

PHYSICO-CHEMISTRY OF CONTINENTAL BENTONITES AND KAOLIN FOR CERAMIC APPLICATIONS

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ABSTRACT:- Growing demand for bentonite and kaolin applications in the ceramic industry motivated this study which aimed at physico-chemically characterizing some selected continental clayey materials from Botswana, Mozambique, Pakistan, Senegal, South Africa and the United States of America. The hydrogen ion concentration (pH), electrical conductivity (EC), cation exchange capacitance (CEC), specific surface area (SSA) and color were determined. The results depicted that the pH values were between 5.8 and 10.5, EC values ranged from 200 \pm S/cm to 6501 \pm S/cm, CEC values occurred between 3.5 meq/100 g and 105 meq/100 g, and the SSA values were between 4 m²g⁻¹ and 19 m²g⁻¹. Raw clay color ranged from white to black with most samples having a greyish-like appearance. These values are in conformity with those obtained by other researchers, and based on their physico-chemical characteristics, the clayey materials were found to be suitable for use in the ceramic industry.

INTRODUCTION

Bentonitic and kaolinitic clay deposits are exploited for very wide varieties of industrial applications, one of which is the ceramic industry (Patterson and Murray, 1983). These clays are used to make claybodies, slips and glazes of different chemical and mineral compositions for ceramic applications. However, their usage in ceramic production is accompanied by significant problems at the different stages of product manufacturing: raw clay mining, processing, claybody formulation, drying, glazing, firing, and cooling of finished product.

The value of any clay mineral deposit for ceramic applications is determined by the mineral genesis, types and amount of mineral and chemical impurities, physico-chemical and granulometric characteristics (Cravero *et al.*, 2000; Patterson and Murray, 1983). Important physico-chemical properties for the evaluation of the suitability of any given clayey material for usage in the ceramic industry include clay pH, electrical conductivity (EC), cation exchange capacity (CEC), specific surface area (SSA) and

clay color. In this regard, an understanding of the physico-chemical characteristics of raw clay materials utilized in the ceramic industry is essential. This study aimed at evaluating some selected clayey materials based on their physico-chemical characteristics for possible ceramic applications.

METHODS AND ANALYTICAL TECHNIQUES

Samples and their sources

Samples analyzed were from Botswana, Mozambique, Pakistan, Senegal, South Africa, and United States of Africa (USA). Samples were coded MT and numbered from 1 to 17 as indicated in Table 1.

pH determination

Van Reeuwijk (1993) and Barnard *et al.* (1990) described the method for pH analysis of soil samples which is also applicable for clay minerals. Finely ground/pulverized clay samples were used for pH determination. 2.5 g aliquots of sample were placed in 3 centrifuge tubes and suspended in

Table 1: Sample code and identification of clayey materials

Sample code	Sample identification
MT1	Bentonite, Mozambique
MT2	Bentonite MD 100#, South Africa
MT3	Bentonite 3%, South Africa
MT4	Bentonite HV 100#, South Africa
MT5	Topi clay, Pakistan
MT6	Sample 1, USA
MT7	Sample 2, USA
MT8	Sample 3, Senegal
MT9	Sample 4, Senegal
MT10	Sample 5, South Africa
MT11	Sample 6, South Africa
MT12	Sample 7, South Africa
MT13	Sample 8, South Africa
MT14	Sample 9, South Africa
MT15	Sample 10, South Africa
MT16	Makoro kaolin, Botswana
MT17	Kgwakgwe kaolin, Botswana

25 ml of distilled H₂O. After shaking on a horizontal shaker for about 30 minutes, the tubes were centrifuged for 5 minutes. The pH of the supernatant of the samples was measured using a Jenway 3020 pH meter. Average values were calculated and recorded for each sample analyzed. The temperature of the samples at the time of analyses was recorded.

Electrical conductivity determination

Finely ground/pulverized clay samples were used for EC determination. Sample preparation as explained by Barnard *et al.* (1990), Okalebo *et al.* (1993), and Van Reeuwijk (1993), was similar to that of pH analyses mentioned above. The EC of the supernatant of the samples were analyzed with a Jenway 4020 EC meter. Average values were calculated and recorded for each sample analyzed. The temperature of the samples at the time of analyses was recorded.

Analysis of exchangeable cations

The CEC of the clay samples was determined using the barium chloride-triethanolamine (BaCl₂ - CH₃-CH₂OH)₃N (Inglethorpe *et al.*, (1993), and Ma and Eggleton, (1999)). Five grams of clay sample were put in 100 ml centrifuge and buffered with BaCl₂ for an hour with four or five stirrings periodically. The centrifuge was set for 15 mins to make 30 000 rounds at 2000 rpm. After centrifuging, the supernatant was decanted and an additional 100 ml of BaCl₂ added.

Unabsorbed ions of Ba were washed off using deionized water. 100 ml of 0.05 M MgSO₄.7H₂O was added to each of the weighed contents in the tube and the mixture was allowed to react for at least 2 hours. Centrifuging of the mixture was done for another hour, and 10 ml of the supernatant was collected into a 100 ml conical flask. Twelve drops of 1M NH₃ of SG 0.91 and 6 drops of catechol violet indicator were added to the suspension, which was titrated with 0.02 M Di-Na EDTA. The CEC was calculated from the titrant values.

Specific surface area (SSA) measurements

The SSA of the clay samples was carried out employing a particle size analyzer (PSA). Water-segregated particles were put in the system supplied cells and analyzed using the 1993 model Shimadzu SA-CP4 automatic PSA. The analyzer was set at 240 revolutions per minute (rpm) for effective segregation of particles. The specific surface area was automatically measured by the PSA.

Color determination

In this study, raw clay samples for color determination were aerated for 24 hours. Clayey aggregates were separated using a mortar and pestle to single particles. With a spatula, the samples were mounted on white cardboard sheets provided by the Munsell Color Company Inc., MD 21218, USA. The color descriptions, which comprised the hue, value/chroma and color of the mounted samples, were obtained by visually comparing them to those of standard soils recorded in the Munsell soil color book (1995).

RESULTS

The results of the pH, EC, CEC and SSA of the clay samples are summarised in Table 2, and the details explained in the

corresponding subsections below. It was not possible to perform these tests on samples MT6, MT7 and MT8 because their quantities were significantly very small. Color determination was carried out for all the samples.

Table 2: Specific surface area, pH, electrical conductivity and cation exchange capacity of some of the clay samples

Sample No	Specific surface area (m ² /g)	PH	EC (̂S/cm)	CEC (meq/100g)
MT1	4	10	6240	58
MT2	6	10.3	4583	105
MT3	19	10	2640	95
MT4	6	10.5	5039	100
MT5	5	9	300	11
MT9	-	5.8	239	-
MT10	5	7.3	141	18
MT11	6	6.6	288	7
MT12	7	7	1408	8
MT13	10	7.6	996	14
MT14	6	8.6	286	12
MT15	10	8	3755	16
MT16	10.5	6.6	275	7
MT17	11	6.6	280	13

pH results

Figure 1 is the pH results of the studied clay samples. The pH of samples analyzed ranged from 5.8 to 10.5 at a temperature of 21 °C. The lowest pH was recorded in sample MT9 and it was 5.8; the sample being classified as acidic. Considering from Figure 1 the pH range of 6.6 to 7.3 recorded for five samples (samples MT10, MT11, MT12, MT16 and MT17), they were classified as neutral or near-neutral clays. Samples MT13 and MT15 were slightly basic at a pH of 7.6 and 8 respectively. The pH values obtained for six of the analysed samples (samples MT1, MT2, MT3, MT4, MT5 and MT14) as reflected on Figure 1, were between 8.6 and 10.5; of which the samples were indicative of a basic nature.

Electrical conductivity

The range of EC of the samples was quite wide. From Figure 2, the samples could be classified into three classes based on their EC values. The first class consisted of samples with EC values being = 1000 ̂S/cm, and these samples were MT5, MT9, MT10, MT11, MT13, MT14, MT16 and MT17. The second class of samples had their EC values between 1001 ̂S/cm and 2500 ̂S/cm, and sample MT12 was the only one in this class. The third class was made up of samples, which had their EC values between 2501 ̂S/cm and 6240 ̂S/cm, and these were samples MT1, MT2, MT3, MT4 and MT15.

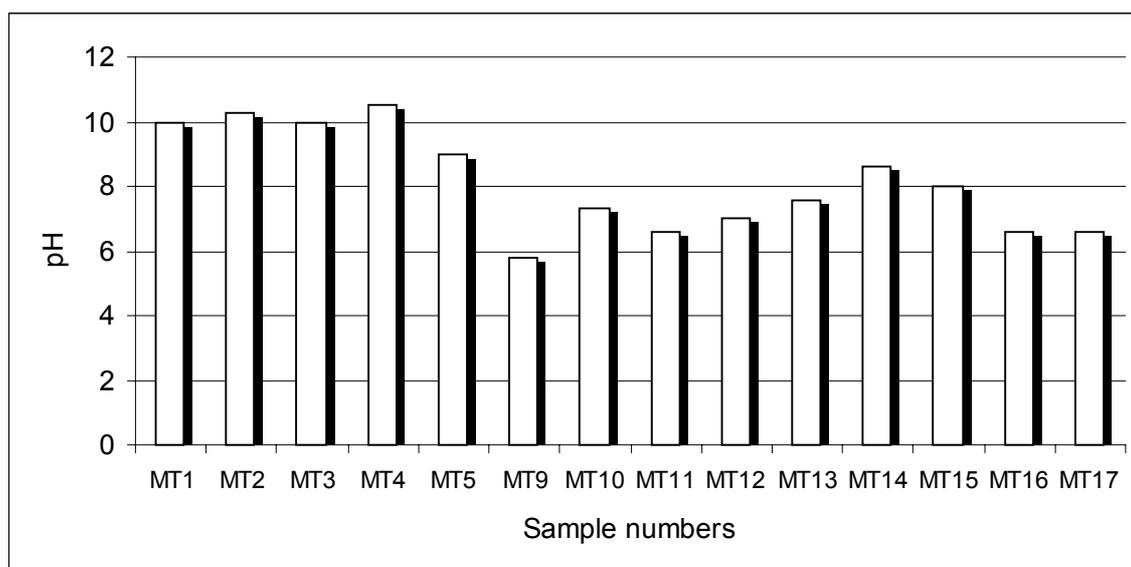


Figure 1: The pH results of the studied clay samples

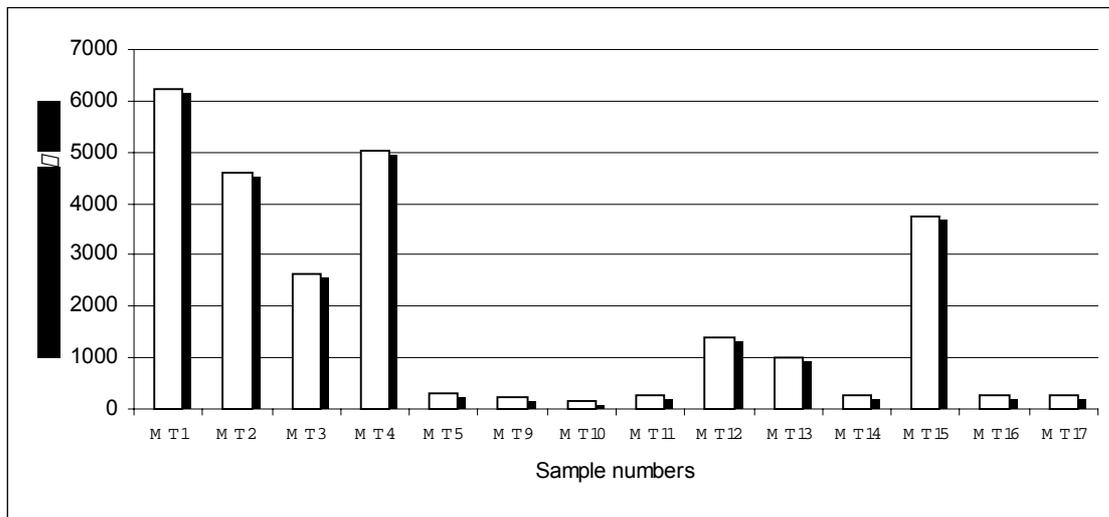


Figure 2: The electrical conductivity results of the studied clay samples

It was observed generally that samples with low pH values also had low EC values, and those with high pH values equally had high EC values. Sample MT1 had the highest EC value recorded, which was 6240 $\mu\text{S/cm}$ as shown in Table 2 and Figure 1. The analyses were conducted at a temperature of 21 °C.

Exchangeable cations

As reflected in Figure 3 below, the results of the samples analysed for CEC could be classified into two classes: samples with CEC values between 0 meq/100 g of clay and 20 meq/100 g of clay constituted the first class whereas samples with CEC values between = 20 meq/100 g of clay

and 120 meq/100 g of clay belonged to the second class. Most of the samples belonged to the first class and these were samples MT5, MT10, MT11, MT12, MT13, MT14, MT15, MT16 and MT17. Sample MT10 had a CEC value of 18 meq/100 g of clay and sample MT15 had a CEC value of 16 meq/100 g of clay. Of the second class of samples, their respective CEC values were as follows: sample MT1 had 58 meq/100 g of clay, sample MT2 had 105 meq/100 g of clay, sample MT3 had 95 meq/100 g of clay, and sample MT4 had 100 meq/100 g of clay. CEC values for the first class of clays were indicative of kaolinitic clays and CEC values for the second class of samples were reflective of smectitic clays.

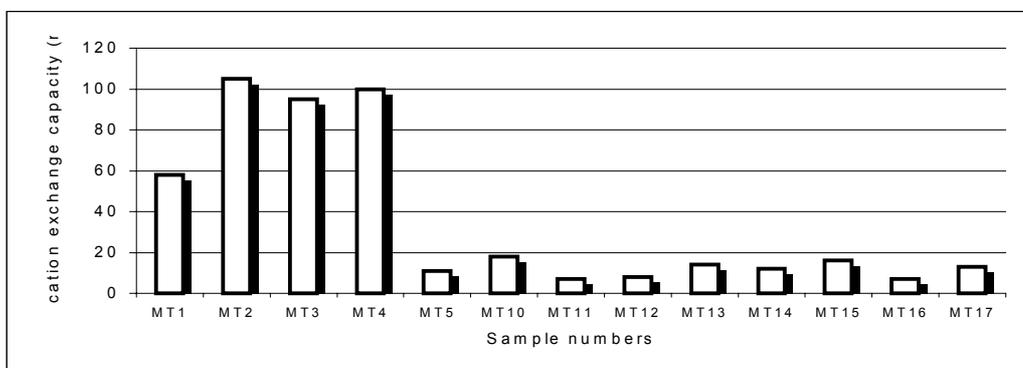


Figure 3: The cation exchange capacity of the studied clay samples

Specific surface area of clay particles

The specific surface area (SSA) of samples obtained ranged from $4 \text{ m}^2\text{g}^{-1}$ to $19 \text{ m}^2\text{g}^{-1}$ and the results are given in Figure 4. Based on values obtained for SSAs of samples analyzed, they could be subdivided into three classes. The first class of samples were MT13 and MT15, which had SSAs of 10

m^2g^{-1} . The second class were samples MT1, MT2, MT4, MT5, MT10 and MT14 having SSAs between $4 \text{ m}^2\text{g}^{-1}$ and $6 \text{ m}^2\text{g}^{-1}$. The third class were samples MT3, MT12, MT17 and MT17, and they had SSAs between $7 \text{ m}^2\text{g}^{-1}$ and $20 \text{ m}^2\text{g}^{-1}$. The SSA of sample MT10 was $10 \text{ m}^2\text{g}^{-1}$ and sample MT17 had a SSA of $11 \text{ m}^2\text{g}^{-1}$. The SSA for sample MT3 was $19 \text{ m}^2\text{g}^{-1}$, and its cumulative wt % of the $\leq 2 \mu\text{m}$ fraction was 59 %.

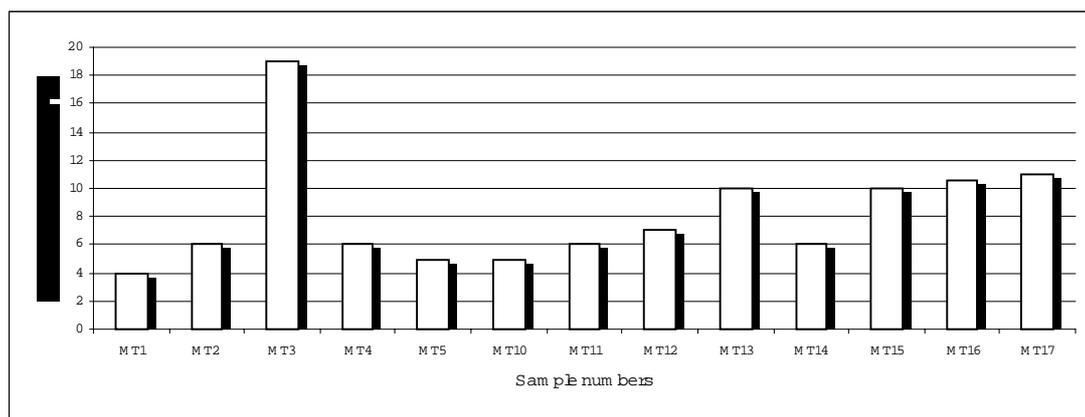


Figure 4: The specific surface area of the studied clay samples

Color of raw clay

A summary of the color of the raw clay samples is given in Table 3. Eleven color shades were obtained from the raw clay samples examined. The color shades covered a wide spectrum from white to black. Most of the samples were however, greyish-like in appearance. Samples MT1 and MT14 were pale yellow. Samples MT2 and MT3 were light yellow brown. The color of sample MT4 was light brown grey and that of sample MT5 was dark yellow brown. Three of the samples were light grey in color, and these were samples MT6, MT13 and MT16. Samples MT7 and MT17 were pinkish grey in color. Sample MT8 was very pale brown in color, and sample MT9 was dark greyish brown. Sample MT10 had a distinct yellow color. Two samples were white and these were samples MT11 and MT12. From Table 3, it could be seen that sample MT15 was uniquely black.

Table 3: Hue, value, chroma and color of raw clay samples

Sample No.	Raw clay sample	
	Hue / value / chroma	Color
MT1	2.5Y / 8/2	Pale yellow
MT2	2.5Y / 6/3	Light yellow brown
MT3	2.5Y / 6/3	Light yellow brown
MT4	2.5Y / 6/2	Light brown grey
MT5	10YR / 3/6	Dark yellow brown
MT6	7.5YR / 7/1	Light grey
MT7	5YR / 7/2	Pinkish grey
MT8	10YR / 8/4	Very pale brown
MT9	10YR / 4/2	Dark greyish brown
MT10	10YR / 7/8	Yellow
MT11	2.5Y / 8/1	White
MT12	2.5Y / 8/1	White
MT13	10YR / 7/2	Light grey
MT14	2.5Y / 7/4	Pale yellow
MT15	2.5Y / 2.5/1	Black
MT16	7.5YR / 7/1	Light grey
MT17	5YR / 7/2	Pinkish grey

DISCUSSION

Influence of raw clays pH in ceramic applications

The kaolinitic clays analyzed had lower pH values compared to the bentonitic clays. The pH of most of the samples exceeded 6.5. High pH values (> 6.5 for kaolin) are indicative of the presence of soluble salts, which could cause severe problems in many applications (Murray, 1986), one of which is drying and firing of ceramic claybodies. The genesis of a clayey material may influence the amount of soluble salts contained in the clay (Velde, 1995). Bentonites because of the environments of formation tend to contain more soluble salts than kaolin (Weaver, 1989).

The total dissolved salts (TDS) of given clayey material may not be reflected by its pH because the salts are generally neutral. The phenomenon of soluble salt surface depositing on raw claybodies is not well understood in ceramics (Drying Ceramics, 2000). Nonetheless, soluble salts have detrimental effects on the finished ceramic body. Soluble salts such as chlorides, phosphates and nitrates that are reflected by pH values exceeding 6.5, affect the surface color of ceramic products, and are potentially dangerous to the integrity of the finished product. Calcium and magnesium sulfates migrate to the surface during drying and leave visible surface-scum after firing (Formulating A Porcelain, 2001). Soluble salts migrate to the surface of vessels where extensive crystallization takes place causing exfoliation and peeling of the surface. Massive needle-like crystals of salts may cover the surface of vessels, and eventually, due to stresses, the vessel breaks (Pottery Conservation, 2001).

Experiments mentioned in Drying Ceramics (2000), have shown that barium carbonate (Ba_2CO_3) precipitates soluble salts so that they do not migrate to the surface with water during drying of raw claybody. In this study, samples MT1, MT2, MT3, MT4, MT5, MT10, MT13, MT14 and MT15 had pH values above 6.5, and at such pH values, clays are considered to contain soluble salts in them (Murray, 1986). For these clays to be utilized in ceramics, there is a need for them to be treated with Ba_2CO_3 .

The pH conditions of a clayey material as explained by Fellman (1996), critically affect the charges along the edge sites of clay minerals, and thus pH conditions often control flocculation and dispersion of claybodies. Under acid conditions, the adsorption of H^+ ions by illitic and kaolinitic clays causes the edge sites to become more positively charged and for the faces and the edges of the clay minerals to stick together. It is not certain whether this phenomenon promotes flocculation. In some cases, OH is introduced to

reduce the acidity. Where clays are very acidic, soap has been added to reduce acidity; and where clays are very basic, vinegar (acetic acid) and/or citric acid has been added to decrease alkalinity (Fellman, 1996). Consequently, the studied clays having high alkalinity may be treated with the suggested acids in order to make them suitable for use in the ceramic industry.

Influence of electrical conductivity of raw clays in ceramic applications

Olhoeft (2001) describes electrical conductivity (EC) as the ability of a material to transport charge through the process of conduction, normalized by geometry in understanding its property. The finer the particles, the higher the EC values of the clayey material (Veris Technologies, 2001). The amount of TDS in the bentonite and kaolin samples can be estimated from their EC values. The EC is used to estimate the presence of dissolved salts such as sulfates, carbonates, phosphates and chlorides. Dissolved salts affect the drying and firing of claybodies

The EC of the studied clay samples had values from 200 iS/cm for sample MT10 to as high as 6501 iS/cm for sample MT1. Samples MT1, MT2, MT3, MT4 and MT15 which are bentonitic clay samples had very high EC (the least value was 2501 iS/cm for sample MT3). Bentonites, because of their environments of formation, generally have high EC values (Weaver, 1989). These clay samples had very fine clayey texture, and would form very good vessels due to binding charges that bring closely together the clay particles. Unfortunately, clayey materials having these properties also exhibit very high shrinkage. Consequently, production waste resulting from cracking due to shrinkage when the claybodies are fired would definitely be very high.

Samples MT5, MT9, MT10, MT11, MT14, MT16 and MT17 had EC values below 1000 iS/cm . Although this group of samples also had fine textures but not as fine as those with very high conductivity listed above. The samples consisted mainly of kaolinitic clayey material. Kaolins are formed in acidic to neutral environments and with little or no soluble salts associated in their depositions (Weaver, 1989). These clay samples had fine clayey texture, and would form a good vessel because the binding charges that bring particles closely knitted together. Conversely, the clayey materials having these properties have low to acceptable shrinkage for ceramic applications. Consequently, production waste resulting from cracking due to shrinkage when the claybodies are fired would definitely be very low.

Samples MT12 and MT13, with EC values between 1001 $\mu\text{S}/\text{cm}$ and 2500 $\mu\text{S}/\text{cm}$, would have moderate shrinkage for ceramic applications. Bentonitic clays with high shrinkage values, as obtained in this study, are equally utilized as additives in augmenting clay conductivity of claybodies having poor cohesion properties. This suggestion has also been advanced by Fellman, (1996) for using clays, which poses very high EC values in the traditional ceramic industry.

Influence of cation exchange capacity of raw clays in ceramic applications

High CEC values are indicative of the presence of ions in solution prepared to exchange sites with other ions. This manifestation could be depicted as soluble salts, which have the tendency of causing severe problems in many applications (Murray, 1986), one of which is drying and firing of ceramic claybodies. The genesis of a clayey material may influence the amount of soluble salts contained in the clay (Velde, 1995). Bentonites because of the environments of formation and their chemical compositions tend to have higher CEC values than kaolin (Weaver, 1989).

The kaolinitic clays analyzed had lower CEC values compared to the bentonitic clays. None of the samples analyzed had CEC values that exceeded 105 meq/100g. Samples MT1, MT2, MT3 and MT4 had CEC values which were between 58 meq/100g and 105 meq/100g. Samples MT5, MT10, MT11, MT12, MT13, MT14, MT15, MT16 and MT17 had CEC values between 5 meq/100g and 20 meq/100g. The CEC values for bentonitic clays range from 50 meq/100g to 150 meq/100g and for kaolinitic clays are from 3 meq/100 g to 15 meq/100 g.

The upper limit of CEC value accepted in the ceramic industry is 11 meq/100g, which is the CEC value of most ball clays (Drying Ceramics 2000), although values as high as 25 meq/100g (Formulating A Porcelain, 2001) have been used. Borchardt (1989) showed that the swelling of smectite is partly due to its exchangeable cations and water. As a result of high swelling capacity which they poses, smectites at drying and firing stages initially expand because of hydration of interlayer cations. This expansion has a tremendous deficiency in product quality. Finished vessels of which smectitic clays constituted the principal clay mineral are not cylindrical, they have blisters, and cracks are common. In this study, all the clays have acceptable CEC values except samples MT1, MT2, MT and MT4. Their recommended usage will be as additives in boosting the CEC of clayey materials with very low CEC values. The rest of the studied samples could be used

directly as major clay mineral phases in formulating claybodies for firing.

Influence of specific surface area of raw clays in ceramic applications

The SSA of any clayey material is a variable used in the determination of clay suitability for ceramic applications. According to M & M Kaolins (2000), the SSA of most commercial clays used for ceramics is 10 m^2/g . The studied samples had SSA values between 4 m^2/g and 19 m^2/g . The SSA of clay particles has a linear relationship with the particle size. Clay particles are flat and are randomly oriented. Particle size and orientation affect the clay's drying shrinkage, which has a consequence on the finished product (Ceramic Terminology, 2001). Finer particles tend to increase the rate at which reactions occur in the kiln/furnace (Bloodworth et. al., 1993). Based on the SSAs of the clay samples analyzed in this study, they were found to be suitable for ceramic applications.

Influence of raw clay color in ceramic applications

The Munsell Soil Color Chart technique was used in assessing the color of raw and fired clay instead of the visual observation technique. The hue of unprocessed clay may vary from 5YR to 5Y, the value are not likely to be < 8 , and the chroma between 0 and 4 (Bloodworth et. al., 1993). The seventeen analyzed samples had hue ranging from 2.5Y to 10YR, value observed were from 2.5 to 8, and the chroma range was from 1 to 8. In terms of color, the samples were from white to black, although most of the samples had grayish and brownish shades. The different colors are considered to have been imparted by color causing elements retained either in the structure of the mineral or as associated oxides occurring with the clay mineral.

Color causing metals held within the structural octahedral sites of clay minerals affect the observed raw color of the claybody. Consequently, clay minerals with a lot of octahedral sites taken over by metals such as Fe and Mg contain less structural water, hence less energy will be required for dehydroxylation (Fellman, 1996), and the amorphous/vitrified state will be achieved at less temperature than usual.

The color of raw clays and finished-fired products has an esthetic significance in ceramics (Bloodworth et. al., 1993; Murray, 1979). Colors of raw claybodies are largely reliant on the presence of certain oxides of Fe and organic matter. Darkly colored claybodies generally are considered to have lower maturing temperature than lighter colored clays as

explained by Fellman (1996). Pure kaolin would require very high temperature for it to vitrify. This observation is contradictory to the mafic-felsic (dark-light) temperature gradient applied in mineralogy. Whereby clays are dark, the Fe oxides act as fluxing agents thereby lowering the temperature for melting and vitrification.

CONCLUSION

The results of physico-chemical analyses revealed that samples MT1, MT2, MT3 and MT4 had higher pH, EC, CEC, and SSA values compared to the other samples analyzed. Except for sample MT15 that had a uniquely black color, the hue/value/ chroma and color of the studied raw clay samples were within the acceptable range for clays used in ceramics. Kaolins are used quite extensively in the manufacturing of different types of ceramic products. Due to its purity, it has high fusion and is used in refractory ceramics. In a separate study, the kaolins from Botswana have been recommended for ceramic usage (Ekosse, 1994; 2000; 2001). Kaolins as ball clays provide good working properties and green strength to ceramic bodies. They have the required casting property, plasticity and dry strength to produce a final ceramic body. Samples MT1, MT2, MT3 and MT4, being identified as bentonitic clays could be conveniently utilized in the ceramic industry. The other samples, which were studied, being primarily kaolinitic, could be gainfully used in the ceramic industry to fulfil any of the functions mentioned in this text. However, the samples will have to be tailored towards specific applications.

For the studied clays to be utilized in the ceramic industry, they will have to be beneficiated so as to avoid process-generated problems such as black coring, bloating, crawling, crazing, exploding, peeling, pinholing, shivering and warping. These problems ensue from claybody formulations, glaze applications, the rate of firing, and the temperature of firing. Failure to eliminate the deficiencies makes the final product weak, unattractive and non-desirable. A lot of the vessels will be discarded, and the economic venture becomes a loss. A further study should therefore be carried out on identifying suitable glazes to be applied on the clay samples. The demand for clays and especially kaolin in the ceramic industry continues to grow at a very steady rate. Increasing demands are being placed on the improvement of clay mineral quality as applied to ceramics (Bloodworth *et. al.*, 1993). Process Technology is now required not only to purify the mineral, but to modify it to be suitable for tailor-guided applications. Market demands and consumer needs dictate cost effective

techniques of producing finished, beautiful and highly desired ceramic vessels.

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