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## METALLURGY OF NONFERROUS METALS

# Nitric Acid Leaching of Polymetallic Middlings of Concentration

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**Abstract**—Investigations into the nitric acid leaching of polymetallic middlings with the purpose of the maximal recovery of copper and zinc into the solution are performed. Using methods of mathematical planning of the experiment, the optimal process parameters are determined: ratio L : S = 5, the consumption of nitric acid is 80 $cm^3$ per 20 g of the charge, and the process duration is 120 min.

*Keywords*: nitric acid leaching, collective middlings, mathematical planning of the experiment

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In recent times, enterprises of the mining-and-smelting complex increasingly pay attention to previously used sources of raw materials for their production. This can be associated both with the depletion of the processed ore raw materials and with a considerable accumulation of various final tailings and middlings of conventional production flow charts of nonferrous metals, which causes the expenses for their storage and utilization. Indeed, the latter definitely negatively affects the environmental situation around the enterprise and often in the region in general [1].

One of current vulnerable spots of mining-and-smelting enterprises is the forced output of difficult-to-break collective middlings (which almost cannot be treated using the conventional flotation selection methods) from the concentration chain [2].

In connection with this, this study is aimed at the development of new production methods for processing similar raw materials which improve the economical characteristics of enterprises and correspond to modern environmental requirements.

The investigations, which had the goal of developing the hydrometallurgical method of breaking the polymetallic middlings formed at some domestic and foreign enterprises, were performed at the Department of Metallurgy of Heavy Nonferrous Metals, Ural Federal University.

The developed method is based on the nitric acid leaching of the copper–zinc raw materials with the subsequent selective stage-by-stage recovery of valuable components.

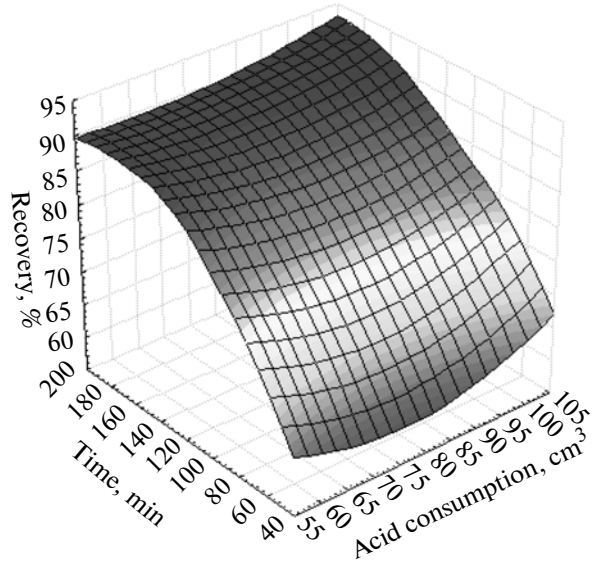
We performed a series of experiments on leaching the collective middlings with the composition, %: 6.1 Cu, 8.7 Zn, 28.8 Fe, 1.1 Pb, 11.5 g/t Au, and 81 g/t Ag. The analyses of solutions and solid products are performed by the methods of the atomic-absorption and X-ray fluorescent spectroscopy.

To attain the optimal characteristic of the production process, we applied the method of mathematical planning of the experiment in a form of a three-level model by three independent parameters.

We processed our experimental results using the Statistica 7.0 computer program in a form of response surfaces (Figs. 1–3), where the resulting functions were the values of recovery of copper, zinc, and iron into the solution ($Y_{Cu}$, $Y_{Zn}$, and $Y_{Fe}$). From the manufacturing reasons, we selected the following factors as the variable parameters: the liquid-to-solid ratio (L : S) in the pulp ($X_1$), which was varied in the limits from 5 to 9; the consumption of nitric acid ($X_2$) of 60–100 $cm^3$ with various volumes of water (40–120 $cm^3$) per 20 g of the concentrate; and the leaching duration ($X_3$) from 60 to 180 min. The pulp in all experiments was stirred using a magnetic stirrer with a rate of 500 rpm. Since the interaction of the sulfide raw material with nitric acid is accompanied by the instantaneous liberation of heat of accompanying exothermic reactions, the process temperature was not taken into account as the controlling factor.

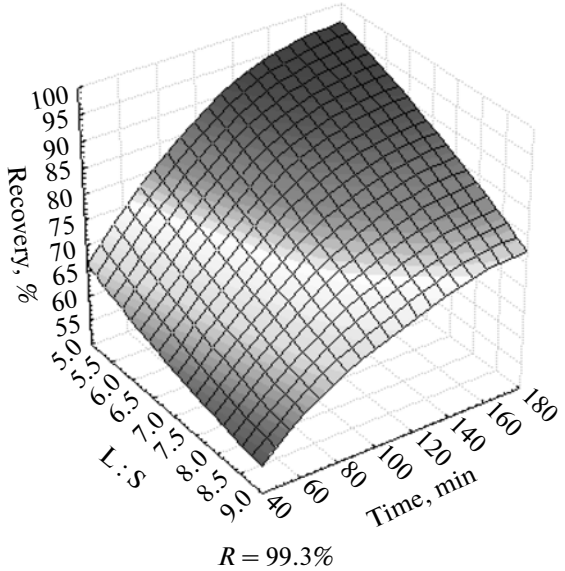
We calculated multiple correlation coefficients $R$ for each surface and derived simple regression equations to reveal the adequacy of our results and determine the most valuable process parameters [3, 4].

An increase in the pulp density (Fig. 1 shows the response surface at L : S = 5 for copper; similar characteristics are found for zinc and iron) makes it possible to attain high characteristics of the recovery of metals into the solution more rapidly, and the same results are




$$R = 97.6\%$$
$$Y = 73.703 - 0.786X_1 + 0.521X_2 + 0.0049X_1^2 - 0.0015X_2^2$$

**Fig. 1.** Dependence of the recovery of copper on time and acid consumption at L : S = 5.




$$R = 99.3\%$$
$$Y = 54.761 - 0.657X_1 + 0.595X_2 - 0.014X_1X_2 - 0.0141X_1^2 - 0.0014X_2^2$$

**Fig. 2.** Dependence of the recovery of zinc on time and the L : S ratio at an acid consumption of 80 $cm^3$.

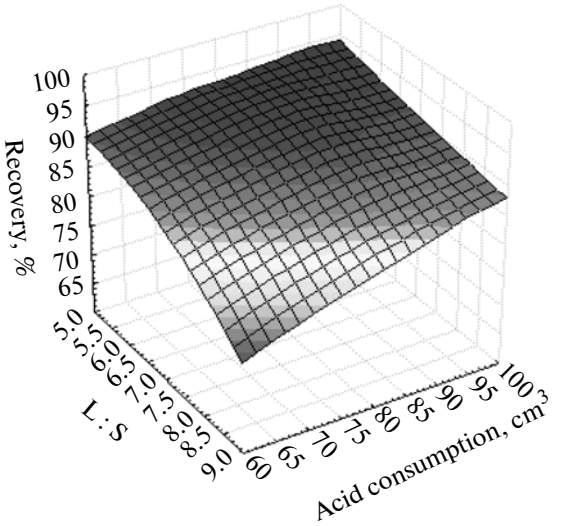
attained by the stoichiometric consumption of nitric acid, while at L : S ≥ 7 an excess of the leaching reagent and prolonged process duration are necessary.

The calculated values of multiple correlation coefficients *R* indicate the adequacy of our models, while the derived regression equations show that the consumption of nitric acid affects the dependent variables of equations most strongly among the criterion parameters.With a deficit of acid, the surfaces become concave, which is associated with the fact that the oxidation potential of the system reduces. The deficit of the leaching agent leads to the formation of surface films, which hamper the oxidant access to the unreacted particles, and intradiffusion limitations appear. However, with stoichiometric acid consumption (80 $cm^3$), the oxidation potential of the system substantially increases, which is indicated by convex shapes of surfaces (see Fig. 2; similar data are found for copper and iron). Consequently, when limiting the consumption of nitric acid, we can attain high leaching characteristics with shorter process durations.

Values of coefficients *R* indicate the correctness of the selection of parameters and ranges in which they vary. According to the results of the regression analysis, the pulp density or the process duration affects the transfer of metals into the solution most strongly depending on the experimental conditions.

The response surfaces, which were found at various fixed process durations, show that 60 min is insufficient to attain a high recovery of metals into the solution. This is associated with kinetic obstacles, i.e., the deficit of time for the interaction between the oxidant and the leached raw material.

However, at a process duration of 120 min (see Fig. 3), it is possible to attain high characteristics of recovery of metals into the solution with the stoichiometric consumption of nitric acid.




$$R = 93.6\%$$
$$Y = 182.115 - 4.792X_1 - 1.245X_2 + 0.121X_1X_2 - 0.551X_1^2 - 0.0031X_2^2$$

**Fig. 3.** Dependence of the recovery of iron on acid consumption and the L : S ratio for a process duration of 120 min.

Lowering the multiple correlation coefficients for copper and zinc indicates that an increase in the process time to 180 min negatively affects the final characteristics of leaching, which is also seen from the regression coefficients.

Our results on mathematical planning of the experiment in the form of a complete polynomial make it possible to evaluate the contribution of each factor to the efficiency of the leaching process:

$$R_{Cu} = 0.915,$$

$$Y_{Cu} = 119.83 - 11.185X_1 - 1.146X_2 + 0.623X_3 - 0.623X_1^2 + 0.004X_2^2 - 0.001X_3^3 + 0.119X_1X_2 + 0.250X_1X_3 - 0.029X_1X_3;$$

$$R_{Zn} = 0.967,$$

$$Y_{Zn} = 101.545 - 6.286X_1 - 0.785X_2 + 0.565X_3 + 0.005X_2^2 - 0.001X_3^2 + 0.041X_1X_2 - 0.001X_2X_3 - 0.18X_1X_3;$$

$$R_{Fe} = 0.948,$$

$$Y_{Fe} = 146.534 - 7.106X_1 - 1.243X_2 + 0.208X_3 - 0.405X_1^2 + 0.003X_2^2 + 0.12X_1X_2 + 0.015X_1X_3.$$

All coefficients of reduced polynomials become valuable, and our values of coefficients $R$ indicate the adequacy of models.

The adequacy of equations is confirmed using the Fischer criterion.

## CONCLUSIONS

The optimal parameters of nitric acid leaching of collective middlings, at which the maximal recovery of copper and zinc into the solution can be attained, were selected by mathematical planning of the experiment.

An analysis of the response surfaces and mathematical models showed that such parameters are ratio L : S = 5, acid consumption of 80 $cm^3$ per middling charge of 20 g, and duration of the experiment of 120 min.

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