



Geochemical Properties of Clays and Clay Pots from Kono Boue and Ara-Ekiti Communities in Nigeria

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Abstract Clays and clay pots samples from two Southern Nigeria communities, Ara-Ekiti and Kono-Boue were studied to ascertain their mineralogical content and physicochemical parameters. The cation exchange capacity (CEC) were 12.06, 5.97, 4.88 and 5.44 Cmol/Kg; bulk density, were 0.94, 1.20, 0.2 and 1.00 g/cm³, organic matter were 8.88, 3.29, 0.74 and 1.48% for Ara-Ekiti clay (AER), Kono-Boue clay (KBR), Ara-Ekiti clay pot (AEP) and Kono-Boue clay pot (KBP) respectively. The percentages of silt, clay and sand were 12.40, 30.00 and 57.60% for AER; 17.40, 24.00 and 58.60% for KBR; 9.40, 14.00 and 76.60% for AEP and 7.40, 12.00 and 80.60% for KBP. The mineralogical content of the clays and clay pots were studied using X-ray diffraction (XRD) instrument. The mineralogy of AER and KBR by XRD analysis showed the presence of kaolinite, quartz, berthierine and almandine while for AEP and KBP XRD results showed the presence of microcline, kaolinite and quartz. The resultant differences in the mineralogy of the clays from the two communities might have resulted due to geological and anthropogenic factors, while the differences between the clays and the clay pots were due to the firing temperature during the clay pots production processes.

Keywords clay, clay pots, mineralogy, X-ray diffractogram, organic matter, cation exchange capacity

Introduction

The term clay refers to a naturally occurring inorganic material with plastic properties and very fine particles size (< 2.0 μm). It is mainly composed of hydrous layer silicates of aluminum and sometimes may contain magnesium and iron. Clay is a natural resource and finds greater use in pottery and in the manufacture of a number of products such as fired bricks, tiles, refractory bricks, well drilling mud etc [1]. Clays are formed from hydrolysis and hydration of silicates, slacking and weathering of clay-rich sedimentary rocks and a product of hydrothermal decomposition of granite rocks [2]. Clay mineral is a constituent of soil and serves as the raw material used in the production of clays. Clay minerals (kaolins) exist in four different forms, namely, kaolinite (Al₂Si₂O₃(OH)₄), dickite (Al₄Si₄(OH)₈), nacrite (Al₂O₃.2SiO₂.2H₂O) and halloysite (Al₂O₃.3SiO₂.4H₂O) [3]. Kaolins are the most widely used clays in pottery, manufacture of paints, cement, plastics among others due to their high purity and brightness. Clay pots are inorganic solids which have been molded into containers of various sizes and shapes, which comprises of compounds formed from metallic and non-metallic elements. Clay pots have good insulating property and have the ability to withstand extreme temperatures [4]. Clay pot is manufactured from clay soil and can be used for cooking and serving of food, water, and other materials, and come in different colours, sizes and shapes. The concentration of iron (Fe₂O₃) and titanium (TiO₂) present in the clay affects the firing colours of the pots.



Clay pottery dates back to 16500-14920 BCE in Odai Yamamoto, Japan at an archeological site [5]. Clay pottery is an old handicraft that has deep root in ancient history [6-7]. The invention of pottery brought about greater and better storage of food than that which was provided by the use of baskets. Due to the wide spread and availability of clay in different regions and times, the invention of pottery became possible at different places and times in many parts of the world [8]. In Nigeria the pottery business is in small scale but widespread, simply because clay deposits have widespread distribution in about 80 different sites and locations [9].

Clay is the major raw material that is involved in the production of clay pots, and includes minerals which possess common crystal structural features and related chemical composition [9]. The major elements present in the earth crust as well as the minor elements are present in clay. Also contained in the soil minerals are trace elements. Clay is a group of microcrystalline minerals made up of aluminum silicates in hydrous form with sheet like structures and contains large quantity of silica and organic matter as impurities [10]. Clay can be distinguished from each other by chemical formula, structure and chemical/physical properties. The three main groups of clay minerals have different chemical structures; Kaolinite, $\text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_4$, montmorillonite (bentonite) $(\text{Al}_2(\text{OH})_2\text{Si}_4\text{O}_{10})$ and Illite, $(\text{K}_{0.2}\text{Si}_4(\text{Si}_{6.8}\text{Al}_{0.2})\text{O}_{20}(\text{OH})_4)$. Studies have revealed regional differences and variations in the composition of clays, which also results in the variation in the properties of the clay pots from different sites [11-13].

This research work is aimed at investigating the geochemical properties of clays and clay pots obtained from Kono-Boue and Ara-Ekiti communities. The research work was set to determine the mineralogical composition of clay and clay pots from the two communities used in pottery production and also conducted physicochemical analysis of the clay soils and clay pots produced from them in order to ascertain the different characteristics of the clay soils from the different communities.

Materials and Method

Description of Study Area

The study areas, Kono-Boue is situated in Khana Local Government Area, Rivers State, South South, Nigeria and is located between latitude $4^\circ 42'$ and longitude $7^\circ 22'$ E [14], while Ara-Ekiti is in Ijero Local Government Area of Ekiti State, South West Nigeria and is located between latitudes $7^\circ 51' 54.15''$ N and $7^\circ 49' 22''$ N and longitudes $5^\circ 02' 17.58''$ E and $5^\circ 5' 22.3''$ E [15]. Both communities are involved in the mining of clay for pottery. The Kono-Boue community in Ogoni land is also influenced by exploration and production of oil [14] while Ara-Ekiti is not influenced by any such anthropogenic activities.

Collection of Clay and Clay Pots Samples

Clay samples were collected from Kono-Boue and Ara-Ekiti with the aid of soil auger, one metre deep in three separate locations at a distance of 1Km apart from the clay mining sites. The clay samples of each location were then mixed together to form a composite sample and stored properly in labelled polyethylene bags. Likewise, the related clay pot samples were bought directly from the potters producing the clay pots with the clay from the various mining sites in Kono-Boue and Ara-Ekiti and were transported to the Chemistry Laboratory of the Department of Chemistry, Rivers State University.

Sample Pretreatment

The clay soils from the two locations were labelled Kono-Boue clay (KBR), and Ara-Ekiti clay (AER) respectively. The clay pots were labelled Kono-Boue clay pot (KPB) and Ara-Ekiti clay pot (AEP) respectively. The clay samples were air dried for seven days on a plastic tray and then ground to powder form with the help of a porcelain mortar, while the clay pots were crushed and pulverized to powder form through the use of a porcelain mortar and then sieved with a 2mm mesh. The sieved samples were then kept in a new polyethylene bag properly labelled and then stored at room temperature for further analysis.



Density Measurement

A portion of the sieved sample was transferred into a previously weighed empty specific density bottle to obtain the weight of the sample and the density bottle. The calculated weight of the sample was obtained and the density of the sample was determined by dividing the weight of the sample by the volume of the specific density bottle. The procedure was carried out in triplicates.

Particle Size Analysis

The hydrometer method (Bouyoucos, [16]) was used for the determination of particle size analysis. Air dried sample (51g) was transferred into a 500ml dispersing cup. Thereafter, 100ml of water was added to the sample on a shaker and then 20g of sodium hexametaphosphate was added. A Bouyoucos hydrometer was then used to measure the density in grams of particles/L of the suspension after selected settling times. Within a 40 second settling time, the buoyant force was due for the concentration of all the particle sizes. However, after 40 seconds settling time, the buoyant force on the hydrometer emanated from the concentration of clay and silt. After a settling time of 2 hours, the silt particles settled at the bottom of the cylinder and the hydrometer reading was recorded. A blank calibration of the hydrometer was done for that containing water and the dispersing agent only. The Bouyoucos hydrometer was calibrated at 20 °C (68°F). Readings were corrected for variation in temperature and density, which were due to changes in viscosity and temperature of water respectively. Then the percentages of clay, silt and sand were calculated thus,

$$\% \text{ clay} = \frac{\text{corrected 2-hr hydrometer reading}}{\text{dry weight of soil}} \times 100$$

$$\% \text{ silt} + \% \text{ clay} = \frac{\text{corrected 40-sec. blank hydrometer reading}}{\text{dry weight of soil}} \times 100$$

$$\% \text{ sand} = 100 - \% \text{ silt} + \% \text{ clay}.$$

Determination of Organic Matter in Clay and Clay Pots Samples

The organic matter was determined by multiplying the organic carbon by 1.724 which is a constant. A 10g sample was transferred into a 500ml conical flask and 10ml of 1N K₂Cr₂O₇ solution was added to the sample. A 20ml concentrated H₂SO₄ was then added to the flask and gently mixed together. The resulting solution was then covered with watch glass and allowed to stand for about 30 minutes to effect redox reactions. Distilled water was added to 125ml mark and 25ml of 0.5N ferrous ammonium sulphate was added. The resulting solution was titrated with 0.5N KMnO₄, and a colour change from green to purple was observed at the end point. In a similar manner blank solution was carried out without the sample.

$$\% \text{ Organic Carbon} = \frac{(T-B) \times 0.390}{w}$$

Where, T = titre value, B = blank titration and W = weight of sample

Therefore % Organic matter in the sample = % organic carbon X 1.724, where 1.724 is a constant.

Cation Exchange Capacity (CEC) of Samples

The procedure for the measurement of CEC is based on the saturation of the exchangeable cations with a particular cation (NH₄⁺) and determining the latter by quantitative removal of the displacing cation. The exchangeable cations were extracted using ammonium acetate extraction procedure. Agitation of the soil sample with ammonium acetate and then filtration results in saturation of soil complex with NH₄⁺. The ammonium extract was then treated with buffer solution, KCN, K₄Fe(CN)₆, NH₂OH, HCl and triethanolamine. Eriochrome black-T indicator was added before titration with EDTA to effect colour change at end point (from wine red to purplish blue). The obtained titre value indicated the amount of calcium and magnesium present in the extract. While for the measurement of sodium and potassium, the flame photometric technique for metal analysis was employed for the determination of the concentration of exchangeable Na and K in the samples.

Mineralogical Analysis

The identification of the unknown crystalline materials (minerals, inorganic compounds, etc.) present in the clay and clay pot samples were carried out using X-ray Diffractometry (XRD) technique. The XRD analysis of the samples



(clay and Clay pots) were carried out at NASENI Centre of Nanotechnology and Advanced Materials, Akure, Ondo State, Nigeria; using GBC Enhance Mini-Material Analyzer (eMMA). Incident radiation CuK α , incident – and diffracted beam slits and a kevox solid-state detector, Si(Li), were used for the analysis. Data were collected from 5° to 70° 2 θ , step size of 0.05° 2 θ , speed of 3°/min and a count time of 4s per step which results in a continuous scan of various major and minor peak intensities. The spectrometer displays diffractograms of the sample which were further analyzed by the instrument's computer into corresponding minerals and their peak intensities.

Results and Discussion

Some physicochemical parameters of the studied clays and clay pots samples collected from Kono-Boue and Ara-Ekiti are shown in Table 1. The cation exchange capacity (CEC) for AER, KBR, AEP and KPB samples were 12.06, 5.97, 4.88 and 5.44 Cmol/Kg, respectively. The density of AER, KBR, AEP and KPB were 0.94, 1.20, 0.72 and 1.00 g/cm³ respectively. The density of the raw clays, were relatively higher than the clay pot samples. The % organic matter in the studied clays and clay pots samples, were 8.88, 3.29, 0.74 and 1.48 % for AER, KBR, AEP and KPB respectively, while the % organic carbon recorded were, 5.25, 1.91, 0.43 and 0.86 % for AER, KBR, AEP and KPB respectively. The percentage silt, clay and sand in the sampled clays and clay pots in AER were, 12.40, 30.00 and 57.60 respectively. In AEP, they were 9.40, 14.00 and 76.60% respectively. In KBR, they were 17.40, 24.00 and 58.60 % respectively. In KPB, they were 7.40, 12.00 and 80.60 % respectively. The textural classes of the clays and clay pots were, sandy clay loam for AER and KBR and loamy sand for AEP and KPB respectively.

The percentage organic matter in the AER and KBr samples were 8.88 and 3.29% respectively, which is consistent with the result obtained by Agbaji *et al.*, [17], on the physicochemical parameters of soil in Kakuri area which ranged between 3.10-5.20%. The relatively higher value of 8.88% organic matter observed for AER might be attributed to the thick vegetation in the region which is influenced by the total organic carbon content in the soil. The indiscriminate disposal of organic waste predominant in the area might have contributed to the higher levels of total organic carbon of the soil. Organic matter is a dynamic component of the soil and varies with time and temperature [18]. Research work revealed that the rate of decomposition of organic matter is proportional to the temperature of the soil, and soil organic matter decreases with temperature [19-20]. This is in agreement with the findings in this work where the organic matter present in the poorly vitrified KPB (1.48%) are higher than the more vitrified AEP (0.74%). The observed lower level of organic matter in AEP compared to AER might be attributed to the relatively higher firing temperature used during the production process of AEP whereas the presence of appreciable amount of organic matter in KPB relative to KBR might be due to the lower firing temperature employed during the production process of the pot. Furthermore, the organic matter content in AER (8.88%) was higher than that of KBR (3.29%) which agreed with the fact that organic matter content is inversely proportional to the density of the soil [21-22]. The observed decrease in the organic matter in the clay pots as compared to the clays might have come from rapid decomposition due to higher temperatures during the production process.

The results of the CEC of the clays and corresponding clay pots from Table 1 indicated that the CEC of clays were 12.06 and 5.97 Cmol/Kg for AER and KBR respectively, while CEC values for clay pots were 4.88 and 5.44 for AEP and KPB respectively. The CEC values obtained in this study were observed to be low when compared with the range of 8.47-49.03 Cmol/Kg reported by Antoniadis and Alloway [18], for sewage sludge treated soils at different temperatures. The observed lower values of CEC in the samples may be due to the presence of kaolin sheets which are held together by hydrogen bonds that prevents expansion and entry of polar molecules [23]. Also clays which contain phyllosilicate minerals such as kaolinite, and large quantity as sand fraction have lower CEC values than clays which consist of illite and smectite and high organic content [24]. The result also showed that the CEC values of clays are higher than that of the corresponding clay pots. This may be due to the relatively higher level of organic matter present in the clays. Reports have shown that soils with high clay fractions and organic matter tend to have higher CEC values as a result of the high concentrations of negatively charged sites on their surface [25]. High CEC soils hold more water than lower CEC soils due to the greater amount of clay and organic matter found in the soil. The higher CEC values in clays correspond to the high amount of clay fractions obtained in the particle size distribution.



The particle size distribution of clays and related clay pots shown in Table 1, indicated that the textural class of both clays (AER and KBR) and clay pots (AEP and KPB) sandy clay loam and loamy sand respectively. The observed particle size of the clays for AER were 12.40 % silt, 30.00 % clay and 57.60 % sand; whereas, the particle size analysis for KBR were 17.40% silt, 24.00% clay and 58.60% sand. The clay pots results showed comparatively lower level of silt (9.40 and 7.40 %) and clay 14.00 and 12.00%. The observed reduction of silt and clay contents of the of the clay pots may be attributed to the loss and conversion of some amounts of the clay pots constituents of the sample during firing. The clay content in this work is less than that reported in the analysis of Kekamega county soil from Ilesis deposits of 26-38% [26]. The result also agreed with previous work which reported 28.08% clay on soils collected from the vicinity of a wood treatment centre factory in Benin City [27]. The level of clay and the mineralogy of the clay sample are important requirements for utility in pottery [1]. The results in Table 1 indicated that % clay in AER and AEP were 30.00% and 14.00% respectively; whereas the percentage clay in KBR and KPB were 24.00% and 12.00%, respectively. Previous research on soil samples reported relative lower levels of clay (6.99%) contained in loamy sand as well as higher levels of clay (40.06%) in clay loam soils [18]. In this study the observed differences in clay content between the clays and the clay pots samples might have resulted from the effect of firing at elevated temperatures. The percentages of sand in AEP and KPB were 57.60% and 58.60% respectively; whereas, the percentages of sand in AEP and KPB were 76.60% and 80.60% respectively. The observed differences between percentage sand contained in the clays and the clay pots might have resulted from the various combination during the clay body formation of the pot production process. Potters mix clay and sand in varying proportions to obtain the desired clay pot body.

Table 1: Some Physicochemical Parameters of Clay and Clay Pot Samples from Kono-Boue and Ara-Ekiti

Sample	CEC	Bulk Density	% Organic Matter	% Organic Carbon	% Silt	% Clay	% Sand	Textural Class
AER	12.06	0.94	8.88	5.15	12.40	30.00	5.60	Sandy clayloam
KBR	5.97	1.20	3.29	1.91	17.40	24.00	58.60	Sandy clay loam
AEP	4.88	0.72	0.74	0.43	9.40	14.00	76.60	Loamy sand
KPB	5.44	1.00	1.48	0.86	7.40	12.00	80.60	Loamy sand

X-Ray Diffraction (XRD) Results for AER, KBR, AEP and KPB Samples

Table 2, showed the clay and non-clay minerals present in the samples and the percentage compositions of the elements. AER and KBR are composed of kaolinite, quartz, berthierine, and almandine. Kaolinite is composed of 20.9% Al, 1.56% H, 55.78% O and 21.76% Si; quartz comprised of 53.26% O, and 46.74%Si; berthierine had 17.32% Al, 0.68% H, 37.84% O, 10.44% Si, 32.08% Fe, and 1.64% Mn; Almandine had 10.84% Al, 38.57% O, 16.93% Si, and 33.66% Fe. However, KPB mineralogy is different from that of AEP; KPB is composed of microcline and quartz whereas, AEP is composed of kaolinite and microcline. The percentage composition of each element in microcline was 9.69% Al, 14.05% K, 45.99% O and 30.27% Si. The observed major clay mineral in both clays is kaolinite, while the main component of clay pot is microcline.

Mineralogical Composition of Clays and Clay Pots from Ara-Ekiti and Kono-Boue

The XRD results shown in Table 2 revealed kaolinite as the main clay mineral present in the clays, while quartz, berthierine and almandine were the major non-clay minerals. Diffractograms for the clays revealed that peaks were at 2θ , 12.362°, 19.985°, 36.387°, 54.924° and 59.979° for both clays. These results agreed with earlier data provided which showed that peaks located within these regions of diffractograms are typically attributed to kaolinite basal planes [28]. The diffractograms showed the predominance of the kaolinite clay mineral and quartz as the major non-clay minerals while berthierine and almandine were recorded as accessory minerals in both clays. The findings from the results is consistent with the mineralogy of clay used in the indigenous processing of pottery and ceramics [29-30]. Gbarakoro *et al* [31] reported that kaolinite is the major clay mineral present in Kono-Boue clay. Diffractogram revealed quartz peak at 2θ , 20.972°, 26.702°, 36.587°, 39.459°, 40.465°, 42.50°, 50.181°, 54.924°, 59.975°, 67.785°,



and 68.357° for AER. Similar quartz peaks were obtained for KBR. The results agreed with the data provided by another study on the physicochemical characterization of raw clay which showed dickite (a form of kaolinite) peaks at 20, 12.4° and microcline at 20, 27.4° and 35° while quartz was at 20, 21° and 26.9° [32]. The major difference noted between the clays is in the intensities of their diffractograms. Berthierine, however showed peaks at 20, 12.393°, 19.985° and 39.541°. Almandine showed peaks at 20, 20.972°, 24.85°, 25.589°, 26.02°, 27.508°, 41.85°, and 42.5°. The results obtained in Table 2 showed that the clay mineralogy of Ara-Ekiti and Kono-Boue to that reported on the mineralogy of local clays from Odupani in South-Eastern Nigeria [32] and the clays from Bentoni, Nigeria [33]. The observed differences in the composition of different clays might be attributed to geological factors. Table 2, also revealed that Ara-Ekiti clay pots (AEP) contained microcline, iron silicate and kaolinite whereas, KBP contained microcline and quartz. The absence of berthierine and almandine in the clay pots might be as a result of firing at elevated temperatures. The kaolinization of the studied clay samples is in line with observation of kaolinitic clays reported by Ojo *et al.*, [29]. The weight percent in Table 2 showed high levels of siliceous sand in the ratio of SiO₂/Al₂O₃ ranging between 1.26-3.88 in the clay pots than the clay soils. The ratio SiO₂/Al₂O₃ present in the Ara-Ekiti clay pots falls below the range of 1.60-3.88, hence the pot is composed of less amount of quartz as compared to the Kono-Boue clay pots. The high resource potential of kaolinitic clay is based on its usefulness as the main raw materials in the ceramic industries and high level of SiO₂/Al₂O₃ and loss on ignition in clays enhances its utility in the ceramic and refractory bricks industries [34].

Table 2: X-ray Diffraction (XRD) Results for AER, KBR, AEP and KP Sample

KBR	Kaolinite (Nacrite)	Aluminium silicate hydroxide	Al ₂ Si ₂ O ₅ (OH) ₄	20.9	-	1.56	55.78	21.76	-	-
	Quartz	Silicon oxide	SiO ₂	-	-	-	53.26	46.74	-	-
	Berthierine	Iron Magnesium Aluminium silicate hydroxide	(Fe ²⁺ , Fe ³⁺ , Al, Mg) ₂₋₃ Si ₂ Al ₂ O ₅ (OH) ₄	17.32	-	0.68	37.84	10.44	32.08	1.64
	Almandine	Iron Aluminium Silicate	Fe ₃ Al ₂ (SiO ₄) ₃	10.84	-	-	38.57	16.93	33.66	-
AER	Quartz	Silicon oxide	SiO ₂	-	-	-	53.26	46.76	-	-
	Kaolinite	Aluminum silicate hydroxide	Al ₂ Si ₂ O ₅ (OH) ₄	20.9	-	1.56	55.78	21.76	-	-
	Berthierine	Iron Magnesium Aluminumsilicate hydroxide	(Fe ²⁺ , Fe ³⁺ , Al, Mg) ₂₋₃ Si ₂ Al ₂ O ₅ (OH) ₄	17.32	-	0.68	37.84	10.44	32.08	1.64
	Almandine	Iron Aluminum Silicate	Fe ₃ Al ₂ (SiO ₄) ₃	10.84	-	-	38.57	16.93	33.06	-
KBP	Microcline	Potassium Aluminum Silicate	KAlSi ₃ O ₈	9.69	14.05	-	45.99	30.27	-	-
	Quartz	Silicon oxide	SiO ₂	-	-	-	53.26	46.74	-	-
AEP	Microcline	Potassium Aluminum Silicate	KAlSi ₃ O ₈	9.69	14.05	-	45.99	30.27	-	-
	Chrysolite	Iron Silicate	Fe ₂ SiO ₂	-	-	-	52.86	45.94	1.21	-
	Kaolinite		Al ₂ Si ₂ O ₅ (OH) ₄	20.9	-	1.56	55.78	21.76	-	-

Conclusion

Clay soil is the major raw material used in the production of clay pots, hence the constituents of the final product of pottery depends on the composition of the raw clay body. Due to natural diversities of clays and anthropogenic factors, its composition changes appreciably from one region to another. Consequently, the composition of the clay pots varies from region to region. Kaolinite and quartz were the major clay and non-clay minerals that were present in the clays used for pottery in Ara-Ekiti and Kono-Boue.



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