Research Article

An Alternative to Clay in Building Materials: Red Mud Sintering Using Fly Ash via Taguchi's Methodology

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“Red mud” or “bauxite residue” is a highly alkaline waste generated from alumina refinery with a pH of 10.5–12.5 which poses serious environmental problems. Neutralization or its treatment by sintering in presence of additives is one of the methods for overcoming the caustic problem as it fixes nearly all the leachable free caustic soda present in red mud. In the present study, feasibility of reducing the alkaline nature of red mud by sintering using fly ash as an additive via Taguchi methodology and its use for brick production, as an alternative to clay, is investigated. The analysis of variance (ANOVA) shows that sintering temperature is the most significant parameter in the process. A pH of 8.9 was obtained at 25–50% of red mud and 50–75% fly ash with water and temperature of 1100°C. Alternatively 50% of red mud can be mixed with 50% of fly ash with water at temperature of 1200°C to get a pH of about 8.4. The mechanism of this process has been explained with also emphasis on chemical, mineralogical, and morphological analysis of the sintered red mud. The results would be extremely useful in utilization of red mud in building and construction industry.

1. Introduction

The Bayer process of extraction of alumina from bauxite remains the most economical process till date. In the Bayer process, the insoluble product generated after bauxite digestion with sodium hydroxide at elevated temperature and pressure to produce alumina is known as “red mud” or “bauxite residue” which is highly alkaline in nature with a pH of 10.5–12.5. Red mud is a mixture of compounds originally present in bauxite and of compounds formed during the Bayer cycle. The main reaction of aluminum oxide dissolution in the Bayer process is as follows:

Depending on the raw material processed, 1–2.5 tons of red mud is generated per ton of alumina produced [1].

An enormous quantity of red mud is generated worldwide (~75 million tons) every year posing a major environmental problem. In India, about 4.71 million tons/annum of red mud is produced which is 6.25% of world's total generation [2]. Red mud is disposed as semidry material in red mud pond or abandoned bauxite mines and as slurry having a high solid concentration of 30–60%. Problems associated with the disposal of red mud waste include its high pH, alkali seepage into underground water, safety in storage, alkaline air borne dust emissions, and the vast area of land required for disposal. Up to 2 tons of liquor with a significant alkalinity of 5–20 g/L caustic (as Na_2CO_3) accompanies every ton of dry mud. Neutralization of red mud will help to reduce the environmental impact caused due to storage activities of the residue and will also open opportunities for the reuse of the residue which till now has been prevented because of the high pH.

It will also reduce degradation of clay or synthetic liners of red mud ponds. The overall risk of groundwater contamination would be reduced significantly by neutralizing red mud.

The literature shows that red mud can be treated with any of these methods to make it environmentally benign: acid leaching, CO₂ treatment, bioleaching, seawater neutralization, and sintering. A review of neutralization processes of red mud and its utilization is given by investigators [3].

A considerable research has been done on the utilization of red mud as a raw material for the production of a range of building products by sintering of red mud. Red mud can be used as a constructional/building material in bricks, blocks, light weight aggregates, roofing tiles, glass ceramics, cement industry as cements and special cements, and concrete industry. Red mud is made into useful ceramics articles by mixing 51–90% of the weight of red mud with 10–49% of the weight of at least one mineral and/or silicate containing material, shaping the mixture and firing it at a temperature of 950°–1250°C [4]. A patent [5] claims a process for manufacturing fired bricks wherein 50–90 wt% of red mud can be used along with clay and a water fixing agent. The raw bricks are dried with heated gases at a temperature below 70°C and subsequently fired at a temperature between 900°–1100°C. Though all these studies are based on sintering of red mud and its use, very little has been stated about the change in pH with the sintering temperature and other parameters. The ways in which these factors may interact in the neutralization or treatment process are poorly understood.

Hence the present study focuses on the study of change in pH in the sintering process due to a wide range of factors. The treatment process involves mixing of silicate material such as fly ash with red mud in presence of water. Fly ash is the by-product of burning of coal and is available from coal-based thermal power plants and other coal burning centers. An estimated availability of fly ash in India is about 80–100 million tons per year [6]. Every thermal power plant generating 1000 MW power on an average produces 3500 tons of fly ash every day [6]. It is estimated that an area of about 28,000 ha of land will be necessary to dump all the fly ash available in the country. The present utilization level of fly ash in India is less than 10% [6]. Fly ash contains 1 to 100 µm suspended particulates affecting plant growth besides being a health hazard [7]. Indian red mud is rich in titania and iron in alkali oxides while the fly ash is rich in silica and alumina. These features contribute to the optimum composition for brick making [7].

Hence use of fly ash has been made as a silicate material in sintering of red mud. In this way both the wastes can be made nonhazardous and can be reused.

Taguchi's experimental methodology has been employed to evaluate the pH values. Taguchi's design of experiments has been used extensively in product quality assessment and several other studies [8–12]. Taguchi's fractional factorial design of experiments has been used to examine the effect of significant parameters such as weight of red mud, weight of silicate material added, volume of water, and reaction temperature on the response characteristic (pH of red mud slurry). The average values and the signal to noise (S/N) ratio of the quality response characteristic for each parameter

at 3 levels of their values have been calculated from the experimental data. The response curves have been graphically represented to reflect any change in the quality characteristic and S/N ratio with the variation in process parameters. These response curves are used to identify the effects of various parameters on the response characteristic. Significant parameters have been identified by using the analysis of variance (ANOVA) on the experimental data.

2. Materials and Methods

2.1. Materials. Red mud from an alumina refinery situated at the eastern coast of India (18°49'9"N, 82°57'52"E) was ground to 100 mesh size and used for the study. Chemical composition of red mud is Al₂O₃ (16–17.5%), Fe₂O₃ (53.5–56%), SiO₂ (6–7%), TiO₂ (5–6%), Na₂O (4–5%), and CaO (2–3%). Mineralogically, red mud contains phases of undigested alumina, aluminosilicates, and phases of iron and titania. Alumina is in form of undigested gibbsite, alumogothite, and sodalite along with silica and sodium (sodium aluminosilicates); iron is in form of hematite, alumogothite, siderite, and ilmenite; titania is in form of anatase, rutile, and ilmenite; and calcium is in form of calcite. Average particle size of red mud is less than 10 microns with specific surface as 20.26 m²/g.

The mineralogical composition of untreated red mud is

gibbsite: Al(OH)₃,

sodalite: Na₂O Al₂O₃·2SiO₂·2H₂O,

hematite: Fe₂O₃,

alumogothite: FeAlOOH,

anatase: TiO₂,

rutile: TiO₂,

ilmenite: FeTiO₃,

siderite: FeCO₃,

calcite: CaCO₃.

Sodium present in red mud (total caustic soda) is in two forms: free or soluble caustic soda and bound caustic soda. Free caustic soda is the entrained liquor in the red mud slurry which gets incorporated during digestion process and remains with red mud in spite of repeated washings. Free soda is in the form of NaOH, Na₂CO₃, NaAlO₂, and so forth. The pH of the red mud is due to the presence of these alkaline solids in red mud. Bound soda is in the form of sodalite complex which can be stated as "NAS" phases: 3(Na₂OAl₂O₃2SiO₂)Na₂X (X = CO₃²⁻, 2OH⁻, SO₄²⁻, 2Cl⁻) (Kurdowski and Sorrentino, 1997) [13]. In red mud, about 20–25% is the free or soluble caustic soda while the rest is in the form of sodalite complex.

Fly ash has the following composition: 57–58% SiO₂, 34–36% Al₂O₃, and 4–6% Fe₂O₃. Mineralogically fly ash contains about 75–80% quartz (SiO₂) with remaining mullite (2Al₂O₃SiO₂), alumina in the form of mullite, and iron as hematite.

2.2. Experimental Setup. Red mud, fly ash, and water were mixed in different proportions as per the experiments designed by Taguchi's methodology and kept in a crucible at different temperatures in a muffle furnace (CHEMINCO, Kolkata). After sintering the mixture for a fixed duration, the crucible was cooled overnight and the mixture was taken in a beaker and 100 mL distilled water was added. The mixture was stirred for 30 min using magnetic stirrer (Eltek M S 204). The pH of the slurry was measured after each test using LabX Light Titrator (Potentiometric Titrator), Mettler Toledo GmbH, Switzerland.

2.3. Taguchi's Design of Experimental Methodology. Dr. Taguchi of Nippon Telephones and Telegraph Company, Japan has developed a method based on "orthogonal array" experiments which gives much reduced "variance" for the experiment with "optimum settings" of control parameters. Taguchi methods [10] use orthogonal array distribution to design an experiment. A traditional experimental design to compare four independent variables at three different levels each requires a large number of individual experiments (eighty-one experiments). The logistical and resource implications of this experimental design make these experiments very difficult to carry out. By using L_9 orthogonal array with Taguchi method to design an experiment, a study involving 4 factors at 3 different levels can be conducted with only nine individual experiments. Taguchi method consists of 3 phases: designing the experiment, running and analyzing, and confirming and validating the experimental results. In an orthogonal array, each array can be identified by the form $L_A(B^C)$; the subscript of L , which is designated by A , represents the number of experiments that would be conducted using this design, and B denotes the number of levels within each column which denotes how many levels could be investigated, while the letter C indicates how many factors or variables could be included in the experiment [10].

L_9 orthogonal array consisting of four control parameters (factors), A, B, C, and D, is given in Table 1 and set at minimum, middle, and maximum value (i.e., levels 1, 2, and 3). The parameters were decided based on the preliminary experimentation carried out. In all nine experiments had to be conducted. Each experimental run was repeated thrice. The pH values (replicated thrice as R_1 , R_2 , and R_3) obtained for each experiment of the L_9 array are shown in Table 2. Subsequently analysis of variance (ANOVA) based on the Taguchi method was carried out using the values to determine the contribution of each parameter to the process of neutralization.

2.3.1. Treatment of Red Mud at High Temperature. The initial pH of the red mud slurry was 10.7 (50 gm of red mud was stirred with 100 mL of distilled water for 30 min). After each experimental run, designed as per Taguchi's experimental methodology, pH of slurry was measured. The slurry sample with the lowest pH value and close to 7.0 (of experimental run number 7) was filtered using Whatman paper no. 40. The filtered red mud was dried and analyzed.

TABLE 1: Experimental layout of parameters and their levels.

Factors	Parameters	Units	Level			Observed value
			1	2	3	
A	Weight of red mud	g	10	20	30	pH
B	Weight of silicate material	g	10	20	30	
C	Volume of water	mL	8	10	12	
D	Temperature	°C	900	1000	1100	

Chemical constituents of red mud mix obtained after sintering at the optimized condition were analyzed by using Wet Chemical Method. Untreated red mud, fly ash, and sintered red mud were analyzed mineralogically for determination of phases using XRD, X-ray diffractometer (PANalytical X-Pert Pro), using Cu $K\alpha$ radiation ($\lambda = 1.54060 \text{ \AA}$). Scanning electron microscopy (SEM) has been conducted to study the morphology of neutralized red mud using Electron Probe Analyzer (MAKE: JEOL, Japan, JXA-840A).

The slurry sample was prepared from red mud sintered at the optimized condition of parameters (of experimental run number 7) and 100 mL distilled water and steered thoroughly. pH rebound phenomena were studied for 2-week time duration with this sample of red mud slurry.

2.3.2. Analysis of Experimental Data. The experimental data generated was analyzed by analysis of variance (ANOVA). Taguchi excel sheet is used for all the analysis which is based on Taguchi method for determination of main effects of the process parameters.

R' values are the ratio of neutral pH (7) to experimental pH value. R'_1 , R'_2 , and R'_3 are the values calculated from R_1 , R_2 , and R_3 . On this basis the "higher-is-better" quality characteristic was used in the analysis of experimental data. The plot of response curves and analysis of variance for raw data and S/N ratio data was used for the analysis of results. The mean of the response characteristic pH (Y_{opt}) at the optimal condition was estimated as

$$Y_{opt} = \frac{T}{N} + \left(A_{1avg} - \frac{T}{N} \right) + \left(B_{3avg} - \frac{T}{N} \right) + \left(C_{3avg} - \frac{T}{N} \right) + \left(D_{3avg} - \frac{T}{N} \right), \quad (2)$$

where T/N is the overall mean of the response, where T is the grand total of all results and N is the number of experiments, and A_{1avg} , B_{3avg} , C_{3avg} , and D_{3avg} represent average values of response at the first level of parameter A and third levels of parameters B, C, and D, respectively.

In order to confirm that the optimal parametric values using Taguchi's methodology are valid, selected confirmatory experiments were carried out under optimal conditions. The average of the results of the confirmation experiment is then compared with the anticipated average based on the optimal parameters and levels tested by Taguchi's methodology. The Y_{opt} value obtained is 0.78 and pH value of which comes out to be 8.969 which is close to the optimized experimental

TABLE 2: L_9 table and observed values of pH.

Run	Factors				Final pH			Ratios of pH			S/N ratio
	A	B	C	D	R_1	R_2	R_3	R'_1	R'_2	R'_3	
	Weight of red mud	Weight of silicate material	Volume of water	Sintering temp.							
1	20	30	8	1000	9.12	9.25	9.3	0.77	0.76	0.75	-2.40
2	20	20	12	900	10.7	10.65	10.54	0.65	0.66	0.66	-3.63
3	30	10	12	1000	9.65	9.62	9.59	0.73	0.73	0.73	-2.76
4	10	20	10	1000	9.58	9.32	9.31	0.73	0.75	0.75	-2.56
5	30	20	8	1100	9.10	9.15	9.18	0.77	0.77	0.76	-2.32
6	30	30	10	900	10.68	10.55	10.61	0.66	0.66	0.66	-3.62
7	10	30	12	1100	8.9	8.92	8.88	0.79	0.78	0.79	-2.09
8	20	10	10	1100	9.27	9.25	9.3	0.76	0.76	0.75	-2.43
9	10	10	8	900	10.69	10.6	10.582	0.65	0.66	0.66	-3.62

TABLE 3: Average and main effects of ratios of pH values—Raw and S/N.

Factors	Raw data, average value			Main effects		S/N data, average value			Main effects (S/N data)	
	L_1	L_2	L_3	L_2-L_1	L_3-L_2	L_1	L_2	L_3	L_2-L_1	L_3-L_2
A	0.74	0.73	0.71	-0.01	-0.02	-1.49	-2.80	-3.01	-1.30	-0.21
B	0.72	0.72	0.74	0.00	0.02	-2.92	-2.89	-2.70	0.03	0.19
C	0.72	0.73	0.72	0.01	0.00	-2.89	-2.79	-2.83	0.10	-0.04
D	0.66	0.75	0.76	0.09	0.01	-3.62	-2.52	-2.37	1.11	0.15

value of 8.8–8.92 in run 7. The deviation between actual and determined pH by Taguchi's methodology is much within 5%, and hence it is confirmed that the optimal parametric values determined by Taguchi's design are valid.

2.4. Utilization of Red Mud. Red mud was used as an alternative to clay in making bricks. Four standard size bricks were made by mixing suitable proportions of red mud, fly ash, and water. The bricks were made in commercial fly ash brick factory machine, that is, Brickman machine. They were pressed at $\sim 200 \text{ kg/cm}^2$, dried overnight in an oven at 110°C , and sintered at 1100°C for 2 hours. The composition of the raw materials used and the properties of the bricks obtained are given in Table 6. The brick samples were tested for cold compressive strength (CCS), water absorption (WA), efflorescence and bulk density.

3. Results and Discussion

3.1. Effect of the Process Parameters on pH. Table 3 provides the raw data for the average value ratios of pH and S/N ratio for each parameter at levels 1, 2, and 3. The sintering temperature from level 1 to level 2 has the greatest influence on value of pH. The difference between the levels also shows the same trend. Figure 1 shows the response curves of the individual parameters on the ratio of pH values, that is, R' values ($7/\text{experimental pH value}$) and S/N ratio.

From Figure 1, it can be seen that the ratio of pH decreases with the increase in the weight of red mud (factor A). It is due to the increase in free caustic soda in the slurry with the increase in quantity of red mud. There is a sharp decrease in the ratio of pH values when factor A changes

FIGURE 1: Main effects of each factor on ratio of pH along with S/N ratio of treated red mud slurry (ANOVA analysis). A: wt. of red mud (g); B: wt. of silicate material (g); C: vol. of water (mL); D: temp. ($^\circ\text{C}$).

from level 1 to 2 and also when the quantity of red mud changes from level 2 to 3. Sintering temperature (factor D) definitely decreases the pH of the slurry as a sharp increase in the ratio of pH is observed from level 1 to 2. Further increase in the ratio of pH values (i.e., decrease in pH) is observed from level 2 to level 3. Not much change in pH is observed at 900°C but the change is seen above this temperature. The increase in temperature decreases the pH substantially due to the fusion of free soda with the silica and alumina present in fly ash. The overall results convey that the pH

TABLE 4: ANOVA table.

Factors	Total variance of each factor	Degree of freedom	Variance	Variance ratio	Pure sum of sq.	Percent contribution
A	0.001	2	0.000	10.5651	0.00062816	1.090
B	0.002	2	0.001	29.82904	0.00189326	3.079
C	0.000	2	0.000	4.087215	0.00020274	0.422
D	0.060	2	0.030	915.4204	0.06005186	94.480
Error	0.001	18	0.000	1	0.00032836	0.929
Totals	0.064	26	0.032			100.000

TABLE 5: Mineralogical phases in sintered red mud.

Compound name	Chemical formula	JCPDS no.
Corundum	Al_2O_3	00-046-1212
Sillimanite	Al_2SiO_5	00-038-0471
Jadeite	$\text{NaAlSi}_2\text{O}_6$	00-022-1338
Burnt ochre	Fe_2O_3	00-033-0664
Aluminum iron oxide	AlFeO_3	00-030-0024
Brookite	TiO_2	00-015-0875
Calcium oxide	CaO	00-037-1497
Calcium silicate	Ca_2SiO_4	00-031-0299
Grossular	$\text{Ca}_3\text{Al}_2(\text{SiO}_4)_3$	00-039-0368
Sodium aluminum oxide	NaAlO_2	00-033-1200

of the slurry is mostly affected by the sintering temperature. The lowest pH is obtained with the weight of red mud at level 1, weight of silicate material at level 3, water at level 3, and temperature at level 3 ($A_1B_3C_3D_3$). The contribution of individual parameters was weighted as shown in Table 4 with analysis of variance (ANOVA) to see their effect on desired response characteristic (pH). The most significant parameters are found to be the sintering temperature with a very high contribution of 94.48% followed by weight of red mud (1.09%) and weight of silicate material (3.08%) and water (0.5%).

3.2. Confirmation Experiment. From Table 2, it can be seen that the experimental condition ($A_1B_3C_3D_3$) was the one in which the lowest value of pH value was achieved (8.88–8.9) which was close to the neutral value of 7 in all the 3 replications. As per the Guidelines of Australian and New Zealand Environment and Conservation Council (ANZEX) and Agriculture and Resource Management Council of Australia and New Zealand (ARMCANZ), the liquor being strongly alkaline with a high pH requires neutralization to a pH below 9 with an optimum value of 8.5–8.9 before becoming environmentally benign [14]. The optimized condition obtained from Figure 1 was $A_1B_3C_3D_3$ having pH value of about 8.90 which is the lowest of all the values. Hence these conditions were fine-tuned by increasing the temperature to 1200°C and the pH achieved was 8.34 which is within the acceptable limit. Hence the final optimized conditions to achieve a pH of 8.9 were 25–50% of red mud and 50–75% silicate material, with water (10%) and a temperature of 1100°C. Alternatively 50% of red mud can be mixed with 50% of fly ash with water at temperature of 1200°C to get a pH

of about 8.4. The results are in accordance with those obtained by investigators [4]. In absence of sintering, studies carried out at Jamaica [15] show that in neutralization of red mud with acidic fly ash, a large amount of fly ash is required with very slow neutralization rate (approx. 150 days to get a pH from 12.5 to 11).

3.3. Red Mud Analysis after Neutralization

3.3.1. Chemical Analysis. The chemical analysis of red mud after sintering is Al_2O_3 : 24.50%, Fe_2O_3 : 31.92%, SiO_2 : 34.50%, TiO_2 : 2.53%, Na_2O : 4.5%, and CaO : 1.09%. Sintered red mud has high alumina content, low iron content, and high silica content due to the addition of silicate material (fly ash) and subsequent sintering of the mixture as compared to the original red mud.

3.3.2. Formation of Different Phases. To investigate the phase structures of the red mud, the X-ray diffraction pattern was measured. Figure 2 shows the XRD pattern which shows a well crystallized sintered red mud powder. The peaks of the red mud show the phases as shown in Table 5. Alumina present in red mud in the hydroxide form has been converted into corundum which is alpha phase of alumina. Alumina has also reacted with silica from fly ash to form aluminosilicates (sillimanite) which impart strength to the fused material. Caustic soda has fused with alumina and silica to form sodium silicates and sodium aluminosilicates. Some of the free soda is still present in the sintered red mud in form of sodium aluminate. Hence pH value of above 8.9 is observed after the process. Calcium aluminosilicates and calcium silicates are also formed.

Some of the reactions taking place in the process are

3.3.3. Morphology of Red Mud. Figure 3 shows the morphological structure of original (untreated) red mud as seen in SEM which indicates scattered fine particles of about 1 micron size. Globular particles with smooth surface which are formed due to the formation of sodium aluminosilicates are observed (Figure 4) in sintered red mud.

TABLE 6: Composition and properties of bricks.

	Brick composition			Properties of bricks			
	Red mud (%)	Fly ash (%)	CCS (kg/cm ²)	WA (%)	Efflorescence	Bulk density (g/cc)	Class of brick
Composition 1	50	50	31.7	24.0	Nil	1.47	Class 3.5
Composition 2	45	55	66.93	24.50	Nil	1.49	Class 5
Composition 3	40	60	86.73	23.8	Nil	1.50	Class 7.5
Composition 4	35	65	134.18	24.10	Nil	1.51	Class 12.5

the same even after two weeks. This means that there is no more caustic soda leaching after its initial leaching from sintered red mud.

4. Conclusion

The paper has discussed in detail the feasibility of sintering of red mud to deal with the caustic soda in it using Taguchi's methodology. The pH of red mud slurry reaches values within the acceptable limits after sintering red mud with a silicate material (fly ash) at a very high temperature of 1100–1200°C. The method of treating red mud with fly ash along with water can be a successful process to tackle the caustic soda problem. Also the resultant material can be reused as a construction material in place of clay. Taguchi's experimental methodology is found to be a valuable tool. The ANOVA analysis using Taguchi methodology shows that the most significant parameters are temperature of calcinations (94.48%) followed by weight of red mud (1.09%) and quantity of fly ash (3.079%). The optimized conditions to achieve a pH value of about 8.9 were 25–50% of red mud mixed with 50–75% of fly ash with water (10%) and a temperature of 1100°C. Alternatively about 50% of red mud with 50% of fly ash and 1200°C can be used where the pH obtained is within the permissible limit of 8.4.

The study shows that treatment of red mud with fly ash at a very high temperature lowers down the pH and hence alkalinity of red mud slurry permanently without pH rebound. This also modifies the physical characteristics as is evident from the morphology of red mud. Sintering of residue can be carried out to fix all leachable soda and to achieve high strength during the sintering process. Thus red mud can be used as an alternative to clay material in building and construction industry. Both red mud and fly ash can be reused. However, the cost would be very high due to the high energy consumption as an elevated temperature is required for sintering. But the process would certainly help in reducing pollution of soil, air, and water and would considerably save resources of mother earth.

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