% lower than in the total; volume of the material. In this manner, to prepare the KB sample for melting by the Romelt process, it is required to apply wash-gravitation technology with the recovery of the size –0.4 mm and then blending for dephosphorization. Melting will produce metal and slag usable in construction. Radioactive and other toxic elements are not detected in the Kamysh-Burun waste.

The comparison of the Mossbauer spectroscopy data on the tailings sample with the low iron content and the dump sample with the high iron content shows that the total content of superparamagnetic phase is not higher than 30% of total iron content. The magnetic fraction yield is round 16%.

Magnetic component of the magnetic concentration rejects was additionally extracted on magnetic table. The process of dissociation of magnetic and nonmagnetic species was stimulated by means of ultrasonic treatment of the pulp.

Ultrasound was successfully tested in stimulation of jigging of the Kerch tobacco ore. Under short-term treatment, cement of tobacco ore is mostly dispergated in acoustic field, while stronger ooolites remain unbroken. The selective dissociation of minerals of all size grades is efficient under ultrasound frequency of 18–22 kHz and intensity of 3–4 W/cm^2. An increase in the intensity to 10 W/cm^2 offers no higher velocity of dispergation [17].

Ultrasonic disintegration of mineral suspensions is assumed as one of the promising processing methods. Researches show that disintegration takes place owing to cavitation erosion. Destructive effect of ultrasound on hard minerals in water is governed by many factors, which explains selectivity of ultrasonic treatment in disintegration of mineral suspensions.

The tests were conducted on ultrasonic machine UZP-100, model NO-376, designed by Aleksandra-Plyus. In a test tank 1 dm^3 in capacity, an air dry sample of rejects after magnetic separation of top and bottom layer materials from the Upper Churbash tailings pond and Kamysh-Burun dump was placed. Then, distilled water was added until the tank was filled. In the tank, beneath the meniscus, needle of ultrasonic sensor was placed. The tank was put on a magnetic table assembled from constant magnet disk fastened between two dielectric plates.

Ultrasonic treatment lasted for 5 min and was limited by the sensor temperature. Resonance transducer created a steady beam of ultrasonic waves with downward cone base in the pulp. Test engineers observed intensive stirring of the pulp with particles soaring at a high rate in the tank.

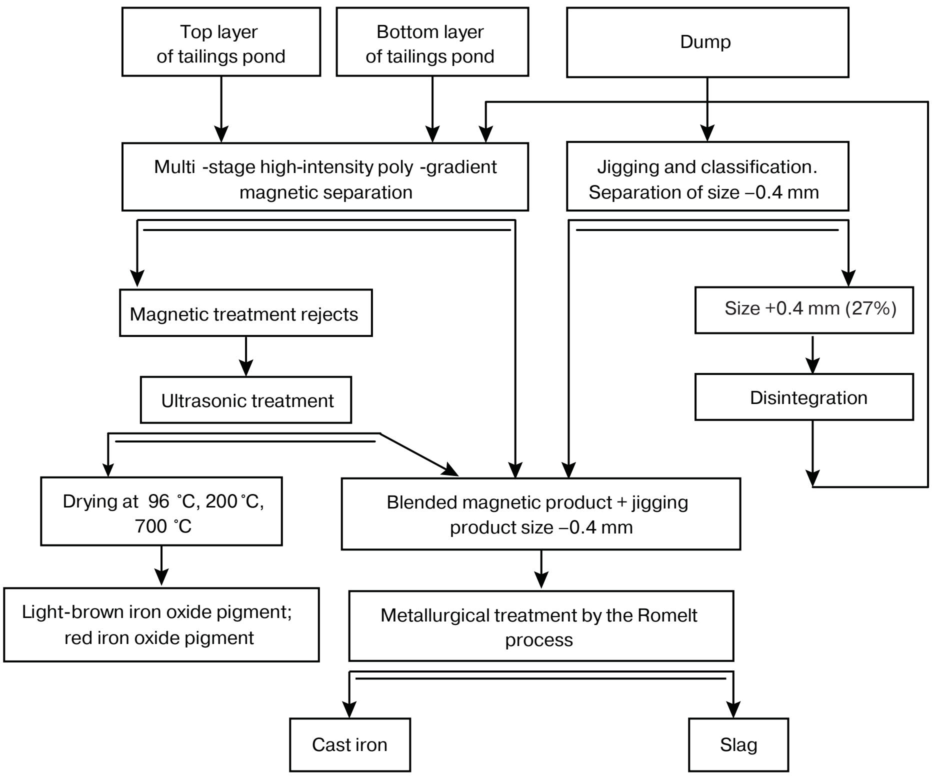


Fig. 4. Process flow diagram for processing of iron-bearing waste of the Kamysh-Burun Iron Ore Industrial Plant

After the ultrasonic treatment, the pulp was settled, and nonmagnetic large heavy particles precipitated by gravity while magnetic fines deposited in magnetic field of the table. As a result, a pulp of a finely dispersed nonmagnetic ferrous product was obtained. The rate of precipitation of the main part of particles was 4.2 mm/h. No total precipitation until clarification took place.

After settlement for 15 min, the material separated into a dark color precipitate and a rich yellow–brown pulp. The pulp was elutriated, added with distilled water and again subjected to ultrasonic treatment on the magnetic table. The cycle was repeated until no yellow–brown pulp appeared. The suspended matter was dried in a baking oven under 96 °C. Higher baking temperature changed the color of the material toward brown.

The dried sediment was sent to the testing laboratory Yaroslavsky Pigment (Yaroslavl).

The chemical analysis of samples before and after the ultrasonic treatment shows the distribution of total, ferric and ferrous iron per processing products. Given the initial total iron content of 34.6%, it grows to 40.8% in the magnetic precipitate and reduces to 26.1% in the dry product. The content of FeO (magnetic material) in the precipitate increases 4 times, from 0.31 to 1.25%. The content of manganese increases in the precipitate and nearly halves from 0.86 to 0.49% in the dry product. Manganese colors the pigment into dark-brown and, thus, its reduction makes yellow richer. Phosphorus concentrates in the magnetic product and decreases in the dry product. Sulfur is detected neither in the original no in the treated samples [10].

The data of the Mossbauer spectroscopy confirm the conclusion that the dry pulp product is a light-brown technogenic iron oxide pigment.

The ultrasonic treatment sink with the iron content increased to 63% is blended with the magnetic product and the dump material jigging product. The resultant iron-bearing material (magnetic and nonmagnetic iron) with the iron content of 40% is sent to metallurgical treatment by the Romelt process.

In this way, as a result of the technological studies, the process flow diagram for processing of old tailings and dump waste of the Kamysh-Burun Iron Ore Plant has been developed (**Fig. 4**). The advantage of this technology is the absence of rejects. As a result, two iron-bearing products and an iron oxide pigment are obtained.

Product no. 1 — composite product of magnetic treatment of the UC and KB samples. Total iron content is 40.4%, phosphorus content on P_2O_5 basis is 1.64%. The minimum limit of the required iron content range for efficient melting by the Romelt process has been reached [16]. Product no. 2 — composite size grade –1.6+0.4 mm after screen analysis of the original air-dry KB sample. For the products, respectively, the basicity is 0.03 and 0.12, the silica module — 3.88 and 4.85. In conformity with the accepted classification, these products are acid (basicity <0.7, which is most often in prac-

a *b* *c*

Fig. 5. The Romelt furnace melting product:
***a* and *b* — product no. 1 and iron cast ingot and slag, respectively; *c* — product no. 2, iron cast ingot**

tice). Experimental melting in a laboratory furnace by the Romelt process produced cast iron and slag suitable for use in construction (**Fig. 5**).

Conclusion

The research has been carried out at an industrial object — Kamysh-Burun Iron Ore Plant put out of operation. The former infrastructure territory accommodates closed processing workshops, waste dump and two Upper Churbash and Lower Churbash tailings ponds. The test samples were taken from the dump, as well as from the top (daylight surface) and bottom layer of the Upper Churbash tailings.

Materials from the top (total iron content 8.54%) and bottom (total iron content 31–38.5%) layers of the tailings pond were subjected to multi-stage high-intensity poly-gradient magnetic separation. This process can be applied to the samples separately, or to a compound of them. The experiments proved the treatment efficiency. The result was a decrease in phosphorus content in the magnetic product and an increase in iron content to 35%.

Washing technology of the waste dump sample allows extraction of an iron-bearing product (weak-magnetic) with iron content to 58%. The lean material larger than 0.4 mm in size is disintegrated and sent to magnetic separation, while the extracted magnetic fraction is blended with the magnetic product obtained after processing of the sample from the top and bottom layers of the tailings pond.

Magnetic separation rejects are subjected to ultrasonic treatment. The extracted pulp is dried under temperatures of 96, 200 or 700 °C. Depending on the drying temperature, pigments obtained can have different colors.

As a result, 2 processing product were obtained:

— composite magnetic product suitable for metallurgical treatment by the Romelt process;

— light-brown iron-oxide pigment.

These products — composite magnetic product and iron-oxide pigment — contain approximately equal iron — 40–41% (the pigment may have lower iron content) but the modes of iron (it is incorrect to speak about mineral composition) are different. The magnetic product mostly contains magnetite and magnetic forms of superparamagnetic phase originating during iron ore treatment and in the course of metamorphic transformations during long-term storage in the tailings pond.

The man-made pigment product contains micron and submicron particles of supermagnetic iron phase and alumosilicate component which is natural for analogous natural pigments. The presented process flow diagram can be considered as a general solution and is to be refined for a specific object.

The magnetic product was melted in a laboratory furnace, and cast iron was experimentally manufactured. The magnetic separation rejects were subjected to ultrasonic treatment on a specially made test bench, which resulted in manufacture of two products: light-brown iron-oxide pigment and magnetic material to be added to the composite magnetic product for melting.

Introduction of the technology to treatment of the Kamysh-Burun Plant waste will enable extra extraction of iron from old dumps and tailings ponds, as well as will essentially mitigate the environmental impact.

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