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X- Ray Diffraction Studies of Patti Claystone Member of the Campano-Maastrichtian Patti Formation, Southern Bida Basin, North Central Nigeria

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ABSTRACT*:* *X-ray diffraction and field studies were carried out on the Patti Clay Member of the Patti Formation in the southern Bida Basin. The studies involved field collection of samples and XRD laboratory analysis of clay samples in order to determine the dominant clay type, subordinate associated minerals, geologic processes and paleoenvironment of deposition. Field study involved standard litho-description by noting textural properties, facies type, fossil presence, structure and diagenetic effect. Laboratory XRD analysis procedure included sample grinding, XRD recording condition, selection and measurement of reference minerals, quantification of clay minerals using XRD processing software, XPert-Highscore Plus to generate results indicating clay mineral type and other minerals peaks. Lithostratigraphic section is dominated by the alternation of sandstone and claystone layers. The sedimentary structures are planar parallel and cross bedding in nature. The sandstone at Ahoko and along Lokoja-Abuja highway is characterized by liesegang ring and bioturbation structures. XRD results indicated that the Patti Clay Members are dominated by kaolinite with subordinate illite, montmorillonite and zincite in rare cases. The clay members were suggested to be deposited by moderate to low energy of transportation, moderate to low rate of deposition through prograding mechanism in a marginal marine setting. The kaolinite clay was formed by meteoric water reaction with the shallow-seated clay members through leaching of K^+, Na^+ and silica ions with resultant kaolinite stable at adequate pH that was not acidic.*

KEY WORDS: Diagenetic effect, liesegang ring structure, kaolinite, marginal marine, prograding and leaching

INTRODUCTION

The area of study lies along the Lokoja-Abuja highway (Kogi State and the Abuja Federal Capital Territory) within Sheets 247 Lokoja (NE and NW), 227 Koton Karfi (NE and SE) and 206 Kwali (SE) located within the southern part of Bida Basin (Fig. 1). The emphasis in this study is on clay facies present in the Patti Formation. Clay minerals have been used in the interpretation of paleoclimate, paleoenvironmental changes, analysis of hydrodynamic processes in coastal environments and to determine the sediment provenance (Bantan *et al*., 2020); paleogeographic reconstructions (Brunovic *et al*., 2020) and to determine sedimentological evolution in litoral environments (Lamoure *et al*. 2017)**.** Clay represents one of the most abundant type of sediment in sedimentary basins worldwide (Pettjohn, 1975) and is considered to represent average crustal provenance composition much better than any other detrital sedimentary rocks (McCulloch and Wasserburg, 1978).

Previous studies on Patti Formation were on the use of shale and claystone members present to determine source rock potential, formation age, lateral formation equivalent and paleoenvironment of deposition using organic geochemical and biostraigraphic techniques (Akande and Ojo, 2002; Akande *et al*., 2005; Ehinola *et al*., 2006; Oladimeji and Ola-Buraimo, 2021). The intercalated sandstone units were investigated for grain size, sorting, geological processes of transportation, deposition and paleoenvironmental reconstruction (Oladimeji and Ola-Buraimo, 2022). However, the application of x-ray diffraction analysis for the evaluation of the claystone member of Patti Formation is rare or not documented. Thus, this necessitates this study using XRD method on Patti Clay Member in order to determine the dominant clay type, transportation processes, and paleoenvironment of deposition.

X-ray powder diffraction was described as the best available technique for the identification and quantification of all minerals present in clay-rich rocks such as claystones, mudstones, and marls (Jan Strodon *et al*., 2001). However, accurate quantitative analysis of clays remains a formidable challenge (Reynold, 1989; Synder and Bish, 1989; Moore and Reynold, 1997). Accurate quantitative mineral analysis is important in petrological studies, engineering, and industrial applications of clay minerals (Jan Strodon *et al*., 2001). Mineral identification using XRD analysis was described to be relatively simple and unambiguous with the application of modern software and availability of good mineral database (Brindley, 1980; Reynolds, 1989; Snyder and Bish, 1989; McManus, 1991; Moore and Reynolds, 1997). The application of XRD analysis is also associated with errors if care is not taken; these could arise from chemical and structural characteristics of clay minerals vis-à-vis variable chemical composition, highly variable structures involving different patterns of layer interstratification including swelling interlayers, and various defects that disturb three-dimensional periodicity. In order to avoid these errors methodology used by Jan Strodon *et al*., (2001) was adopted whereby the selection of insensitive analytical reflections offers a better chance for success.

Geologic Setting

Bida Basin is an inland basin in Nigeria located geographically in the central part of Nigeria. It is bounded to the north by Sokoto Basin, to the east by Benue Trough, southward by Basement Complex and southeastward by Anambra Basin. The evolution of the Bida Basin was placed on different pivots such as rifting and drifting apart of African and South American plates (king, 1950 and Kennedy, 1965); wrench-fault tectonic model (Ojo, 1984 and Adeniyi, 1985); tension-built-up during rifting and separation of African and South American land masses during Mid-Cretaceous (Agyingi, 1991).

Bida Basin is characterized by three different stratigraphic facies (Adeleye, 1974; Akande et al., 2005; Oladimeji and Ola-Buraimo, 2021, 2022). The basin is marked at the base by Lokoja

Formation sitting unconformably on the basement crystalline rock, dated Campanian-Maastrichtian age. Patti Formation overlies the Lokoja Formation, characterized by shale, clay and sandstone lithofacies sequence. The Patti Formation was previously dated Maastrichian by Adeleye (1971) and Akande *et al*., (2005). However, the work of Oladimeji and Ola-Buraimo (2021) redefined the stratigraphic age of the formation based on the application of pollen and spores retrieved from the shale and claystone members, thus, put the age at Late Campanian-Early Maastrichian age.

Patti Formation was also correlated, having mutual stratigraphic age with Mamu Formation in Anambra Basin (Oladimeji and Ola-Buraimo, 2021; Fig. 2). The sandstone member of the Patti Formation was investigated, classified into coarse, medium and fine grain sandstones deposited in fluviatile and marine paleoenvironments (Oladimeji and Ola-Buraimo, 2022). The vertical and lateral facies relationship from shale through clay to various sandstone sizes was described as retrogradational system depositional style of Patti Formation (Oladimeji and Ola-Buraimo, 2022). Stratigraphically, the topmost formation is Agbaja Formation uniquely characterized dominantly by oolitic ironstone of about 5-20 m in thickness (Braide, 1992; Ladipo *et al*., 1994).

Field observations show that Patti Formation directly overlies the Lokoja Formation and consists of interbedding of siltstones, claystone, sandstone, shales and bioturbated ironstones. It is laterally equivalent to the Enagi Siltstone in the northern part of the basin. The exposures of the formation in Ahoko and Abaji show preponderance of kaolinitic and carbonaceous mudrocks containing woody plant debris where sandstones units are subordinate. The siltstones are parallel laminated and in places exhibit wavy ripples, as well as such soft sediment deformation structures as slumps, convolute lamination and load casts (Oladimeji and Ola-Buraimo, 2022). Trace fossils especially *Thalassinoides* are present. Biostratigraphic and paleoecologic studies by Petters (1986) revealed the occurrence of a cosmopolitan assemblage of arenaceous foraminifera- *Ammobaculites*, *Milliamina*, *Trochamina* and *Textularia* spp.

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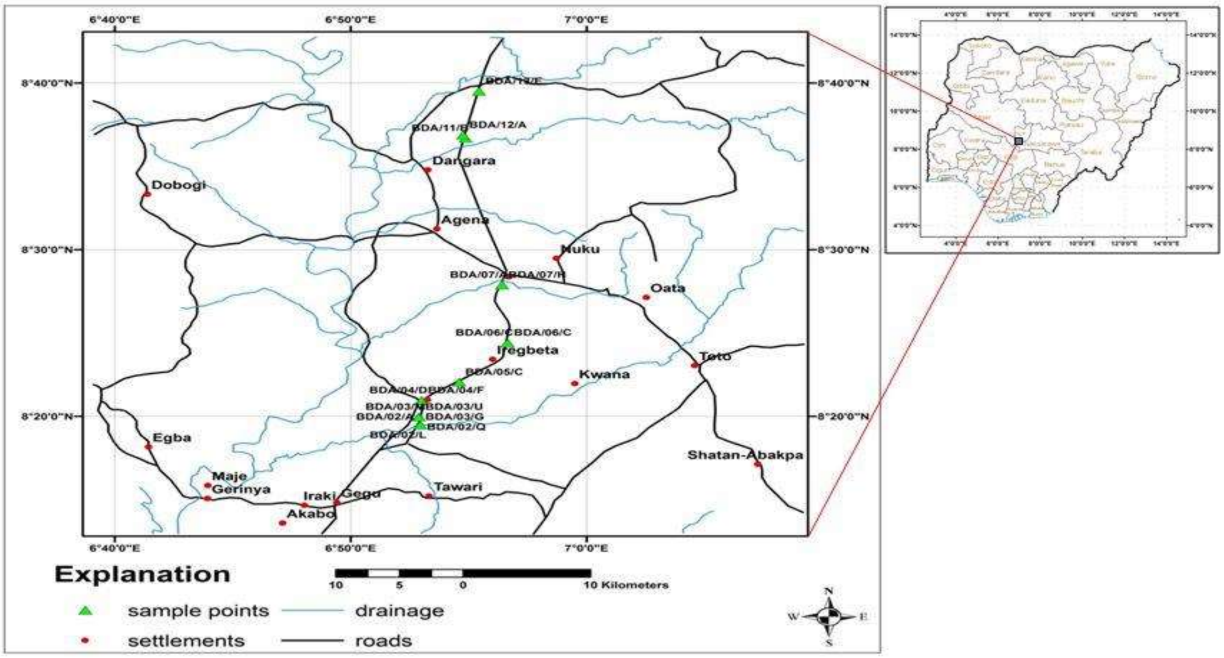

Figure 1. Map showing the location of the study area (After Oladimeji and Ola-Buraimo, 2021)

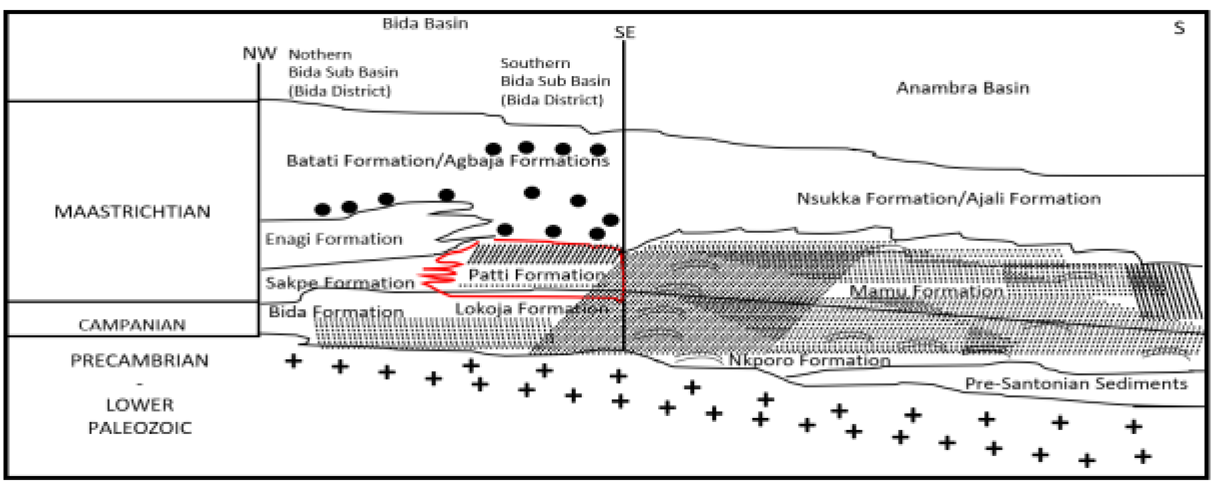

Figure 2. Stratigraphy of Maastrichtian-Campanian formations in the Bida Basin correlated with the Anambra Basin; Patti Formation highlighted (After Oladimeji and Ola-Buraimo, 2021).

METHODOLOGY

The methodology involved fieldwork and laboratory study. The field study was carried out as described succinctly in the work of Oladimeji and Ola-Buraimo (2021) whereby, physical, biological and chemical properties of the lithofacies section were considered. The laboratory analysis was carried out by using XRD. The laboratory procedures included sample grinding, XRD recording conditions, selection and measurement of reference minerals and quantification of clay minerals using XRD equipment (Plate 1).

Sample grinding is very important in order to get a precise and accurate results especially when it involves bulk rocks (Bish and Reynolds, 1989). Alexander and King (1948) suggested that a grain size less than 20µm is ideal for XRD analysis. However, the analyzed materials were finely ground into powder, then compressed using preparation block, flat sample holder to create a flat, smooth surface. The sample was later mounted on the sample stage in the XRD cabinet (Plate 2).

The samples were analyzed using reflection-transmission spinner stage at Theta-Theta setting. Two-Theta starting position was 5 degrees and ends at 80 degrees with a two-theta step of 0.026261 at 30 seconds per step. Tube current was 40mA and the tension was 45VA. A Programmable Divergent Slit was used with a 5mm Width Mask and the Gonio Scan was used.
The intensity of diffracted X-rays is continuously recorded as the sample and detector rotate through their respective angles. A peak in intensity occurs when the mineral contains lattice planes with d-spacings appropriate to diffract X-rays at that value of θ. Although each peak consists of two separate reflections ($K\alpha_1$ and $K\alpha_2$), at small values of 2θ the peak locations overlap with $K\alpha_2$ appearing as a hump on the side of $K\alpha_1$. Greater separation occurs at higher values of θ. Typically these combined peaks are treated as one. The 2λposition of the diffraction peak is typically measured as the center of the peak at 80% peak height.

Results are commonly presented as peak positions at 2θ and X-ray counts (intensity) in the form of a table or an x-y plot (shown above). Intensity (*I*) is either reported as peak height intensity, that intensity above background, or as integrated intensity, the area under the peak. The relative intensity is recorded as the ratio of the peak intensity to that of the most intense peak (*relative intensity* = $I/I_1 \times 100$).

The d-spacing of each peak is then obtained by solution of the Bragg equation for the appropriate value of λ. Once all d-spacings have been determined, automated search/match routines compare the *d*s of the unknown to those of known materials. Each mineral has a unique set of d-spacings, matching these d-spacings provides an identification of the unknown sample. A systematic procedure is used by ordering the d-spacings in terms of their intensity beginning with the most intense peak. Files of d-spacings for hundreds of thousands of inorganic compounds are available from the International Centre for Diffraction Data as the Powder Diffraction File (PDF). The peaks obtained from this analyses were matched with the minerals phases from the PDF 2 ICDD database which is attached to the XRD processing software, **XPert Highscore Plus** also from Panalytical. Therefore, the XRD processing software generated results with interpretation of dominant clay suites and other minerals present.

Plate 1. XRD machine **Plate 2. Internal section of the XRD equipment**

RESULTS AND DISCUSSIONS

Field Investigation and Sampling

The lithologic sections of the Patti Formation revealed alternating sequences of shales, claystone, siltstone and sandstone with ferruginous mudstone interbeds as shown in Figure 3. The shale units are dark-grey to black in colour with abundant plant remains, though very fissile but some are flabby.

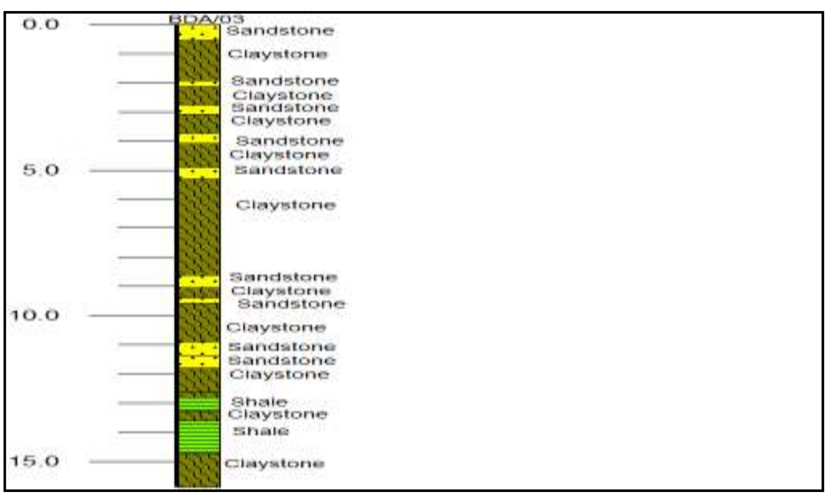

Figure 3. 2D strip log and lithofacies of BDA/03 location along Lokoja- Abuja highway

The clay units are whitish grey to yellow and reddish to purple in colour, while the sandstone units are fine to coarse grained in size with beds of 0.17 to 6 m in thickness. The sandstones exhibit a variety of colours ranging from whitish through greishy to yellowish and reddish brown in some cases (Fig. 3).

Liesegang ring structure is prominent in sandstone concretions at Ahoko along Lokoja-Abuja highway. The liesegang ring structures are in concentric bands with load cast within (Plate 3). The structure was described to form as a result of chemical precipitation of authigenic minerals under conditions of fluid concentration separated by spaces arranged in a regular repeated pattern (Plate 3). Liesegang ring structure was described to form under the condition of spatiotemporal reaction of polymer-analogous transformation (salt to base) of chitosan (Tatiana *et al*., 2020). Several authors advanced different phenomenon responsible for the formation of liesegang ring structure, however, fluid mineral rich concentration, phase differentiation and crystallization are the controlling factors (Kurochkina *et al*., 2021, Min-Kyung *et al*., 2022; Yu Seob et al., 2022; Jean-Yves *et al*., 2022 and Chen *et al*., 2022). Siltstone facies is light to reddish brown with bed thickness ranging from 0.11 to about 7 m. However, the claystone units are of utmost interest for the x-ray diffraction in this study.

Plate 3. Liesegang ring structure in sandstone concretions at Ahoko, along the Lokoja–Abuja highway

Claystone is the most frequently occurring rock type in the study area; observed in almost all outcrops. It occurs as planar cross bedding and parallel beds in the study area exhibiting whitish grey to yellowish and reddish to purplish colours (Plates 4 and 5). The
claystone is fairly to heavily bioturbated in some outcrops and exhibits heterolitic nature in relationship with sandstone in some outcrops.

Plate 4. Showing concretionary sandstone with wavy geometry, along the Lokoja–Abuja highway (Oladimeji and Ola-Buraimo, 2021)

Plate 5. Showing heterolithic beds of sandstone-claystone along lokoja-Abaji-Abuja highway (Oladimeji and Ola-Buraimo, 2021, 2022)

Laboratory result

X-ray diffraction analysis

X-ray diffraction analysis was carried out on six claystone samples. The essence was to determine bulk mineral assemblages of the claystone samples and dominant clay mineral type that typified Patti Clay Member. The XRD machine used is self-interpretative; indicated the dominant clay mineral types and associated minerals present in the claystone samples. The samples collected from the field were coded as BDA/03/A, BDA/05/A, BDA/06/A, BBB/01/A, BBB/02/A and BBB/07/E. Results from the X-ray diffraction analysis revealed minerals such as quartz and kaolin as dominant minerals in all the samples. Samples BDA/03/A and BBB/07/E of Figures 7 and 12 respectively are characterized by preponderance of quartz mineral peaks in association with relatively low amount and low peaks of kaolinite. The dominance of the quartz mineral peaks in the XRD result might suggest the relationship between the amount of quartz grains to kaolinite particles present in the samples. The paleoenvironment of deposition of the clay samples was suggested to be of a relatively moderate energy of transportation and moderate rate of deposition in a marginal marine setting. Figures 8-10 are depicted by strong quantitative peaks of kaolinite and relatively low quantitative peaks of quartz. There is a relatively greater amount of clay/sand ratio from the XRD peaks for the samples in Figures 8-10, depictive of relatively low energy of transportation and slow rate of sedimentation in the basin in a prograding marginal marine setting. The paleoenvironment deduced here is in tandem with paleoenvironment indicated for Clay Member in Patti Formation of same Bida Basin through the application of palynology and in the work of Oladimeji and Ola-Buraimo (2021). Sample BBB/2/A represented in Figure 11 was interpreted to contain quantitative strong peaks of kaolinite and moderately quantitative peaks of quartz. Subordinate amount and low quantitative peaks of rare minerals such as montmorillonite in sample BDA/03/A (Fig. 7); zincite in sample BDA/05/A (Fig. 8); illite in sample BDA/06/A (Fig. 9) is present. The presence of the clay mineral types in the analyzed claystone samples could suggest diagenetic transformation stages in the weathering of feldspatic rich and mica associated sediments from montmorillonite through illite to stable kaolinite clay facies in the Patti Formation

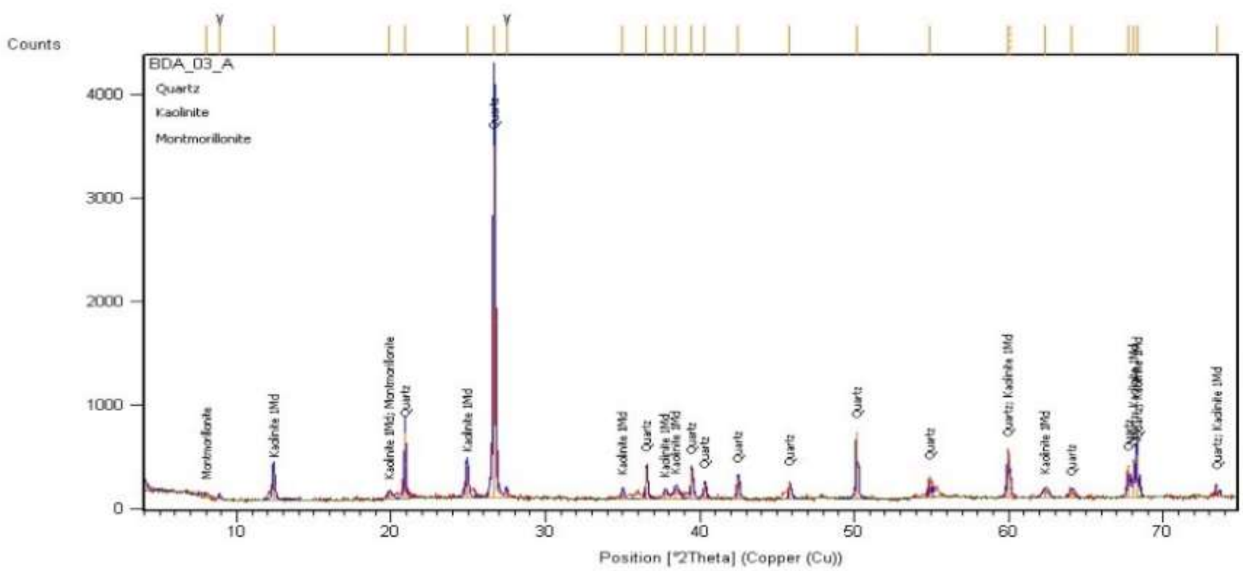

Figure 7: XRD result of sample BDA/03/A

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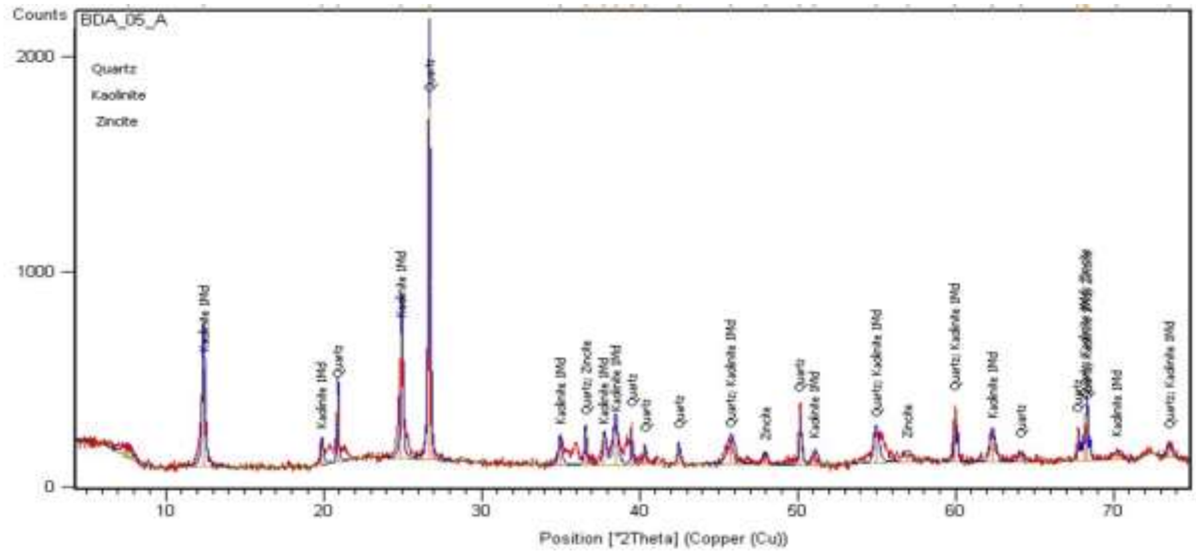

Figure 8. XRD result of sample BDA/05/A

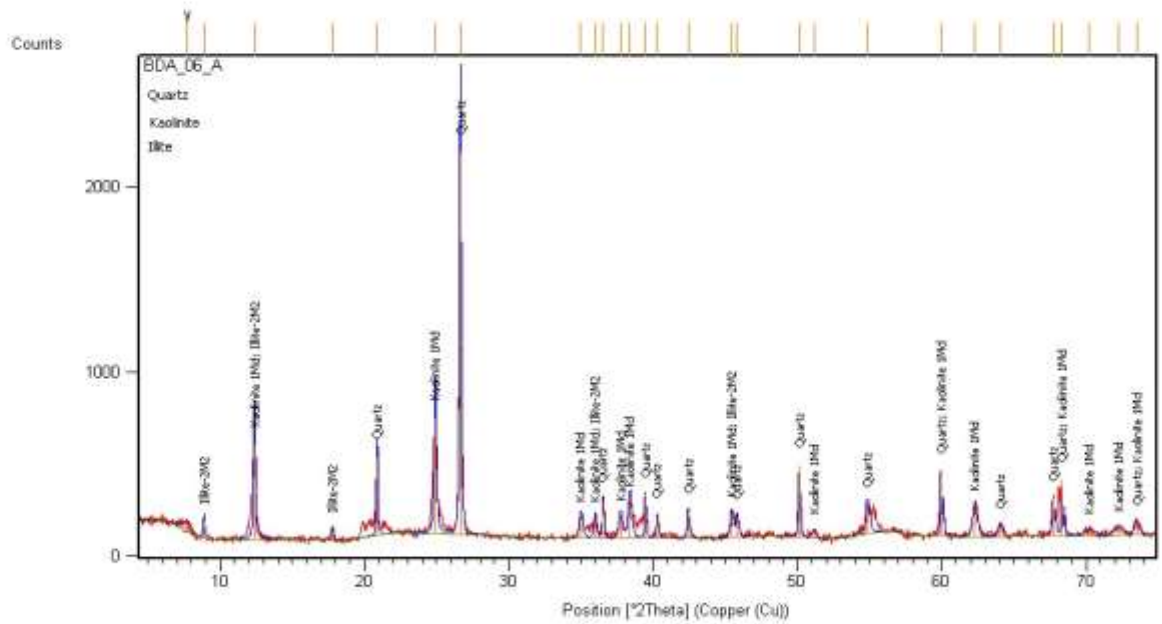

Figure 9: XRD result of sample BDA/06/A

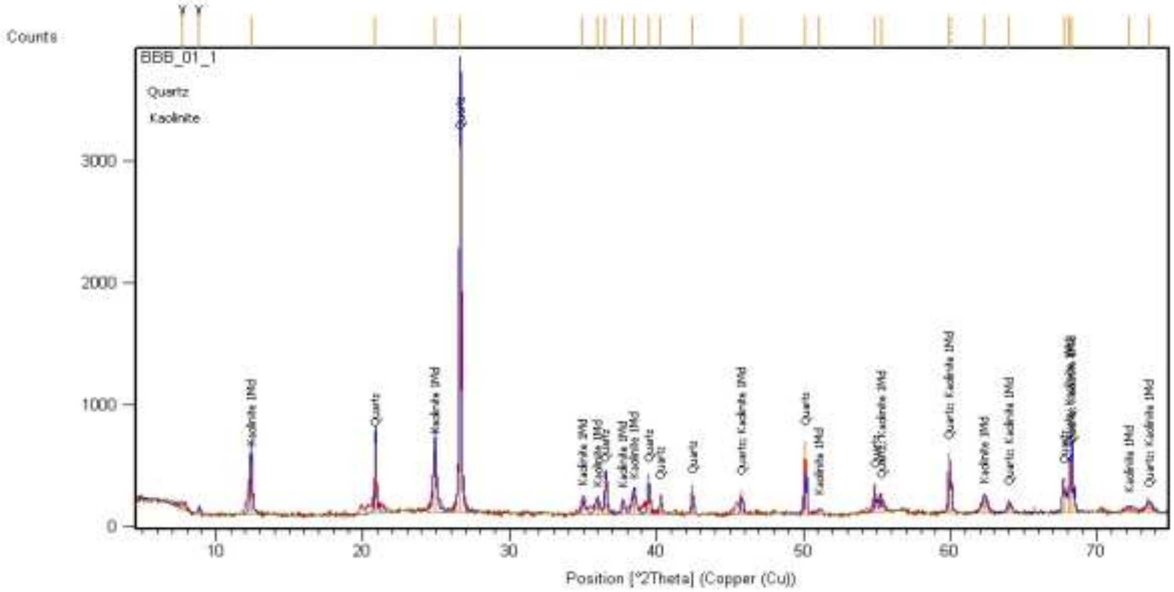

Figure 10: XRD result of sample BBB/01/A

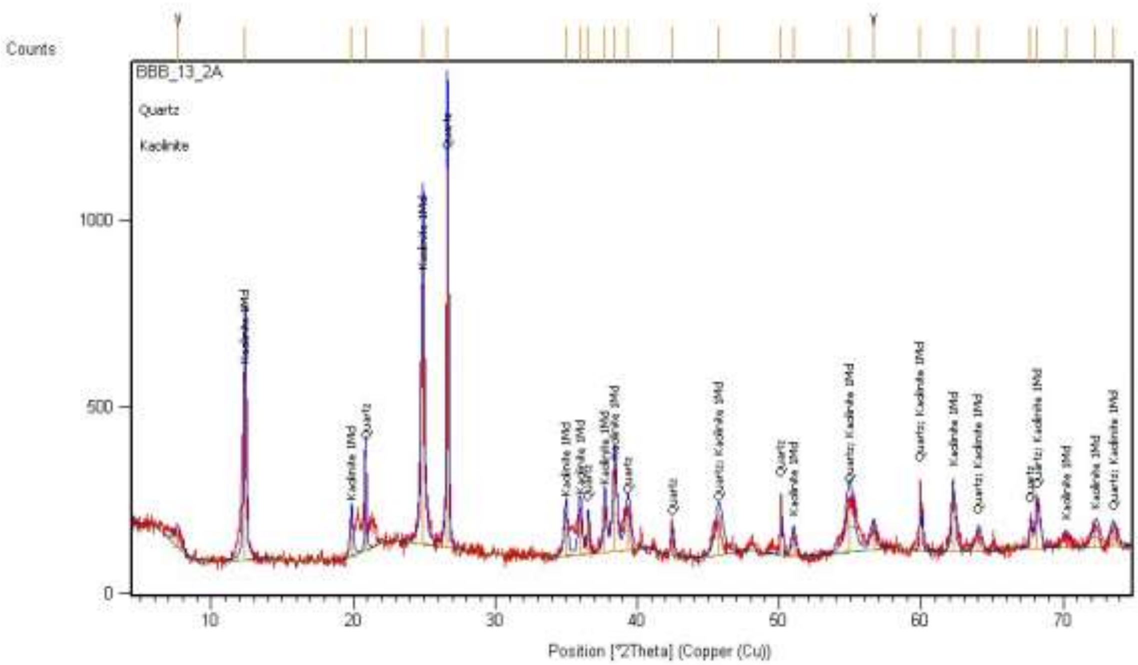

Figure 11: XRD result of sample BBB/2/A

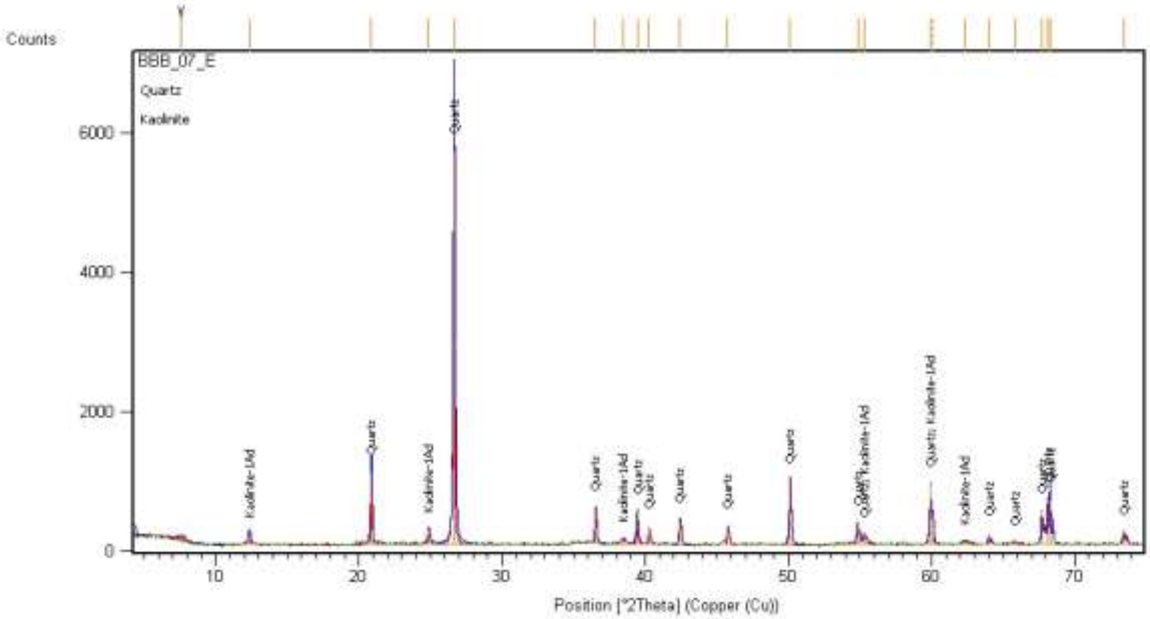

Figure 12: XRD result of sample BBB/07/E

A correlation between the Patti Formation and Asata /Nkporo Shale in terms of geologic age was put at Campanian–Maastrichtian (Oladimeji and Ola-Buraimo, 2021; Ola-Buraimo and Akaigbobi, 2013). Similar correlation in this study was found in the clay mineralogy suite of the Patti Formation in Bida Basin and Nkporo Shale in the adjacent Anambra Basin with dominance of kaolinite when compared with the work of Ikoro *et al*. (2012).The formation of the Kaolinite resulted from the dissolution of feldspar and mica as a result of flow of meteoric water within to remove cations such as Na^+ and K^+ and silica as shown in the chemical reaction below (Bjorlykke, 1997).

$$2K(Na)AlSi_3O^8 + 2H^+ + 9H_2O = Al_2SiO_5(OH)_4 + 2H_4SiO_4 + 2K(Na^{+)}$$

This mechanism as shown in the chemical reaction above is usually aided by leaching towards formation of authigenetic kaolinite like that of Patti Clay Members. Therefore, the Patti Formation kaolinitic clay members are suggested to have resulted from burial diagenesis, whereby, meteoric water flow reacted deep into the Patti Formation of the Bida sedimentary basin driven by the ground water. However, the in-situ weathering that took place in the Patti Formation allowed removal of K^+, Na^+ and silica ions in such a way that the pore-water within the clay minerals remained in stable condition of the resultant kaolinite minerals. The pore-waters that retained in the Patti Formation kaolinite clay members were not acidic but perhaps have a low $K^{+/}H^+$, Na^+/H^+ and Si^+/H^+ ratios.

CONCLUSIONS

Outcrop section of the Patti Formation have alternation of sandstone and claystone facies; characterized by planar parallel, cross-bedding and bioturbation stuctures. The sandstone beds at Ahoko along Lokoja-Abuja highway is marked by presence of liesegang ring structure having within it load cast. The liesegang structure was formed as a result of high concentration of fluid, crystallized in uniform rings.

The XRD results suggested variable sand/clay ratio depictive of different energy regime in terms of transportation and depositional rate in a marginal marine setting. The Patti Clay Member is dominated by kaolinite clay formed in-situ as a result of meteoric water reaction with feldspar and mica rich rock. The Patti Clay Member was diagenized through leaching of K^{+}, Na^{+} and Si^{+4} ions; left with resultant stable kaolinite mineral which constitute the Patti Clay Member.

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