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Mineralogical and major oxide characterization of Panyam clays, North - Central Nigeria

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Clay minerals are form over long periods of time, through weathering as a result of gradual chemical breakdown of rocks rich in feldspar. The extent of weathering is determined by many factors. Most common constituent mineral of clays is often one of the members of the mica mineral group. They include illite, sericite, muscovite, and biotite. Others are kaolinite, smectite, mixed-layer clays and chlorite. This paper characterizes eight clay samples from Panyam, North-Central Nigeria. The characterization is based on their mineralogy and major oxide chemistry. This is aimed at determining the extent of weathering and/or maturity and origin or source of the clay materials. Compositional mineralogical and oxide analysis was performed using X-Ray Diffraction (XRD) and X-Ray Fluorescence (XRF) respectively. The X-ray data revealed that the primary constituents of the rocks such as feldspars, biotite and other minerals have been altered to Illite and kaolinite. Nesbitt and Young weathering trend model was used for characterizing the samples using their major oxides; four samples classified as muscovite, two as kaolinite and two as illite. From the Nesbitt and Young model, it was discovered that all samples had a granitic origin with a slower rate of weathering.

Key words: Panyam, clay characterization, smectite, kaolinite, oxides.

INTRODUCTION

The term “clay” refers to a natural occurring material, composed primarily of fine-grained materials having one or more clay minerals with traces of metal oxides and organic matter that is plastic at appropriate water content and hardens when dried or fired. Clays are distinguished from other fine-grained soils by differences in size and mineralogy. Silts, which are also fine-grained soils that do not include clay minerals, tend to have larger particle sizes than clays. Geologists and soil scientists usually consider clay to be finer than silt, with particle size of <2 µm. Geotechnical engineers distinguish between clays and silts based on the plasticity properties of the soil, as measured by the soils' Atterberg limits (Guggenheim and Martin, 1995).

The character of the clay mineral found in a given soil depends on the nature of the parent material, climate, topography, vegetation and time during which these factors have operated (Grim, 1968). Clay minerals resemble the micas in chemical composition, except they are very fine grained, usually under microscope.

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Like the micas, clay minerals are shaped like flakes with irregular edges and one smooth side.

Most clay minerals with the possible exception of halloysite are synthesized from combination of hydroxides or oxides and water at fairly low temperatures and pressures. For example, Kaolinite can be formed in alumina-silica systems in the absence of alkaline earth. Illite cal also be formed with the addition of potassium. Vermiculite can be formed by a prolonged reaction, where potassium or mica is exchanged with any hydrated alkali or alkaline earth cation. Chlorite minerals can be formed by precipitation of hydroxide between layers of vermiculite or montmorillonite.

Clays are composed predominantly of hydrous aluminium silicate (Vitra, 2004). When wet, clay minerals exhibit plastic behaviour. These minerals are sheet silicates (or phyllosilicate) structures formed most commonly from combinations of Al-OH octahedral (gibbsite sheet) and Si-O tetrahedral (silica sheet). Additionally formed in few minerals is a brucite sheet comprising of Mg and Fe partially substituted for Al in Al-OH octahedral (gibbsite sheet). The most common types of clay minerals include kaolinites, Illites and Smectites (montmorillonites) (Al-Ani and Sarapaa, 2008).

Clay minerals are formed over long periods of time through the weathering processes which is a result of gradual chemical breakdown of rocks, usually silicate-bearing, by low concentrations of carbonic acid and other diluted solvents (Robb, 2005). The extent of weathering is determined by the nature of the parent rock, topography, climate, vegetation and the time period in which these factors operated. If the parent rock is soluble, weathering will be enhanced. Additionally, topography, climate, and vegetation can enhance weathering by controlling the character and direction of water moving through the weathered zones (Robertson, and Eggleton, 1991). Oliva et al. (2003) suggested that the positive impact of temperature on chemical weathering is obvious only when runoff is higher than 1000 mm/yr. Basin relief apart from run-off, is regarded as another insensitive control of chemical weathering in catchments (White and Blum, 1995). Since clays occur in different environments their source material varies and may include glacial, alluvium, loess, shale, weathered and fresh schist, slate and argillite. The most common constituent mineral of clays is often one of the members of the mica mineral group. Mica clays include Illite, sericite, muscovite, and biotite. Other frequently occurring minerals of clay are kaolinite, smectite, mixed-layer clays (interlayers of smectite and illite), and chlorite (Deer et al., 2003; Keith and Murray, 2006).

Clays are best studied and identified using the following: X-ray diffraction, X-ray fluorescence, energy-dispersive X-ray analysis, electron diffraction, differential thermal analysis, infrared spectroscopy and electron microscopy (WHO, 2005). This paper is therefore aimed at characterizing clay materials based on their mineralogy and major oxide chemistry, to determine the extent of weathering and/or maturity of the clay materials in the Panyam village of north central Nigeria.

Geology of the area

The study area is located in Panyam village, north-central Nigeria. It is geographically located between latitudes N9°21′ and N9°26′ and longitudes 9°11′E and 9°15′30″E. the area is accessible via Mangu-Shendam road Plateau State, in north-central Nigeria (Figure 1).

The geology of Nigeria is made up of three major litho-petrological components, namely, the Basement Complex, the Younger Granites and the Sedimentary Basins. The Basement Complex which is widely accepted to be Precambrian in age, is made up of the Migmatite-Gneiss Complex, the Schist Belt and the Older Granites. The Younger Granites comprises of several Jurassic Migmatite ring Complex centered on Jos and other parts of the north central Nigeria (Obaje, 2013).

Geologically, the area is composed of four major rock units, which can chronologically be listed as Granite gneiss, fine/medium-grained biotite granite, older basalt and new basalt as shown in Figure 2. While the granite gneiss are the oldest rocks which is part of the basement complex series, the fine to medium grained biotite granite are of the Jurassic non-orogenic alkaline younger granite series. The older and younger basalts are Tertiary and Quaternary volcanics, respectively. The basalts are distinguished on the basis of their period of emplacement and textural differences (Macleod, 1971; Lar and Tsalha, 2005). The older basalts occur as decomposed boulders, plugs or dome-like outcrops, while the newer basalts occur as cones and lava flows (Lar and Tsalha, 2005). Spatially, the newer basalt occupies about 85% of the area covering the Northern portion of the studied area. The fine to medium-grained biotite granite occupies about 10% of the area occurring along the entire southern portion of the studied area. The granite gneiss which covers about 4% occurs along the southwestern part of the area while the remaining 1% of the area far north is occupied by the older basalt (Figure 1). Given their relative ages, the basalts overlie directly the granitic rocks. The fines to medium-grained biotite granite outcrop with elevation reaching peak at about 1180 m (Figure 2), compared to the basalts.

SAMPLE CHARACTERIZATION AND METHODS

Sampling sites was carefully selected based on physiographic zones of the area; high ground (volcanics) and plains (FAO, 1998) with samples collected to properly represent the parent materials that would constitute mainly of volcanic rocks and basement system rocks. Sampling design involves the selection of the most efficient method for choosing the samples used to estimate the properties of the population. Probability sampling was adopted that selects sampling points at random locations using a range of specific

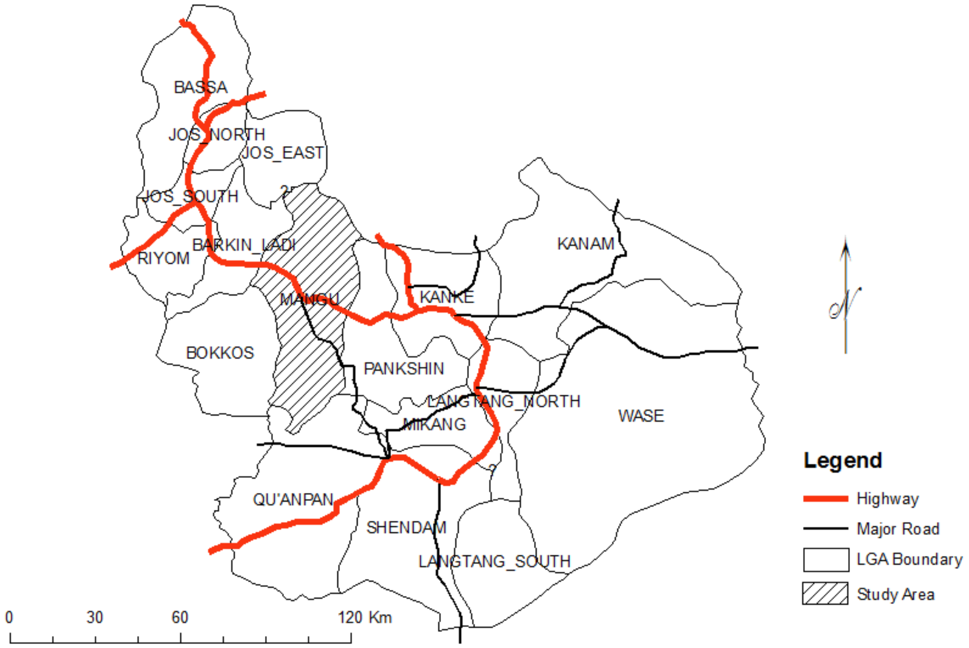


Figure 1. Location map of the area.

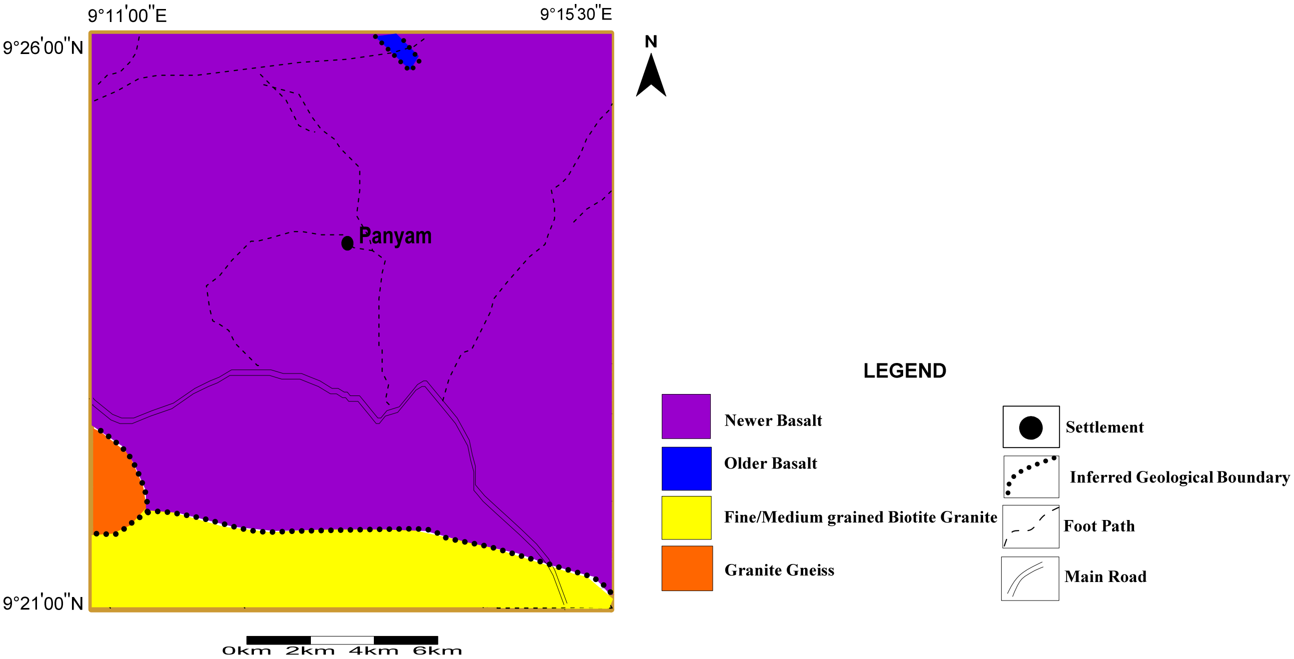


Figure 2. Geologic map of the study area.

sample layouts. The soils were retrieved from the area using the hand auger and were then plastic wrapped and air tight.

Eight soil samples were collected randomly around Panyam village, North-Central Nigeria (Figure 1). Samples were taken at an average depth of 1.5 m. The samples were characterized on the basis of their mineralogical and elemental (trace and oxides) composition. Each of the samples was split into two halves with each half used for mineralogical and elemental analysis. The compositional and mineralogical analyses were done using X-Ray Diffraction (XRD) and X-Ray Fluorescence (XRF) equipment respectively.

Compositional and mineralogical analyses were performed at the

Table 1. XRD mineralogical analysis data of samples.

Sample ID	Illite	Kaolinite	Muscovite
UAA 6		+	
UAA9			+
UAA14		+	
UAA15	+		
UAA16	+		
UAA24			+
UAA26			+
UAA36			+

Figure 3. Typical nature of soil in the area.

Nigerian Geological Survey Agency (NGSA) laboratory, Kaduna Nigeria. The XRD mineralogical analysis was done using the empyrean PANalytical schimadzu 6000 XRD model machine with CuKα radiation. The elemental analysis was carried out using PANalytical's minipal 4. This is a portable bench-top Energy Dispersive X-ray Fluorescence (EDXRF) spectrometer.

Elemental compositions of samples were determined using the XRF. The samples were pulverized using arget pulverizing machine. The ground samples were ensured to pass 150 micro mesh sieves. This was to ensure homogeneity of the samples. About 5 g of the pulverized sample was weighed into a beaker and 1 g of binding aid (Starch soluble) was added. The mixture was thoroughly mixed to ensure homogeneity, which was pressed under high pressure to produced pellets ready for the analysis. The pellets were carefully placed in the respective measuring positions on a sample changer of the machine to determine the elemental composition using a current of 14 kV for the major oxides with a selected filter; "kapton" for the major oxides. Time of measurement for each sample was 100 s and the medium used was air throughout. The machine was then celebrated by the machines gain control, after which the respective samples were measured by clicking the respective positions of the sample changer. For the XRD analysis, the powder was mixed with ethanol that serves as a dispersant solution and then spread evenly over the slide. The samples were then carefully pressed into an XRD sample holder using a glass plate. The operation was set at a voltage of 40 kV with current of 40 mA, a range of 5-80 deg 2θ and a step size of 0.017 deg 2θ. A time per step of 50.165 s with a divergence slit at fixed angle of 0.5°. Crystalline mineral phases were identified in X'Pert High Score Plus using the PDF-4 Minerals 2013 ICDD database. The quantities of the crystalline minerals were determined using the Rietveld method.

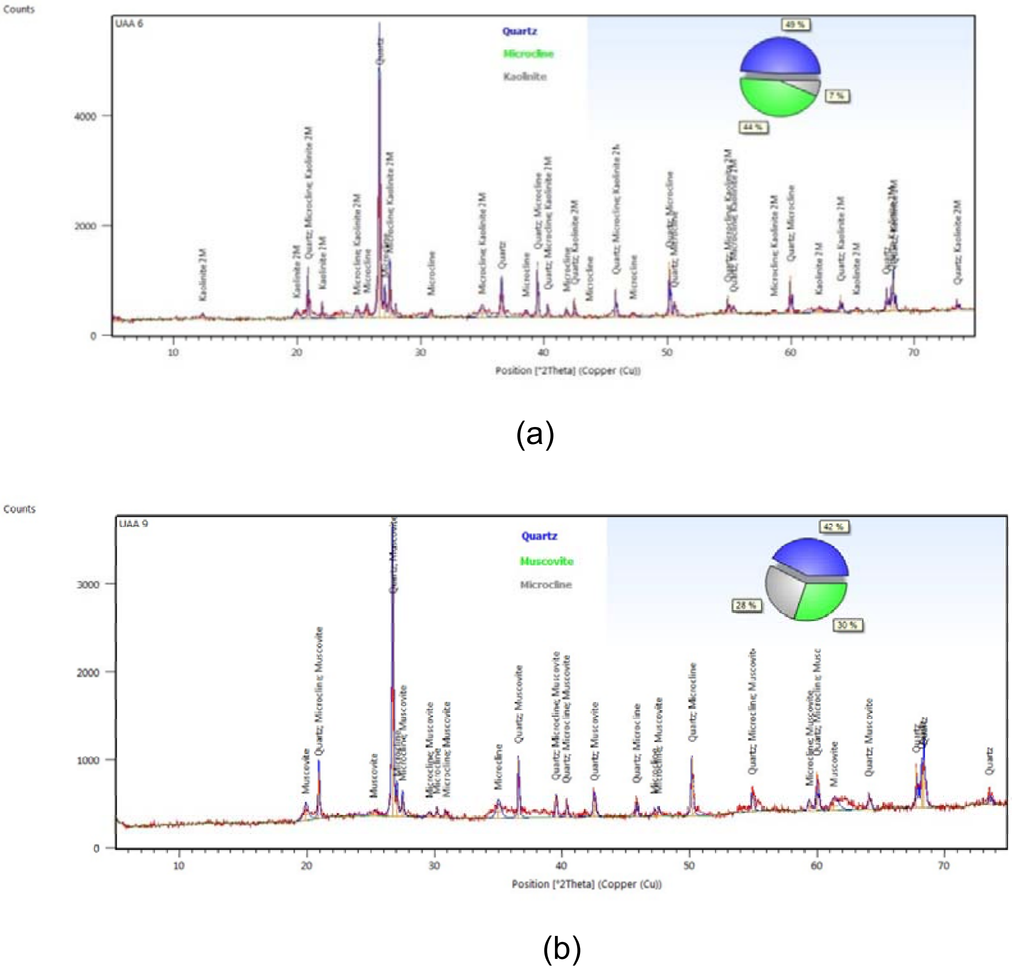
RESULTS AND DISCUSSION

Compositional analysis

Mineralogy

The XRD mineralogical data is presented in Table 1. The data indicates the presence of some mineral phases. The limited number of minerals measured could be as a result of equipment detection limit, which could mean that low concentrations are hard to be picked up by the equipment or could be due to the dominance of these mineral phases in the samples, given that they are clay samples. (Moore and Reynolds, 1997).

Of the eight samples analyzed, kaolinite was detected in two samples (sample ID UAA 6 and 14). illite in two samples (sample ID. UAA15 and 16). Muscovite was also detected in four samples as well (sample ID UAA 9, 24, 26 and 36). The area chosen was as a result of the infrastructural collapse because of the nature of the soil due to it mineralogy (Figure 3). The analysis shows that



(a)

(b)

Figure 4a and b. XRD results showing some mineral composition of some of the soil samples.

Table 2. XRF major oxide data of samples, values in percentage.

Sample ID	K_2O	Al_2O_3	$CaO + Na_2O$
UAA 6	2.69	24.97	1.03
UAA 9	3.78	16.45	1.11
UAA14	2.36	23.86	1.13
UAA15	3.01	16.49	1.06
UAA16	2.97	16.21	1.01
UAA24	3.86	16.01	1.21
UAA26	3.32	16.16	1.00
UAA36	2.99	16.02	0.90

all measured minerals belong to the silicate group of minerals and are of the phyllosilicate family (Figure 4a and b). To understand the trend of weathering in the area, a triangular diagram (Na_2O+CaO)-Al_2O_3-K_2O after Nesbitt and Young (1984, 1989) was plotted using the major oxides data from the XRF results (Table 2). This triangular diagram is interpreted as a measure of the extent of conversion of feldspars to clay.

The Nesbitt and Young triangular diagram show the trend of weathering in an average granitic rock. The weathering advances from K-feldspars to muscovite, Illite through to kaolinite. Therefore, from Figure 5, it can be

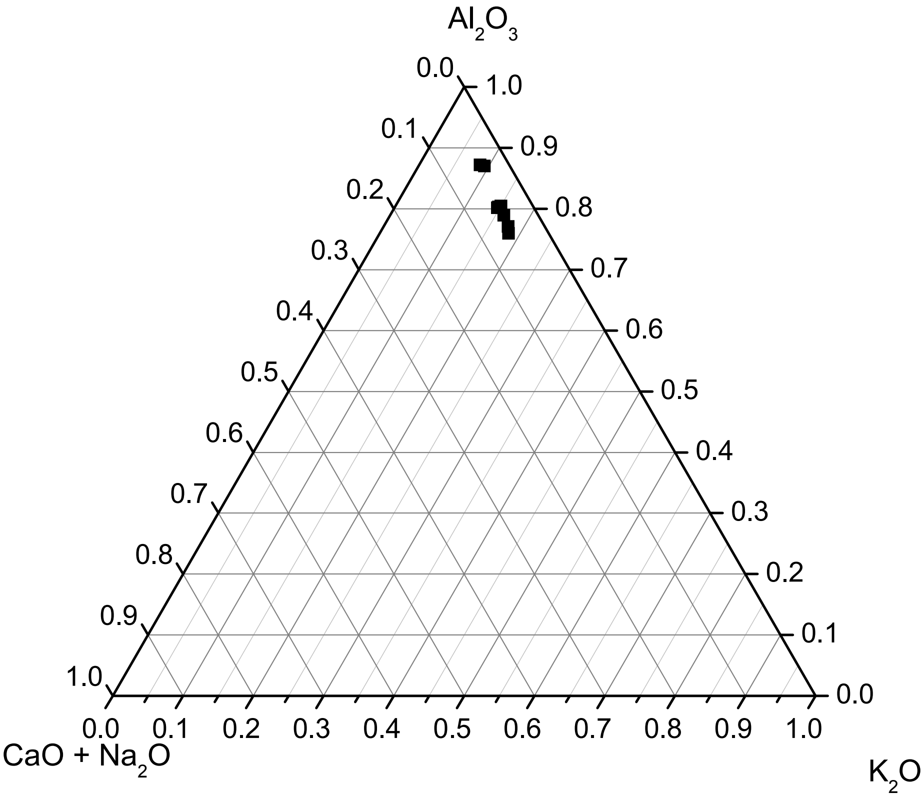
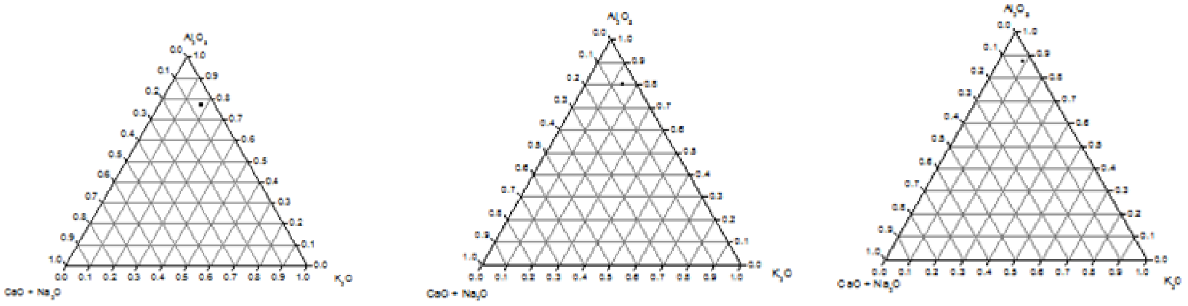


Figure 5. The (Na_2O+CaO)-Al_2O_3-K_2O diagram of all samples from the study area (after Nesbitt and Young 1984, 1989).

Table 3. Weathering trends in samples after Nesbitt and Young (1984, 1989).

Sample	Average Gabbro		Advanced weathering	Average granite		
ID	**Plagioclase**	**Smectite**	**Kaolinite**	**K-feldspars**	**Muscovite**	**Illite**
UAA 6			+			
UAA9					+	
UAA14			+			
UAA15						+
UAA16						+
UAA24					+	
UAA26					+	
UAA36					+	

observed that the plot revealed that samples are deficient in CaO+Na_2O, thus indicating advanced weathering within the plagioclase phase. The trend of enrichment can be shown as Al_2O_3 > K_2O> CaO+Na_2O. From the position of the samples on the Nesbitt and Young graph, the classification of the samples can be summarized as shown in Table 3. From the original Nesbitt and Young (1984, 1989), at 50-50% CaO+Na_2O vs. Al_2O_3, samples are classified as plagioclase, while at 80% Al_2O_3 samples are classified as Smectite. Along the K_2O - Al_2O_3 axis, at 50 to 60% concentrations, samples are classified as K-feldspars. Muscovite is reached at 75% Al_2O_3 concentration. Between 75 and 80% of Al_2O_3 concentration is Illite. From both axes, that is, Smectite and Illite samples advance to kaolinite which is indicative of advanced weathering. From Figure 5, it can be observed that samples UAA 9, 24, 26 and 36 plots between 75 and 80% Al_2O_3, 10 and 20% K_2O, 5 and 10% CaO+Na_2O. From the position of the plot, samples are classified as muscovite having progressively advanced



UAA9 (muscovite) UAA15 (illite) UAA 6 (kaolinite)

Figure 6. Trends in the weathering of different clays as shown from muscovite – illite – kaolinite in the different samples.

from K-feldspar due to depletion in K_2O (Figure 5 and Table 2). Also, Samples UAA 6 and 14 plots have Al_2O_3 concentration between 80 and 90, 5 and 10% concentration of K_2O and 2 and 8% concentration of $CaO+Na_2O$. All samples showed advancement in weathering and classified kaolinite.

Samples UAA 15 and 16 revealed concentration of 80 and 85% Al_2O_3, 10 and 15% concentration of K_2O, 1 and 8% concentration of $CaO+Na_2O$, the point of plotting coincided with that of Nesbit and Young (1984, 1989) corresponding to Illite. Hence, as Table 3 shows, the samples are classified into three dominant mineral phases as follow (Table 3):

(i) Kaolinite - samples UAA 6 and UAA 14
(ii) Illite - samples UAA 15 and UAA 16
(iii) Muscovite - samples UAA 9, UAA 24, UAA 26 and UAA 36 (Figure 4a and b).

Thus from the Nesbitt and Young (1984, 1989) weathering trend analysis, it has been concluded that the samples obtained from Panyam are mainly of granitic sources which is in agreement with the geology of the area. On a diagram of this type (Figure 5), initial stages of weathering form a trend parallel to the ($CaO + Na_2O$) - Al_2O_3 side of the figure as shown, whereas advanced weathering showed a marked loss of K_2O as the composition of the samples move towards the Al_2O_3 apex (Table 2), showing the degree of weathering from the basic (less advanced) to the advanced stage; muscovite – illite – kaolinite in the trend of the weathering (Figure 6). The trends followed mixing lines representing the removal of alkalis and calcium in solution during the breakdown of the first plagioclase and then potassium feldspars and ferromagnesian silicates.

IMPLICATION AND CONCLUSION

From the Nesbitt and Young weathering model, it is been confirmed that the Panyam clays are mainly from granitic source showing advanced weathering in some of the places. The clays of the study area are largely mica (50% of samples analyzed and classified). The other minerals phases present include kaolinite and illite. The dominance of biotite and with the presence of Illite, the soils of the area may have limited engineering applications.

CONFLICT OF INTERESTS

The authors have not declared any conflict of interests.

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