

CONVERTER SLUDGE DEZINCIFICATION BY HYDROMETALLURGICAL METHOD

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Preliminary Note – Prethodno priopćenje

The article presents the results of studying the process of sludge dezincification by hydrometallurgical method: leaching of zinc from sludge with hydrochloric acid solutions. The object of the study was converter sludge with the zinc content of 1,24 %. On the basis of experimental data, the optimal conditions of sludge leaching were established: the concentration of hydrochloric acid in the solution was 15 %, the leaching time was 90 min, the liquid : solid phase L:S ratio was 6:1. The separated iron cake contained 0,43 % zinc and could be returned to the production cycle at the stage of agglomeration of iron ore raw materials.

Keywords: hydrometallurgy, converter sludge, zinc transition into the solution, leaching, hydrochloric acid

INTRODUCTION

At the ArcelorMittal Temirtau JSC (Kazakhstan, Karaganda) a large amount of solid waste is generated and dumped in the two-stage steel production based on the smelting of pig iron and its subsequent conversion to steel in oxygen converters. Solid wastes accumulated in dumps is a source of environmental pollution, in this regard the task of their disposal becomes urgent. Such wastes include iron-containing sludge from blast-furnace and converter production [1, 2]. The best option for the disposal of sludge characterized by a high iron content, should be considered its return to the production cycle as an additive to the sinter charge of iron ore raw materials. However, utilization of the entire volume of sludge by this method does not seem possible due to the increased content of zinc (more than 0,5 %). [3-8].

In the conditions of the ArcelorMittal Temirtau JSC, hydrochloric acid leaching can be one of the acceptable methods of sludgedezincification. The source of hydrochloric acid at the plant can be liquid waste of the pickling department of the sheet-rolling production: the waste pickling solution and wash water [9].

EXPERIMENTAL PART

To carry out the studies, there was used converter sludge of the ArcelorMittal Temirtau JSC of the following composition, wt. %: 1,24 Zn; 0,56 Pb; 33,70 Fe_{MET}; 32,13 FeO; 20,69 Fe₃O₄; 0,54 MnO; 3,65 CaO; 1,59 MgO; 1,20 SiO₂; 0,57 Al₂O₃; 0,54 C. The experiments with leaching converter sludge were carried out using a five-factor experiment plan at four levels [10] on a labo-

ratory setup consisting of a mechanical stirrer and a flask with the volume of 0,5 dm³. The variable factors in the plan of the experiment were the concentration of hydrochloric acid in the leaching solution (from 5 to 20 %), the duration of leaching (30 - 120 min) and the L : S phase ratio = 3 - 6 with a step of 1.

RESULTS AND THEIR DISCUSSION

The conditions and results of the experiments with leaching of converter sludge in a laboratory setup and the degree of transition of zinc into the solution according to the experimental (e) and calculated (c) data are presented in Table 1.

From Table 1 it follows that in order to obtain a cake with the zinc content of no more than 0.5 %, the degree of zinc transition into the solution during leaching of sludge must be at least 79 %.

By means of sampling, there were obtained point plots of the degree of zinc transition into the solution dependence on the factors under study. Then the obtained point dependences were described by particular equations, the adequacy of which was established by calculating the coefficient R of nonlinear multiple correlation and its significance t_R . In this case, the equation is considered adequate if the condition $t_R > 2$ is met. Figure 1 shows a graphical dependence of the degree of zinc transition into the solution on the concentration of hydrochloric acid in the leaching solution.

It follows from the graph that, with increasing the concentration of hydrochloric acid, the degree of zinc transition from the sludge to the solution increases. The required degree of zinc transition into the solution is achieved only when the concentration of hydrochloric acid in the solution is not less than 19 %.

The need to use solutions with such high concentrations of HCl is most likely dictated by a high content of

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Table 1 The conditions and results of the experiments with converter sludge leaching

Experiment No.	$X_1 - C_{HCl} / \%$	$X_2 - \tau_{LEACH} / \text{min.}$	$X_3 - L/S$	X_4	X_5	$Zn_{END} / \%$	$\eta_{Zn}^{EXP} / \%$	$\eta_{Zn}^{CALC} / \%$
1	5	30	3	1	1	1,12	24,19	24,31
2	10	60	4	2	1	0,95	51,61	47,60
3	15	90	5	3	1	0,30	87,90	72,74
4	20	120	6	4	1	0,34	87,10	99,53
5	5	60	5	4	2	1,00	39,52	36,95
6	10	90	6	1	2	0,60	72,58	64,54
7	15	120	3	2	2	0,46	79,03	57,71
8	20	30	4	3	2	0,62	71,77	60,88
9	5	90	3	3	3	0,98	33,87	29,99
10	10	120	4	4	3	0,73	64,52	54,33
11	15	30	5	1	3	0,70	66,94	58,97
12	20	60	6	2	3	0,44	81,45	87,21
13	5	120	5	2	4	0,76	54,84	42,17
14	10	30	6	3	4	0,85	56,45	52,33
15	15	60	3	4	4	0,72	65,32	50,56
16	20	90	4	1	4	0,35	84,68	75,09

metallic iron in sludge. The partial dependence of the degree of zinc transition into the solution on the concentration of hydrochloric acid is as follows:

$$\eta_{Zn} = 15,81C_{HCl}^{0,546}, R = 0,95, t_R = 13,78 > 2 \quad (1)$$

The dependence of the degree of zinc transition into the solution on the duration of leaching is shown in Figure 2. The longer the duration of leaching, the more fully the reaction of interaction of zinc oxide with hydrochloric acid proceeds. Accordingly, the degree of zinc transition into the solution also increases with increasing the duration of leaching.

The partial dependence of the degree of zinc transition into the solution on the duration of leaching has the following form:

$$\eta_{Zn} = 28,64\tau_{LEACH}^{0,191}, R = 0,94, t_R = 11,08 > 2 \quad (2)$$

Figure 3 shows a graphical dependence of the degree of zinc transition into the solution on the liquid : solid phase ratio.

The graph shows that the degree of zinc transition into the solution increases with increasing the L : S ratio. However, among all the factors studied, this factor turned out to be the least significant; its effect on the degree of zinc transition into the solution is insignificant in comparison with the acid concentration and the duration of leaching. The low significance of this factor looks quite logical, given that additional increasing the mass of hydrochloric acid solution during leaching is less likely to affect increasing the amount of zinc removed from sludge than increasing the concentration of the acid itself in the leaching solution. The partial dependence of the degree of zinc transition into the solution on the liquid : solid phase ratio is as follows:

$$\eta_{Zn} = 27,35(L:S)^{0,56}, R = 0,71, t_R = 2,01 > 2 \quad (3)$$

The obtained partial equations (1) - (3) were combined to obtain a generalized dependence of the degree of zinc transition into the solution:

$$\eta_{Zn} = 2,85C_{HCl}^{0,546} \times \tau_{LEACH}^{0,191} \times (L/S)^{0,56} \quad (4)$$

$$R = 0,78, t_R = 23,84 > 2$$

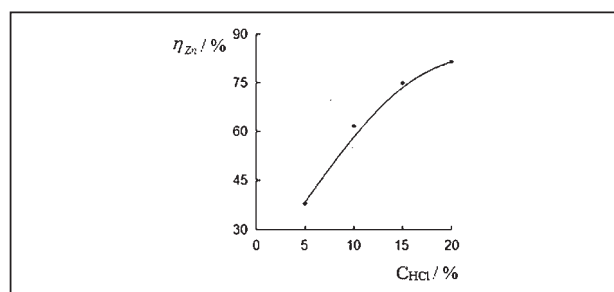


Figure 1 The dependence of the degree of zinc transition into the solution on the concentration of hydrochloric acid with converter sludge leaching

Using equation (4), the optimal parameters of converter sludge leaching were determined, which ensure the required degree of zinc transition into the solution: $C_{HCl} = 15 \%$; $\tau_{LEACH} = 90 \text{ min}$; $L : S = 6 : 1$. With these parameters, the calculated degree of zinc transition into the solution is 80,53 %.

Sludge leaching in a large-scale laboratory unit.

For the purpose of determining the experimental degree of zinc transition into the solution at optimal parameters, an experiment was carried out on converter sludge leaching in a large-scale laboratory set consisting of a 3 dm³ thermostat and a laboratory stirring device MR25 with a two-blade stirrer. For the experiment, a sample of converter sludge weighing 0,15 kg was taken and 0,9 kg of the 15 % hydrochloric acid solution was consumed. Leaching was performed at the room temperature. After the end of leaching, the pulp was filtered off, the resulting filtrate and cake were sent for the chemical analysis. The filtrate obtained during leaching (0,85 dm³) is a solution of metal chlorides of the following composition wt. %: 2,01 HCl; 0,33 ZnCl₂; 15,25 FeCl₃; 0,10 PbCl₂; 1,20 CaCl₂; 0,54 MgCl₂; 0,06 MnCl₂; 0,19 AlCl₃. The filtrate can be further processed in order to extract iron and zinc from it.

Leaching resulted in 85,18 g of cake of the following composition, wt. %: 0,43 Zn; 0,15 Pb; 55,22 FeO; 36,43 Fe₃O₄; 0,55 MnO; 1,35 CaO; 0,21 MgO; 2,11

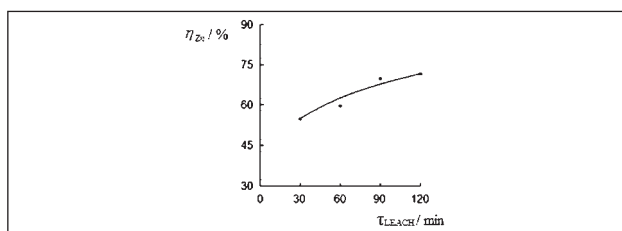


Figure 2 The dependence of the degree of zinc transition into the solution on the leaching duration with converter sludge leaching

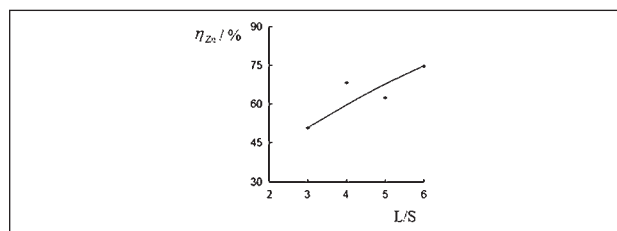


Figure 3 The dependence of the degree of zinc transition into the solution on the L : S phase ratio with converter sludge leaching

SiO₂; 0,17 Al₂O₃; 0,96 C. In this case, the degree of zinc transition into the solution was 80,32 %. The resulting cake in terms of zinc content meets the requirements of the blast furnace process and can be returned to the production cycle at the sintering stage.

CONCLUSION

Reducing the zinc content in sludge released during the wet cleaning of gases at ferrous metallurgy enterprises is the main task for their disposal by returning to the production cycle. Dezincated sludge returned to metallurgical processing must contain no more than 0,5 % zinc. In this work, the process of dezincification of converter sludge containing 1,24 % zinc was studied by the hydrometallurgical method: zinc leaching with hydrochloric acid solutions. Based on the results of the leaching experiments, the possibility of separating cake with the zinc content of no more than 0,5 % was established. The optimal parameters of sludge leaching were determined ($C_{\text{HCl}} = 15\%$; $\tau_{\text{LEACH}} = 90\text{ min}$; L : S = 6 : 1) at which the degree of zinc transition from sludge into the solution was 80,32 %, and the released iron cake contained 0.43 % zinc.

The results obtained make it possible to recommend hydrochloric acid leaching as an effective method of dezincification of converter sludge before returning it to the production cycle at the stage of agglomeration of iron ore raw materials.

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