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Bessong Moïse
Institute for Geological and Mining Research, Yaounde – Cameroon.

Mbesse Cecile Olive
University of Douala-Cameroon.

Ntsama Atangana Jacqueline
Department of Science, University of Ngaoundere – Cameroon

Hell Joseph Victor
Institute for Geological and Mining Research, Yaounde - Cameroon

Abderrazack EL Albani
University of Poitiers-France.

Claude Fontaine
University of Poitiers-France

Nolla Junior Désiré
Institute for Geological and Mining Research, Yaounde – Cameroon.

Dissombo Edimo André Noel
Institute for Geological and Mining Research, Yaounde – Cameroon.

Eyong John Takem
Institute for Geological and Mining Research, Yaounde – Cameroon.

Mvondo Owono Francois
University of Douala-Cameroon.

Correspondence:
Bessong Moïse
Institute for Geological and Mining Research, Yaounde – Cameroon.

Mineralogy and clay minerals distribution in the Benue Trough, Northern Cameroon (W. Africa): Diagenetic Significance

Bessong Moïse, Mbesse Cecile Olive, Ntsama Atangana Jacqueline, Hell Joseph Victor, Abderrazack EL Albani, Claude Fontaine, Nolla Junior Désiré, Dissombo Edimo André Noel, Eyong John Takem, Mvondo Owono Francois

Abstract
Putting aside contents, mineralogy generally varies slightly regarding its main constituents which include quartz, potassic feldspars and kaolinite. Added to these we have iron oxides (hematite and goethite) and manganese oxide (cryptomelane). Clays are mainly dominated by the kaolin group. Illites/micas, interstratified illites/smectites and smectites are present in little quantities in the locality of Boumedje but occur as traces in Ndjola. The mineral assemblages are mainly controlled by diagenetic processes (burial, circulation of fluids). Nevertheless, the present climatic conditions are equally responsible for the formation of some minerals of the kaolinite type. A mineralogical study has led to the putting in place of mineral assemblages.

Keywords: Benue Trough, Clay minerals, Garoua-Sandstone, Mineralogy, Diagenesis

1. Introduction
According to [1], the Benue Trough (Figure 1.A) is the most interesting sedimentary basins of West Africa mainly because of the tectonic movements that affected the marine and continental sediments in it and so due to the presence of volcanism and plutonic intrusions. The study area is in Cameroon and is an extension of the Benue trough or rift (Yola - Garoua branch). It is located precisely in the formation commonly known as the "Garoua sandstone". Many geologists have carried out studies in North Cameroon, amongst whom the following can be cited: [2-14] and other researchers of the MGRC (Mining and Geological Research Centre). This formation representing the extension of the sandstone petroleum reservoir of Nigeria in Cameroonian territory has not been well studied during the last few years especially regarding the mineralogy of the sedimentary assemblages. The main objective is therefore to define mineralogy of Garoua Sandstone by the clay assemblages and their evolution in order to bring out evidence and to interprete the markers of the depositional palaeoconditions and also to estimate the degree of burial.

2. Materials and methods
Samples were obtained from nine representative sectors in the study zone (Figure 1.B) mainly because of the impressing number of sandy hills or buttes present.
Sampling was done along geologic sections from the bottom/base to the summits of the buttes/hills and also along the streams in a direction perpendicular to the beds. Only samples presenting fresh surfaces were conserved for laboratory analyses. The study of the lithofacies mainly consists of the survey of the outcrop section and description of reference hills/buttes; in order facilitate reconnaissance, interpretation and comprehension of their spatio-temporal variation in view of reconstituting palaeoenvironments. For macroscopic description, the classification of [15] was adopted in order to designate the sizes of the elements/materials. The vertical succession of the facies was represented as to facilitate sequence analysis. X- ray diffraction (XRD) was carried out on all the samples. Their powders were obtained by

automatic crushing of part of the massive samples. Disoriented preparations were done using [16] protocol, in such a way as to avoid the orientation of the crystallites. The defractometry is a Barker D8 Advance (A25) with a copper anticathode. The acquisition of diffractograms is managed with the software "Diffract" V11.0.8 treated with the software EVA. The conditions of analysis are Cukα, 40 kv, 40 mA. The identification of minerals on disoriented powders is done by comparing with files from JCPDS (Joint Committee on Powder Diffraction Standards).

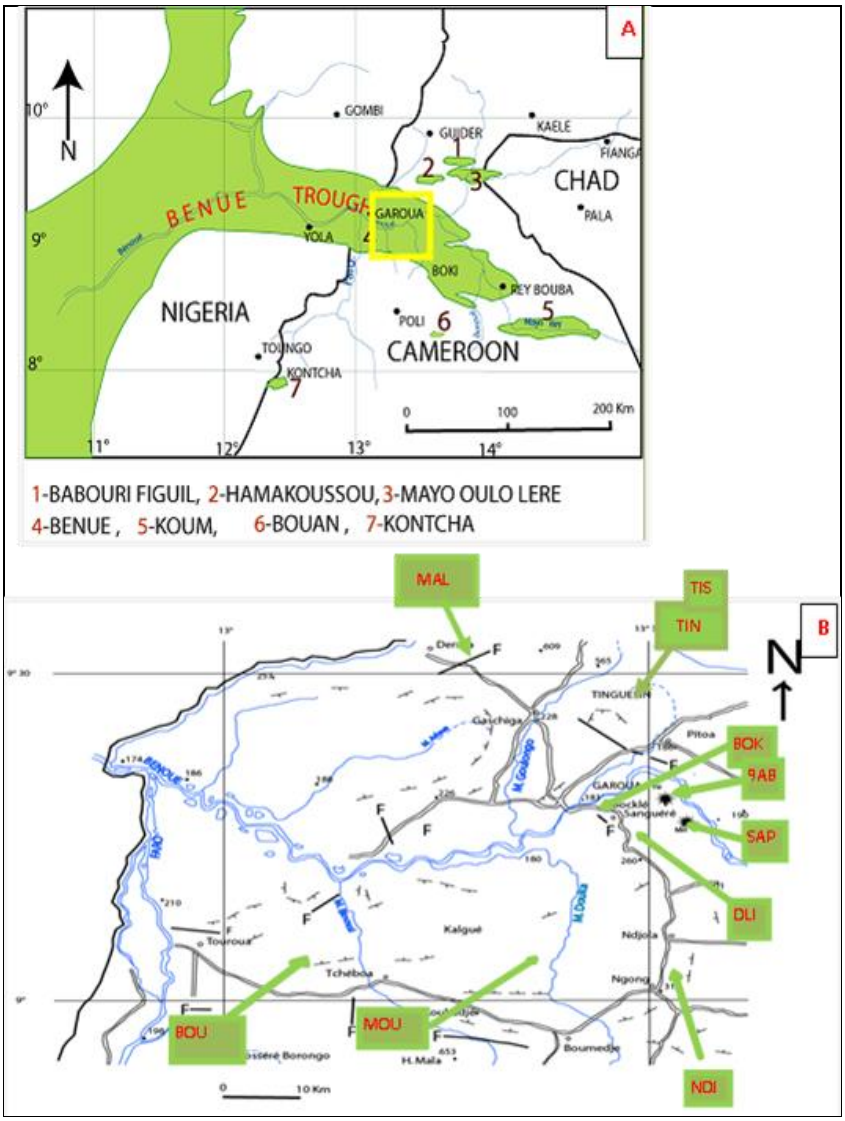

Fig 1: Location of the Benue Trough (A) and the different sampling sites (B).

MAL*: Malmakabay;* **TIN***: Tinguelin;* **TIS***: Tinguelin basement;* **BOK***: Bocklé;* **BAB***: Babla;* **SAP***: Sanguéré;* **DLI***: Douli;* **NDJ***: Ndjola;* **MOU:** *Mourbeli ;* **BOU** *: Boumedjé.*

The Separation of the clay fraction is a function of the rock's hardness. For the unlithified/loose rocks, mixing and average breaking is carried out on the more hardened rocks. In this case, the obtained fragments are disaggregated using liquid nitrogen by alternate freezing and defrozing.

The Separation of the clayey fraction (<2µ m) has been done as follows:

- 20 to 50 g of the mixed or disaggregated rock is put in a 700 ml pot half filled with osmosed water. The obtained suspension is subjected to repeated ultra sound treatment. This is to accelerate the dispersion of particles. After this, it is agitated for about 4hours in a turning system.
- The obtained suspension is kept at 20 ˚C for about 8 hours minimum so as to control its stability. The clayey fraction by configuration is later on separated and oven dried at 60 ˚C. The centrifuge used is the JOUAN GR 422 type which does 100 spins/minute for 2 minutes and 41secondes at 20 ˚C.
- Before drying, the separated fraction in suspension is analyzed by Laser granulometry, in order to know if there exist different sizes in the fine fraction. The Laser granulometry used is a MALVERN Mastersize IP100.

For the analysis of the suspensions (<50µ m just as <20µ m), a lens of 45 mm focus is mounted (the domain investigated is between 0.1 to 80 µ m). The optimum concentration of the suspension is determined by measuring the optical density. The distribution curve obtain is a function of a chosen calculation model (passage of a measurement in 2D to a result in 3D), and notably the ovoid form of the chosen particle, which is commonly used.

In view of differentiating the minerals of the kaolin group, we were interested in the fraction comprised between 5 and

10 µ m according to [18]. The extraction method is as follows:

- We first of all proceed in a similar manner with extraction of the fraction < 2 µ m.
- This suspension is put in a glass beaker of 1litre and are allowed to settle/sediment at 20˚C for a given time interval (of 2 hours 35 minutes for particles <5µ m, and 38 minutes for those that are <10 µ m) which correspond to the settling of particles obeying Stocke's law. The clayey fraction is obtained by pipetting and the process is repeated several times, such that at 20 cm of the height of the sedimentation level, the solution becomes clear and it is then oven dried at 60 ˚C

The characterization of clay phases is effectuated with X-ray diffractograms on oriented preparations. The identification of existing phyllosilicates is based on the study of their reflection angle. Diffractograms obtained with disoriented powder preparation are used to determine the reflection position (060) and the relative intensity of the polytypes.

The acquisition duration of the X-ray diffraction spectra has been fixed in relation to the investigated objectives, even though its increase considerably ameliorates the quantity of the spectra obtained. A semi-quantitative analysis was done using the same minerals of the JCPDS reference (Table 1).

Diffractograms of oriented preparations**:** were acquired at a natural state (unsaturated), after being air dried, saturated with ethylene glycol for 24 hours and finally heated at 550 °C (in order to follow up the evolution of some phases). In order to realize a good preparation of the disoriented powders, the dry products are moderately crushed again in an agate mortar so as to obtain the ''powder''. The powder is placed without any pressing/compaction in a hollowed support and the surface of the material is then levelled with a thin slide in order to obtain a sample surface which is quite plane and without any orientation. Care is taken so that the powder should not be pressed or compacted in order to avoid the reorientation of the particles especially along their basal surfaces (001 plane).

Small sample blocks previously chosen during optic microscopic observation, were observed in a secondary electron mode with a Scanning electron microscopy (SEM): (tension: 15 KV, intensity: 1 nA) after metallization with gold. The objective of this observation is to characterize the morphology of the different mineral phases. The apparatus used is the scanning electron microscope JOEL JSM 6400 equipped with an energy dispersion photon spectrometry (EDS). This spectrometry possesses a diode SiLi detector with an oxide weight analytic error of 1.5 %, after calibration using different standards (albite, forsterite, orthoclase, wollastonite, manganese, titanium and pyrite metals).

Fourier Transformed Infrared spectrometry (FTIR) has been used in this case, associated to diffractometry in order to characterize and differentiate the clay mineral groups. The wavelength domain (MIR = 400 to 4000 cm^{-1}), precisely the vibration domains of the valences of hydroxide groups (3750 to 3600 cm^{-1}). The spectrometry used in a Nicolet 510-FTIR whose resolution power is about 4 cm. Pellets composed of 1 mg powder (clay fraction) of finely crushed sample and mixed in 150 mg of potassium bromide (KBr) were used. They are compressed at 5 $t.cm^{-2}$ and then at 12 t.cm-2 for 5 minutes. After drying at 100 ˚C for 12 hours, the pellets are analyzed by transmission at room temperature.

Table 1: JCPDS referential minerals used for the semi quantitative analysis of diffract grams

Name	Formula	N° JCPDS	RIR
Quartz	SiO_2	01-079-1910	3,07
Microcline	$KAlSi_3O_8$	01-084-1455	0,58
Orthoclase	$KAlSi_3O_8$	01-075-1592	0,81
Albite	NaAlSi3O8	00-009-0466	2,10
Goethite	FeO(OH)	01-081-0464	2,67
Hématite	Fe_2O_3	01-072-0469	3,24
Cryptomélane	KMn_8O_{16}	00-029-1020	3,12
Anatase	TiO2	01-071-1168	4,86
Calcite	$CaCO_3$	01-083-1762	3,25
Montmorillonite-15A	$Na_{0.3}(Al,Mg)_2Si_4O_{10}(OH)_2,4H_2O$	00-029-1498	*0,3 -0,5*
Montmorillonite-14A	$Na_{0.3}(Al,Mg)_2Si_4O_{10}(OH)_2,xH_2O$	00-013-0259	*0,3 -0,5*
Beidellite-12A	$Na_{0.3}Al_2(Si,Al)_4O_{10}(OH)_2,2H_2O$	00-043-0688	*0,3 -0,5*
Chlorite (clinochlore)	$(Mg_{3.0}Fe_{1.7}A_{l1.3})(Si_{2.6}Al_{1.4}O_{10})(OH)_8$	01-079-1270	1,00
Illite	$KAl_2Si_3AlO_{10}(OH)_2$	00-026-0911	0,50
Muscovite	$K(Al_{1.9}Fe_{0.1})(Si_3Al)O_{10}(OH)_2$	01-072-0496	0,41
Muscovite	$KAl_2Si_3O_{10}(OH)_2$	01-075-0948	2,94
Muscovite-Mg	$K(Al_{1.8}Mg_{0.2})(Al_{0.4}Si_{3.6})O_{10}(OH)_2$	01-070-1868	0,79
Kaolinite	$Al_2Si_2O_5(OH)_4$	01-080-0885	1,04

3. Results

The mineralogy as indicated in Table1 shows a marked diversity. At Ndjola, the sandstones are essentially made up of quartz. Potassic feldspars are quasi absent except for sample NDJ05 containing goethite. Other constituents are clay minerals and rare traces of illites and mica relics. On a more elevated topographic position, precisely at Morelia, the materials are mainly constituted of quartz and kaolinite. No feldspar indice or a 13Å phase was put into evidence. The oxide present is hematite and not goethite. Further to the West at a more similar altitude, the Boumedje section is made up of quartzo-feldsparthic deposits, with the feldspars mainly being potassic. The clay fraction is only made up of kaolinite. At a certain level with less compact clayey pebbles (BOU 08), the presence of anatase (TiO_2), traces of illites or mica and goethite were put into evidence underlining local

concentration (anatase and mica) and syn-sedimentary consolidation phenomena. Finally, this section is also characterised by the presence of calcite especially at some levels where it is present in the form of Cement.

The diffractograms of natural glycolated and heated oriented sections of Tinguelin show that the preponderant clay phase is kaolinite with the presence of the other minerals such as illite and quartz. Kaolinite is the most abundant clay mineral characterised by well individualised and crystallised peaks. It is associated to the relatively high amount of quartz and to illite. At Malmakabay, the clay mineral assemblages are characterised by kaolinite, illite and/or mica and a clayey phase at 11.40 Å which becomes displaced at 13, 1 Å after treatment ethylene glycol, synonymous to interstratified illite/smectite (Table 3).

The compilation of diffractograms of natural oriented sections show that samples from Malmakabay have a similar mineralogic composition except for MAL 02 which does not present this mineral phase around the 11.40 Å. The form and position of this pic vary and at times seem to get closer to 10 Å. The intensity ratio between the reflections of this phase at 11Å and illite/mica at 10 Å can be reversed. Quartz is equally in the fine fraction. At Bocklé, the clayey minerals observed on the diffractograms of glycolated natural oriented sections are represented essentially by kaolinite and illite (Table 3). Quartz and feldspars are also observed in little quantities

At Babla, the compilation of the difractograms show that the mineral assemblages are identical in the different treated samples and are made up of kaolinite and illite. Some minerals such as goethite, quartz and feldspar are also present in this fraction. At Sanguéré, kaolinite and illite/mica are the main clay phases in its diffractograms. Some minerals such as quartz and feldspars are equally present in the fine fraction (Table 3). In Douli, the clay minerals present on the diffractograms of glycolated heated, and natural oriented sections are mainly kaolinite and illite/mica (Table 3). At Ndjola, these diffractograms are represented by kaolinite, illite and smectite (Table 3). At Mourbeli, they are mainly represented by kaolinite and illite (Table 2). Hematite and quartz is observed alongside these clay minerals. At Boumedje, the minerals observed are mainly kaolinite, di-octahedric smectite and illite (Table 2). The presence of quartz and feldspars can also be noted in this fraction. Illite occurs discretely around kaolinite and is seen as an alteration product resulting from the diagenesis (Figures 2D, E and F). X-Ray diffraction on the fine fraction indicates the simultaneous presence of kaolinite/Quartz/Illite/Potassic feldspar phases. Microcrystalline quartz is well crystallized under the SEM; this could lead to the conclusion that these three minerals were formed during diagenesis. The presence of di-octahedric smectite is indicated in the Boumedje sector.

Table 2: mineraligical composition of whole rock samples, Tinguelin (Tin), Malmakabay (Mal), Ndjola (NDJ), Mourbeli (MOU), Boumedjé (BOU), Bocklé (BOK), Sanguéré (SAP), Babla (BAB) and Douli (DLI).

Sample	Qz	FK	P	A	He	Go	Crypt	Ca	Ka	I/M
Douli										
DLI 19	+++++++								++	tr
DLI 18	+++++++			+					++	tr
DLI 17b	+++++++								++	
DLI 15	+++++++		+						++	
DLI 13	+++++++					+			++	
DLI 12	+++++++					+			+++	
DLI 11	+++++++								++	
Babla										
BAB 03'	+++++++	++						tr (?)	++	
BAB 02'	+++++++	++						tr (?)	+	
BAB 01'	++++++	+++				++		+	+++	+
Sanguere										
SAP 26b	++++++	+++			++	+		+	+	
SAP 26a	++++++	+++			++	+		tr (?)	+	
SAP 25	+++++++	++						+	++	
SAP 23	+			++	+	+			+++++++	
SAP 22	+++++++	++						+	++	
SAP 21	+++++++	++						tr	++	
SAP 20	+++++++	++				+	tr (?)		++	
Bockle										
BOK 43	++++++	+++				++			+	
BOK 40	++++++	+++				++			tr	
BOK 36b	++++++	+++				+++		tr	++	
BOK 36a	++++++	+++			tr (?)	++		tr	+	
BOK 35n	++++++	+++					+++		++	
BOK 33	++++++	+++				+			+++	tr
BOK 32	++++++	+++				++		tr	+++	

(Quartz : Qz, K-Feldspars : FK, Plagioclase : P, Anatase : A, Hematite : He, Goethite : Go, Cryptomelane : Crypt., Calcite : Ca, Kaolinite : K, Illite/Mica : I/M

Sample	Qz	FK	P	A	He	Go	Ca	Ka	I/M
Boumedje									
BOU 09		+++++++							
BOU 08c gal.	++++++	++		+		+	+	++++	tr
BOU 08c mat.	++++++	++		+		+		+++	tr
BOU 08b	++++++	++		+		+		+++	+
BOU 08a	++++++	++		+		+	+	++++	tr
BOU 07	++++	++	+				++++	+	

BOU 06	+++++++	++				+		+	
BOU 05	+++++++	++					+	+	
BOU 04	+++++++	+++					+	+	
BOU 03	+++++++	+++					+	+	
BOU 02	+++++++				+			+++	+
BOU 00	+++++++	++						+	
Mourbeli									
MOU 03	+++++++				+			+++	
MOU 01	++++++			tr	+			++++	
Ndjola									
NDJ 10	+++++++							+++	
NDJ 07	+++++++							+++	tr
NDJ 06	+++++++							+++	+
NDJ 05c	++++++	++				++		++	
NDJ 05b	++++++	+++				+		+++	
NDJ 05a	+++++++	+				+		+	
NDJ 04	+++++++							++	

Sample	**Qz**	**FK**	**A**	**He**	**Go**	**Ka**	**I/M**	**Ch**
Malmakabay MAL 07	+++		**tr**		+	++++		
MAL 06	++++++	++++		+		+++		
MAL 05	++++++	++++		+		+		**tr**
MAL 04	++++++	+++		+		+++	**tr**	
MAL 03	++++++	++++		+		+++	**tr**	
MAL 02	++++++	++++		+		++	**tr**	
MAL 01	+++++++	+++		**tr**		+		

Sample	**Qz**	**A**	**He**	**Go**	**Crypt**	**K**
Tinguelin TIN 06	+++++++		tr	+		+
TIN 05	+++++++	tr	++			++
TIN 04	+++++++	tr				+++
TIN 01	+++++++				tr (?)	++

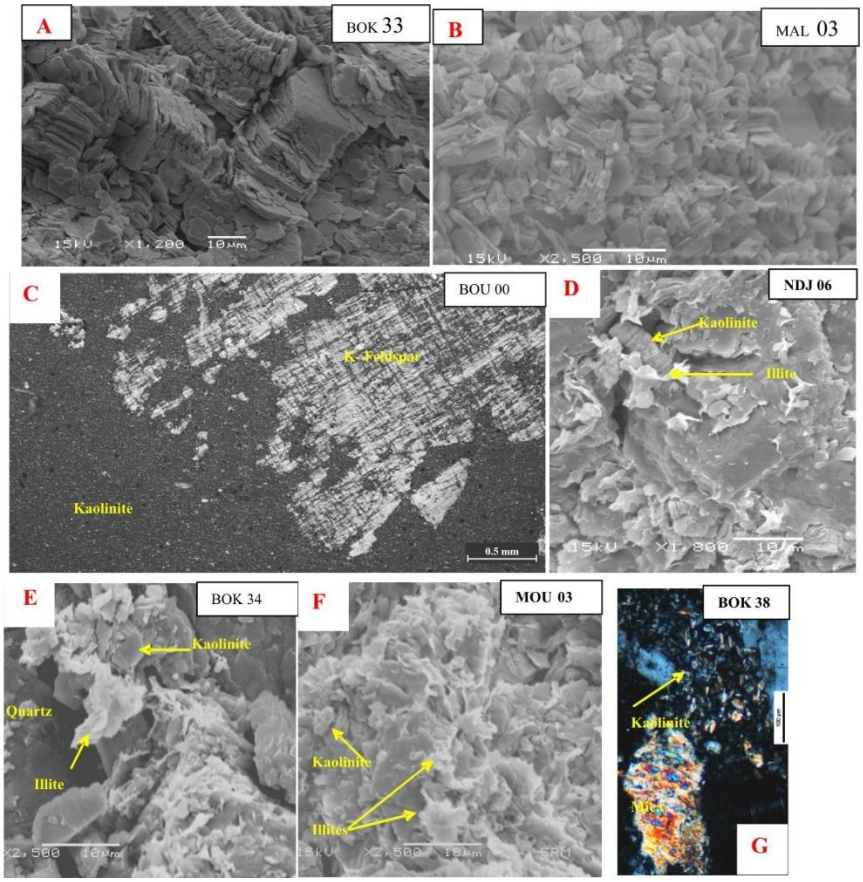

Fig 2: Morphology of kaolin group minerals showing vermicular kaolinite disposed in accordion (A) kaolin showing stocky crystals and sometimes elongated baguettes (B). Formation of kaolinite by alteration of potassium feldspar (C). Illite growing above kaolinite (D, E, and F). Transformation of mica into kaolinite (G).

Table 3: Mineralogical composition of whole rock samples, Tinguelin (TIN), Malmakabay (MAL), Ndjola (NDJ), Mourbeli (MOU) Boumedjé (BOU), Bocklé (BOK), Sanguéré (SAP), Babla (BAB) and Douli (DLI)

Sampling Site	X-Ray Diffracrion on oriented sections (infra 2µm)			
		Clay minerals		
	Sample	kaolin	illite	smectite
Northern part of the basin				
Hosséré Tinguelin	TIN 01	XXXX	X	
	TIN 04	XXXX	XX	
	TIN 05	XXXX	XX	
	TIN 06	XXXX	X	
Hosséré Malmakabay	MAL 01	XXXX	XX	
	MAL 02	XXXX	XX	
	MAL 03	XXXX	XX	
	MAL 04	XXXX	XX	
	MAL 05	XXXX	XX	
	MAL 06	XXXX	XX	
	MAL 07	XXXX	XX	
Central part of the basin				
Hosséré Bocklé	BOK 36	XX	X	
	BOK 37	XX		
	BOK 38	XXXX	X	X
	BOK 43	XXX		
Hosséré Babla	BAB 01	XXXX	X	
	BAB 02	XXXX	X	
	BAB 03	XXXX	X	
Hosséré Sanguéré	SAP 21	XXXX	X	
	SAP 22	XXXX	X	
	SAP 23	XXXX		
	SAP 25	XXXX	X	
	SAP 26	XXXX	X	
Hosséré Douli	DLI 12	XXXX	X	
	DLI 13	XXXX	X	
	DLI 15	XXXX		
	DLI 17	XXXX		
	DLI 18	XXXX	X	
	DLI 19	XXXX		
Southern part of the basin				
Hosséré Ndjola	NDJ 04	XXX		
	NDJ 06	XXXX	X	X
	NDJ 09	XXXX	X	
	NDJ 10	XXXX	X	
Hosséré Boumedjé	BOU 00	XX	X	XXX
	BOU 01	XX	X	XX
	BOU 02	XXX	XX	
	BOU 05	XXX	X	
	BOU 08	XX	X	
Hosséré Mourbelli	MOU 01	XXX	XX	
	MOU 02	XXX	XX	

XXXX : + 50%
XXX: between 50% and 30%
X : Mineral traces

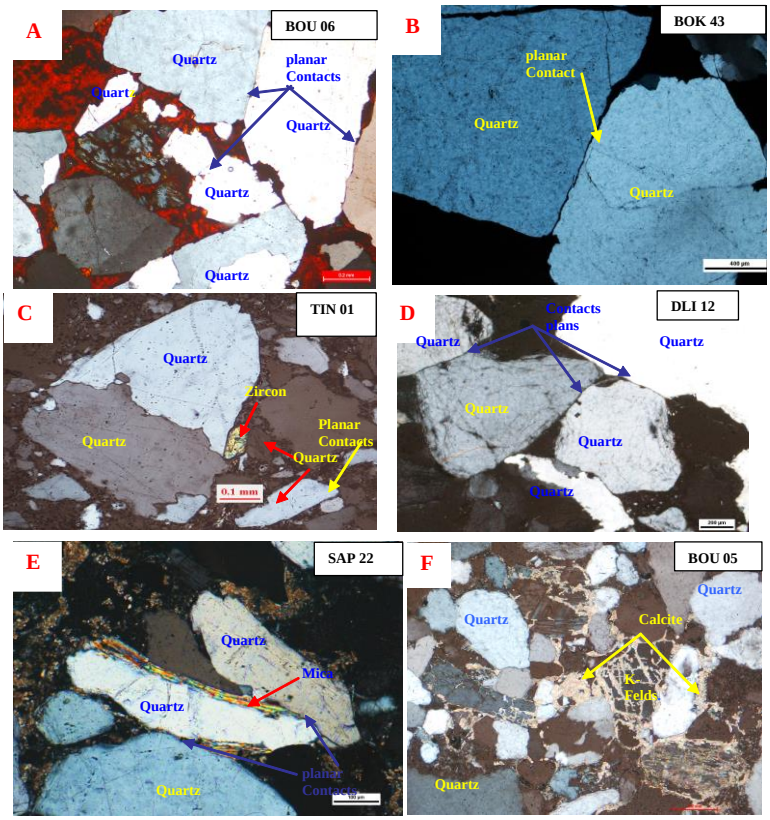

Fig 3: Mechanical compaction (3A and B) is illustrated not only by different types of contacts (3D and E), but also by interlocking grains contacts to concavo-convex quartz grains impressed (3D). Quartz grains under the effect of pressure (punching micas, Fig 3E). There may also be heavy minerals caught between the quartz grains (3C) carbonate cement (calcite) (3F)

4. Conclusion

Mineralogical studies have led to the delineation of the following mineralogical assemblages:

Kaolin group minerals are present in most of the samples. They are the products of the weathering of feldspars. This observation reveals that these minerals are of diagenetic [17]. The polytypes of kaolin enable the identification of dickite. The weathering of feldspars brings about the supply of Al and Si needed for the growth of the stable phase of dickite [18]. The transformation of kaolinite to dickite is controlled by diagenesis while structural and textural transformations depend on temperature [18, 19]. According to theses authors, dickite appears at between 2000-2500 m. This depth can be envisages in the Garoua sandstone from geophysical data which gives a thickness of 4000 m [20]. Based on the structure of the clays, we can note that the crystallinity index around 1.5 is indicative of well-structured materials. The R2 index is closer to 1.2 and confirms this observation. The degree of organization of kaolinite is higher than that of dickite [21, 22]. From the results of Infra-red spectra analysis, X-ray difractograms, the different treatments with hydrazine and from the morphological observation of kaolin using SEM, we could conclude that we are in the presence of a mixture of two phases (kaolinite and dickite) with greater proportion of kaolinite.

Illite develops on the kaolin group minerals as observed with the scanning electron microscope. The formation of dickite and illite takes place during deep diagenesis at temperatures between 100-130 °C [23, 24, 25]. This transformation takes place around the depth of 3500 m [23, 26]. A petrographic study of thin sections from some of the samples enables the degree of the compaction to be estimate. This was possible through an analysis of the contacts between the detritic fragments. We identified, planar contacts, concavo-convex contacts, sutured contacts and triple point contacts. This enables us to conclude that, the Garoua sandstone was subjected to a very intense degree of compaction.

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