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Assessment of Calcined Ededimeji Termite Mound for Potential Alumina Recovery via Nitric Acid Route

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Abstract: *The potential of extracting alumina from Termite Mound with Nitric acid was examined in this study. The termite mound was collected from Ededimeji in Ede, Osun state, Nigeria. The optimization of Al_2O_3 leaching from termite clay was carried out using Nitric acid. Some process parameters such as calcination temperature (550-900 ℃) and time (60-240 min) were considered for alumina leaching based on the I-optimal Design under the Response Surface Methodology of the Design Expert software (12.0.1). X-ray fluorescence (XRF) was used to determine the chemical composition of the beneficiated, calcined and dealuminated clay sample. The analysis indicated the presence of Alumina in the raw termite mound and the dissolution rate of alumina from the meta clay was up to 64.88 %. Optimum alumina was achieved at 750 ℃ and 60 min. Therefore, abandoned termite mounds can be used extensively to produce alumina.*

Keywords: Alumina, Dealumination, Leaching, Nitric acid, Termite mound
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1. INTRODUCTION

The global depletion of bauxite ore deposits and the increasing demand for aluminium has elicited the need to develop alternative technologies for the production of aluminium, especially from low-grade ores. Comprehensive studies on the potential for extracting alumina from other non-bauxitic materials, like clays, have been published in several nations [1-3].

Termite mound has lower soil bulk density and elevated minerals such as carbon, nitrogen, phosphorus, magnesium, calcium, and clay content compared to the surrounding adjacent soils [1,3]. Termite mounds, a naturally occurring clay mineral, are made up of large amounts of Silica, Alumina and basic oxides of other elements [2]. Therefore, the possibility of extracting alumina from thermal-treated termite mounds is high.

The extraction of alumina from termite mound involves several intricate steps, typically starting with beneficiation process to remove impurities and enhance the purity of the termite mound. Subsequently, the beneficiated termite mound undergoes a leaching process with HCl, HNO_3, or H_2SO_4 solutions to extract alumina.

2. MATERIALS AND METHODS

2.1 Materials

The termite mound used for this research work was procured from Ededimeji in Ede, Osun state, Nigeria. The termite mound was separated from dirt, washed with distilled water and oven-dried at 120 ℃ until constant weight was achieved. The beneficiated sample was ground and sieved with a mesh size of less than 75µm. After that, it was subsequently calcined in a muffle furnace (English Labscience SX-4-10 Model) at temperature values of 550-900 ℃ for a time range of 1-4 h to improve the leaching operation. The concentrated nitric acid (≥ 98 %) used as a leachant was of analytical (BDH) grade.

2.2 Methods

Leaching Studies

Preliminary studies were conducted on calcined clay samples to determine the optimal calcination temperature and time. For every leaching experiment, 0.5 g of the metaclay from each run (as generated by the Optimal Design of the Response Surface Methodology of the Design of Expert (DOE 12.0.1) was separately mixed with 50 ml of 0.1M Nitric acid in a 100 ml conical flask. The mixtures were constantly stirred with a magnetic stirrer at 1000 rpm and allowed to react for 1 h at a constant temperature of 80℃. The supernatant was decanted and centrifuged at 1500 rpm for 30 mins and was collected for

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Al analysis using atomic absorption spectrophotometer (AAS) "AAS Buck Scientific 211 VGP". The concentration of Al_2O_3 leached was recorded [11].

XRF Analysis

The elemental composition of the raw, calcined and dealuminated termite mound used in this study was examined with X-ray Fluorescence (XRF). All measurements were carried out on an energy dispersive X- ray fluorescence spectrometer Shimadzu EDXRF-702HS operated at 40 kV and 18 mA. The current was automatically adjusted (maximum of 1 mA). A 10 mm collimator was chosen and the counting time was 100 seconds for all measurements. The intensity of element Kα counts per second (cps/μA) was obtained from the sample X-ray spectrum using the Shimadzu EDX software package. XRF was performed on samples prepared in the form of glass disks with a sample/melt ratio of 0.5/5; Li 2 B4O 7 was used as the melt. The reference materials GSS8 and BCS-CRM 354 were used to produce standards; mixtures of these with an oxide content including that of the samples to be analyzed were used to produce a calibration curve. The reference materials were mixed with a 5% Au/Pt ZGS glass rod and melted in a furnace at a temperature of 1100 °C for 15 min. The melt was taken from the furnace and stirred after 10 min and then replaced to eliminate bubbles. It was then poured into a Pt/Rh 30-mm diameter mold to form a glass disk, of 30 mm diameter and 9 mm thickness were used for the analyses

3. RESULTS AND DISCUSSION

3.1 Characterization of Raw, Calcined and Dealuminated Samples

The elemental composition of the raw, calcined and dealuminated termite mound used in this study was examined with X-ray Fluorescence (XRF). The result revealed the following elemental composition for the raw sample: SiO_2 (35.73 %), Al_2O_3 (15.84 %), MgO (3.11 %), CaO (5.08 %), Fe_2O_3 (9.94 %), K_2O (4.21), P_2O_5 (1.48 %), TiO_2 (1.01 %), MnO_2 (0.51 %), NiO (0.02 %), ZnO (0.03 %), CuO (0.03 %), SO_3 (0.02 %) and the loss on ignition (LOI) (20.31 %). The summary is given in Table 1. This result affirmed that raw termite clay contains Al_2O_3 which makes it a good source of Alumina. After calcination, it was observed that the alumina and silica contents in the calcined sample increased from 15.84 to 23.11 % and 35.73 to 54.10 % respectively. Other basic oxides also got reduced except the oxides of Fe, Mn, Zn and S which got increased. Calcination has increased the proportion of alumina in the calcined termite clay [7] and has changed the crystalline structure of the mound, resulting in a metaclay. This has reduced the volatile matter content of the raw clay sample and improved the susceptibility to acid leaching [4]. Additionally, it was evident that the oxides of Si, Ca, K, and P increased after dealumination, making the dealuminated mound a silica-rich mound.

Table 1: Chemical composition of Ededimeji termite mound before and after calcination and dealumination

Oxide	SiO_2	**Al_2O_3**	MgO	CaO	Fe_2O_3	K_2O	Na_2O	P_2O_5	TiO_2	MnO	NiO	ZnO	CuO	SO_3	*LOI
Raw%	35.73	**15.84**	3.11	5.08	9.94	4.21	2.70	1.48	1.01	0.51	0.02	0.01	0.03	0.02	20.31
Calcined	54.10	**23.11**	1.78	0.23	12.24	0.38	1.12	0.06	1.78	0.94	0.02	0.17	0.02	0.07	3.98
Dealuminated	68.96	**14.97**	0.36	1.86	5.35	3.79	0.27	0.08	1.07	0.07	0.03	0.03	0.02	0.03	3.01

* LOI Loss on Ignition

3.2 Response from Experimental Data for the Alumina Leaching from Ededimeji Termite Mound

The study category used for the experimental design in the leaching of Aluminum from termite clay was I-optimal Design, under Response Surface Methodology (RSM). The factors considered were temperature (A) and time (B) while the responses were Al conc in ppm and percentage. Twelve (12) experimental runs were generated. The response at different calcination temperatures and times as generated by the software (DOE 12.0.1) is represented in Table 2. The highest Al concentration of 16.85 ppm at 64.88% was obtained at Run 3 (750°C, 60 min). From the result, the optimal leaching condition of aluminum occurs at Run 3

Table 2: Experimental Responses for the calcination process

Run	Factors		Responses	
	Temperature (°C)	Time (min)	Alumina removal	
			ppm	%

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1	700	180	14.08	54.22
2	550	60	13	50.06
3	750	60	16.85	64.88
4	800	240	12.78	49.21
5	550	180	15.12	58.22
6	650	120	9.84	37.89
7	550	120	9.34	35.96
8	900	120	15.02	57.84
9	800	240	11.58	44.59
10	700	150	9.84	37.89
11	900	240	9.08	34.96
12	550	240	14.95	57.57

3.3 Effect of Process Variables

3.3.1 Effect of Calcination Temperature on Alumina Leaching

Raw termite clay samples used for this study are calcined at various temperatures (550-900 ℃). The results displayed in Table 2 showed that alumina leaching rates of 13 ppm (50%), 16.85 ppm (65%) and 10.43 ppm (40%) were achieved with calcination temperatures of 550, 750 and 900 ℃ respectively in 60 min at leaching temperature of 70 ℃, stirring rate of 1000 rpm and HNO_3 concentration of 0.1 M. Therefore, 750 ℃ was used as the temperature for the remaining samples [9].

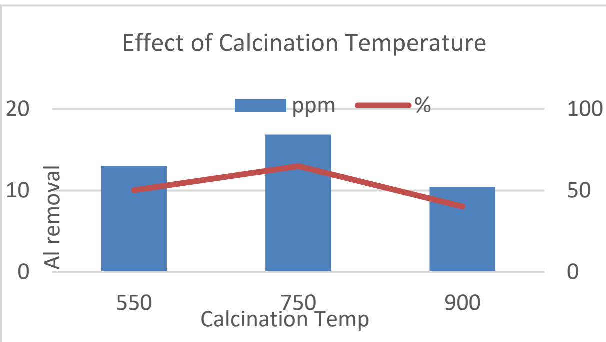

Fig 1: Effect of Calcination Temperature

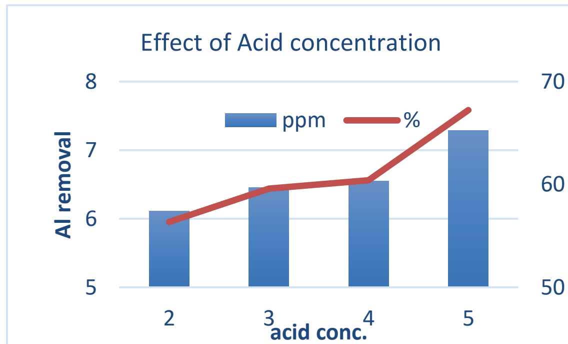

Fig 2: Effect of HNO_3 Concentration

3.3.2 Effect of Acid concentration on alumina leaching

Acid concentration plays a crucial role in the extraction of Alumina from Ededimeji termite mound. In this study, concentrations of 2 M, 3 M, 4 M and 5 M HNO_3 were used. The result obtained is shown in Figure 2. Alumina recovery rates of 67 %, 60%, 59 % and 56% were recorded within 60 min of leaching using HNO_3 concentrations of 5, 4, 3 and 2 M respectively. The result indicated that an increase in the acid concentration increases the alumina yield. [6] reported that an increase in acid concentration can enhance the yield of alumina. The solubility of Al atoms is affected by the concentration of H+ ions. The amount of H+ ions in the solution increases with the acid content. Additionally, it is hypothesized that as the concentration and diffusion of hydroxonium ions rise, so does the diffusion rate of Al^{3+} from solid to solution [12]. According to [3], more aluminum ions (Al^{3+}) are converted to alumina (Al_2O_3), boosting the alumina output because the acid is more prevalent at this point. The same observation was made by [10]

3.3.3 Effect of Calcination time on alumina leaching

Calcination time plays a vital role in the extraction of Alumina from termite mounds. It was observed that the colour of the sample changes with time during calcination. From this study, the alumina extraction increases with an increase in calcination time up to 120 min. Thereafter, the alumina removal behaves irrationally as time passes. The calcination time that gave the best Alumina removal was 120 min.

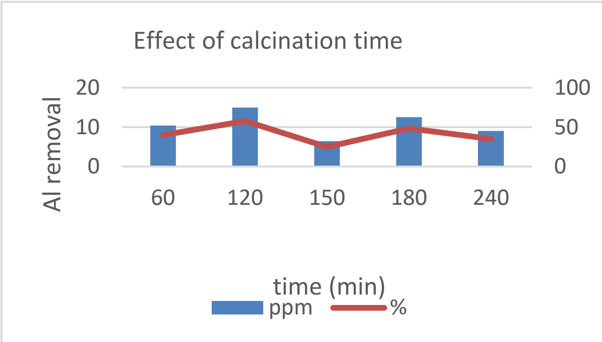

Fig 3: Effect of calcination time

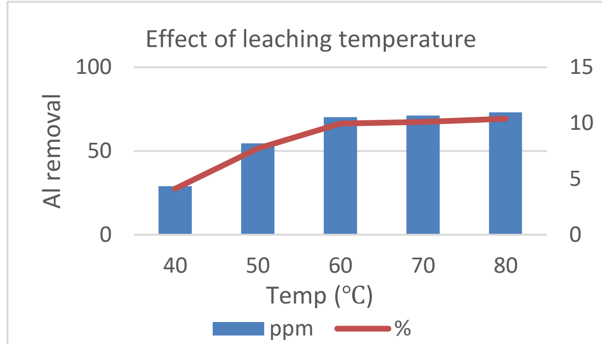

Fig 4: Effect of leaching Temperature

3.3.4 Effect of leaching temperature on alumina leaching

Temperature plays a critical role in efficient leaching. This is because reacting molecules have a tendency to collide with one another and the walls of the vessels in which they are housed, producing a perfect elastic collision, as their temperature rises. The effect of leaching temperature on alumina leaching from the Ededimeji termite mound was studied at varying temperatures (40, 50, 60 70 and 80 ℃). Alumina`s dissolution rate increases as the temperature increases as shown in Fig 4. This occurs because greater temperatures give the interacting particles more kinetic energy, which causes more direct collisions and a rise in the yield of alumina. This is consistent with [11, 14] who discovered that the reason for the notable increase in the leaching rate is probably because a higher temperature accelerates the molecule's thermal motion, increasing the contact surface between a single particle and the leaching agent, as well as because a higher temperature causes the barrier of products layer to thin and the diffusion flux to increase. From this study, 80 ℃ was chosen as the optimum temperature indicating that temperature had a positive influence on the leaching of alumina from Ededimeji termite mound [13].

4. CONCLUSION

The results of this investigation on the thermal treatment of termite mound leachability suggest that termite mounds might be a good source of alumina production via an acidic pathway. The availability of termite mounds and the moderate conditions required in the leaching process for the complete dissolution of Al content are sellable benefits. The optimal conditions for the alumina to dissolve in HNO_3 include calcination temperature of 750 ℃ for 60 min at a leaching temperature of 80℃. This study shows that Ededimeji termite mounds can be used as the primary raw material for Alumina production.

5. ACKNOWLEDGEMENTS

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