

Conference Paper

Iron Ore Dephosphorization of Gara Djebilet Deposit By Hydrometallurgical Method

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Abstract

For industrial use, iron-ore concentrates shall correspond to definite requirements regarding both basic substance content – iron as well as phosphorus content. Decreased phosphorus content in iron-ore concentrate can be received by the method of hydrometallurgy (desalinization of phosphorus by mineral acid). On the experimental base of OJSC VNIIMT, a laboratory research was carried out regarding iron ore dephosphorization; as a result maximum possible content of phosphorus was obtained in a final product. The optimal technological parameters of sulphuric acid desalinization were defined that have an effect on quality of phosphorus removal of iron ore magnetite concentrate: size degradation of roasted concentrate; phosphorus removal duration; pulp heating temperature effect; sulphuric acid-specific consumption.

Keywords: desalinization, iron ore, concentrate, pulp, sulphuric acid, phosphorus content, optimal parameters

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1. Introduction

In the Algerian People's Democratic Republic there is iron ore deposit Gara Djebilet, one of the largest deposits in the world. Development of this deposit is suppressed due to high (up to 0.75–1.0%) content of phosphorus in the ore.

Research works on iron ore beneficiation of Gara Djebilet deposit were carried out before many times starting from the 1960s. In the research mainly mechanical methods of beneficiation were used (degradation and subsequent operations of separation of ore constituents from mining waste – magnetic separation, floatation etc.). In this case

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iron ore concentrate could not be obtained with acceptable for subsequent metallurgical process phosphorus content that basically transferred to the concentrate during the beneficiation as presumably it was mainly tied chemically to iron.

For the subsequent use of the iron ore concentrate, for example, in the blast-furnace process phosphorus content is required 0.2% or less, and phosphorus removal in the steelmaking process is an economically expensive procedure. In this regard it is relevant to carry out laboratory research to determine, test and select effective process of the Algerian iron ore processing of Gara Djebilet deposit with obtaining of maximum possible beneficiation of ore on iron content by high-temperature magnetizing roasting and maximum possible reduction of phosphorus content in a final product after the stage of sulphuric acid desalinization.

2. Research Objectives

To achieve objectives of this research work the following tasks shall be completed:

1. To develop technique and equipment for a laboratory experiment on dephosphorization of iron ore concentrate after high-temperature magnetizing roasting by hydrometallurgical processing method.
2. To determine optimal parameters of sulphuric acid desalinization of phosphorus: process duration, sulphuric acid concentration in aqueous solution, ratio of solid substance to liquid phase in the pulp, pulp's temperature.

3. Laboratory Research and Results Analysis

The work on desalinization of preliminary roasted concentrate was carried out in a chemical laboratory of OJSC VNIIMPT [1-4] with using of laboratory stirring shaft DAIHAN-HS-120A (Figure 1) for the pulp's mixing.

Technological stages totally influenced on dephosphorizing of the concentrate are: preliminary high-temperature magnetizing roasting of the material, degradation up to defined by testing optimal size, magnetic separation and subsequent phosphorus desalinization by light aqueous solution of sulphuric acid. Quality of each stage has an effect on dephosphorizing extent of the material in whole.

In the first series of testing influence of size on residual content of phosphorus after dephosphorizing process was studied. Desalinization of the roasted concentrate at temperature of 950°C was made under the following parameters of sulphuric acid

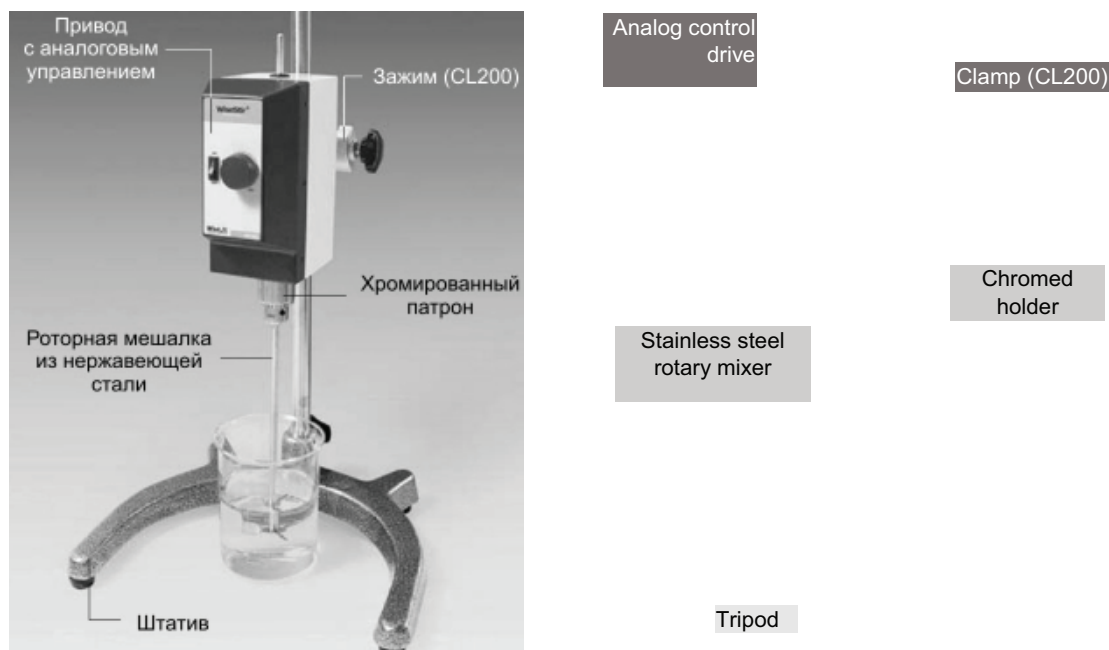


Figure 1: General view of laboratory stirring shaft.

processing: solid-to-liquid ratio – 1:2, sulphuric acid concentration in aqueous solution 20 gr per 100 gr of the concentrate, desalination time 120 minutes, pulp's temperature 65°C. Technological parameters of dephosphorization (temperature, concentration and time) in research works were selected in experimental way of the hydrometallurgical processing of Lisakovsk ores [5, 6] and are overrated on purpose in order to estimate only influence of the material degradation extent before desalinization on residual content of phosphorus in a final product.

By 2–0 mm size of iron ore magnetite concentrate the residual content of phosphorus in the leached concentrate was obtained as 0.45% (experiment 3), in this case phosphorus content in the base ore makes up 0.75%. The material's leaching in 2–0 mm size reduces phosphorus insignificantly. For the leached concentrate of 0.071 mm size the best result with residual content of phosphorus 0.17% is observed.

With decreasing of fraction size of the roasted iron ore magnetite concentrate up to minus 0.044 mm under the same technological parameters the residual content of phosphorus made up 0.19% (experiment 1). Based on data of the Table 1 it is seen that the residual content of phosphorus in the concentrate after sulphuric acid treatment minus 0.044 mm in size and minus 0.071 mm in size does not differ much (makes up on an average 0.18%, error limit of chemical analysis 0.024%). Based upon the obtained results given in Table 1 as optimal degradation size of the roasted ore 0.071 mm is accepted.

In the second series of tests research works on influence of dephosphorizing duration on phosphorus content in a final product were carried out. Desalination time was selected in the range of from 20 to 120 min. The results are given in Table 2.

TABLE 1: Influence of degradation size of roasted ore on phosphorus content in dephosphorized concentrate.

No.	Roasting	Desalination	Size Fr., mm	Fe	Growth%
1	$T_{roas} = 950^{\circ}\text{C}$, $T_{roas} = 80''$	S:L = 1:2, $\text{C H}_2\text{SO}_4 = 20 \text{ gr}/100 \text{ gr T}$, $T_{deph} = 60''$ $t_{sol} = 65^{\circ}\text{C}$	-0.044	63.0	0.19
2	$T_{roas} = 950^{\circ}\text{C}$, $T_{roas} = 80''$	S:L = 1:2, $\text{C H}_2\text{SO}_4 = 20 \text{ gr}/100 \text{ gr T}$, $T_{deph} = 60''$ $t_{sol} = 65^{\circ}\text{C}$	-0.071	63.8	0.17
3	$T_{roas} = 950^{\circ}\text{C}$, $T_{roas} = 80''$	S:L = 1:2, $\text{C H}_2\text{SO}_4 = 20 \text{ gr}/100 \text{ gr T}$, $T_{deph} = 60''$ $t_{sol} = 65^{\circ}\text{C}$	2-0	57.6	0.45

Legend: t_{roas} – ore roasting temperature; τ_{roas} – ore exposure time; t_{sol} – pulp temperature (solution + ore); T_{deph} – dephosphorization time; S – solid phase (ore); L – aqueous solution of sulphuric acid; $\text{C H}_2\text{SO}_4$ – concentration of sulphuric acid in aqueous solution.

TABLE 2: Influence of dephosphorizing duration on phosphorus content in dephosphorized concentrate.

No.	Roasting	Magnetite Concentrate		Dephosphorization	Dephosphorized Concentrate		
		Content, %			Content, %		
		P	Fe		Size Fr., mm	Fe	P
1	$T_{roas} = 900^{\circ}\text{C}$, $T_{roas} = 120''$	0.52	63.3	S:L = 1:2, $\text{C H}_2\text{SO}_4 = 20 \text{ gr}/100 \text{ gr T}$, $T_{deph} = 20''$, $t_{sol} = 65^{\circ}\text{C}$	-0.071	65.5	0.20
2	$T_{roas} = 900^{\circ}\text{C}$, $T_{roas} = 120''$	0.52	63.3	S:L = 1:2, $\text{C H}_2\text{SO}_4 = 20 \text{ gr}/100 \text{ gr T}$, $T_{deph} = 80''$, $t_{sol} = 25^{\circ}\text{C}$	-0.071	65.3	0.18
3	$T_{roas} = 900^{\circ}\text{C}$, $T_{roas} = 120''$	0.55	63.3	S:L = 1:2, $\text{C H}_2\text{SO}_4 = 20 \text{ gr}/100 \text{ gr T}$, $T_{deph} = 120''$, $t_{sol} = 65^{\circ}\text{C}$	-0.044	65.3	0.16

Legend: t_{roas} – ore roasting temperature; τ_{roas} – ore exposure time; t_{sol} – pulp temperature (solution + ore); T_{deph} – dephosphorization time; S – solid phase (ore); L – aqueous solution of sulphuric acid; $\text{C H}_2\text{SO}_4$ – concentration of sulphuric acid in aqueous solution.

With increasing of dephosphorization process duration of from 20 to 120 minutes insignificant reduction of the residual content of phosphorus in the dephosphorized

concentrate from 0.20 to 0.16% is observed. Based upon the obtained results as optimal and sufficient duration of dephosphorizing the time equal to 20 minutes is accepted.

In the third series of tests pulp heating temperature influence on the quality of the dephosphorized magnetite concentrate was determined. In each experiment the aqueous solution of sulphuric acid was heated up to 25, 45 and 65°C. The magnetite concentrate with the optimal size of 0.071 mm was treated by the leaching procedure with light aqueous solution of sulphuric acid of mass concentration of 20 gr per 100 gr of the concentrate and ratio of solid material to liquid phase S:L = 1:2. The results of the tests are given in Table 3.

TABLE 3: Influence of temperature of pulp heating on residual content of phosphorus in dephosphorized concentrate.

No.	Roasting	Magnetite Concentrate		Dephosphorization	Dephosphorized Concentrate		
		Content, %			Content, %		
		P	Fe		Size Fr., mm	Fe	P
1	$T_{roas} = 900^{\circ}\text{C}$, $T_{roas} = 120''$	0.55	63.6	S:L = 1:2, C H ₂ SO ₄ = 20 gr/100 gr T, $T_{deph} = 120''$, $t_{sol} = 65^{\circ}\text{C}$	-0.071	65.3	0.16
2	$T_{roas} = 900^{\circ}\text{C}$, $T_{roas} = 120''$	0.45	63.6	S:L = 1:2, C H ₂ SO ₄ = 20 gr/100 gr T, $T_{deph} = 120''$, $t_{sol} = 45^{\circ}\text{C}$	-0.071	66.0	0.15
3	$T_{roas} = 900^{\circ}\text{C}$, $T_{roas} = 120''$	0.45	63.6	S:L = 1:2, C H ₂ SO ₄ = 20 gr/100 gr T, $T_{deph} = 120''$, $t_{sol} = 25^{\circ}\text{C}$	-0.071	65.9	0.15

Legend: t_{roas} – ore roasting temperature; T_{roas} – ore exposure time; t_{sol} – pulp temperature (solution + ore); T_{deph} – dephosphorization time; S – solid phase (ore); L – aqueous solution of sulphuric acid; C H₂SO₄ – concentration of sulphuric acid in aqueous solution.

As it is seen in Table 3 in the range of temperatures of pulp heating from 20 to 65°C the residual content of phosphorus in the dephosphorized concentrate was received almost the same (0.15–0.16%). In this range of temperature of dephosphorizing the iron content in the dephosphorized concentrate made up 65.3–66.0%. In accordance with results of tests of sulphuric acid desalinization of phosphorus the pulp temperature of 25°C was defined as optimal value.

In the next series of tests the cold roasted concentrate after magnetic separation was treated by the leaching procedure by light aqueous solution of sulphuric acid of mass concentration of from 5 to 30 gr per 100 gr of the concentrate under ratio of

solid material to liquid phase S:L = 1:2. The pulp was actively mixed within 120 minutes at temperature of 65 °C. Upon the completion of the experiment a sample of leached concentrate was taken for analysis of phosphorus and content of iron and phosphorus in a final product was determined (Table 4).

Based on data of Table 4 it is seen that for the roasted concentrate at temperature of 900 °C and size of minus 0.071 mm with the increase of specific consumption of sulphuric acid of from 10 to 30 gr per 100 gr of the concentrate the iron content makes up 64.5% on an average; the residual content of phosphorus is reduced in the range of from 0.38% to 0.16%. By roasting at 950° C and degradation size of minus 0.044 mm with the increase of specific consumption of sulphuric acid of from 5 to 20 gr per 100 gr of the concentrate the iron content makes up 62.5% on an average, and the residual content of phosphorus is reduced in the range of from 0.47 to 0.19%. As it is seen from the series of experiments the concentration of 5–15 gr of sulphuric acid per 100 gr of the concentrate is not enough as the residual content of phosphorus remains rather high and makes up 0.4% on an average.

Also an experiment on dephosphorization of the roasted concentrate was carried out using as chemical reagent of nitric acid HNO₃ under the following technological parameters: ratio S:L = 1:2 and specific consumption of HNO₃ 20 gr per 100 gr of the magnetite concentrate, desalinization time 20 minutes, pulp temperature 25°C. Due to this experiment the iron content in the dephosphorized concentrate 66.7% and the residual content of phosphorus 0.17% were obtained. As alternative chemical reagent for dephosphorization of iron ore magnetite concentrate the nitric acid can be used but up to date the sulphuric acid is more available and less expensive product.

4. Summary

On the base of OJSC VNIIMT experimental tests on the roasted concentrate dephosphorization of Gara Djebilet deposit by hydrometallurgical method were conducted. As a result of research works main technological parameters of sulphuric acid treatment of magnetite concentrate were defined under which the content of iron in the concentrate was increased from initial 52.1% to 64.0–65.3%, and the residual content of phosphorus in the concentrate was reduced from initial 0.75% to 0.15–0.20%. It was determined that for more complete dephosphorization of magnetite concentrate ($P_{res} \leq 0.2\%$) by sulphuric acid method it is required to carry out preliminary high-temperature magnetic roasting of ore at temperature not lower than 900° C and with duration not less than 40 minutes. Then the roasted ore degradation follows, wet magnetic separation with

TABLE 4: Influence of specific consumption of sulphuric acid on phosphorus content in dephosphorized concentrate.

No.	Roasting	Magnetite Concentrate		Dephosphorization	Dephosphorized Concentrate		
		Content, %			Content, %		
		P	Fe		Size Fr., mm	Fe	P
1	$T_{roas} = 900^{\circ}\text{C}$, $T_{roas} = 120''$	0.55	63.6	S:L = 1:2, $\text{C H}_2\text{SO}_4 = 20 \text{ gr}/100 \text{ gr T}$, $T_{deph} = 120''$, $t_{sol} = 65^{\circ}\text{C}$	-0.071	65.3	0.16
2	$T_{roas} = 900^{\circ}\text{C}$, $T_{roas} = 120''$	0.45	63.6	S:L = 1:2, $\text{C H}_2\text{SO}_4 = 30 \text{ gr}/100 \text{ gr T}$, $T_{deph} = 120''$, $t_{sol} = 65^{\circ}\text{C}$	-0.071	64	0.19
3	$T_{roas} = 950^{\circ}\text{C}$, $T_{roas} = 80''$	0.6	61.4	S:L = 1:2, $\text{C H}_2\text{SO}_4 = 5 \text{ gr}/100 \text{ gr T}$, $T_{deph} = 120''$, $t_{sol} = 65^{\circ}\text{C}$	-0.044	61.5	0.35
4	$T_{roas} = 950^{\circ}\text{C}$, $T_{roas} = 80''$	0.6	61.4	S:L = 1:2, $\text{C H}_2\text{SO}_4 = 10 \text{ gr}/100 \text{ gr T}$, $T_{deph} = 120''$, $t_{sol} = 65^{\circ}\text{C}$	-0.044	62.6	0.47
5	$T_{roas} = 950^{\circ}\text{C}$, $T_{roas} = 80''$	0.6	61.4	S:L = 1:2, $\text{C H}_2\text{SO}_4 = 15 \text{ gr}/100 \text{ gr T}$, $T_{deph} = 120''$, $t_{sol} = 65^{\circ}\text{C}$	-0.044	62.6	0.39
6	$T_{roas} = 950^{\circ}\text{C}$, $T_{roas} = 80''$	0.6	61.4	S:L = 1:2, $\text{C H}_2\text{SO}_4 = 20 \text{ gr}/100 \text{ gr T}$, $T_{deph} = 120''$, $t_{sol} = 65^{\circ}\text{C}$	-0.044	63	0.19

Legend: t_{roas} – ore roasting temperature; T_{roas} – ore exposure time; t_{sol} – pulp temperature (solution + ore); T_{deph} – dephosphorization time; S – solid phase (ore); L – aqueous solution of sulphuric acid; $\text{C H}_2\text{SO}_4$ – concentration of sulphuric acid in aqueous solution.

obtaining of magnetite product and tails, concentrate drying and its sulphuric acid treatment by aqueous solution of sulphuric acid.

The main technological parameters of sulphuric acid treatment of iron ore concentrate.

1. In a series of experiments with various degradation of the roasted concentrate before magnetic separation it was shown that the optimal size is 0.071 mm. In such fraction the dephosphorized concentrate was obtained with iron content of 63.8% and the residual content of phosphorus of 0.17%.
2. In a series of tests with different specific consumption of sulphuric acid (5-30 gr per 100 gr of the concentrate) it was shown that the optimal mass concentration

of sulphuric acid in aqueous solution is 20 gr per 100 gr of the concentrate, in this case in the dephosphorized concentrate the content of iron made up 65.3%, and the residual content of phosphorus made up 0.16%.

3. In a series of tests with different temperature of pulp heating (25–65°C) it was shown that the optimal pulp temperature is 25°C. In this case in the dephosphorized concentrate the content of iron made up 65.9%, and the residual content of phosphorus made up 0.15%.
4. In a series of experiments with different duration of the concentrate leaching it was shown that the optimal duration of dephosphorization process is 20 minutes. In such duration of dephosphorizing the concentrate was obtained containing iron 65.0% and the residual content of phosphorus 0.17%.
5. As a leaching reagent the nitric acid was tested (HNO_3). In this case the content of iron in the dephosphorized concentrate made up 66.7%, and the residual content of phosphorus made up 0.17%, but significant minus of nitric acid use is that it is difficult to get and its rather high price.

References

- [1] Belikov, V. V., Ogorodov, V. B., Yadryshnikov, A. O., et al. (27 June 2002). Method of purification of iron ore concentrate from phosphorus impurities. Patent of the Russian Federation No. 2184158.
- [2] Karelin, V. G., Zainullin, L. A., and Epishin, A. Y. (2017). Kinetics of high-temperature dehydration of Lisakovsk iron ore concentrate. *Izvestiya Vysshikh Uchebnykh Zavedenij. Chernaya Metallurgiya*, vol. 60, no. 8, pp. 656–661.
- [3] Epishin, A. Yu., Zainullin, L. A., and Karelin, V. G. (2011). On dephosphorizing of Lisakovsky brown ironstone with leaching methods with pre-roasting. *Digest of the VIII Congress of Dressers of CIS Countries*, vol. I, pp. 83–86.
- [4] Karelin, V. G., Zainulin, L. A., Epishin, A. Y., et al. (2013). The Modern Techniques of Involvement of Phosphorous. Containing Sedimentary Production. *The 12th China-Russia Symposium on Advanced Materials and Technologies*, pp. 388–391. Kuming.
- [5] Karelin, V. G., Zainulin, L. A., Epishin A. Y., et al. (2015). Combined pyro-hydrometallurgical technology of dephosphorizing brown ironstone of Lisakovsky deposit. *Ferrous metallurgy, Bulletin of Scientific and Technical and Economic Information*, no. 2, pp. 10–15.



- [6] Karelin, V. G., Zainulin, L. A., Epishin, A. Y., et al. (2015). Features of pyro-hydrometallurgical technology of dephosphorizing brown ironstone of Lisakovsky deposit. *Steel*, no. 3, pp. 8–11.