Studies On The Chemical And Physical Characteristics Of Selected Clay Samples

By

Sani Aliyu, Bashir Garba, B. G. Danshehu and A. D. Isah*

Sokoto Energy Research Centre, Usmanu Danfodiyo University, Sokoto

*Department of Forestry and Fisheries, Usmanu Danfodiyo University, Sokoto

Abstract

The chemical composition as well as the refractory properties of some selected clay samples obtained from three different Local Government Areas of Sokoto State, Nigeria was studied. The clay samples were coded A, B, C, D and E. The chemical analysis was performed using X-Ray Fluorescent – (XRF) method, while the physical properties investigations were carried out following American Society for Testing and Material (ASTM) stipulates standards. at the National Metallurgical Development Centre (NMDC) Jos, Plateau State, Nigeria. Test specimens were prepared using each of the samples by standard methods. They were then tested for properties such as apparent porosity, bulk density, cold crushing strength, thermal shock resistance, firing shrinkage, and refractoriness. The result of the chemical composition analysis shows that the, the clay is rich in Oxides of Silica (SiO_3), Aluminum (Al_2O_3), and Iron (Fe_2O_3) with other oxides in trace amount. The overall experimental analysis carried out, shows that all the samples are siliceous in nature and of the alumino-silicate refractories that are classified as kaolinitic fireclay with appreciable and reasonable values of the refractory properties that are comparable to the standards. It was shown in these studies that on the basis of the physio-chemical characteristics of this kaolinitic fireclay deposit, it can successfully be processed for use in the paper industry as filter, in furnace lining and in the development of improved wood burning stoves in addition to usual pottery activities.

1.0 INTRODUCTION

Clay is regarded by an ordinary person as a kind of natural earth. When mixed with water; it becomes plastic and mouldable and becomes hard again on drying and firing [1]. Clay is a complex inorganic mixture, whose composition varies widely depending on the geographical location. It is a natural substance occurring in great abundance in nature, being constantly formed on the earth's surface as a result of rock weathering [2]. Clay is composed of silica (SiO_2), Alumina (Al_2O_3) and water (H_2O) plus appreciable concentration of oxides of iron, alkali and alkaline earth, and contains groups of crystalline substances known as clay minerals such as quartz, feldspar and mica [1]. Clay deposits occur as a mixture of different clay types, one group or type normally being dominant. The term "Clay" applies to both materials having particle size of less than 2 micrometers and to the family of minerals that have similar compositions and structural characteristics. [3]. Clays have several properties; both chemical and physical properties. These properties are responsible for the various areas of application of clays. Clays are used as ceramic and refractory materials, used in the formulation of drilling fluids, as binders in foundry moulds, as binders for iron ore pellets in metallurgy, as catalyst in petroleum refining and production of petrochemicals, soil remediation, bleaching of oils, clarification of wines etc[4]. The properties of clay units depend on the mineralogical compositions of the clays used to manufacture the unit, the manufacturing process and the firing temperature [5]. Clay is composed mainly of alumina–silicates, but also contain fine grained deposits of non- aluminosilicates such as shale and some argillaceous soil [2].

The percentage of the minerals oxides (Fe_2O_3, MgO, CaO etc) in the clay ultimately determine the areas of applications of the clay such as in bricks, floor tiles, paper etc, while the presence of the alkali metals oxides (Na_2O, K_2O, CaO, etc) determine their suitability for making ceramic products[5]. Clays are usually classified into two basic types according to their geological origin, primary clay (Kaolins) and secondary clay. They are then sub-divided by particle size and variation of impurities inherent in the native form of the clay. The forces of nature and numerous geological upheavals have caused the various impurities in the form of the minerals and metal compounds have been added, and others, being soluble, have been leached out. Thus, it is a variation of the impurities in the basic formula that accounts for the different types of clay, and consequently, of clay bodies. A theoretical formula for this substance is as shown [6,7].

$Al_2O_3.2SiO_2.2H_2O$………………… (1)

According to Raw Materials Research and Development Council of Nigeria [8], clays are one of the major Nigeria minerals deposits cover with an estimated proven reserves of billions of tones, and these clays deposits are scattered all over the states in the country.

One of the best-known applications of clay is in the manufacture of such articles as pottery. Another major application of kaolinite clay is in the paper industry where it is used either as filler or for coating paper. The metallurgical industry employs clay mixed with sand to form moulds that are used for casting operation. In the oil industry, clays are used in the formation of drilling fluids in drilling of oil wells and also as important constituent of catalyst for refining of crude petroleum and also in the separation of gasoline, gas and coke. [9]. Clay based Refractories are the fireclay materials and are made from clays containing the aluminosilicates mineral-kaolinite ($Al_2[Si_2O][OH]_5$) plus impurities such as alkalis and iron oxides. [10, 11] .Refractories are classified based on the impurity content and the alumina-to-silica ratio (Al_2O_3/SiO_2) [12].

A lot of research has been carried out on the conversion of clays for industrial uses and other applications. Omowumi [6] observed that the properties of Refractories produced from Onibode, Ara- Ekity, Ibamajo and Ijoko clay samples compared favourably with imported fire clay refractories. Authors [13] found that Onibode refractory clays are suitable for the production of refractory bricks for furnace building. Akinbode [14] carried out an investigation on the properties of termite hills as refractory material for furnace lining. In his report, he observed that the refractory properties of termite hill material which include porosity, density, dimensional change and permeability are very similar to known refractory materials for furnace lining. Investigation on the refractory properties of clays from Kuru, Alkaleri, Barkin-ladi and Bauchi, revealed that refractory clays from Alkaleri and Barkin-ladi are suitable for furnace construction due to higher thermal shock resistance, crushing strength, bulk density and refractoriness values[9]. Research work by an author, [15] revealed that kaolinitic clays from Kankara, Jos and Oshiele are of the residual variety and also very suitable for paper, paint and pharmaceutical production. Experimental analysis on the refractory properties of local clay materials for furnace building by an author, [16] revealed that all the clay samples were classified as low alumina with kaolinitic nature. An investigation carried out by Hussani [17] on the refractory properties of bricks produced from five Nigerian clays revealed that all the samples were found to possess good refractoriness (P.C.E) with excellent firing shrinkage value that fall within 3-5% range of the imported bricks. [18]They also exhibit different refractory characteristics with respect to thermal shock resistance, refractoriness, porosity, permeability, bulk density, modulus of rupture and water absorption. Hassan [19] investigated the effect of additives like saw dust, graphite and asbestos on Kankara clay. He discovered that a good thermal insulating fire clay brick could be produced from this clay by the addition of graphite and saw dust (15%).This study is aimed at characterizing selected clay samples to determine their chemical and refractory properties for industrial applications.

Theoretical Background

***Bulk Density**:* This is responsible for the overall weight coming upon the foundation of a refractory structure. Density of all ceramic materials is an indirect ions measurement of their capacity to store heat – a particularly useful property in heat exchanger installations [20].. Bulk density is the mass per unit volume of the materials ignoring the volume occupied by pores. It depends upon the true specific gravity and the porosity [21] .

Apparent Porosity: The ability to be impervious to gases and liquids. Bricks have pores formed as water and gases are given off during firing process [21]. Clay samples with low apparent porosity have greater resistance to penetration by slags and fluxes, resistance to corrosion and erosion and usually lower gas permeability than those with high porosity. There is an inverse relationship between thermal conductivity and porosity of refractory bricks.

***Shrinkage on Firing**:* This is a property of clay material which makes them to undergo least structural changes and disintegration while being heated. The firing shrinkage always gives an indication of the ability of clay bricks to withstand thermal shock.

Cold Compressive Strength*:* This is the ability of clay to bear load. This is an important indicator of the ceramic materials to withstand handling or shipping and impact or abrasion at low temperature. It does not, however, give an indication of the clay's strength at a given temperature

***Refractoriness**:* This is the resistance of clay to fusion and softening at high working temperatures. It is the maximum temperature a material can withstand after which it will fail (break). Refractoriness is measured by a standard technique and practically reported in pyrometric cone equivalents, PCEs. The test measures the softening point of a refractory material by comparing the behaviour of its test cone with reference cones of standard composition designated by PCEs values between 12 and 42 in the Orton series.

***Thermal Shock (Spalling) Resistance**:* This is the ability of sample to withstand heating and cooling cycles before failure. Is the ability of the sample to withstand sharp (sudden) temperature changes without fracture (spalling). It is the number of thermal cycles (i.e. heating and sharp quenching in water or air) a sample can undergo before failure.

2.0 Materials and methods

2.1 Materials

Five clay samples coded A(Sokoto), B(Dange1), C(Dange 2), D(Wurno1) and E(Wurno 2),were obtained from Yargabas in sokoto North Local Government, Dange in Dange/Shuni Local Government, and Wurno in Wurno Local Government areas of sokoto state, Nigeria. In these studies, Samples (A and D) of the clays were wet and in large chunk they were moulded into balls of about 30 mm in diameter and then exposed to ambient sun dried at temperature 35^oC for 7 days. On the other hand, samples (B, C, and D) were in dried form initially. About 2.0 kg of each sample was collected and placed in small polythene bags for various analyses.

The equipment and tools used include sieve, basin, containers, wooden moulds, mechanical dryer, electric furnace, measuring tape, Vernier calliper, measuring cylinder, matter balance, electric oven and pyrometric cone.

2.2 Methods

The chemical composition of the clay was determined using X-ray fluorescence (XRF) method. This is a non-destructive analytical technique used to identify and determine the concentration of elements present in solid, powdered and liquid samples.

2.2.1 Determination of the Chemical Composition.

The chemical composition of the raw clay samples in wt % of (SiO_3, Al_2O_3, Fe_2O_3, etc) was examined using X-ray fluorescence/gravimetric methods, carried out at National Geoscience Research Laboratories Center, Kaduna, Nigeria.

Sample Preparation: The sample was ground and sieved to -75 µm particle size. 4g of the sieved clay particles were intimately mixed with 1g of lithium tetra borate binder ($Li_2B_4O_7$) and pressed in a mould under a pressure of 10-15 ton/m^2 into pellets. The pressed pellets were dried at 110 ^0C for 30 minutes in an oven to get rid of absorbed moisture and were finally stored in desiccators for analysis.

Analysis:

The spectrometer was switched on and allowed to warm up and also to stabilize the optics and the X-ray tube. It was then calibrated to determine the expected element present in the sample. The sample was run using the prepared program and the concentrations of the elements present in the sample were automatically calculated and displayed by the spectrometer.

2.2.2 Determination of the physical properties

Evaluation of the refractory properties of the various clay samples was carried out using standard methods and procedures. The refractory properties determined include; apparent porosity, bulk density, cold crushing strength, thermal shock resistance, linear shrinkage and refractoriness.

Determination of Apparent Porosity

For each of the clays, the specimen of dimensions, 5cm x 5cm x 4cm size was tested. The specimens were dried over 12 hours at 110^{0}C in preparation for the test; they were taken directly from the oven for the test. The dry weight in air (D) of each specimen was measured. The specimens were then transferred into a 250ml beaker in empty vacuum desiccators. Water was then introduced into the beaker until the tested pieces were completely immersed. The specimens were allowed to soak in boiled water for 30 minutes being agitated from time to time to assist to release trapped air bubbles. The specimens were transferred into empty vacuum desiccators to cool. The soaked weights (W) were recorded. The specimens were then weighed suspended in water using beaker place on balance. This gave suspended weights (S). The apparent porosity was determined according to [22]

ASTM Standards C 20/2007 using equation (2)

$$\textbf{\textit{Apparent Porosity}} = \frac{W-D}{W-S} \times 100 \;\ldots\ldots\ldots\; (2)$$

Where, W = Soaked weight

D = Dried weight

S = Suspended weight

Determination of Bulk Density

Representative samples of each measuring (6cm ×6cm × 1.5cm) were cut from the fired test samples. The specimens were air dried for 24 hours and then dried at 110 ^{o}C, cooled in a desiccators and weighed to the accuracy of 0.008(dried weight) after which the specimens were transferred to a beaker and heated for 30 minutes to assist in releasing the trapped air. The specimens were cooled and soaked weight (W) taken. The specimens were then suspended in water using beaker placed on a balance. The suspended weight (S) was taken. The bulk density was calculated from the equation.

$$\textbf{\textit{Bulk Density}} = \frac{DP_W}{W-S} \text{ g/cm}^3 \;\ldots\ldots\ldots\ldots\ldots\; (3)$$

Where, D = Dried weight

W = Soaked weight

S = Suspended weight

P_w = Density of water

Determination of Cold Crushing Strength Tests

Pieces of the test bricks produced using the clay samples were prepared to standard cube sizes, of dimensions 50mm wide and 50mm high. They were then air dried for 24 hours and oven dried at a temperature of 110 ^{0}C for 12 hours and then fired in a furnace at a temperature of about 1200 ^{0}C for 6 hours. They were then cooled to room temperature. Each specimen was then taken to the tensiometer where load was applied axially to the piece until crack was noticed. The load at which the specimen cracked was noted, which represents the load required for determining cold crushing strength of the test specimen. Cold crushing strength was then determined using equation (4)

$$CCS = \frac{\text{Maximum load (KN)}}{\text{Cross-sectional area (m2)}} = P/A \ldots(4)$$

Where,

C.C.S = Cold Crushing Strength

P = Applied Load

A = Area of Load Applied

Determination of Thermal shock Resistance

Specimen measuring 5cm x 5cm 4cm was used for this test. The prepared samples were inserted in a furnace which has been maintained at 900 ^{o}C. This temperature was maintained for 10 minutes. The specimens were removed with a pair of tongs from the furnace one after the other and then cooled for 10 minutes and observed for cracks. In the absence of cracks (or fracture),the specimen were put back into the furnace and reheated for a further period of 10 minutes and then cooled for another 10 minutes. The specimens were returned to the furnace for further 10 minutes. The process was continued until the test pieces were cracked. The number of cycles of heating and cooling before cracking for each specimen was recorded as its thermal shock resistance.

Determination of Linear Shrinkage

To determine the shrinkage properties of the specimen, the specimen were pressed in a fabricated wood box of size 5x5x4cm. The rectangular test pieces were marked along a line in order to maintain the same position after heat treatment. The distance between the two ends of the slab was measured with vernier calliper. The samples were air dried for 24 hours and oven dried at 110^{O}C for another 24 hours. They were then fired for 6 hours. The test pieces were cooled to room temperature and measurements taken. The linear shrinkage was calculated from equation (5).

$$\text{Linear Shrinkage} = {D_l - F_l}/{D_l} \times 100 \quad \ldots (5)$$

Where, D_L = Dried Length, F_L = Fired Length

Determination of Refractoriness (PCE)

The refractoriness or softening point was determined using the method of pyrometric cone equivalence (PCE). Test cones were prepared by mixing each clay sample aggregate with sufficient quantity of water to make the clay become plastic and molded by hand into a cone shape. The samples were dried and fired to a temperature of 900 ^{0}C in a muffler furnace. Pyrometric cones designed to deform at 1300 ^{0}C, 1400 ^{0}C, 1500 ^{0}C were placed round the samples and the temperature rose to above 1000 ^{0}C at 10 ^{0}C per minute. The heating was discontinued when the test cone bent over and leveled with the base of the disc. The pyrometric cone equivalent (P.C.E) of the samples was recorded to be t number of standard pyrometric cone corresponding to the time of softening of the test cone.

3.0 Results and Discussion

3.1 Results

3.1.1–Chemical Composition

Chemical compositions in terms of oxides for each clay sample were shown in Table 1.0. In general, three groups of oxides were of interest; silica, alumina and alkaline-containing oxides. The results (table1&2) shows that the various components of the chemical analysis are high including the loss on Ignition (L0I) values, which indicates the correctness of the analysis and the lack of considerable content of other components as occasionally observed in Clays. The analysis shows a great similarity in the silica (SiO_3) and Alumina content - (Al_2O_3) in the clay samples, while a great difference in the Iron oxide content is observed. Sample C is had the highest silica (SiO_3) content – (63.1%) followed by sample E which had a value of 60.2%. Simple A had the lowers value (48.8%) while samples B and D are had the moderate values (54.4% and 50.3%) respectively.

Table 1.0: Chemical Composition (wt %) of the Clay Samples

Clay Samples	% Al_2O_3	% SiO_2	% K_2O	% CaO	% Ti_2O	% MgO	% V_2O_3	% Cr_2O_3	% MnO	% Fe_2O_3	% Ag_2O	% Na_2O
A	16.00	48.80	3.12	0.28	2.23	ND	0.10	0.04	0.22	23,56	4.41	ND
B	15.00	54.40	1.57	1.36	3.63	ND	0.17	0.05	0.09	16.90	4.91	ND
C	18.70	63.10	0.67	0.07	4.86	ND	0.20	0.05	ND	6.39	5.59	ND
D	15.40	50.30	2.44	1.17	2.26	0.81	0.13	0.04	0.11	17.13	ND	0.11
E	18.00	60.20	0.94	0.62	5.89	ND	0.21	0.05	0.02	6.77	6.12	ND

A=Sokoto B=Dange 1 C=Dange 2 D=Wurno 1 E=Wurno 2, ND= Not detected

3.1.2 Determination of the physical properties

The physical properties of the clay samples were determined with respect to apparent porosity, bulk density, cold crushing strength, thermal shock resistance, linear shrinkage and refractoriness. The results of these tests are as presented in Tables 3.

Table 3: Mean and Standard Deviation of the Physical Properties of Clay Samples

Clay Samples	Apparent Porosity (%)	Bulk Density (g/cm^3)	Cold Crushing Strength (Kg/cm^2)	Thermal Shock Resistance (cycles)	Linear Shrinkage (cm)
A	24.65 ±0.25	1.87 ±0.11	295.65±2.01	20 ±1.01	2.0 ±1.02
B	20.40 ±1.02	1.80 ±0.12	286.96±3.24	20 ±1.05	4.5 ±1.23

C	25.35 ±1.02	1.85 ±0.01	269.57±12.90	18 ±1.11	0.8 ±0.11
D	21.00 ±0.92	1.97 ±0.02	246.95 ±2.02	20 ±0.02	2.0 ±0.01
E	19.00 ±0.19	1.91 ±0.11	304.35 ±1.02	20 ±1.04	1.5 ±0.10

A=Sokoto, B=Dange 1, C=Dange 2, D=Wurno 1, E=Wurno 2

Table 2: Lost on Ignition Values

sample	**A**	**B**	**C**	**D**	**E**
LOI	0.93	1.64	0.23	9.87	0.92

A=Sokoto, B=Dange 1, C=Dange 2, D=Wurno 1,E=Wurno2

Table 4: Reference Data on the Chemical Composition of Fireclay

Sample	**SiO_2**	**Al_2O_3**	**Fe_2O_3**	**MgO**	**CaO**	**Na_2O**	**K_2O**	**LOI**
*Fireclay	46 -62	25 – 39	0.4 – 2.7	0.2 – 1.0	0.2 – 1.0	0.3 – 3.0	0.3 – 3.0	8 - 18

*Source -Nnuka and Agbo, (2000)

Table 5: Refractoriness of Clay Samples

S/N	Samples	Refractoriness			pyrometric cone Equivalent (PCE)
		PCE No.	Range/Limit	Temperature (^{0}C)	
1	A	15	>	1400	Intermediate PCE
2	B	19	>	1500	High PCE
3	C	16	<	1500	Intermediate PCE
4	D	15	>	1400	Intermediate PCE
5	E	19	>	1500	High PCE

A=Sokoto B=Dange 1 C=Dange 2 D=Wurno 1 E=Wurno 2

3.2 Discussion

3.2.1 Determination of chemical composition

The Alumina Content of all the clay samples agreed with what was reported by literature that in Nigeria, the major refractory clay deposits containing alumina – silicate are kaolinitic and fireclay in nature with alumina content of less than 45% [23]. Table 1 shows the chemical composition of the clay samples. From its chemical composition, all the sample fall under Aluminum-silicate type of clay because of their high value of aluminum-oxide and silicon oxide which are highest among other chemical composition of the clay. The silica in the clay samples is present in a different form as a free form (SiO_2) and in the form of compounds when mixed with other elements such as aluminium oxide (Al_2O_3) to form kaolinite ($Al_2(Si_2O_5)(OH)_4$) in the feldspar group.

3.2.1 Determination of refractory properties

The results of the investigation carried out were discussed under the key physical properties of the clay samples determined as shown in Table 3.0.

Apparent Porosity

The values of the porosity of the clay samples in percentages were also determined as shown in Table 3. Sample C, has the highest porosity of 25.35 % against 19.00 % for Sample E, 24.65 % for sample A, 20.40% for sample B, and 21.00 % for sample D. respectively. The porosity of refractory clay material is directly related to the air pockets contained in it, hence, the higher the porosity of the clay material, the higher its insulating properties. All the five samples gave apparent porosity value which is within the acceptable range (10-30%) suggested for refractory clays [24], This implies that the clay samples have common major compositions in terms of oxides, which constitutes the highest percentage of the clay mineral compositions .However, the variation in values of the porosity account for the variation of clay types.

Bulk Density

Looking at the Table, sample C, had the lowest bulk density, (1.80g/cm^3) while sample D had the highest value of 1.97g/cm^3 this can be attributed to low percentage of porosity of the sample. Therefore, load bearing structures developed using sample D would likely have low thermal conductivities. The average value of the bulk density of the clay samples were within the range of 1.08-1.97 g/cm^3. This makes them suitable for siliceous fireclays as reported [6] ,and fireclays as also reported [25].

Cold Crushing Strength

The cold compressive strength (C.C.S) of the clay samples were determined as reported in Table 2. Samples A and E have the highest compression strenght of 295.65kg/cm^2 and 304 kg/cm^2 respectively and a better advantage over others with respect to rigidity and load bearing capability. The implication of this is that the samples could have more tendencies to bear load at low temperatures, this is desirable in the productions of load bearing structures such as clay made improved wood burning stoves. Sample D, has the lowest value of 246.95 kg/cm^2.This could be improved upon by the addition of additives. Samples B and C values were found to be in between those of A and E. These values are comparable to standard values of fireclay as reported by an author [24]. This shows that the test bricks have been properly fired. This shows the ability of the clays to withstand abrasion and loading at low temperatures

Thermal Shock Resistance

Thermal shock resistance for all the clay samples obtained gave the number of cycle to failure for samples A, B, D and E to be 20 cycles. This value fall within the 20-30 numbers of cycles recommended by an author [24] for fireclay refractories. Sample C had the lowest value of 18 cycles, which is just below the recommended value. The practical implication of this is that their use is restricted to lining of ladles and slag pots which are early mended at short intervals. However, this value (18 cycles) can be used for refractory clay as thermal shock characteristics of a material may be improved by the introduction of some relatively large pores [26].

Linear Shrinkage

The results of the linear shrinkage for all the clay samples had highest value in sample B and this is within the recommended range of 4 – 10 % for fireclay refractories as reported by [6]. The values found for other samples (A, C, D and E) were lower than the recommended range. This is more desirable, as higher shrinkage values may result in warping and cracking of the brick and this may cause loss of heat in the furnace. The high shrinkage value obtained for sample B, is an indication of its high water content and plasticity.

Refractoriness

The refractoriness of the clay samples was determined as shown in Table 3. The values for samples A and D were found to be 1400 ^{0}C while those of samples B, C and E were greater than 1500 ^{0}C..The high values obtained for samples B.C and E is an indication of very good refractoriness, because the normal range for fire clay refractoriness is 1500 ^{0}C – 1700^{0}C. This may be due to appreciation of the alumina content in the fired sample. The refractoriness values for samples A, and D were lower than the recommended range for fireclay Refractories of 1500 ^{0}C-1700 ^{0}C as reported by literature [25]. These low values of refractoriness were as a result of the high silica content of the clays. This means that their use is restricted to the processing of materials whose melting points do not exceed 1400 ^{0}C or non ferrous materials.

Conclusions

Looking at the properties of the clay samples tested and analyzed in this studies, the following conclusions can be deduced.

(1) From the chemical analysis, (Table1) all the clay samples had silica, aluminina. And iron as the predominant substances, hence it could be concluded that they are siliceous in nature and are of the alumino-silicate Refractories that are classified as kaolinitic fireclay. .In addition the percentage of iron III oxides (Fe_2O_3) in samples C and E were the least of all other samples as reported in table 1, making them better insulators than others. This is because the percentage of Iron III oxide contained in the refractory clays is a major factor that determines the clays' refractoriness.

(2) A comparison of the clay samples with specification of some industrial clays (Table 2) shows that the samples have appreciable and reasonable values of the

physical properties tested that are comparable to standards

(3) Samples (B and E) have the best refractory property among the five samples. It could be used for the lining of furnaces that could operate at temperature well above 1500°C without fear of thermal deformation of the furnace wall.

(4) On the basis of the physio-chemical characteristics of this kaolinitic fireclay deposit, it can successfully be processed for use as refractory materials furnace lining, in the paper industry as filter, and in the development of improved wood burning stoves in addition to usual pottery activities.

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