Optimisation of process parameter using Response Surface Methodology to recover ceramic materials from bauxite mining waste rocks

[1]Ranjita Swain Mohanty, [2]Sunita Routray and [3]Rudra Narayan Mohapatro
[1,2,3]Assistant Professor, C V Raman College of Engineering, Bhubaneswar 752054, India

Abstract

The bauxite mining waste rocks is also referred as Partially LaterizedKhondalite (PLK) a rock that is generated during mining of bauxite. PLK rocks that associated with kaolinized khondalite rocks are being generated and dumped at the mine site as waste material or sometimes used for back filling of abandoned mines.These rocks contain 36.14% Al_2O_3, 29.57% SiO_2, 15.87% of Fe_2O_3, 2.37% of $TiO_2$and 15.60% LOI.This rock cannot be used as a valuable material for any industry due to the presence of high iron content as well as not suitable for Bayer's process as it contains reactive silica. Physical methods such as magnetic separation, flotation, scrubbing etc. are not fully successful for removal of iron from PLK rocks. Leaching techniques are the only alternative method to minimize iron content in it. This present paper deals with leaching studies of mining waste PLK rocks during bauxite mining, is carried out on different parameters like temperature, concentration, pulp density and time. The brightness of the product is increased to 80% which is desirable for high valued ceramicproducts. Hence, the optimized parameters of the leaching can be scaled up to the industrial trials for further viability.

Keywords: Bauxite mining waste (PLK) rock, Leaching, Pulp density, Concentration, Temperature, Time

1. Introduction

NALCO is a major aluminium producer in Odisha, India which is located at Panchpatmali, Koraput, Odisha. In order to produce around 2.1 million tonnes of alumina per year by National Aluminium Company (NALCO), about 6.34 million tonnes per annum of bauxite is being mined. About 6.3 million tons per annum of Partially Laterised Khondalite (PLK) rock is also generated during bauxite mining at NALCO, Damanjodi mine area. Bauxite mining waste contains on an average 41–57% of Al_2O_3, 1–30% of Fe_2O_3, 1–30% of TiO_2, 20–35% of SiO_2 and 20–35% of loss of ignition (LOI). This waste is not used or recycle in the present period. It is also a problem that it cannot be used for the production of alumina. Hence, it can be used for other industrial applications and a major resource for the industries like paint, pigment, rubber, cosmetics, ceramics, refractory and fillers. This can be converted as a valuable material after a suitable beneficiation to remove iron, illimanite etc.

The less number of work has been carried out on this sample, hence the literature is collected on the basis of bauxite and clay materials. The researchers worked on bauxite and clay for removal iron oxide using different methods like physical and chemical process techniques.

Kuys et al. (1990) studied aboutthe acid leaching of ultrafine materials such as kaolinite, boehmite and gibbsite etc. The surface iron was removed by this process. It is also found that the removal depends on the geological part of minerals. Goethite and hematite removed by reductive leaching. The total iron removed of 67% using HCl from present in ROM without affecting the structure.

Rao et al. (2005) reported in their paper about the leaching of iron from PLK rock samples. The fine grade sample leached using HCl at higher concentration and with high temperature. It is also shown that it can be suitable for the industries.

Swain and Rao (2012) studied on kinetic study on leaching of PLK rocks to remove iron oxide. This study was carried on leaching of fine sample containing less iron content about 5.39%. This is suitable for filler industries as the brightness is around 80%.

The aim of this work is to optimise the leaching study using ANOVA at different conditions such as pulp density, acid concentration, time and temperature. The developed product from bauxite mining waste containing 15.87% of iron oxide content. The raw material produced from the bauxite mining waste can be used in the industries like high valued ceramicmaterial.

2. MATERIALS AND METHODS

Bauxite mining waste (PLK) rock collected from the mine site of NALCO, Damanjodi from Easternghat of Odisha. Figure 1 indicates the sample from mining to lab and prepared for leaching study.ROM was crushed and screened through 150 µm sieve.

(a) (b)

Fig.1 (a) ROM Sample, (b) Homogonise sample

Leaching experiments were carried out in leaching reactor attached with a magnetic stirrer and a thermometer. The variables were pulp density, acid concentration, time and temperature. Iron analysis was carried out by using wet chemical method.

<u>Conditions for leaching experiments</u>

Conc. of HCl, M -[x_1]:	1, 2, 3and 4
Pulp density (S/L) - [x_2]:	5, 10, 15
Volume of the solid liquid solution:	100
Temperature, °C - [x_3]:	40, 70,100
Time period, minute - [x_4]:	60, 120,180, and 240

3. RESULTS AND DISCUSSIONS

The size analysis is shown in Fig.2 which indicates 50% of the sample passed in a screen size below 100-micron. The physio-chemical analysis of PLK rock sample is given in Table 1that indicates the bulk density is 1.28 gm/cc, true density is 2.6 gm/cc whereas the porosity is 52.3% and angle of repose is 37.9º.The chemical analysis is represented in Table 1 shows that sample contains 36.14% Al_2O_3, 29.57% SiO_2, 15.87% of Fe_2O_3, 2.37% of $TiO_2$and 15.60% LOI.

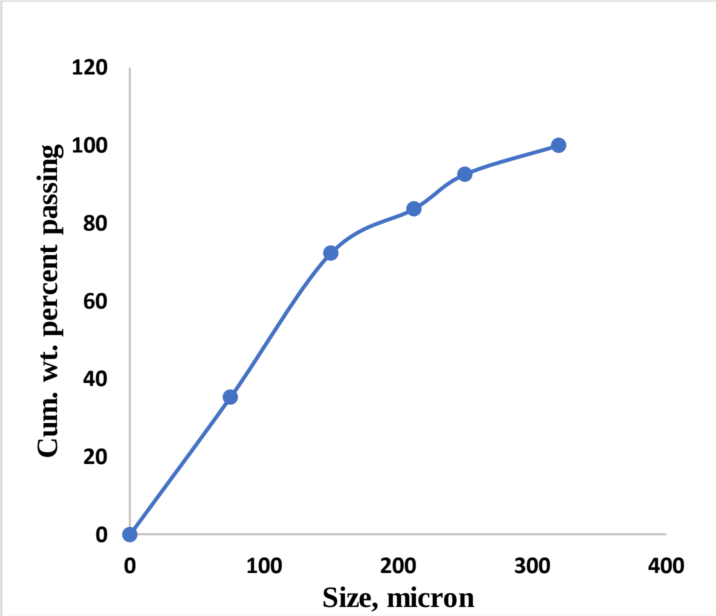

Fig.2 Cumulative percent passing of ground sample of PLK rocks

Table 1. Physico-chemical analysis of bauxite mining waste rock samples

Details	Physical properties of the samples			
	Bulk density, g/cc	True density, g/cc	Porosity, %	Angle of repose, °
PLK rock bulk material	1.28	2.6	52.3	37.9
	Chemical analysis of the samples				
	Al_2O_3%	SiO_2%	Fe_2O_3%	TiO_2%	LOI%
PLK rock bulk material	36.14	29.57	15.87	2.37	15.60

During the leaching period, following minerals are being leached out with HCl.

$Fe_2O_3 + 6HCL \rightarrow 2FeCl_3 + 3H_2O$ ……………………………..[1]

$Al_2O_3 + 2HCl \rightarrow 2AlCl_3 + 3H_2O$ ……………………………...[2]

$TiO_2 + 2HCl \rightarrow TiOCl_2 + H_2O$ ………………………………...[3]

The leaching study at different variables is tabulated in Table 2. This experiment is designed using RSM in Design Expert software. As per the design, the experiments were carried out at different conditions. Then, the predicted result is compared to actual result by using ANOVA software.

The statistical design shows one regression analysis for the removal of iron oxide. The equation is being presented in the coded form:

$$Removal\ of Fe2O3 = +84.00 + 17.01 \times x_1 + 9.07 \times x_2 + 35.12 \times x_3 + 3.82 \times x_4 - 4.32 \times {x_1}^2 - 11.08 \times {x_2}^2 - 24.98 \times x_3^2 - 12.22 \times x_4^2 - 0.62 \times x_1 \times x_2 - 4.83 \times x_1 \times x_3 - 8.78 \times x_1 \times x_4 - 8.80 \times x_2 \times x_3 - 12.28 \times x_2 \times x_4 - 1.97 \times x_3 \times x_4$$

....(1)

The data indicate that the regression analysis shows the actual and predicted values match each other. This can be very sure by seeing the R^2 value. The R^2 value is 0.91 which is approaching 1. This value shows a good relation between the predicted and actual value, this shows in Fig.3.

Table 2. Leaching study of bauxite mining waste rockswith different variables

Si. No.	Pulp density, % [x_1]	Concentration of HCl, M [x_2]	Temperature, °C [x_3]	Time, hr [x_4]	Fe_2O_3, %
1	5	3	70	4	62.6
2	10	3	100	4	72
3	10	3	70	3	84
4	10	4	70	4	53
5	15	2	70	3	65.2
6	10	2	40	3	2.6
7	5	3	40	3	6.3
8	15	4	70	3	99.4
9	10	2	70	4	65.6
10	10	3	40	2	6.7
11	10	2	70	2	45.5
12	15	3	40	3	25.2
13	10	3	40	4	20.2
14	10	2	100	3	95.6
15	10	3	70	3	84
16	10	3	70	3	84
17	10	3	100	2	66.4
18	5	4	70	3	62.3
19	10	3	70	3	84
20	10	4	70	2	82
21	10	4	40	3	27.2
22	15	3	70	4	99.5
23	10	4	100	3	85
24	10	3	70	3	84
25	15	3	100	3	95.1
26	15	3	70	2	99.2
27	5	3	70	2	27.2
28	5	3	100	3	95.5
29	5	2	70	3	25.6

The data shown in Table 2 indicates that the pulp density plays an important role to dissolve the iron in HCl. At higher pulp density with lower leaching time and medium temperature the iron dissolution is maximum. It is clearly seen that with 15% PD and at 3M HCl concentration for two hours leaching duration, 99.2% of iron leached out. The tabulated data also indicates that the iron leaching is constant with increase in molarity and leaching time at constant temperature.

Optimisation of the process parameter has been carried out by using MATLAB 7.0. The data from equation 1 is being used for the optimisation. In the programme the optimisation is done on quadratic programme for the process parameters by achieving the maximum value of the response (removal of Fe_2O_3%). The minimum and maximum value for the parameters is also given in the software for optimum removal of Fe_2O_3 from bauxite mining waste rocks. The optimum conditions achieved for 99% of removal of iron oxide is 15% PD with 3 M HCl concentration at 70°C for 2 hours of time duration.

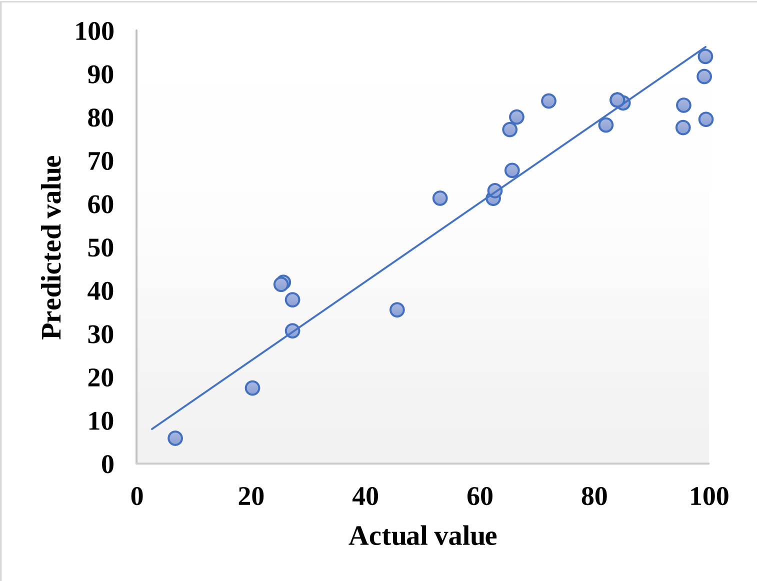

Fig.3 ANOVA analysis for leaching of bauxite mining waste at different conditions

Table 3. Evaluation of the optimised result from the model

Si. No.	Pulp density, % [x_1]	Concentration of HCl, M [x_2]	Temperature, °C [x_3]	Time, hr [x_4]	Fe_2O_3, %	Brightness, %
1	15	3	70	2	99.1	80.08
2	15	3	70	2	99.4	79.98
3	15	3	70	2	99.8	80.69

It is shown in Table 3 that the results obtained from the experiments at the optimum leaching process parameter. It is observed that the brightness value depends on the removal of iron from the sample. The experimental data may vary slightly with respect to the same process parameter. The reason may be the residence time and leaching reaction inside the reactor or may be analysis by the analyser. The approximate value of iron by using three consecutives is 99.4%. The brightness is approximately 80.25%. The leached material is very much suitable as a high valued raw material for ceramic object preparation.

Table 4. Properties of leached residue and comparison with standard of ceramic property used by industries

Details	Product (Leached residue)	Standard ceramic property*
Plasticity using water, %	11.4%	32%
Shrink due to drying, %	3.45%	7% (max.)

Shrink due to firing, %	10.2%	14% (max.)
Colour after calcination	White	Perfectly white
Grit present, %	Nil	2% (max.)
Fe_2O_3, %	0.79%	1% (max.)

*As per BIS

The product contains 0.79% of $Fe_2O_3$and brightness value is 80.25% as given in Table 4. It also shows that the product has the plasticity within limit. The shrinkage after firing and drying is also shows a good result. Hence, the product satisfies the ceramic standard used by BIS. This is one of the high valued ceramic material can be used for preparing This can be used for preparation of capacitors, cupolas and insulators etc.

4. Conclusions

The bauxite mining waste containing PLK rocks are being generated during bauxite mining. This waste contains reactive silica and iron content as a discolouring mineral. The optimum conditions for removal of iron content are PD: 15%, HCl Conc.: 3 molars, Time: 2 hours and Temperature: 70°C. The acid concentration is less required in the leaching processis more approachable for the industries as well as in favour of the environment. The high valued leached product can be a new resource for ceramic industry. This leached product satisfies the Indian standard properties. This ceramic raw material can also be used for manufacturing of capacitors, cupolas and insulators etc.

5. References

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6. Acknowledgement

Authors are thankful to Chairman of C. V. Raman group of Institutions, Bhubaneswar, Odisha, India for providing support to carry out the research work. One of the authors Dr.Ranjita Swain is thankful to NALCO for funding and JNARDDC for the support to carry out the work.