GYPSUM IN THE DUKAMAJE AND DANGE FORMATIONS SOUTHEAST OF THE RIMA RIVER, SOKOTO BASIN NIGERIA

BY

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DECLARATION

I, Mr. Patrick, C. Agbata declare that this Thesis is my own work and has not been submitted in any form for another degree or diploma at any other University or Institution. Information derived from published or unpublished work of others has been acknowledged in the text.

Signature of Candidate

CERTIFICATION

This Thesis entitled "Gypsum in the Dukamaje and Dange Formations, Southeast of the Rima River, Sokoto Basin, Nigeria" by Patrick, C. Agbata meets the regulations governing the award of the degree of Master of Science of Ahmadu Bello University, and is approved for its contribution to knowledge and literary presentation.

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ABSTRACT

The area covered by this project is located on the the southeast bank of the Rima River area of the Sokoto Basin.

Gypsum occurs in the Sokoto Basin in the brackish water Dukamaje and the marine Dange Formations of Maastrichtian and Upper Paleocene ages respectively.

Gypsum occurs in fibrous forms as discontinous thin beds and veins,in septaria, and as minute disseminated crystals; it also occurs as large selenite crystals.

X-ray diffractometry of clay minerals in shales and fluid inclusion studies in gypsum indicate an alKaline reducing environment for the Dukamaje and Dange Formations and a low temperature (40°C) of gypsum formation respectively. Chemical analysis of the gypsum from Dukamaje and Dange Formations shows that the Ca:Mg ratio is 12:1 and 14:1 respectively. This ratio indicates that the fluids that formed gypsum are marine in origin and were later concentrated diagenetically to form gypsum once the solubility product was exceeded.

A diagenetic mode of origin is proposed for the gypsum in the study area based on the above data. This involves a continous diffusion of SO^{2-} ions into sediments from the overlying seawater and the dissolution of

Carbonate particles which releases Ca^{2+} ions, thus increasing the concentrations of Ca^{2+} and SO^{2-} ions in the sediments. Gravitational compaction, evidenced by the presence of two lignite seams in the Taloka Formation at Taloka village, further increased the Ca^{2+} and SO^{2-} ions concentrations by ion filteration through charged-net clay membranes. Both the Ca^{2+} and SO^{2-} ions combined to form gypsum once the solubility product of this mineral was exceeded.

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ABBREVIATIONS AND SYMBOLS

NW	-	Northwest
SE	-	Southeast
^{o}C	-	degree centigrade
Fm	-	Formation
Lat	-	Latitude
long	-	Longitude
Sp.	-	Species

Chapter 1

1. INTRODUCTION

1.1 General

Gypsum occurs in the Sokoto Basin mainly in the Dukamaje and Dange Formations of Maastrichtian and Upper Paleocene age respectively. The Sokoto Basin is part of the southeastern portion of a wide intracontinental sedimentary basin known as the Iullemeden Basin which covers part of Mali, Niger, Benin and Nigeria (Fig.1.1)

The environment of deposition of the Sokoto Basin sediments range from continental through marginal marine to normal marine. However, the formations of interest in this work are the Upper Paleocene marine Dange Formation and the Maastrichtian Dukamaje Formation which was deposited during the Maastrichtian marine incursion (Table 2.1).

The Dukamaje Formation separates the Taloka and Wurno Formations of the Rima Group of sediments while the Dange Formation is the oldest formation of the Sokoto Group of sediments.

1.2 Location and Extent

The project area is located in Sokoto State in Northwestern Nigeria, particularly in the Rima valley area of the Sokoto Basin, where gypsiferous beds are exposed (Fig. 1.2). The area is about 400 kilometers,

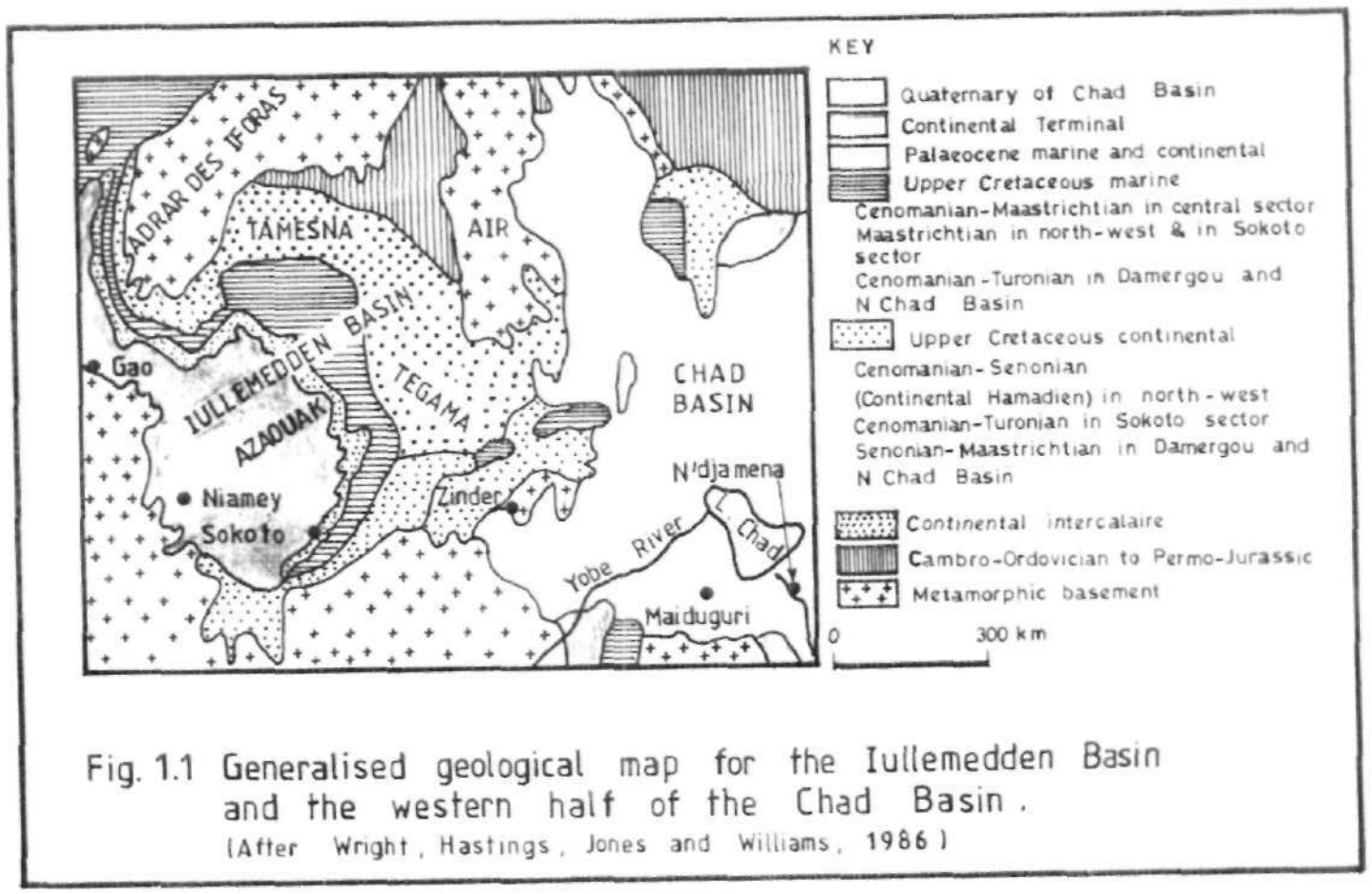
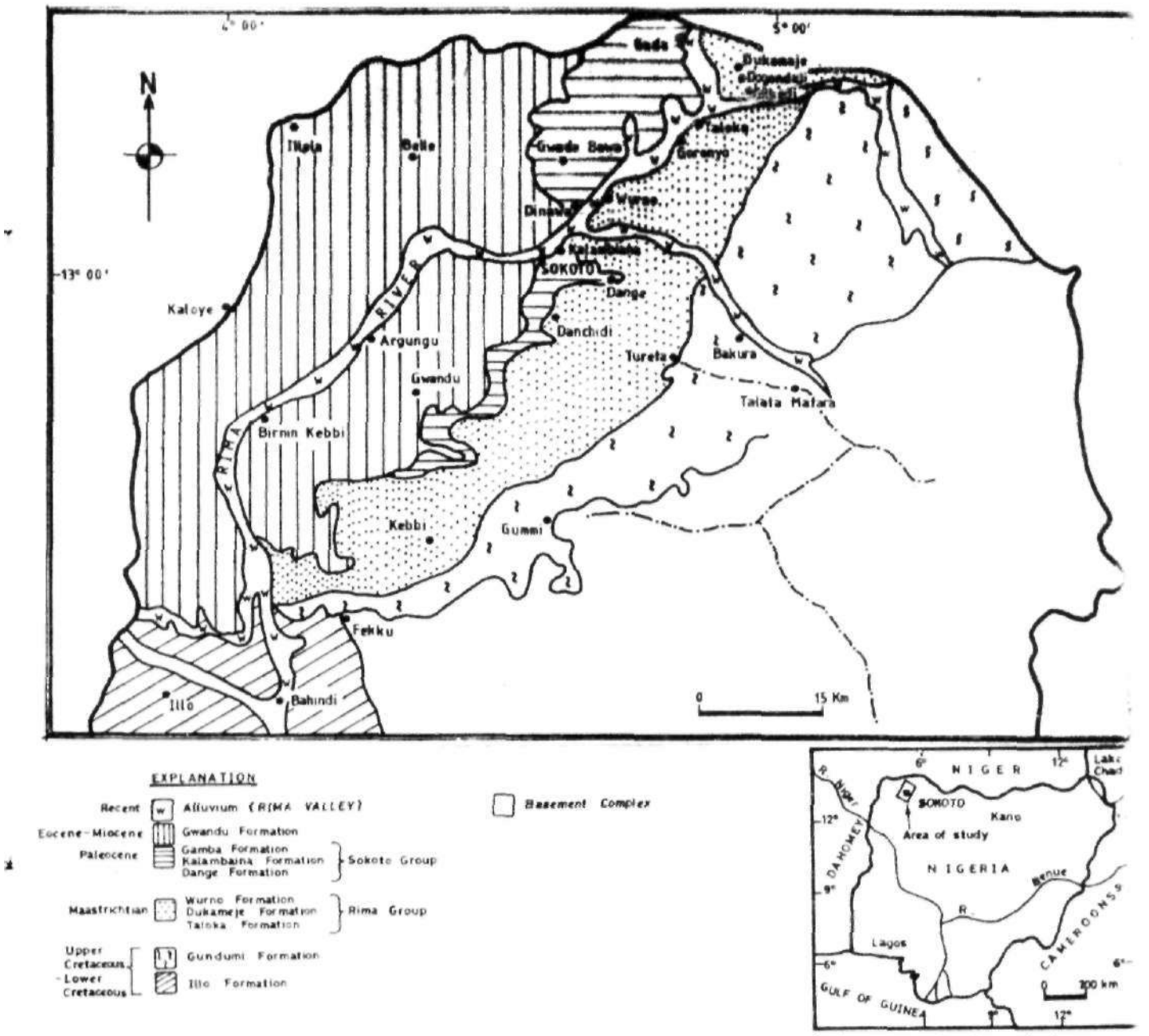


Fig. 1.1 Generalised geological map for the Iullemedden Basin and the western half of the Chad Basin.
(After Wright, Hastings, Jones and Williams, 1986)

Age	Formation	Group	Environment	Type Locality	Brief descriptions at type locality
Quaternary	Sands, laterites and lateritic sands		Continental	Widespread	
Eocene / Miocene	Gwandu Formation	Continental Terminal	Continental	Dutsen Badua ~4 km North of Birnin Kebbi	Massive clays, clayey grifts and sandstones
Upper Paleocene	Gamba Formation	Sokoto Group	Shallow Marine	Cement Quarry	Greenish-grey thinly laminated phosphatic shale
	Kalambaina Formation		Shallow Marine	Cement Quarry	White clayey limestone and shale
	Dange Formation *		Shallow Marine	Dange village ~24 km South of Sokoto	Pale, greenish-grey, thinly laminated shale, gypsiferous
Maastrichtian	Wurno Formation	Rima Group	Deltaic / Brackish water	Wurno village	Pale coloured friable, fine-grained sandstones siltstone and mudstones
	Dukamaje Formation *		Marginal Marine	Dukamaje	Shales, limestones and mudstones with vertebrate fragments and limb bones phosphatic & gypsiferous
	Taloka Formation		Deltaic / Brackish water	Taloka ~2km East of Goronyo	White, grey, brown siltstones, friable sandstones, claystones, biogenic structures
Lower most Cretaceous and Older	Illo and Gundumi Formations	Continental Intercalair	Continental lacustrine / fluviatile	Dutse Dambo ~near Bakura	False-bedded, massive, feldspathic, clayey grifts, clays, pebbly sand
Pre-cambrian	Basement complex				

* Gypsum bearing formations studied in detail.

Table 2.1 Stratigraphic framework of the Sokoto Basin. (after Kogbe, 1979)



Index map showing study area.

Fig. 1.2 Geological sketch map of the Sokoto Basin
(Inset: Index map showing the location of study area) [Kogbe, 1979].

northwest of Zaria. The Rima valley area is a rather narrow Northeast - Southwest trending belt with an average width of about 20 kilometers. The belt extends from Dukamaje, a border village with the Niger Republic in the north to Sokoto in the south with a distance of about 100 kilometers.

Places where gypsum is found include Dukamaje, Gilbedi, Dogondaji, Dange, Wurno, Rabbah, Gada, Tunga, Taloka and Shinaka.

The area covered in this project is located on the southeast banks of the Rima River centered about Latitude $13^{\circ}10'N$ and Longitude $4^{\circ}80'E$. The project area covers about 1200 square kilometers over a length of 60 kilometers and a width of about 20 kilometers.

1.3 Previous Work

Geological work in the Sokoto Basin dates back to the late 1900's, but most of the early published materials deal with the general geology and descriptions of fossil localities.

Falconer (1911) published the result of his pioneering study of the geology and geography of northern Nigeria which included the Sokoto Basin.

Jones (1948) carried out the first detailed stratigraphic study of the Sokoto Basin and his work has been published as Bulletin 18 of the Geological Survey of Nigeria. Subsequent investigations include those by Parker (1964), Asseez and Fayose (1972), Kogbe (1972, 1974, 1976, 1977, 1978, 1979,

1981), Kogbe and Adetunji (1986), Petters (1976, 1977a, 1977b, 1978a, 1978b, 1980, 1982) and Alam (1986).

Falconer (1911) first reported the occurence of gypsum in the shalley sediments of the Rima valley. Jones (1948) distinguished two horizons of gypsiferous shales belonging respectively to the Mosasaurus shales of the Upper Cretaceous and the Clay-shale Group of the Upper Paleocene. These two formations were later confirmed by Parker (1964) to host gypsum and were renamed the Dukamaje and Dange Formations respectively.

An investigation of the gypsiferous horizons of the Dukamaje Formation was carried out on a hill southwest of Taloka between 1967 and 1969 by the Geological Survey of Nigeria (Consultancy Report of the Cement Company of Northern Nigeria, Sokoto) in which seven trenches were dug and gypsum was extracted from them and analysed. The results of the investigation indicated gypsum grade of 5.5 kg/m^3 or a tonnage of just over 8000 tons. Other reported works on gypsum include Kogbe (1979), Oteze (1982) and Petters (1986).

1.4 Geography

The physiography of the Sokoto Basin consists essentially of a gently undulating plain with an average elevation of 350 meters above sea level. This monotonous topography is interrupted at intervals by mesas, isolated, steep-sided, flat-topped hills, and also by a low escarpment named the 'Dange Scarp' by earlier workers (Kogbe, 1972).

The plains are basically erosional landforms carrying a mantle of sandy drift averaging about two meters in thickness.

The character of the landscape in the basin (Ologe, 1979) is attributed to

a) The gently dipping nature of the rocks (less than 2^{o} in places) towards the northwest.

b) Lithological differences amongst the rocks.

c) The widespread occurence of lateritic ironstone forming resistant cappings to underlying less resistant rocks.

The Rima River is the main drainage system in the area. It flows southwestwards before joining the Sokoto River near Wamako. The middle part of this gypsum belt is occupied by the flood plain of River Rima which has been cut down through the gypsum bearing Dange and Dukamaje shales thereby exposing the gypsum mineralization. The flood plain is several kilometers wide and extends almost through the entire length of the gypsum belt. It is bounded by gentle slopes which merge into the surrounding plains. The flood plain is also characterized by fringing swamps, marginal lagoons, ox-bow lakes and several abandoned channels.

The Rima River receives water from many tributaries which flow in a general North - South (N-S) direction. These rivers usually originate as stream flows from the face of the flat-topped hills and are dry during the dry season.

The natural vegetation is a typical Sudan Savana, comprising grasses, shrubs and a few trees which seldom exceed eight meters in height. The dum palm (Hyphaese thebaica) the baobab (Adansonia digitala), and Acacia (Acacia albida) are the commonest tree species in the area.

1.5 Accessibility

The study area is fairly accessible through a few tarred and untarred roads. In the flood plain area, adequate access to the field is provided by a network of motor tracks and footpaths. However these motor tracks are covered by sand or fragments of lateritic ironstones making travel difficult. Because of the intricate nature of the network of tracks and paths and absence of road signs, the use of field guides and the compass is imperative. During the wet season (Mid May - Mid September) the rivers are usually flooded, thereby rendering the flood plain inaccessible except on foot.

1.6 Aims and Methods of study

The work is to carry out a detailed study of the lithological units, and other features of geological importance in the area, noting specifically the formations in which gypsum occurs, the mode of occurence of the gypsum and other physical features so as to indicate a possible origin of the gypsum

In order to define more clearly the parameters of the gypsum occurences, selected samples of gypsum were analysed,

the clay minerals of the shales were determined by
iffractometer and a study of fluid inclusions in
gypsum was undertaken.

A literature review of the project area was first done. This was followed by field visits to Dukamaje and Dange outcrop areasmainly where there are road cuts, trenches, pits etc. and the stratigraphic and lithological units were observed and noted. Particular attention was paid to the mode of occurences and the extent of the gypsum while the gypsum was sampled. Sample preparation was followed by X-Ray diffractometry of minerals of clays in shales, fluid inclusion studies in gypsum and chemical analysis of gypsum. The results obtained from the studies were interpretted.

Chapter 2.

2.0 OUTLINE GEOLOGY OF THE SOKOTO BASIN

2.1 Introduction

The Sokoto Basin (Reyment 1966a) forms the Nigerian and Southeastern part of the Iullemedden Basin (Radier, 1957; Greigert, 1961), which extends from Mali and the western boundary of Niger through northern Benin and northwestern Nigeria (Fig. 1.1).

The Iullemedden Basin is an intercontinental basin which does not seem to be directly related to Mesozoic rifting, but its origin is not well understood. Faure (1973) suggested that these intracontinental basins were caused by vertical movements resulting from horizontal relative translation of the lithosphere over ridges in the asthenosphere.

Geotectonically, the Nigerian part of the Iullemedden Basin, of which the Sokoto Basin is a part, is represented by a broad, shallow basin with small gravity faults at its margin (Alam, 1986).

The Iullemedden Basin includes Paleozoic rocks which now underly the northern part of the much larger Mesozoic basin (Fig. 1.1).

The sedimentary deposits of the Sokoto Basin were laid down unconformably on the Precambrian Basement complex of gneisses, quartzites, and granitic complexes

(Table 2.1) during the Upper Cretaceous and Tertiary. The sediments of the Sokoto Basin cover an area of about 29,900 square kilometers.

The sediments of the Sokoto Basin were deposited during three main phases of deposition: Continental pre-Maastrichtian and Tertiary phases with an intervening marine Maastrichtian to Paleocene phase. The Illo and Gundumi Formations unconformably overlie the Pre-cambrian basement and are the oldest rocks in the Sokcto Basin. They are continental in origin and form part of the "continental intercalaire" of the French-speaking neighbouring countries.

In the Sokoto Basin, the Illo Formation lies to the southwest whereas the Gundumi Formation occurs in the north-east. It is supposed that the two formations form one continous unit partially separated by a basement ridge located in the area around Fekku (Fig. 1.2).

The Illo and Gundumi Formations are lacustrine/ fluviatile deposits and are very poorly fossiliferous. Only fragments of silicified wood have been recovered from both formations (Kogbe, 1972). The Illo Formation is overlain successively from east to west by the Rima Group, the Sokoto Group and the Gwandu Formation while the Gundumi Formation is overlain by the Rima Group of Maastrichtion age (Fig. 1.2) (Kogbe 1979).

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The Maastrichtian Rima Group consists of mudstones and friable sandstones (Taloka and Wurno Formations) separated by the fossiliferous shaly Dukamaje Formation.

The Paleocene Dange and Gamba Formations (mainly shales) are separated by the calcareous Kalambaina Formation. The continental Gwandu Formation of Eocene to Miocene age overlie the Gamba Formation.

These sediments dip gently and thicken gradually toward the northwest with a maximum thickness of over 1000 meters near the frontier of the Niger Republic (Kogbe, 1979).

2.2 Stratigraphic Framework

The stratigraphic framework of the Sokoto Basin fits neatly into three major depositional episodes. The first episode was a continental depositional phase during which the deposits of the Gundumi and Illo Formations were deposited. This episode occurred during the uppermost Jurassic to lowermost Cretaceous. Northwestern Nigeria was part of an uplifted continental landmass far removed from the limits of the Southern or Tethys seas and the deposits are probably fluviatile or lacustrine in origin (Kogbe 1979).

The second episode began with the Maastrichtian transgression when the sea moved northwards from the Gulf of Guinea through the mid-Niger Basin (Kogbe 1979) and the Tethys sea moved southwards from North Africa through the Sahara. The transgression extended into the Paleocene during which the Rima Group (Maastrichtian) and the Sokoto

Group (Paleocene) were laid down.

The third and final episode began during the Eocene with the regression of the sea. Thus continental conditions resumed again and continued till the present day.

The Gwandu Formation of Eocene to Miocene age was deposited during this episode and was followed by a period of lateritization which was very widespread in Nigeria and is still in progress today.

2.2.1 Continental Intercalaire

The succession begins with poorly exposed fluviatile and lacustrine sediments, mainly sands and clays, with a locally well-developed basal conglomerate. These are called the Gundumi Formation in the north-east of the sector and the Illo Formation in the south-west; the formations are considered to be laterally equivalent. Towards the top of the Illo Formation there is a discontinous horizon, up to 8m thick, of pisolitic and nodular kaolinitic clays.

2.2.2 The Rima Group

The Maastrichtian marine transgression was the second episode in the depositional history of the Sokoto Basin. The sediments were deposited disconformably on the Gundumi and Illo Formations during this transgression period and form the Rima Group of sediments, which consists of three formations.

c) Wurno Formation

b) Dukamaje Formation

a) Taloka Formation

The Taloka Formation forms the basal unit of the Maastrichtian transgressive sequence in the Sokoto Basin and consists of pale coloured, friable siltstones, very fine-grained sandstone, and thinly interbedded mudstone and silt-shale and carbonaceous horizons. In outcrop the unit shows a cyclic pattern of alternating groups of thick and thin bedded sandstone, siltstone and mudstone beds. The unit is bioturbated in places, the most common biogenic structures being skolithos.

The siltstone and sandstone show frequent ripple drift cross laminations and parallel laminations. The mudstone shows occasional mud cracks. The sediments are finely bedded and deposited on a low lying coastal plain periodically inundated by the sea, when bioturbated tidal flat siltstones were deposited, producing cyclothem-type alternation of sediments. The type locality of the Taloka Formation is at the village of Taloka about two kilometers east of Goronyo. Two lignite seams occur within brown carbonaceous mudstones.

The marine Maastrichtian Dukamaje Formation outcrops only in a small area north-east of Sokoto and wedges out to the south and west. Where it is absent, the non-marine formations above and below it cannot be distinguished, because both are dominated by siltstones and contain no diagnostic fossils.

The shales of the Dukamaje Formation are often gypsiferous and contain fish and other vertebrate remains, especially turtles and crocodiles, including the famous Mosasaurus nigeriensis that was recovered from the formation (Kogbe, 1979). The formation is notable for thin but persistent bone bed at the base, in which teeth of fish and sharks, fragments of turtle and primitive crocodiles, and coprolites are concentrated. It is a type of lag deposit, probably formed in shallow waters where currents and waves winnowed away the finer sediment particles.

The Dukamaje Formation was deposited during the Maastrichtian marine incursion. This is confirmed by the presence of the Upper Maastrichtian ammonite (Libycoceras sp.) at the type locality in Dukamaje (Jones, 1948).

At Taloka village, the Dukamaje Formation overlies the Taloka Formation with its characteristic bone bed lying directly at the base of the formation and thins out westwards towards Goronyo and disappears about 1 km from Taloka.

The Wurno Formation appears to represent similar environmental conditions with the Taloka Formation, with extreme shallow water and an unconformity at the top. The Wurno Formation is well exposed at its type section at Wurno, but elsewhere exposures are quite rare. The formation is about 23 meters thick at Wurno and consists of whitish-grey coloured friable fine grained sandstones, siltstones and mudstone intercalations. The sediments are

very loosely consolidated and susceptible to weathering. Where both the Taloka and Wurno are preserved in outcrop, they are usually protected by the more resistant layers of the overlying formations.

2.2.3 The Sokoto Group

The Sokoto Group of sediments were also deposited during the second episode of Maastrichtian to Upper Paleocene transgression. The group is made up of three formations of Upper Paleocene age (Reyment, 1965, Kogbe, 1979). These are, beginning with the oldest, the Dange Formation, the Kalambaina Formation and the Gamba Formation. Sediments of the Sokoto Group are entirely marine in origin and are richly fossiliferous. The formations within the Sokoto Group increase in thickness northwestwards and attain maximum thickness towards the centre of the Iullemedden Basin in the Niger Republic.

The Dange Formation consists of slightly indurated bluish-grey shale interbedded with thin layers of yellowish - brown limestone. The shale is bluish-grey and thinly laminated and gypsiferous and plastic when wet. It becomes light grey, pale yellow and marly towards the top where it is overlain by the Kalambaina Formation. It is characterized by abundant phosphatic nodules and macrofossils, most importantly some echinoids, gastropods and lamellibranchs. Ostracods and Foraminifera have been described by Reyment (1960, 1963, 1965, 1966), Kogbe (1976, 1979) and Peters (1979c, 1982).

The Kalambaina Formation consists of a marine, white, clayey limestone and shale. The formation is rich in invertebrate fossils, mainly echinoids, corals, nautiloids, lamellibranchs and gastropods. Foraminifera and ostracods have also been described from the formation (Reyment, 1965; Kogbe et al., 1972; Petters, 1978; Obaje 1987). The formation contains a rich, but low-diversity, shallow-water late Paleocene benthonic foraminiferal assemblage that suggests water depths of probably less than ten meters (Petters, 1978). The benthonic foraminiferal assemblage comprises Operculinoides bermudezi, Thalmannita madrugaensis, Boldia Cubensis, Pararotalia tuberculifera, P. perclara, Rosalina koeneni, Elphidiella africana, rotaliids, cibicids and nonionids.

The limestone contains shallow water invertebrate fossils (e.g. Lucina Pharaoinis), as well as considerable amounts of detrital mud suggesting that sedimentation took place in an inner shelf environment close to shore.

The Kalambaina Formation is very argillaceous at the top and changes gradually into the Gamba Paper shales.

The Gamba Formation consists of thinly laminated Phosphatic shale with a greenish-grey colour and bears striking similarity to the Dange Shale.

An impoverished arenaceous foraminiferal assemblage comprising Ammobaculites, Haplophragmoides and Trochammina

occur in the Gamba Shale. The Gamba Formation marks the final withdrawal of the sea from the Sahara.

The Dange and Gamba Formations represent marginal marine environments, while the Kalambaina marl and limestone originated in a warm shallow epeiric sea (Petters, 1979).

2.2.4 The Gwandu Formation

The third episode in the depositional history of the Sokoto Basin is the deposition of the post-Paleocene continental sediments of the Gwandu Formation. The Gwandu Formation outcrops most extensively in the Sokoto Basin, covering an extensive area and overlying the Paleocene marine shales of the Gamba Formation.

The Gwandu Formation is characterized by some prominent ranges and groups of flat-topped steepsided hills and usually capped by lateritic ironstones. The variable continental clays, fine-grained sandstones and coarse, cross-bedded red sandstones are known as the Gwandu Formation, or the "continental terminal" in French-speaking West African countries (Table 2.1). It is of fluviatile, lacustrine origin (Jones, 1948).

The Gwandu Formation overlies the Gamba Formation unconformably in the northern and central parts of the Sokoto Basin. To the south, the formation overlaps the Paleocene Formations and rests directly on the Pre-Maastrichtian Rima Group. Palynologic determination by Shell B.P. indicates a possible Eocene age (Reyment, 1965) for the Gwandu Formation and this has been confirmed by

subsequent palynologic determinations by Kogbe and Sowunmi (1975) in which an Eocene - Miocene age was assigned.

Chapter 3

3.0 THE DUKAMAJE AND DANGE FORMATIONS OF THE PROJECT AREA

3.1 Introduction

The second stage in the depositional history of the Sokoto Basin began during the Maastrichtian, when the Rima Group was deposited disconformably on Pre-Maastrichtian continental beds. The transgression continued into the Paleocene with the deposition of the Sokoto Group of sediments (Table 2.1).

The deposits of the Upper Paleocene marine cycle comprise the Dange Shale, the Kalambaina Marl, and the Gamba Shale and constitute a single depositional package. This cycle represents a marine transgression more extensive than that of the earlier Maastrichtian transgression (Petters, 1977a).

Lithologically and faunally, the Dange - Kalambaina - Gamba sequence (Sokoto Group of Geological Survey of Nigeria, 1964) replicates the lower sediments of the Dukamaje Formation, suggesting that essentially the same sedimentary environments reoccupied the area during the deposition of the sediments.

The greater thickness of the Sokoto Group, 22 meters in outcrop, (Petters, 1979a) compared to about 10 meters for the Dukamaje Formation, reflects the longer duration of the Paleocene transgression, with greater sediment influx during that time.

The Maastrichtian marine and Paleocene deposits in which appreciable gypsum has been observed are the Dukamaje Formation (Rima Group) and the Dange Formation (Sokoto Group).

3.2 Lithology

3.2.1 Dukamaje Formation

This formation was originally named the "Mossasaurus Shales" by Jones (1948) after the new species Mossasaurus nigeriensis. The Dukamaje Formation consists predominantly of bluish-grey shales with some limestones and mudstones. The shales of the Dukamaje Formation are often gypsiferous and contain fish and other vertebrate remains, especially turtles and crocodiles.

Bone beds, most of which are lateritized or encrusted with gypsum, occur at the base of the Dukamaje Formation. It is an excellent marker horizon in the field. The bone bed consists of teeth of fish and sharks, fragments of turtle and primitive crocodiles and coprolites.

The gypsum is intercalated within the bluish-grey shales of the Dukamaje Formation. Gypsum occurs in the Dukamaje Formation as discontinous thin beds, septarian concretions, selenite crystals and minute crystals.

3.2.2 Dange Formation

The Dange Formation consists of slightly indurated bluish-grey shale, interbedded with thin layers of yellowish-brown limestone. The shales include bands of fibrous gypsum with numerous irregular phosphatic nodules. The nodules are

The Maastrichtian ammonite libycoceras sp. occurs at the top of the marl band. The ammonite was found at Dukamaje and Gilbedi (Kogbe, 1980). The foraminiferal assemblage of the marl includes middle and late Maastrichtian species such as Guembelitria cretacea and Orbignya inflata and species of Nonion, Nonionella, and Gavelinella.

The upper unit of the formation has yielded only an impoverished arenaceous foraminiferal microfauna dominated by Ammodiscus.

The shales of the Dukamaje, Dange and Gamba Formations contain a rich but low diversity arenaceous foraminiferal assemblage that is completely devoid of calcareous forms (Petter, 1978a). In contrast, the marls of the middle Dukamaje and lower Dange Formations contain both arenaceous and calcareous benthonic species with a preponderance of calcereous forms (Petters, 1978a).

3.3.2 Dange Formation

The vertebrate fossils observed in the Dange include remains of sharks, fish bones, skull fragments, limb bones, scutes of crocodiles and turtle carapaces. White (1934) and Halstead (1973, 1975) described vertebrate fossils from the Dange Formation. These include teeth of Odontaspis and remains of nurse sharks, there are fish bones, skull fragments, vertebrae, limb bones, scutes of crocodiles and turtle carapaces.

The foraminiferal microfauna of the Dange Shale is very sporadic and consists of simple arenaceous species of Ammobaculites, Miliammina, Trochammina, Haplophragmoides and Ammodiscus while the marl band contains cibicides simplex.

The bottom shale of the Dange Formation contains vertebrate remains and the foraminiferal assemblage is entirely arenaceous and rather impoverished.

Kogbe et al. (1972) reported Ostracod fauna from the subsurface of the Dange Formation.

The gypsiferous shales in the Sokoto Basin contain abundant fossil vertebrates and arenaceous foraminifera. Fossil crocodiles, turtles, and fish occur in profusion in the more gypsiferous parts of the Dukamaje Formation at Taloka, Dukamaje and Gilbedi where a prominent bonebed, 10 cm. thick occurs (Halstead 1979, Petters, 1979, Walker, 1979).

Similarly, the most prolific vertebrate fossil localities in the Dange Formation, at Gada and Wurno, are the most gypsiferous parts of the formation.

There is, therefore, some relationship between the distribution of gypsum and vertebrate fossils in Sokoto Basin.

Black, grey or green colouration signifies reducing conditions with at least partial preservation of organic material (Patnode, 1941). The greyish nature of the gypsiferous shales of the Dukamaje and Dange Formations

and abundant vertebrate fossils associated could imply that gypsum was emplaced under reducing conditions which supports the preservation of the fossils.

Gypsum shows an intimate association with organic matter.

It is noteworthy that, unlike the Dukamaje Formation with a distinct bone bed at the base, the Dange Formation has no bone bed. Although the Dange Formation is richly fossiferous, the vertebrate fossils are not concentrated to a particular horizon but occur randomly throughout the formation.

3.4 Stratigraphy

3.4.1 Dukamaje Formation

The Dukamaje Formation constitutes the Maastrichtian marine unit and is about ten meters thick in outcrop (Petters, 1979). It occurs between two coastal plain sandstones, the Taloka and Wurno Formations. The Dukamaje Formation wedges out northeast of Sokoto town (Fig. 3.1).

Peters (1979) divided the Dukamaje Formation into a lower gypsiferous, fissile, grey shale of three to four meters in thickness, a middle fossiliferous marl band of about four meters thick, interbedded with thin carbonaceous and gypsiferous fissile, grey shale of two to three meters in thickness, sparsely gypsiferous and poorly preserved in outcrop.

At the base of the Dukamaje Formation is a thin but persistent bone bed which is an excellent marker horizon

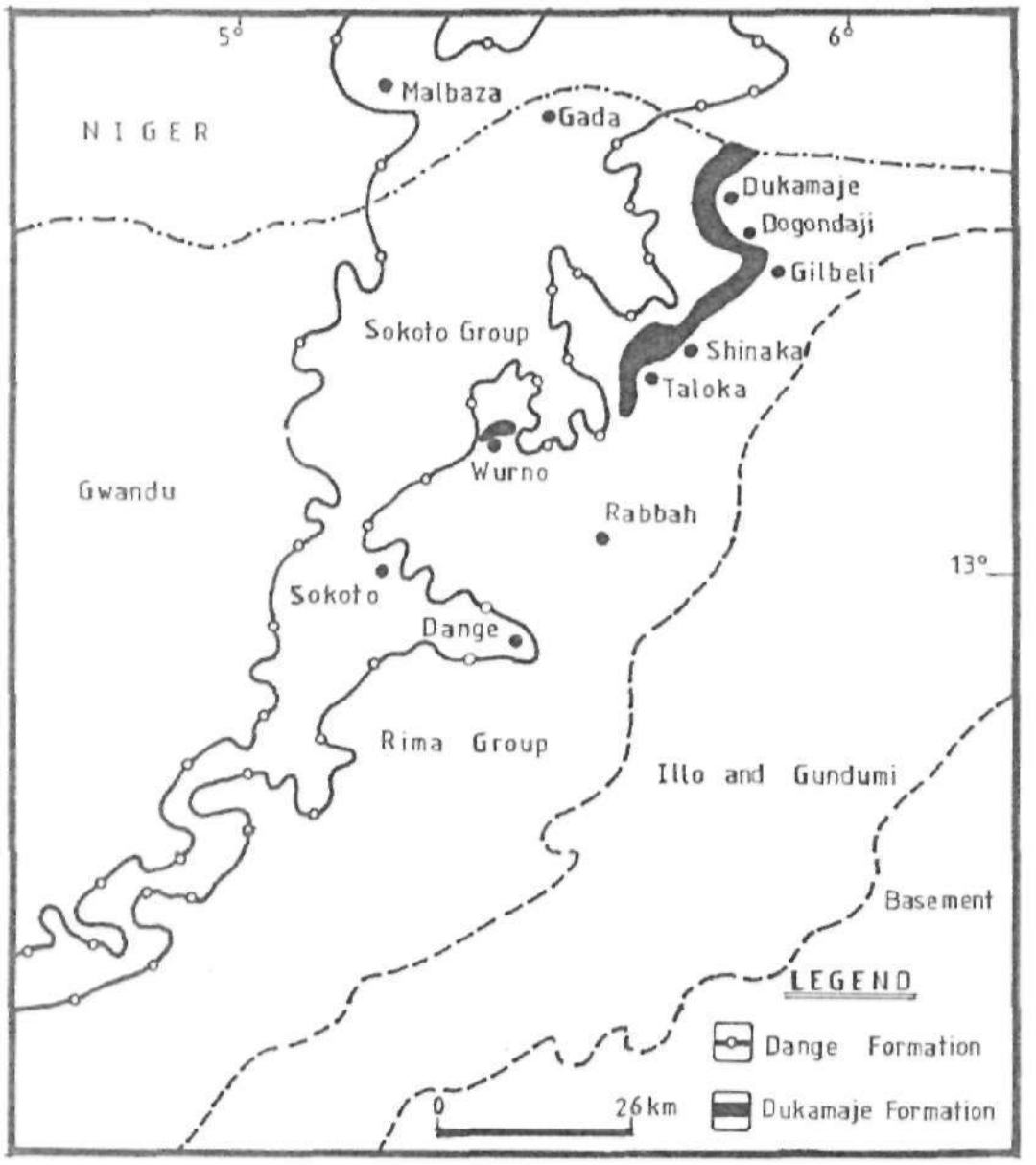


Fig. 3.1 Geological sketch map showing location of the area in which Dange and Dukamaje Formations outcrop in the Sokoto Basin.

in the field.

On the hill south-west of the village of Dukamaje, the base of the Dukamaje type section is exposed (contrary to the assertion of Halstead, 1979) and the sharp contact with the underlying Taloka Formation was observed in several sections at the foot of the hill south-west of the village. The bone bed is evident (Fig. 3.2). The formation varies from about 12 meters at the type area to less than 0.5 meters 50 kilometers further south (Kogbe, 1980).

An excellent exposure of the Dukamaje Formation was observed at the village of Dogondaji about three kilometers south of Dukamaje (Fig. 3.4). Here, the base of the formation does not outcrop.

The thickness of the Dukamaje Formation at Taloka is about six meters (Fig. 3.5). Here, the lower gypsiferous shale of the Dukamaje Formation flanked by lateritic layer was observed. It thins out rapidly westwards towards Goronyo and disappears about one kilometer from Taloka (Fig. 3.1).

On the hills south of Gilbedi, an excellent section of the Dukamaje Formation was observed (Fig. 3.3). At Gilbedi, two horizons of bone beds at the base of the Dukamaje Formation were observed. Kogbe (1974) earlier reported the presence of two horizons of bone beds in this area .

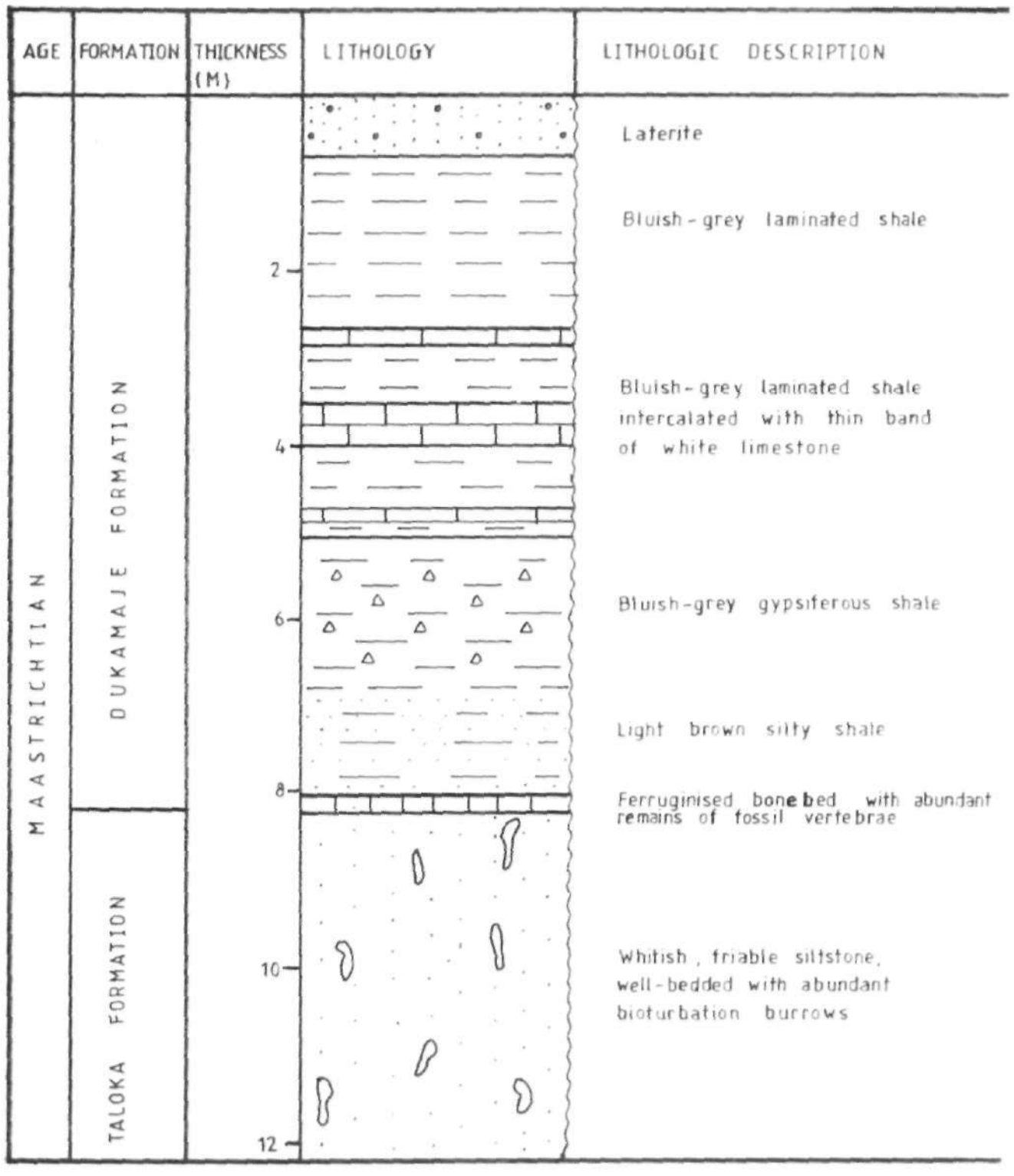


Fig. 3.2 Dukamaje type section, south-west of Dukamaje village.

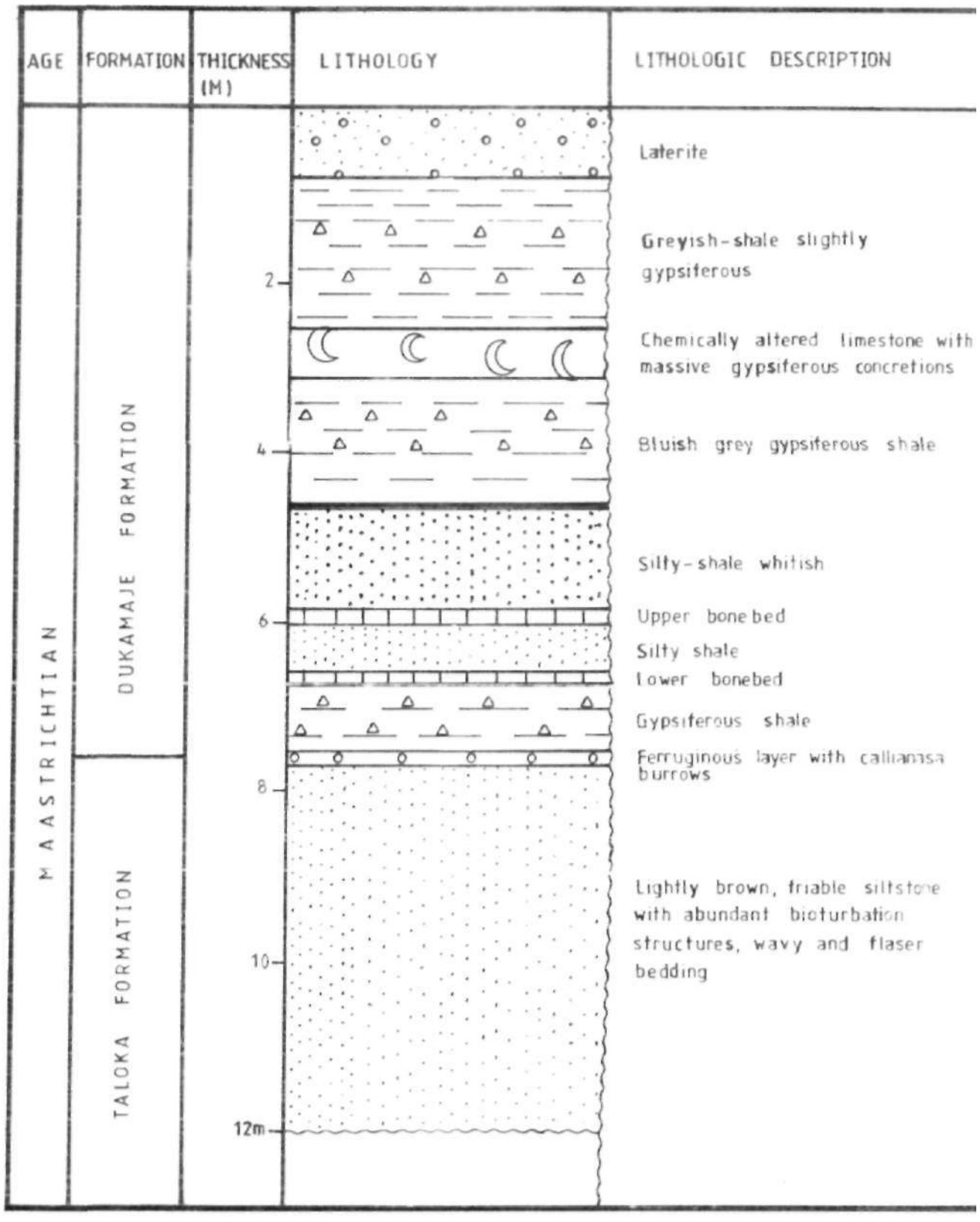


Fig. 3.3 Dukamaje type section at Gilbedi village.

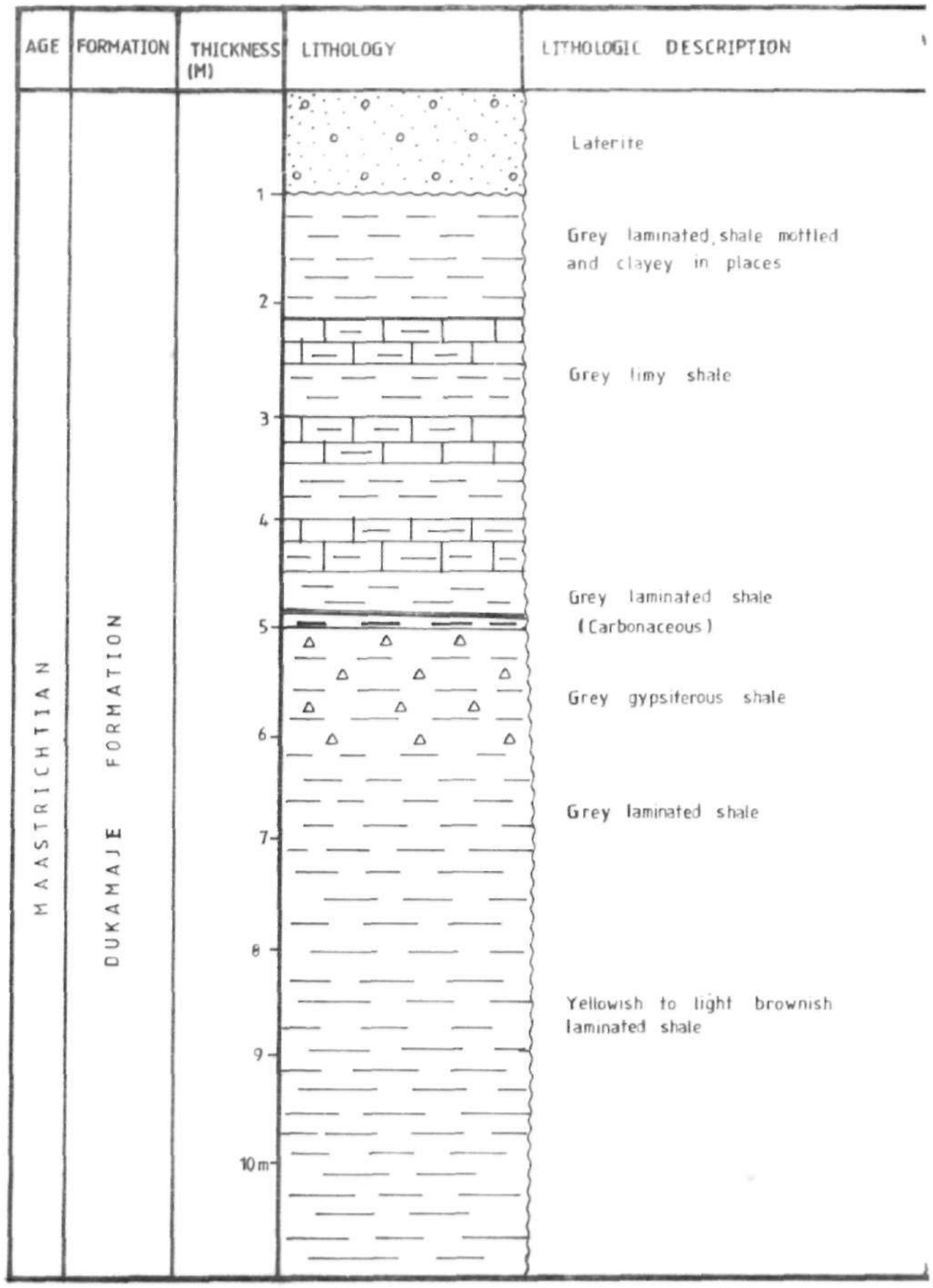


Fig.3.4 Dukamaje type section at the village of Dogondaji , 3km South of Dukamaje.

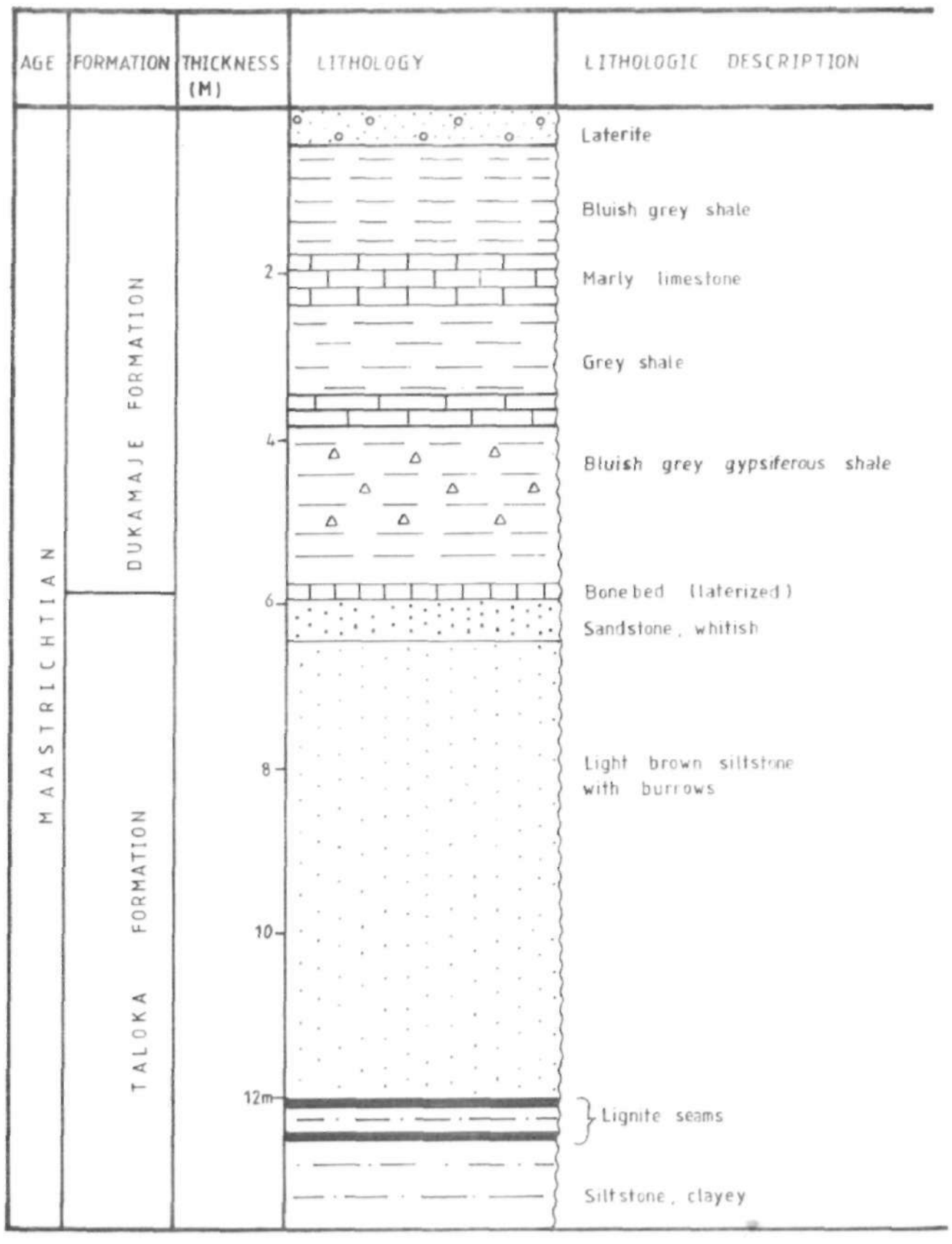


Fig. 3.5 Section of Dukamaje Formation at Taloka village.

3.4.2 Dange Formation

The Dange Formation occupies the base of the Sokoto Group of sediments of Upper Paleocene age. The group is made up of three formations:

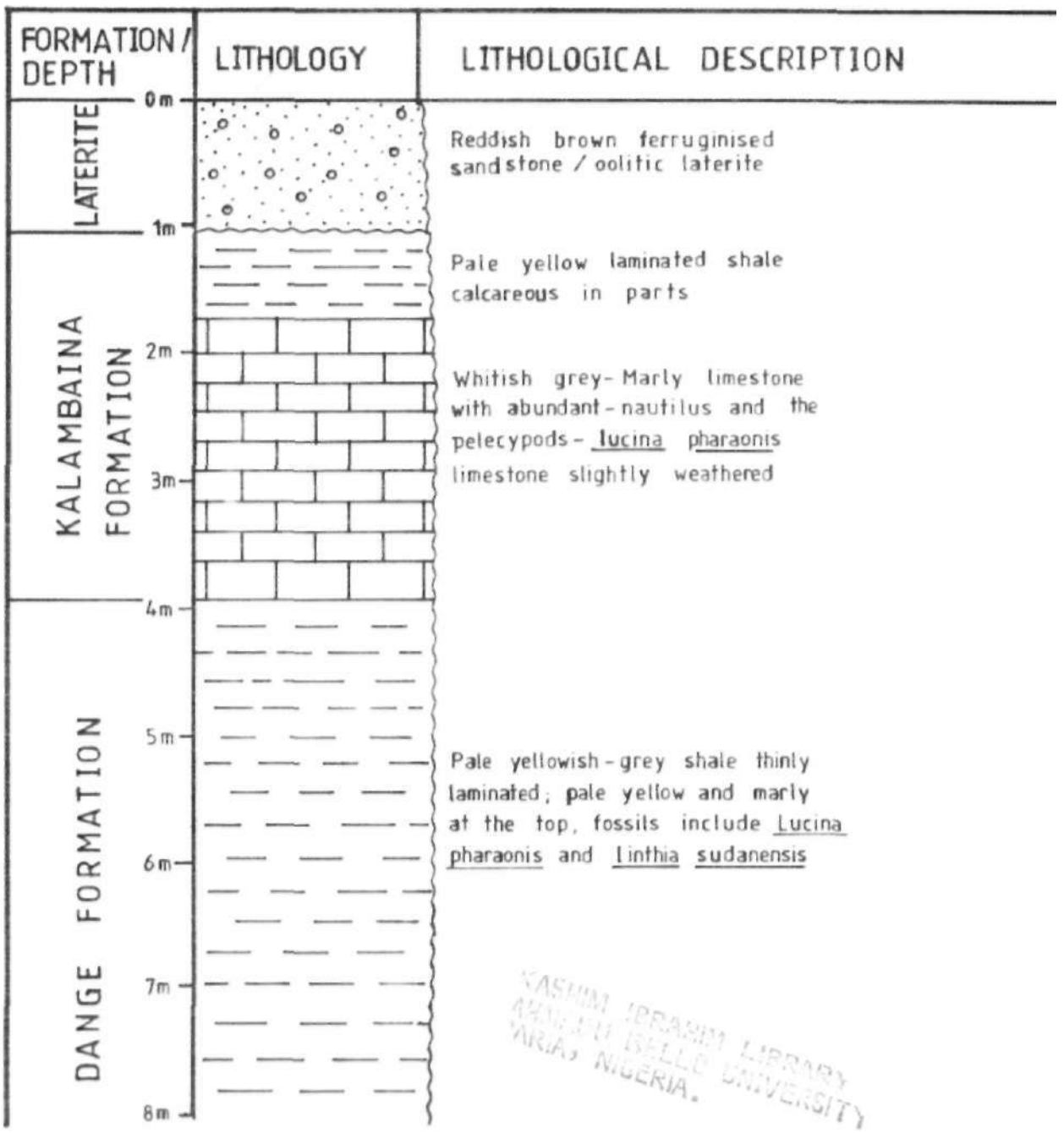
Dange Formation (oldest)	Sokoto Group. Upper Paleocene.
Kalambaina Formation	
Gamba Formation (youngest)	

In surface outcrops, the maximum thickness of the formation is about 22 meters near Sokoto but in subsurface wells it attains a thickness of over 45 meters (Kogbe, 1981). Generally, outcrops of the Dange Formation are restricted to the slopes of the Dange Scarp.

There is an erosional plain represented by a thin bed of conglomerate at the base of the Dange Formation. This erosional horizon now constitutes the Cretaceous - Tertiary boundary in the Sokoto Basin.

The ostracod fauna reported by Kogbe et al. (1972) from the subsurface supports a late Paleocene age for the Dange Formation and further indicates that there is no real age difference between the Dange, the Kalambaina and the Gamba Formations.

The type localities for the type section of the Dange Formation are at Dange, Wurno and Gada. The type section of the Dange Formation at Dange village, about 28 kilometers south of Sokoto, is along a road-cut on the Sokoto - Gusau road (Fig. 3.6). The section described by Kogbe (1972) has suffered considerable erosion but still shows the essential



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Fig. 3.6 Road-cut section at Dange, (Sokoto-Gusau road).

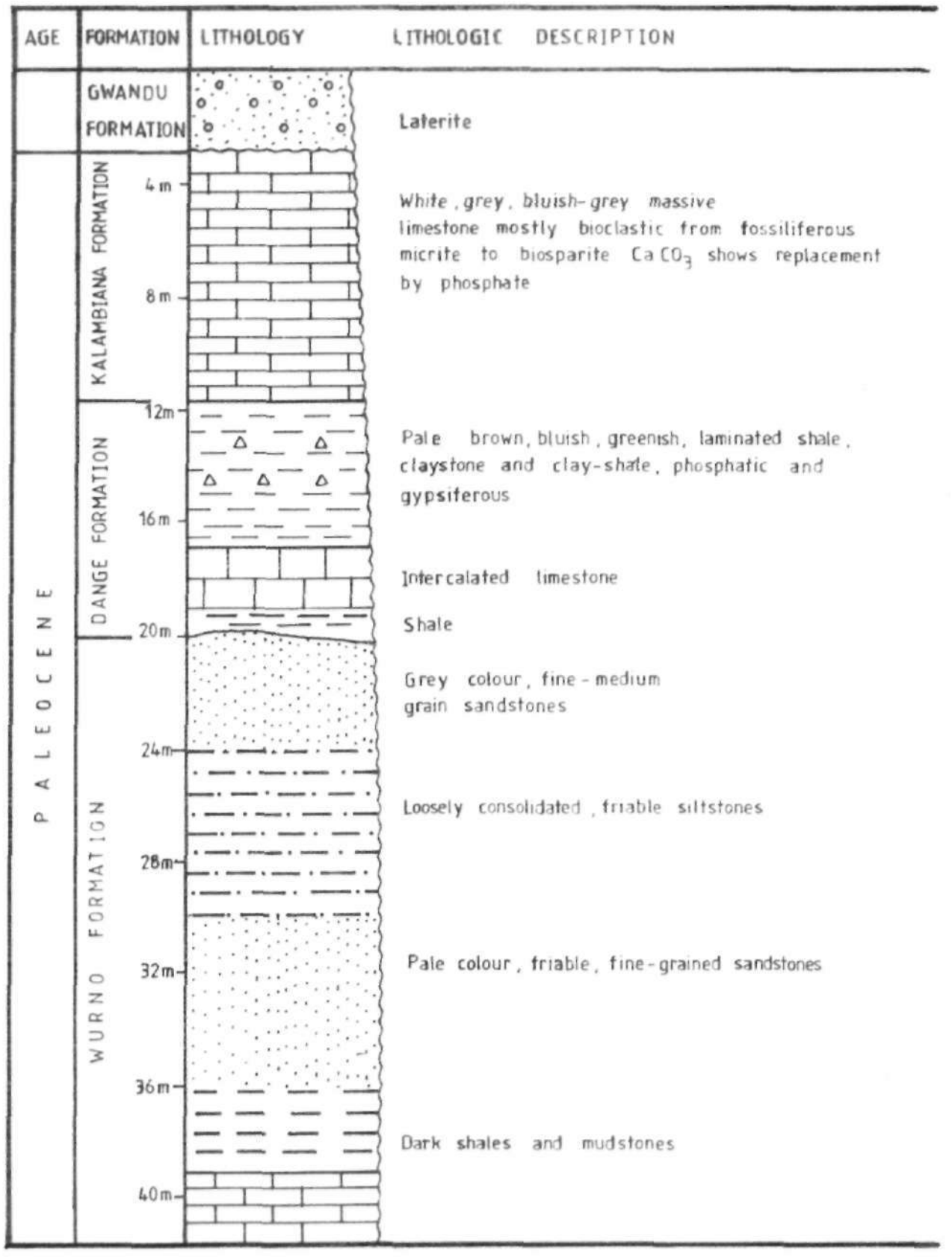


Fig. 3.7 Dange type section at the Wurno hill near the village of Wurno Lat = 5° 26', Long.=13° 17' .

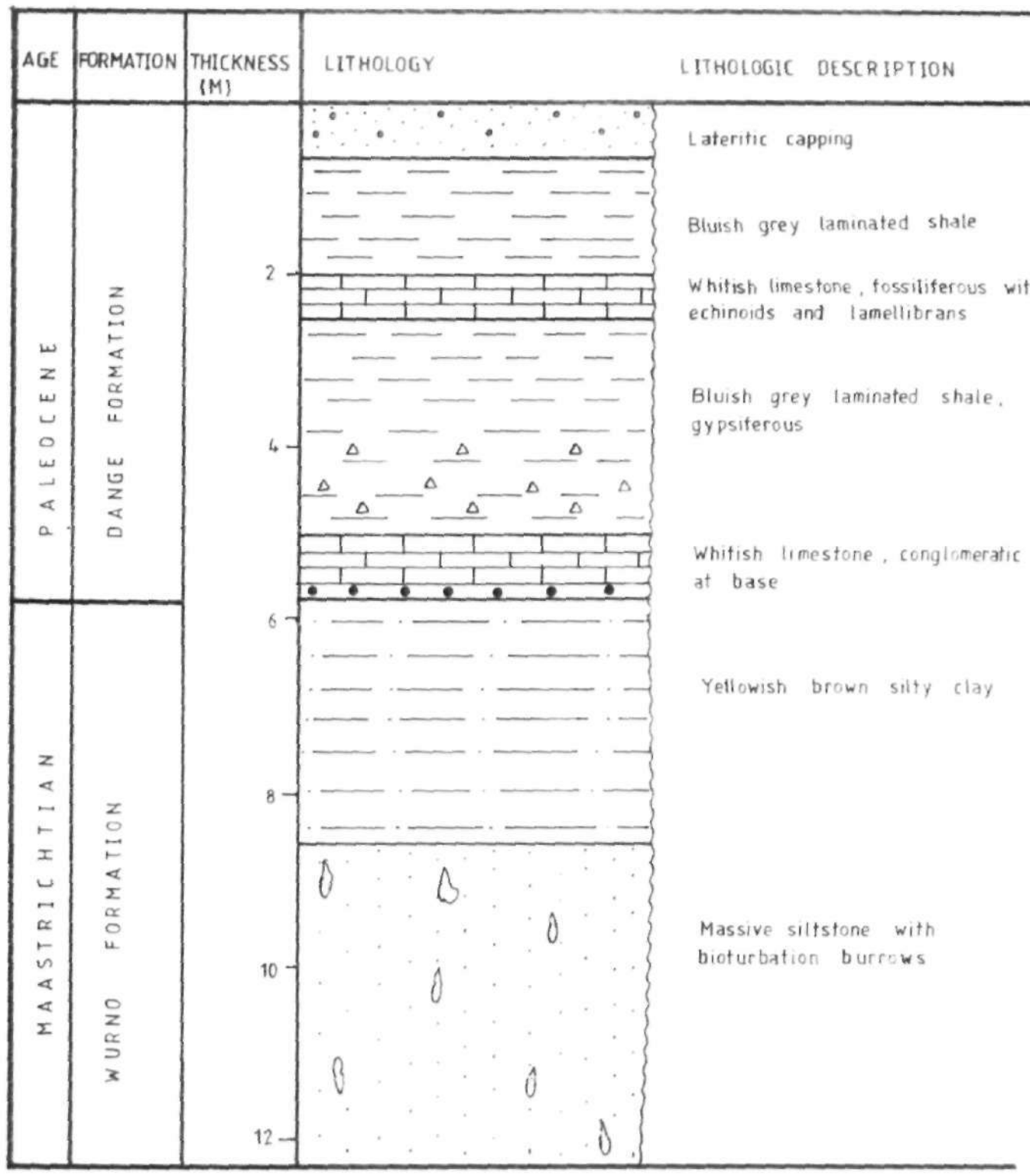


Fig. 3.8 Section of Dange Formation at Gada (Lat. 13° 44'N, Long. 5° 40'E).

features of the formation. The base of the formation does not outcrop at Dange but at Wurno (Fig. 3.7) and at Gada (Fig. 3.8).

Limestone beds about 0.5 - 1.0 m in thickness overlie the erosional horizon at the top of the Wurno Formation and are unfossiliferous. They are overlain by grey, gypsiferous shales rich in macrofossils and fossil vertebrates.

Geological survey borehole No. 3512 drilled at Dange village, reveals the thickness of the formation to be over 23 meters. South of Birnin Kebbi , the Dange Formation is about 15 meters thick in Geological survey borehole No. 2483. The Dange Formation attains a thickness of 9 meters at Kaloye, 50 meters at Balle, 4 meters at Argungu, and 21 meters at Sokoto.

3.5 Phosphate Association

Sheldon (1983) has shown that the 40 - 95my period has been a time of enhanced phosphate deposition and accumulation worldwide.

Phosphate occurences in the Sokoto Basin sediments have been reported by Jones (1948), Kogbe (1981) and others. Typically, such phosphate occurences comprise phosphatic nodules, pellets or bone beds which occur in shaly units of the Rima and Sokoto Groups (Table 2.1).

In the Dange Formation, individual nodules and pellets contain over 30% P_2O_5 (Jones, 1948) but they occur

sporadically and widely separated. More recent work has shown that they are restricted to bands within the shales (Odibo, Personal Communication)(Fig. 3.9). The phosphate nodules range in colour from dirty white to grey and brown with the later containing higher phosphate content. The phosphate nodules are present throughout the Dange Formation, and shows a stratigraphic relationship with gypsum. In several areas of the Sokoto Basin, weathering and erosion of the Dange Formation has led to the surface accumulations of the phosphatic nodules in stream beds and valley floor.

The Kalambaina Formation contains a distinct horizon characterized by abundant phosphatic pellets and small coprolites from 1 mm to 1 cm in size. This phosphatic unit is sufficiently persistent to be termed a marker horizon with a thickness of over three meters. The phosphate nodules in the Kalambaina Formation and near the top of the Dange Formation, may have formed directly from sea-water at the sediment-water interface. This is supported by the presence of unaltered planktonic fossils with phosphate nodules and occasional growth of small phosphate crystals on bedding planes (Alam, 1986).

The Dukamaje Formation, which contains a rich bone bed as its basal unit, is also reported to include phosphatic coprolites (Jones, 1948) and though no essay data is reported, it has been considered to be phosphate rich. Thicknesses

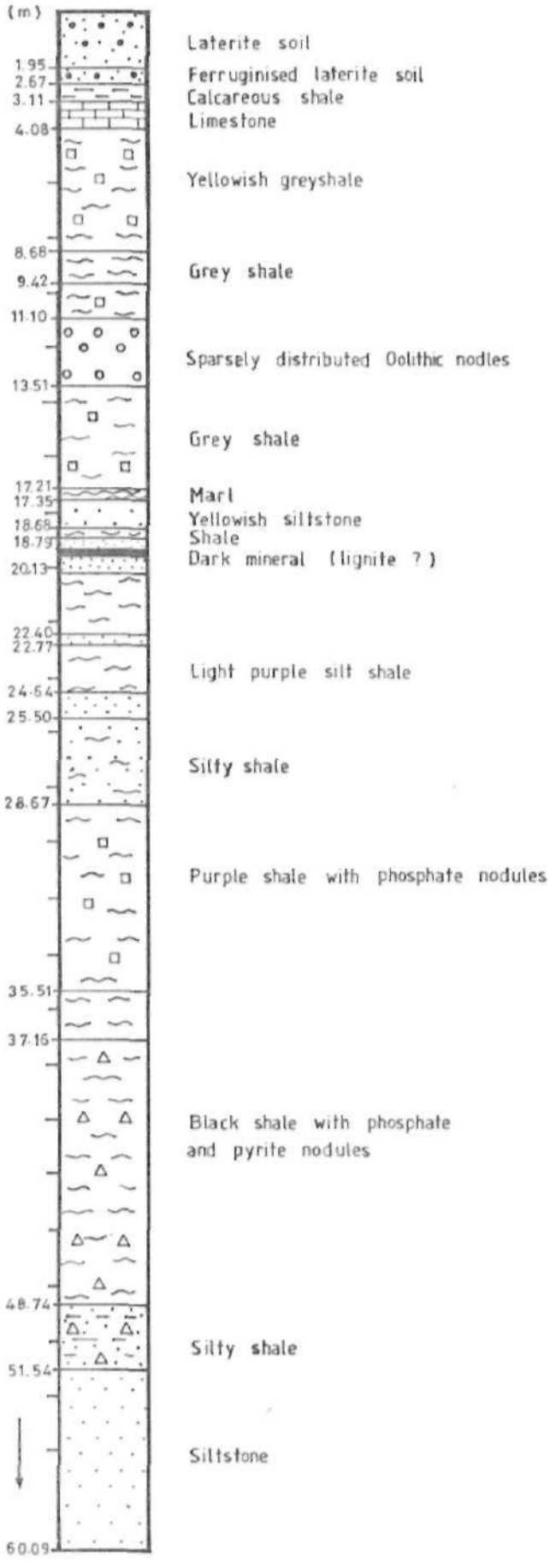


Fig. 3.9 Stratigraphic section of borehole intersecting phosphate mineralization, Dange Sokoto State.

(Woakes and Olade, 1981)

of up to 0.6 meters have been recorded (Beltaro, 1970).

Recent exploration core drilling in the Sokoto Basin by the Geological Survey of Nigeria, in cooperation with the Nigerian Mining Corporation, has been reported to have located commercial grade phosphate suitable for the Kaduna superphosphate plant and in large quantities (Nehikhare, 1986). This work has been based on a comparative paleoecological study of the basin to select the most favourable localities but details of the work have not been released.

3.6 Sedimentology

3.6.1 Dukamaje Formation

The Dukamaje Formation represents a transgression of the sea over the area. Initially, the sea was shallow and probably brackish or even almost fresh water, due to the influx of fresh water from the adjacent land areas, and was inhabited by elasmobranchs and other fish, turtles, mososaurs and crocodiles. The fauna contains species likely to have been tolerant of low salinity and it is unlikely that the area was a site of evaporite deposition.

The fauna of the Dukamaje Formation confirms more fully marine conditions, as shown by foraminifera and echinoids. However, it is likely the salinity was still rather low, as indicated by the rather restricted fauna, the dominance of hardy types among the foraminifera.

The presence of Haplophragmoides, a robust arenaceous foraminifera, could suggest quite turbulent nearshore waters. Stainforth (1952) suggested that where assemblages of foraminifera are dominated by large, robust species, they may be controlled by turbulence and gave an example of a Haplophragmoides assemblage among others occuring in the North Alaskan Cretaceous and elsewhere.

Marginal marine conditions prevailed in the Sokoto Basin during Maastrichtian times, when the Tethys sea extended to the south-east of the interior of western Africa. This resulted in the deposition of (gypsum - bearing shales) the Dukamaje Formation, well known for its reptilian and fish remains. This formation contains abundant arenaceous foraminifera but an extremely limited calcareous microfauna (Kogbe, 1980). Pelagic faraminifers are absent in these sediments, this attests to the shallow and marginal conditions under which sedimentation took place.

The bone beds could be formed by catastrophic changes in the environment, as described by Ager (1963), but it seems more likely that this deposit represents winnowed deposits of animal relics that were living in the sea and associated with the transgression of the sea over the area (Kogbe, 1975, 1981).

3.6.2 Dange Formation

The Dange Formation represents the transgression of the sea over the area during the upper paleocene, leading to the deposition of the shales of the Dange Formation, disconformably on the Maastrichtian Wurno.

The bottom shale of the Dange Formation contains vertebrate remains and the foraminiferal assemblage is actively arenaceous and rather impoverished. The ostracod fauna reported by Kogbe et al. (1972) from the subsurface supports a late paleocene age for the Dange Formation and further indicates that there is no real age difference between the Dange, the Kalambaina and the Gamba Formations. The arenaceous microfauna would support alithofacies interpretation that the Dange Formation is the transitional facies equivalent of the open marine calcareous Kalambaina Formation.

An erosional plain represented by a thin bed of conglomerate occur at the base of the Dange Formation. The presence of coprolites, gypsum as well as the remains of molluscs (lamellibranchs) in the conglomeratic bed (Kogbe, 1980), suggests that the base of the Dange Formation must have been affected by erosion. This erosional horizon now constitutes the Cretaceous - Tertiary boundary in the Sokoto Basin.

Chapter 4

4.0 GYPSUM IN THE PROJECT AREA

4.1 Introduction

Gypsum ($CaSO_4.2H_2O$) is a common evaporite mineral like anhydrite ($CaSO_4$) and halite (NaCL). It is usually the first evaporite to be precipitated by the partial or complete evaporation of marine brines.

Gypsum forms under the following depositional settings.
(a) Evaporite type deposit which is subdivided into the brine-filled basin and the sabkha type deposits.
The older, well established concept of deposit formation idealizes a brine-filled basin having restricted circulation (Branson, 1915; King, 1971) which permits limited replenishment of the brine as it evaporates. The resulting concentration causes precipitation of the contained salts in the inverse order of their solubility (eg. Calcium sulphate) and a substantial thickness is visualized as being derived from this type of regime. An example of this type is the A subzone of the Mississipian Windsor Series of Southeastern Canada (Appleyard, 1975). That this is the mechanism of formation of some economic calcium sulphate deposits is unquestioned. Another common depositional setting for the evaporite type of calcium sulphate minerals is the sabkha, a fairly level, salt-encrusted surface that only occasionally is inundated by the sea.

(b) A second mode of gypsum formation is the diagenetic mode of origin. Diagenetic forms of gypsum were observed in the Mediterranean (Robert and Chamley, 1974) and off the slope of southwest Africa (Siesser and Rogers, 1976). Gypsum forms by accompanying the dolomitization of limestone at the seafloor, and also by the formation of calcium sulphate by the action of sulphuric acid, generated by the decomposition of pyrite , on the calcium carbonate of shells, etc., in clays. The good crystals of selenite found in many clay formations, such as the London Clay and Oxford Clay (Read, 1970) are formed by this process.

4.2 Distribution of Gypsum in the Project Area

As previously described, gypsum occurs mainly in the shales of both the Dukamaje and Dange Formations. The mudstones and siltstones of the Wurno Formation contain small lumps of gypsum (Kogbe, 1979; Petters, 1986). A section at the Sokoto cement factory quarry site (Kogbe, 1979, Fig. 4.1) indicates the presence of gypsum at the base of the Gamba Formation. However, the occurence of gypsum in this formation has not been reported elsewhere.

Although gypsum is found in four formations within the Sokoto Basin, namely, the Dukamaje, Wurno, Dange and Gamba Formations, the quantity of gypsum in the Wurno and Gamba Formations is too small to be of any economic significance. Consequently, only the gypsum in the Dukamaje and Dange Formations are considered in evaluating gypsum mineralization in the Sokoto Basin.

x = 5° 11′ y = 13° 03′

AGE	FORMATION	(M)	SECTION	LITHOLOGIC DESCRIPTION	FAUNA
	LATERITE	5		Ferruginised sandstone and Oolitic ironstone or laterite	
PALEOCENE	GAMBA FORMATION	10		Ironstone concretion in shales Grey laminated shale Phosphatic pellets & small coproliths Laminated shale with gypsum	Foraminifera Ostracods Lamellibranch
	KALAMBIANA FORMATION	15		White limestone with crevices in-filled with none-calcareous clay Shally limestone white to whitish yellow White yellowish limestone slightly shally Shally level within limestone bed carbonaceous Limestone with shally inclusion 3mm thick Shales laminated within limestone	Foraminifera Ostracods Lamellibranch Gastropods Crinoids Corals Nautilus Echinoderms
		20			

Fig. 4.1 Kalambaina and Gamba Formations section at the cement quarry near Sokoto, (after Kogbe, 1979)

4.3 Modes of Occurence of Gypsum in the Dukamaje and Dange Formations

Gypsum is quite common in the exposures of the Dukamaje and Dange Formations, where it occurs as fibrous crystals in septarian concretions, thin discontinous beds and veins, and minute crystals; it also occurs as selenite crystals. The gypsum appears intercalated within the shale host rock in both the Dukamaje and Dange Formations.

4.3.1 Gypsum Beds and Veins

Thin discontinous beds of gypsum are intercalated within the fissile shales of the Dukamaje Formation at Dukamaje, Dogondaji, Gilbedi and Taloka. At Taloka, the Dukamaje type section (Fig. 3.5), a thin discontinous bed of gypsum being cut across by a thin vein of gypsum was observed. In the lower parts of the Dange Formation at Wurno and Gada, thin discontinous beds of gypsum are intercalated within the fissile shales. At Wurno, the Dange type section (Fig. 3.7) shows a thin discontinous bed of gypsum being cut across by a thin vein of gypsum. The gypsum beds are up to 4 cm. thick and 20 cm. long (Plate 1.) They easily weathered out of the shales and disintegrated into white gypsum flakes which are carried down the barren hillsides and gullies where they reflect flashes of sunlight and are easily seen.

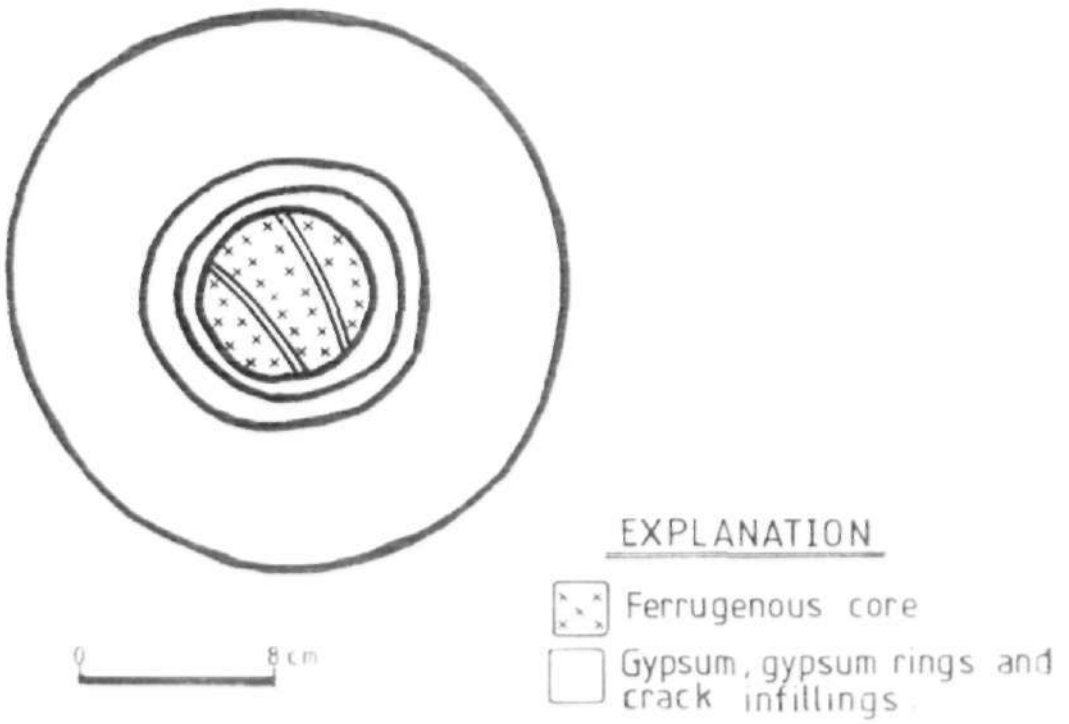
4.3.2 Gypsiferous Septarian Concretions

Spherical and oviod gypsiferous septarian concretions (Plate 2) up to a maximum of 40 cm. in diameter, are common in the lower shale of the Dukamaje Formation at Taloka and Gilbedi where they appear to fill desication cracks. The

Plate 1. Gypsum discontinous thin bed being cut across by a thin vein of gypsum at Wurno.



Plate 2. Gypsiferous septarian concretion from Dukamaje Formation at Taloka village.



large nodules have massive ferruginous cores with thin rapidly attenuating crack which are filled with tiny crystals of gypsum. Thin outer concentric rinds of gypsum surround the cores, and on the surface radiating blades of gypsum define a weak boxwork structure. The smaller concretions with multiple concentric layers of gypsum are marly and friable.

4.3.3 Selenite Crystals

Gypsum also occurs as crystals averaging between 3-4 cm. in length. Transparent crystals showing cleavage are called selenite. Tabular monoclinic crystals of selenite weather out of the lower shale of the Dukamaje Formation at Gilbedi. These crystals exhibit well formed clinopinacoids (010) and prisms (110), and excellent cleavages parallel to the(010) crystal face. Although transparent, the crystals are poikilitic and contain shaly inclusions which are randomly arranged. The selenite crystals were found occuring along the shale bedding planes.

4.3.4 Disseminated Aggregates of minute gypsum crystals

A distinctive layer of green mudstone with disseminated aggregates of minute crystals of gypsum are observed at the base of the Dange Formation at Gada village. The gypsum weathers out easily leaving cavities which give the mudstone a superficial cellular structure. The bone bed near the base of the Dukamaje Formation often contains vertebrate fragments encrusted with gypsum.

Under the binocular microscope, small white gypsum aggregates were observed in washed residues of mudstone samples from the lower part of the Wurno Formation which is exposed behind Wurno village. The mudstone is silty and contains small lumps of gypsum which easily disintegrate into tiny crystals when touched with a picking needle. Small gypsum aggregates were also observed in washed residues of the mudstones which are intercalated within the shales of the Dukamaje Formation.

In the gypsiferous horizons, gypsum may fill up fractures, bedding planes, joints and cavities as veins and veinlets.

Chapter 5

5.0 LABORATORY STUDIES

5.1 X-Ray Diffractometry of Clay Minerals in Shales of the Dukamaje and Dange Formations

5.1.1 Methods

The shales of the Dukamaje Formation and the Dange Formation were both analysed for clay mineral using X-ray diffractometer.

Clay mineral X-ray diffraction traces were obtained on a Philips 1140 diffractometer using Cu K α radiation at a scan speed of $2^{o}min^{-1}$, using oriented samples on glass slides. The samples were heated to $550^{o}C$ permitting more precise identification of the clay minerals present. The percentages of the clay minerals were estimated from the heights of the XRD peaks above the background radiation (Appendix 1).

5.1.2 Clay mineral analysis

X-ray diffractometric analysis was carried out on some of the shale samples collected from the Dukamaje and Dange Formations. X-ray diffraction patterns for the shales of the Dukamaje and Dange Formations show strong, sharp peaks for chlorite, and rather weak ones for illites. Kaolinite occurs in trace amount.

The lack of dominance of kaolinite indicates a non-oxidizing acid depositional environment.

Illite is commonly produced from the alteration of alkali feldspars and micas under alkaline conditions. Diagenetic changes might also have led to the conversion of kaolinite to illite.

The Maastrichtian Dukamaje and the Upper Paleocene Dange sediments in northwestern Nigeria are known to contain gypsum, which is usually formed in an alkaline reducing environment. The chlorite and illite minerals are probably aluminous varieties which are known to be relatively stable in any environment.

5.2 Fluid inclusions

5.2.1 Introduction

Fluid inclusion studies of gypsum samples from Dukamaje and Dange Formations were carried out using the optical microscope.

Fluid inclusions are small volumes of fluid which are trapped within crystals by any of several mechanisms, either during their growth, or by later processes.

Basically, inclusions are samples of fluids that were present when the enclosing minerals grew or recrystallized usually from these same fluids. Important but relatively rare exceptions are the gas bubbles that occasionally are trapped in minerals growing from gas-saturated or boiling liquids and the globules of immiscible fluids, such as oil, or carbondioxide, that are sometimes trapped along with the water phase.

Fluid inclusions may be trapped by:

(a) Crystal growth irregularities or fluid in homogeneities during the formation of the enclosing mineral; these are called primary inclusions and

(b) healing fractures formed in the crystal during its growth - pseudo-secondary inclusions or by

(c) healing of fractures formed at some subsequent time - secondary inclusions.

Primary or pseudo-Secondary inclusions should reflect the character of the fluids present during growth, where as secondary inclusions represent later fluids possibly unrelated to those responsible for primary growth. In samples that have suffered multiple stages of fracturing and re-healing, many different generations of secondary inclusions may be present.

Sorby (1858) was able to show from a series of careful experiments on the thermal expansion of salt solutions and from observations on inclusions formed at various temperatures that the tiny vapour bubbles in many of the inclusions were the result of differential shrinkage of the liquid and the enclosing mineral during cooling from the higher temperature of trapping to the temperature of observation. Sorby showed that the coefficient of expansion on heating (and conversely, the coefficient of contraction on cooling) for a variety of liquid solutions resembling the fluids in inclusions was one or two orders

of magnitude greater than the coefficients for the enclosing or "host" mineral. Sorby thus reasoned that the temperature of trapping can be estimated by heating the sample to the point that the bubbles disappear. The most abundant type of inclusion consists of a liquid of low viscosity and a gas or vapour bubble having a volume generally less than that of the liquid.

Another important observation made by Sorby was that the higher the proportion of vapour, the higher the trapping temperature. This established the potential for using fluid inclusions as geothermometers.

5.2.2 Methods

A study of fluid inclusions using small fragments of gypsum immersed in oil with a refractive index close to that of the mineral (Hollister et al., 1981) was carried out under the optical microscope. An introductory study was carried out by grinding and sieving the gypsum sample. The grains retained by 125 micrometer sieve were immersed in the oil on a clear slide and viewed under the optical microscope.

Wafers of gypsum samples having doubly polished surfaces with thickness of about 50 mm were prepared and a microscopic study to further identify fluid inclusions was undertaken. A perfect polish is made first on one side of the sample by first sawing, grinding and polishing. The temperature during sample preparation was kept below 100^{o}C to avoid decrepitation. The polished side is attached

to a glass slide with a cement that can easily be dissolved or melted at a very low temperature. The specimen is then sawed and ground down to the desired thickness and the second side is so polished. As polishing was going on the sample was viewed under the microscope from time to time to check when the inclusions are clearly seen.

5.2.3 Fluid inclusion study of gypsum samples SS - 1, SS - 2, SS - 5, SS - 6, SS - 7 SS - 8, and SS - 11

The following samples SS - 1, SS - 2, SS - 5, SS - 6, SS - 7, SS - 8 and SS - 11 where ground and sieved. The grains retained by 125 micrometer sieve were immersed in the oil on a clear slide and observed under an optical microscope.

Primary fluid inclusions filled with only liquid were observed. The inclusions generally show spheriodal to oval shapes.

Wafers of samples SS-2, SS-5, SS-7 and SS-11 were prepared and studied under the microscope. Observation shows primary fluid inclusions, filled with only liquid (Plate 3) predominate in the samples and the frequency of occurence of the fluid inclusions in the samples was low.

The sizes of the inclusions range between 100 - 120 um.

5.2.4 Implications of the Fluid inclusion study

According to Roedder (1979), fluid inclusions filled with only liquid indicate that they probably grew at a temperature less than 40°C. This suggests that the processes leading to gypsum formation in the Dukamaje and Dange

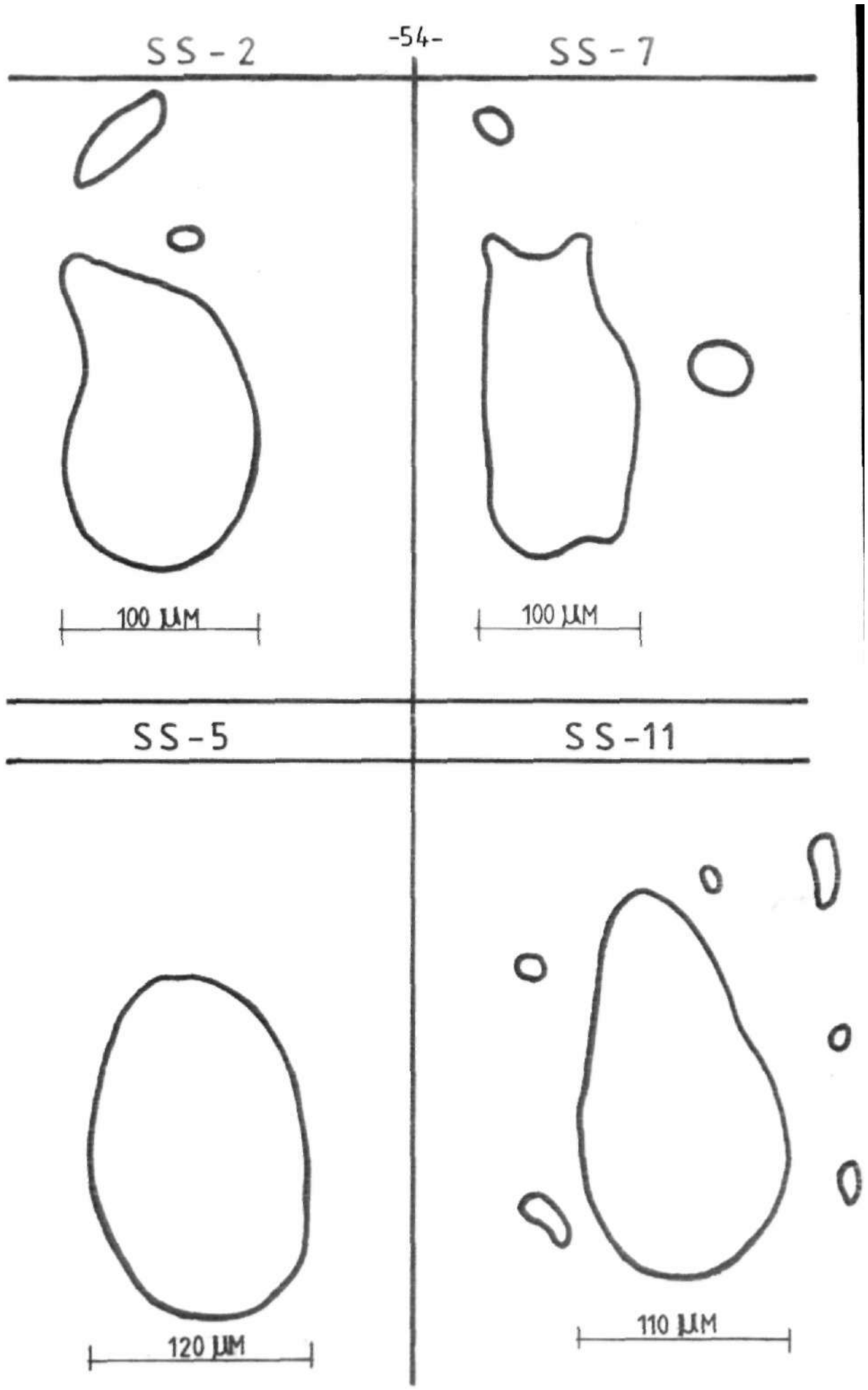


Plate 3. Fluid inclusion study of Wafers of SS-2, SS-5, SS-7 and SS-11.

Formations might be a shallow phenomena.

Carbonaceous shales and thin horizons of lignite are common in the Taloka Formation (Parker, 1966) and are well exposed at Taloka and Gilbedi. Teichmüller and Teichmuller (1967) pointed out that the rank of coal increases with depth if the geothermal temperature is greater than 50°C. High geothermal temperatures accelerate geochemical coalification. A strong heating of short duration can have the same effect as slow heating over longer periods of time. Thus, the rank of a coal is indicative of temperature only if the duration of the heating of the coal is known (Teichmüller and Teichmüller, 1967).

The early transformation of vegetable matter into peat and then to lignite is accompanied by a tremendous reduction in bed thickness. Falini (1965) estimated that reduction in bed thickness when peat is transformed to lignite would be about tenfold ($\frac{1}{10}$ of the original thickness). The fact that lignite, the lower rank coal occurs in the Maastrichtian Taloka formation, indicates a low geothermal temperature of less than 50°C and supports a low temperature of entrapment of the fluid included in the gypsum from Dukamaje and Dange Formations.

Hanshaw and Bredehoeft (1968) indicated the pressure - temperature relationship between gypsum and anhydrite where gypsum would be deposited from saturated calcium sulphate solutions below 42°C and anhydrite above the temperature

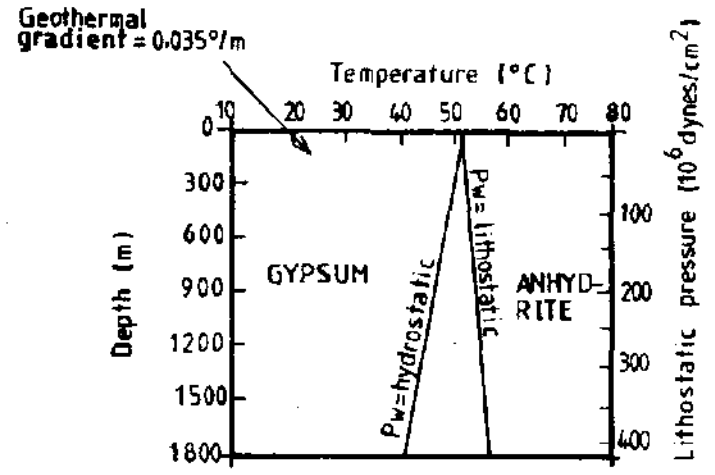


Fig. 5.1 Pressure - temperature relationship between gypsum and anhydrite where activity of $H_2O = 0.93$ (= 2 m Nacl). (After Hanshaw and Bredehoeft, 1968).

(Fig. 5.1). The stable phase is determined by the activity of water (related to salinity) and temperature (Hardie 1967).

The reduction in bed thickness as a result of peat transformation to lignite shows that the sediments were under gravitational compaction and that there was fluid entrapment due to gravitational compaction.

5.3 Geochemistry

5.3.1 Introduction

The major and trace element contents of gypsum from the Dukamaje and Dange Formations were determined by Laboratory analysis. The distribution and association of such elements in the gypsum are discussed in this section.

5.3.2 Analytical methods

5.3.2.1 Sample preparation - A systematic selection was made of samples from various levels of the Dukamaje and Dange Formations at Gada, Rabbah, Wurno, Taloka, Dukamaje, Dogondaji, Gilbedi and Shinaka. The selected samples were cleaned by washing to remove host rock particles that cling to the samples and then allowed to dry. The samples were later crushed and ground to powder with a grinding machine. These samples were then weighed and heated in a muffle furnace at 450^{o}C for 30 minutes and reweighed to determine the ignition loss. 1g of the samples were mixed with 2g of solid ammonium chloride (NH_4C^{l}) in 150 ml beaker. To the mix, 10 ml concentrated hydrochloric acid was added, mixed and the beaker was covered. The mix was heated up on the sand bathe for 30 minutes. The content was stirred

repeatedly during this time with the glass rod. After time mentioned above, 50 ml. of hot dilute hydrochloric acid was added and the mix was heated for another few minutes and then filtered through filter quality 589 blackband into 500 ml graduated flask. The residue on the filter paper was washed with hot dilute hydrochloric acid. The beaker was cleaned and then washed with hot water. The residue contained precipitated SiO_2 and acid-insoluble components of the gypsum. The filterate is cooled down to room temperature and used for further analysis.

5.3.2.2. Chemical analyses - Analyses for Fe, Ti and Mg were carried out using the PERKIN ELMER (sp 400) AAS of the Department of Chemistry, A.B.U., Zaria and later cross checked with the Atomic Absorption Spectrometer (AAS) of the Soil Science,Department, A.B.U., Zaria.

Ca, Na and K in the gypsum were analysed using the flame spectrometer of the Department of Chemistry, A.B.U., Zaria. The samples were diluted to a factor of 1000 for the major elements and 100 for trace elements. The readings were compared against standard graphs drawn by plotting absorbance against concentration.

Analyses for the Al, Si and SO_3 contents in the gypsum samples by complexometric titrations were made by the Northern Nigerian Cement Company Limited, Sokoto.

The values of the chemical analysis of the gypsum samples are given in tables 5.1, 5.2, 5.3, and 5.4 and the average values in table 5.5. The behaviour of these elements as shown in figures 5.2 - 5.11 are discussed.

5.3.3 The distribution of elements in the gypsum

Results of the chemical analysis of Si, Al, Ti, Fe, Mg, Ca, Na, K and SO_3 in 18 gypsum samples of different modes of occurence, and from different formations and localities are discussed in this section.

5.3.3.1 Silica (Si) - The trend of silica is shown in Figure 5.2. The average value of silica is highest in the Septaria mode. The average value of the value of silica in the septarian and selenite modes is close, being 0.31 and 0.30 percent respectively. The silica trend corresponds with those of Ti, Al and Fe.

The silica value is very low in the Dange discontinous thin bed mode when compared with the other gypsum modes.

5.3.3.2 Aluminium (Al) The average value of Aluminium in the gypsum modes is highest in the septarian mode having 1.38 percent. This decreased through 1.10 percent in the selenite mode, 1.01 percent in the Dukamaje

Table 5.1 CHEMICAL ANALYSIS OF THE DISCONTINOUS THIN BEDS Mode OF GYPSUM, DANGE FORMATION, SOKOTO BASIN (%)

Sample No.	Locality	Si	Al	Ti	Fe	Mg	Ca	Na	K	SO_3	Ignition Loss	Sum Total
SS - 1	Wurno	0.01	0.43	0.04	0.50	1.43	23.00	2.40	0.34	49.30	20.90	97.35
SS - 2	Wurno	0.08	1.12	0.10	0.24	2.60	24.65	2.25	1.30	45.20	20.10	97.64
SS - 3	Rabbah	0.00	0.03	0.05	0.10	1.81	29.91	2.09	0.08	46.60	19.20	99.87
SS - 4	Gada	0.06	0.19	0.20	0.07	2.70	28.57	0.70	0.71	48.10	18.40	99.54
SS - 5	Gada	0.02	0.15	0.20	0.40	1.91	28.00	1.80	0.60	46.31	19.01	98.22
AVERAGE OF SS-1, SS-2 SS-3, SS-4 AND SS-5		0.03	0.38	0.08	0.26	2.09	26.83	1.85	0.61	47.00	19.52	

Table 5.2 CHEMICAL ANALYSIS OF THE DISCONTINOUS THIN BEDS Mode OF GYPSUM, DUKAMAJE FORMATION, SOKOTO BASIN (%)

Sample No.	Locality	Si	Al	Ti	Fe	Mg	Ca	Na	K	SO_3	Ignition Loss	Sum Total
SS - 6	Shinaka	0.11	1.52	0.21	0.62	1.80	26.80	1.62	0.30	45.46	17.00	95.44
SS - 7	Taloka	0.09	1.00	0.01	1.34	0.60	24.60	1.20	1.40	43.80	20.80	94.80
SS - 8	Taloka	0.23	0.81	0.07	0.21	1.30	24.23	0.52	0.10	47.5	19.00	93.97
SS - 9	Dukamaje	0.04	1.22	0.40	0.08	3.00	26.98	1.80	0.30	43.13	18.80	95.75
SS - 10	Dukamaje	0.18	0.51	0.30	0.60	1.50	24.12	0.94	0.50	46.00	20.40	95.06
AVERAGE OF SS-6, SS-7 SS-8, SS-9 AND SS-10		0.13	1.01	0.20	0.57	1.64	25.34	1.42	0.53	45.18	19.20	

Table 5.3 CHEMICAL ANALYSIS OF THE SELENITE ~~mode~~ OF GYPSUM, DUKAMAJE FORMATION, SOKOTO BASIN (%)

Sample No.	Locality	Si	Al	Ti	Fe	Mg	Ca	Na	K	So_3	Ignition Loss	Sum Total
SS - 11	Dukamaje	0.21	0.68	0.18	0.08	1.42	25.00	1.68	0.51	46.00	19.00	94.86
SS - 12	Dukamaje	0.06	1.50	0.20	1.11	0.75	23.40	0.82	0.80	44.80	17.80	93.24
SS - 13	Dukamaje	0.44	1.98	0.05	0.34	2.68	27.00	1.74	0.20	43.00	20.56	96.99
SS - 14	Gilbedi	0.50	0.24	0.42	1.33	1.55	24.4	1.06	0.42	45.2	18.00	93.82
AVERAGE OF SS-11, SS-12 SS-13, AND SS-14		0.30	1.10	0.21	0.72	1.6	25.00	1.33	0.48	44.80	18.84	

Table 5.4 CHEMICAL ANALYSIS OF THE SEPTARIAN CONCRETION Mode OF GYPSUM, DUKAMAJE FORMATION, SOKOTO BASIN (%)

Sample No.	Locality	Si	Al	Ti	Fe	Mg	Ca	Na	K	SO_3	Ignition Loss	Sum Total
SS - 15	Taloka	0.21	0.84	0.09	1.75	1.30	24.56	1.90	0.05	44.00	19.60	94.30
SS - 16	Dukamaje	0.09	1.50	0.06	2.00	0.80	22.40	0.58	0.30	43.9	18.00	89.27
SS - 17	Taloka	0.81	2.20	0.30	1.30	2.00	20.00	2.21	0.18	43.5	17.20	90.7
SS - 18	Shinaka	0.14	0.90	0.75	1.50	1.75	23.30	0.31	0.62	42.6	19.10	90.79
AVERAGE OF SS-15, SS-16 SS-17 AND SS-18		0.31	1.38	0.30	1.64	1.46	22.57	0.80	0.29	43.5	18.25	

Table 5.5 AVERAGE VALUES OF THE ELEMENTS IN THE GYPSUM Modes

Mode	Si	Al	Ti	Fe	Mg	Ca	Na	K	SO_3	Ignition Loss
A	0.03	0.38	0.08	0.26	2.09	26.83	1.85	0.61	47	19.52
B	0.13	1.01	0.20	0.57	1.64	25.34	1.42	0.53	45.18	19.20
C	0.30	1.10	0.21	0.72	1.60	25.00	1.33	0.48	44.8	18.84
D	0.31	1.38	0.30	1.64	1.46	22.57	0.80	0.29	43.5	18.25

A = The Dange discontinous thin beds.

B = The Dukamaje discontinous thin beds.

C = The selenite Mode

D = The Septarian Mode

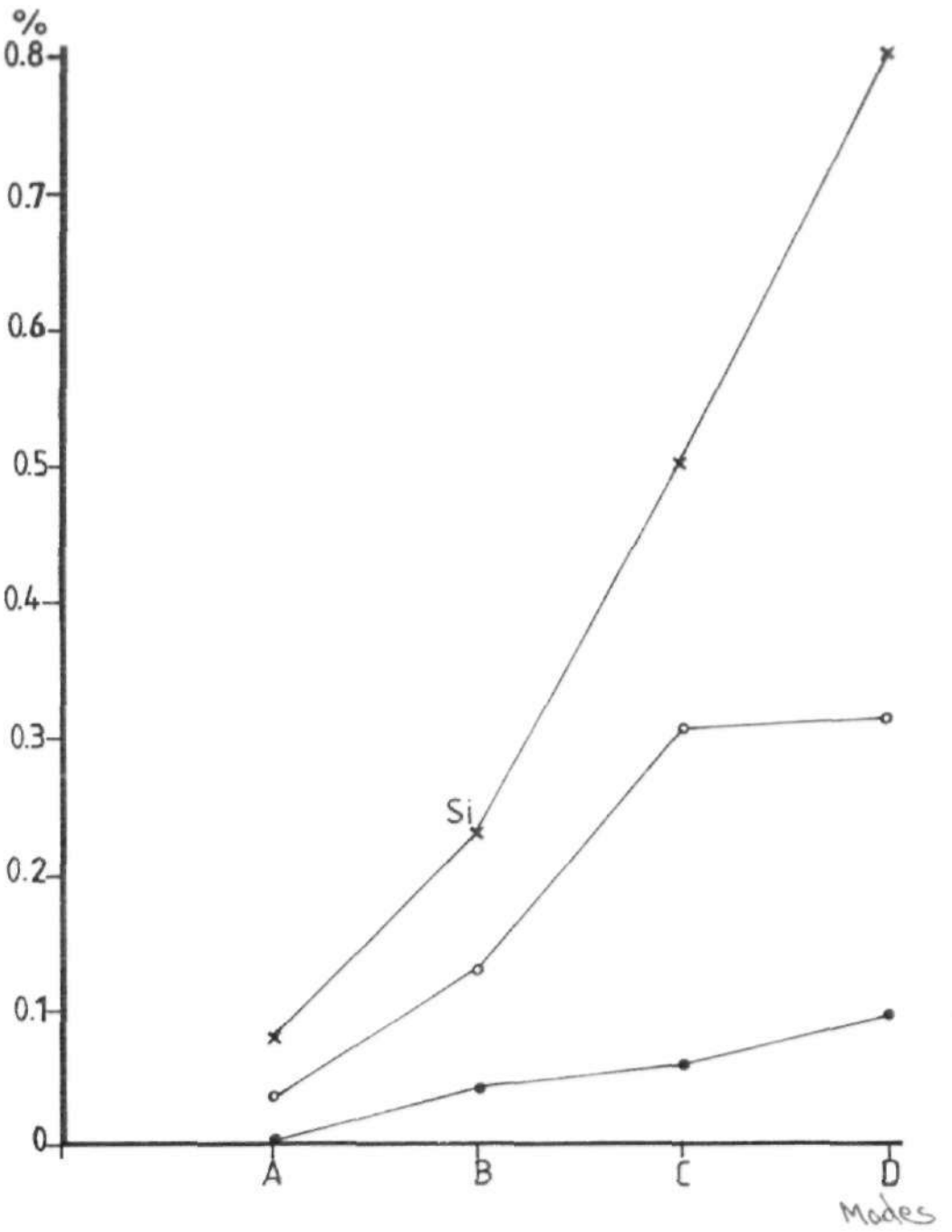


Fig 5.2 The distribution of Si in the gypsum Modes.

A = The Dange discontinous thin beds
B = The Dukamaje discontinous thin beds
C = The Selenite
D = The Septaria

×—× *Highest* values
o—o Average values
●—● Lowest values

discontinous thin bed mode to 0.38 percent in the Dange discontinous thin bed mode (Fig. 5.3).

Aluminium shows inverse relationship with Na, K, Mg, Ca and SO_4.

5.3.3.3 Titanium (Ti) Titanium is a trace element in the gypsum samples. Of the gypsum modes, the septarian mode has the highest average value of 0.30 percent titanium. The value decreased through 0.21, 0.20 and 0.08 percent titanium in the selenite mode, Dukamaje discontinous thin bedded mode and Dange discontinous thin bedded mode respectively.

The titanium trend shown in figure 5.4 indicates that the amount of titanium decreases with the marine Dange mode,until the Dukamaje discontinous thin bedded and the selenite modes where a close average value is maintained. This trend of decreasing titanium content towards the marine Dange Gypsum mode indicates a shift towards gypsum purity.

Titanium shows a similar trend with iron and an inverse relationship with Ca, SO_4, Na, Mg and K.

5.3.3.4 Iron (Fe) - The amount of iron in the gypsum is highest in the septarian mode having 1.64 percent (average of SS-15, SS-16, SS-17 and SS-18). This decreases through 0.72 percent in the selenite mode, 0.57 percent in the Dukamaje discontinous thin bedded mode to as low as 0.26

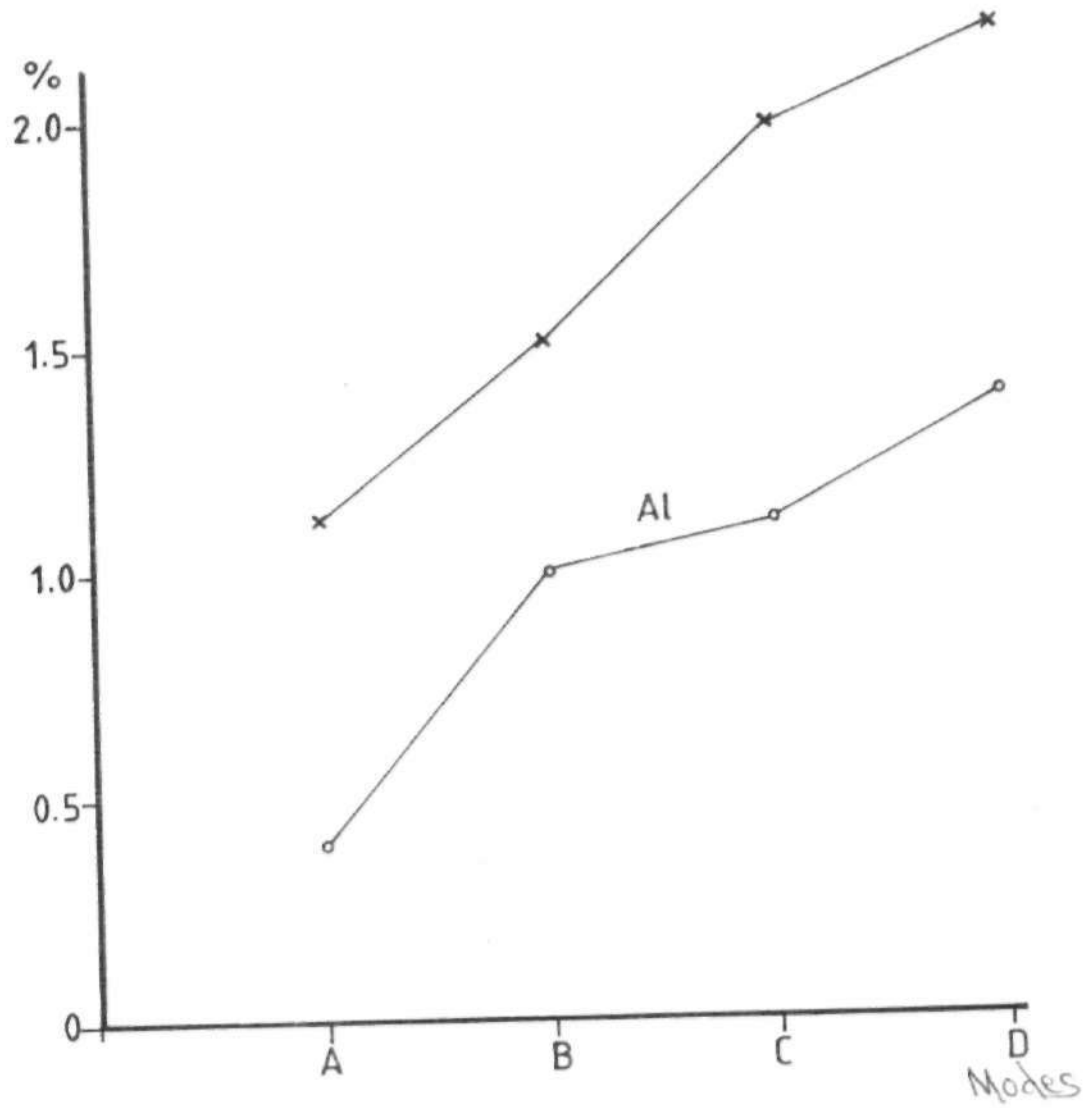


Fig. 5.3 The distribution of Al in the gypsum Modes.

A = The Dange discontinous thin beds
B = The Dukamaje discontinous thin beds
C = The Selenite
D = The Septaria

×——× Highest values
o——o Average values

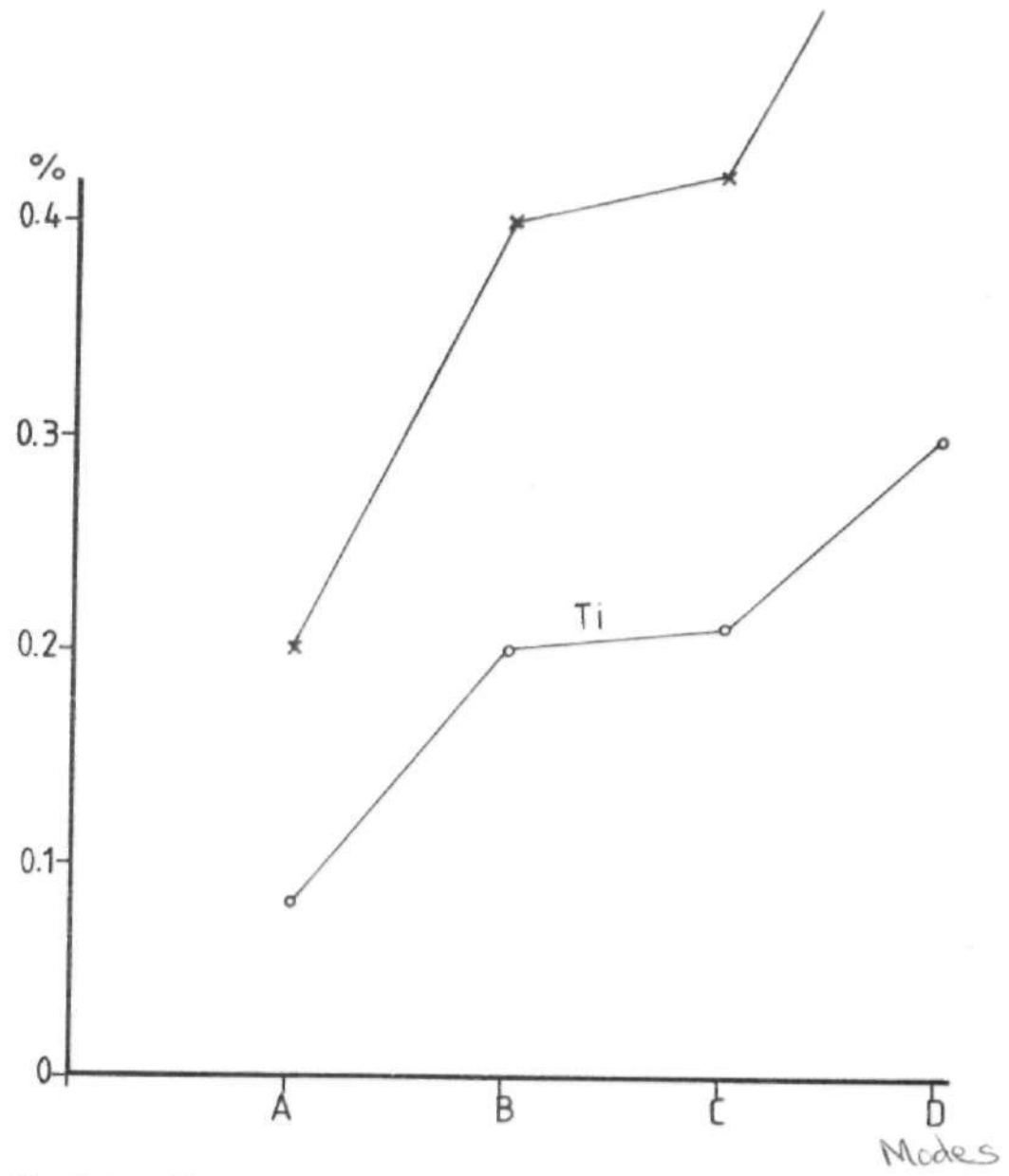


Fig. 5.4 The distribution of Ti in the gypsum modes.

A = The Dange discontinous thin beds
B = The Dukamaje discontinous thin beds
C = The Selenite
D = The Septaria

×——× Highest values
o——o Average values

percent in the Dange discontinous thin bedded mode. The high value of iron in the septarian mode corresponds with the macroscopic description of the septarian variety in which gypsum surrounds an iron core.

The trend of iron (Fig. 5.5) in the gypsum modes show an inverse relationship to those of Na, K, Ca, Mg and SO_4.

5.3.3.5 Magnesium (Mg) - Magnesium values vary from 2.09 to 1.46 percent (average values) in the Dange discontinous thin beds mode and the septarian mode respectively. The magnesium trend (Fig. 5.6) shows that the geochemistry of magnesium is quite similar to that of calcium.

Magnesium is generally found in lesser concentrations in freshwaters than calcium despite the higher solubility of most of its compounds. The higher concentration of magnesium in the Dange discontinous thin beds mode could be due to paleo-environment and the large ionic charge and radii for magnesium.

5.3.3.6 Calcium (Ca) - Calcium is a major element in all the samples. The trend of calcium is shown in figure 5.7. The value of calcium is highest in the Dange discontinous thin bedded mode with average value of 26.83 percent and lowest in the septarian mode with average value of 22.57 percent.

The calcium values for the Dukamaje discontinous thin beds and selenite modes vary very slightly.

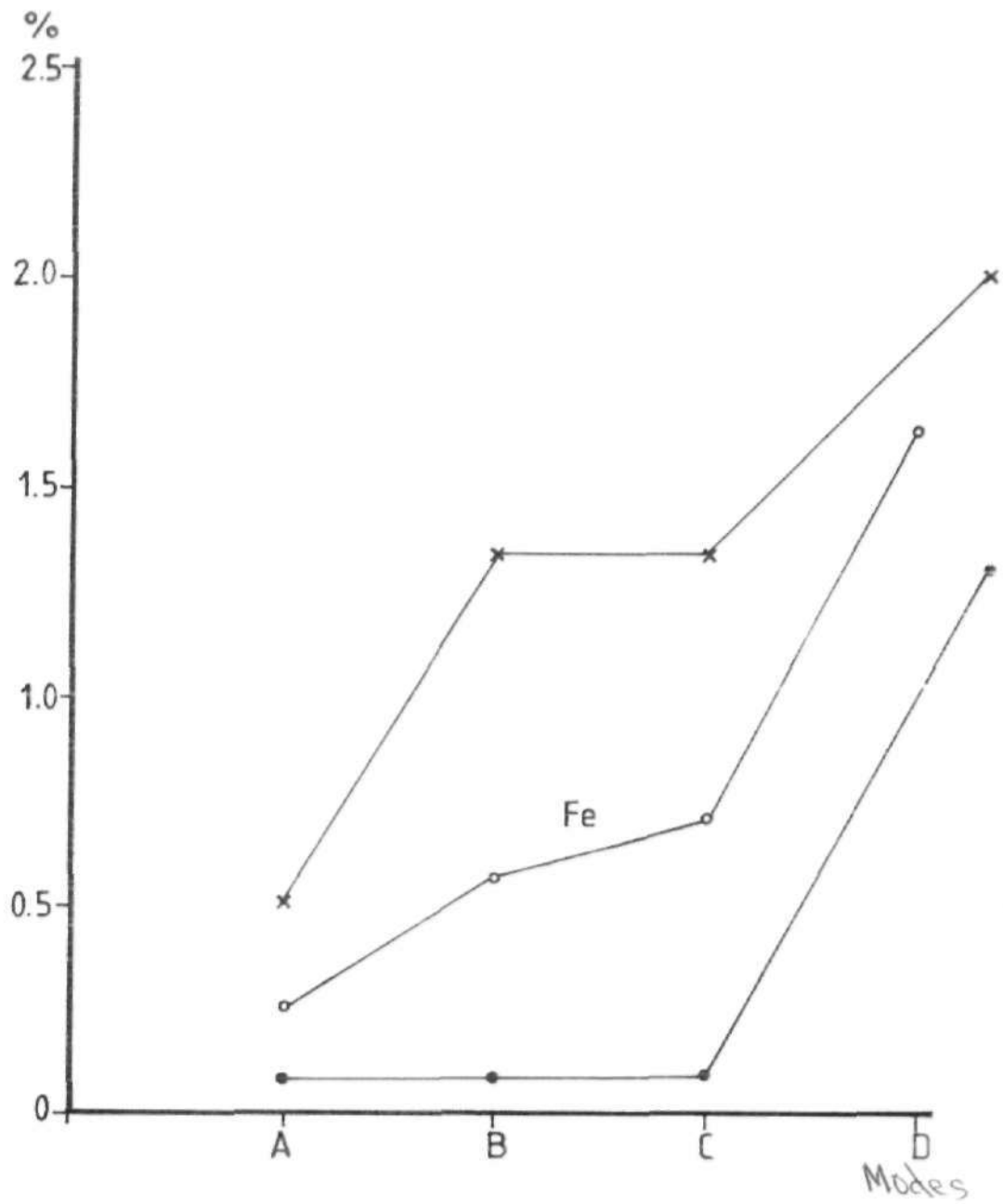


Fig. 5.5 The distribution of Fe in the gypsum Modes.

A = The Dange discontinous thin beds
B = The Dukamaje discontinous thin beds
C = The Selenite
D = The Septaria

×——× Highest values
o——o Average values
●——● Lowest values

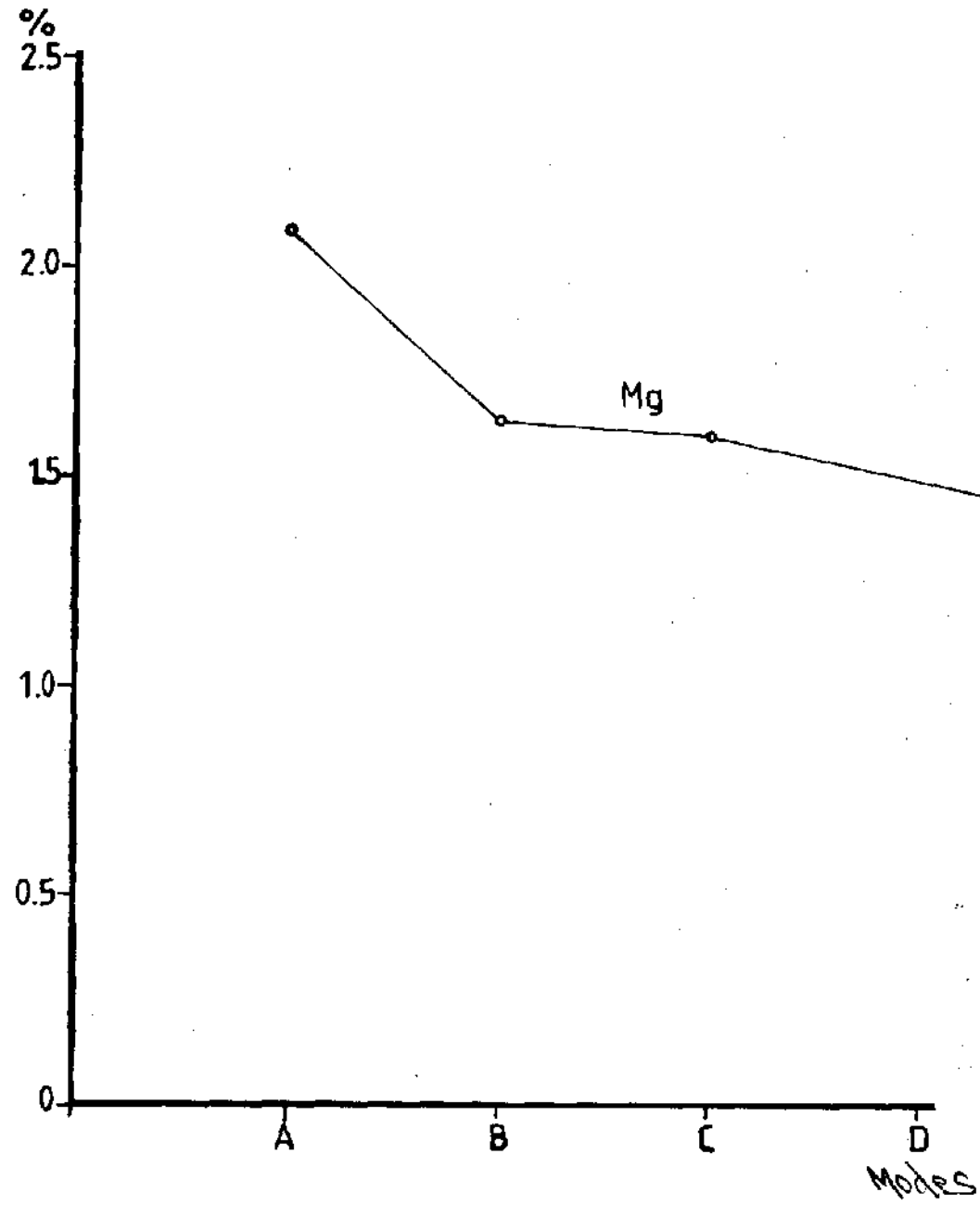


Fig. 5.6 The distribution of average Mg in the gypsum modes.

A = The Dange discontinous thin beds
B = The Dukamaje discontinous thin beds
C = The Selenite
D = The Septaria

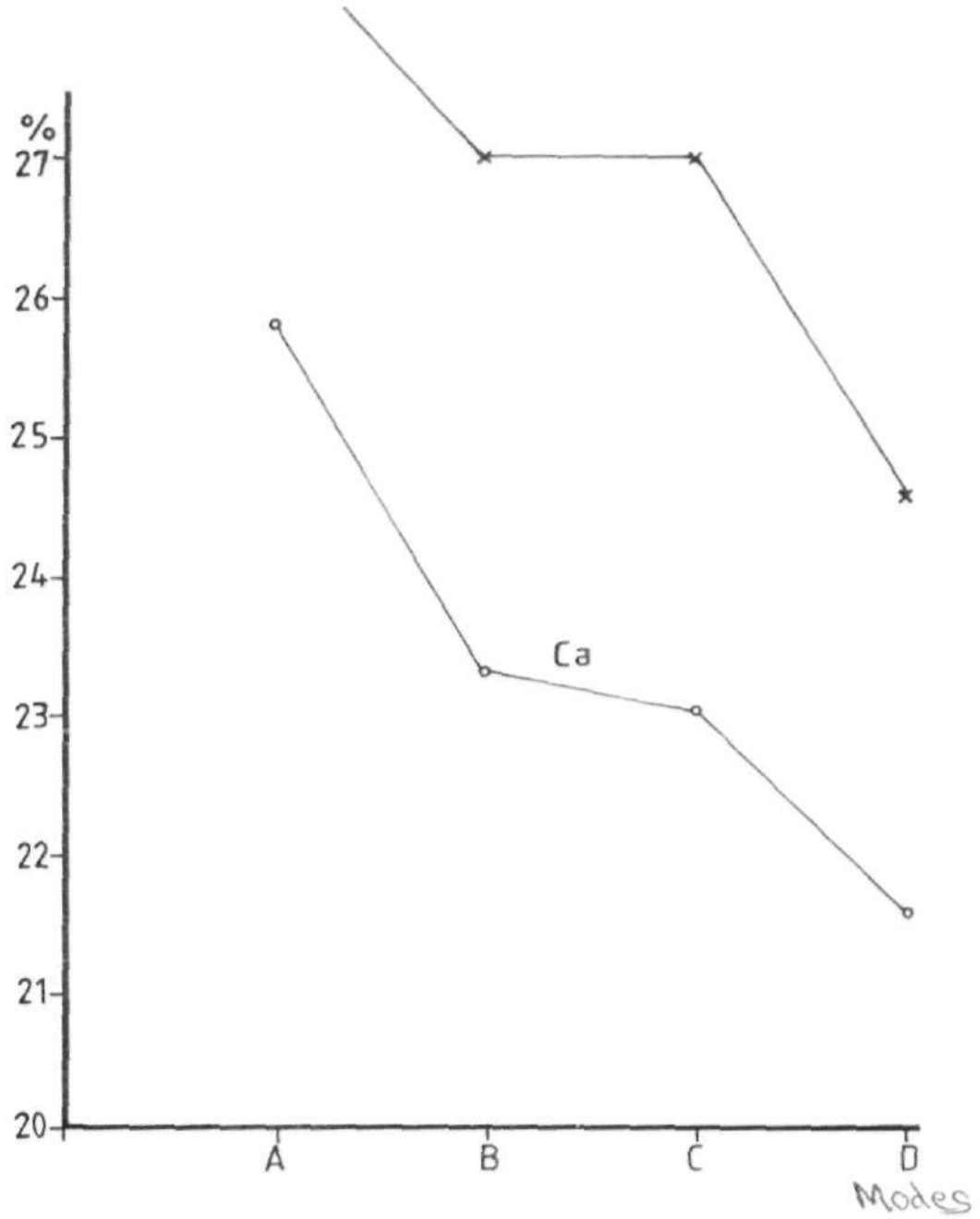


Fig. 5.7 The distribution of Ca in the gypsum modes.

A= The Dange discontinous thin beds
B= The Dukamaje discontinous thin beds
C= The Selenite
D = The Septaria

×—× Highest values
o—o Average values

The ratio is 12 and 14 to 1 for calcium to magnesium. This ratio indicates that the brines are marine in origin (Davies and Dewiest, 1966).

5.3.3.7 Sodium (Na) - The trend of sodium is shown in figure 5.8. The average value of sodium in the gypsum modes vary between 0.80 and 1.85 percent in the septarian and Dange discontinous thin beds of gypsum respectively. The selenite mode and the Dukamaje discontinous thin gypsum beds have average values of 1.33 and 1.42 percent respectively.

The values of sodium in the gypsum samples are moderately high (See table 5.1 - 5.4). The high content of sodium (> 1%) in some of the gypsum samples indicates a more marine environment in line with affinity sodium has for the oceanic environment.

5.3.3.8 Potassium (K) - The trend of potassium is shown in figure 5.9. The value of potassium is highest in the Dange discontinous thin bed mode and lowest in the septarian mode with average values of 0.61 and 0.29 percent respectively. The average potassium values for the Dukamaje discontinous thin bed and selenite modes are 0.53 and 0.48 percent respectively.

Potassium shows a similar trend with sodium. The ratio of sodium to potassium is very high in favour of sodium in all the localities. This trend could be due to differences in the source and ionic charge and radii between the two elements.

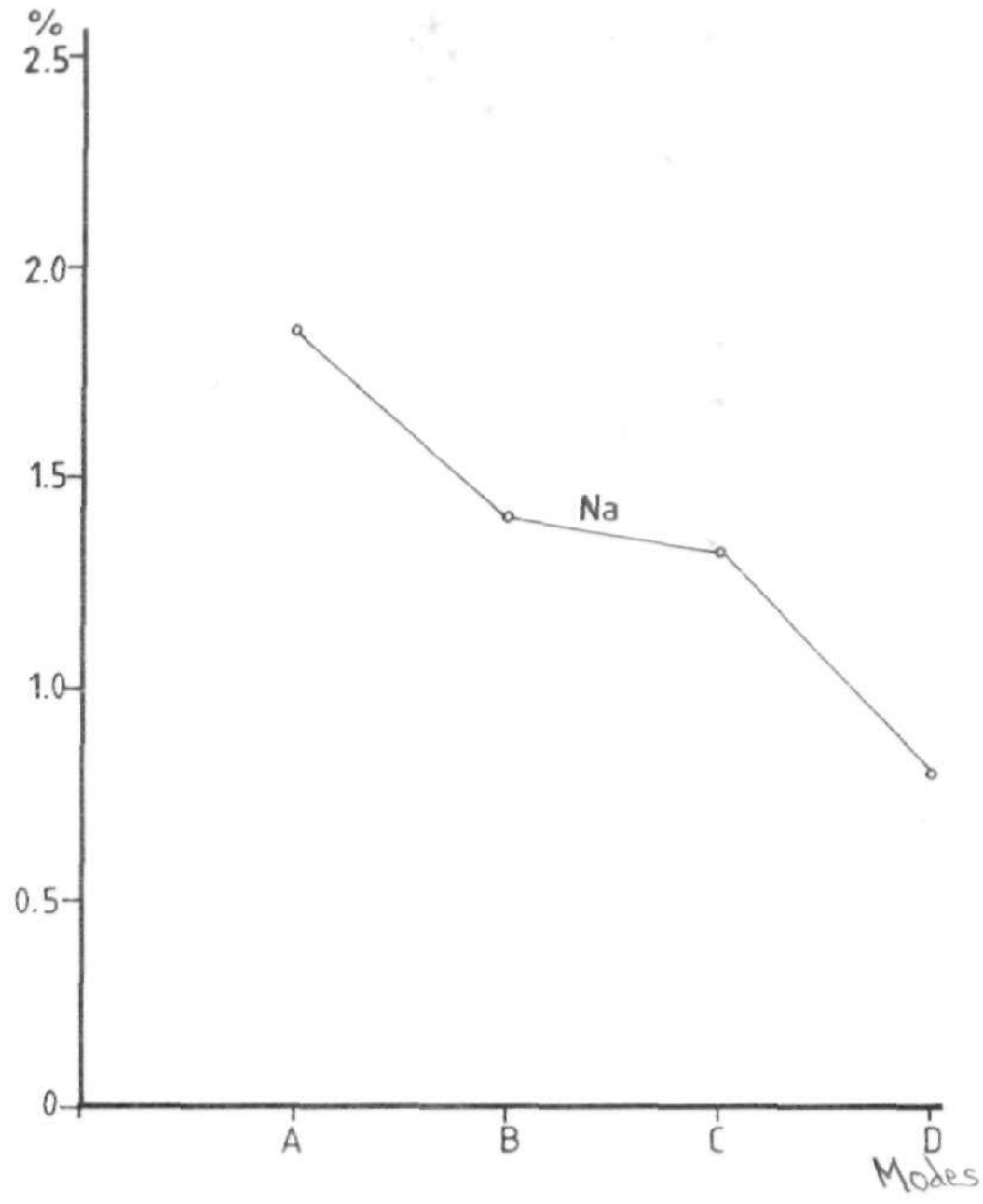


Fig. 5.8 The distribution of average Na in the gypsum modes.

A = The Dange discontinous thin beds
B = The Dukamaje discontinous thin beds
C = The Selenite
D = The Septaria

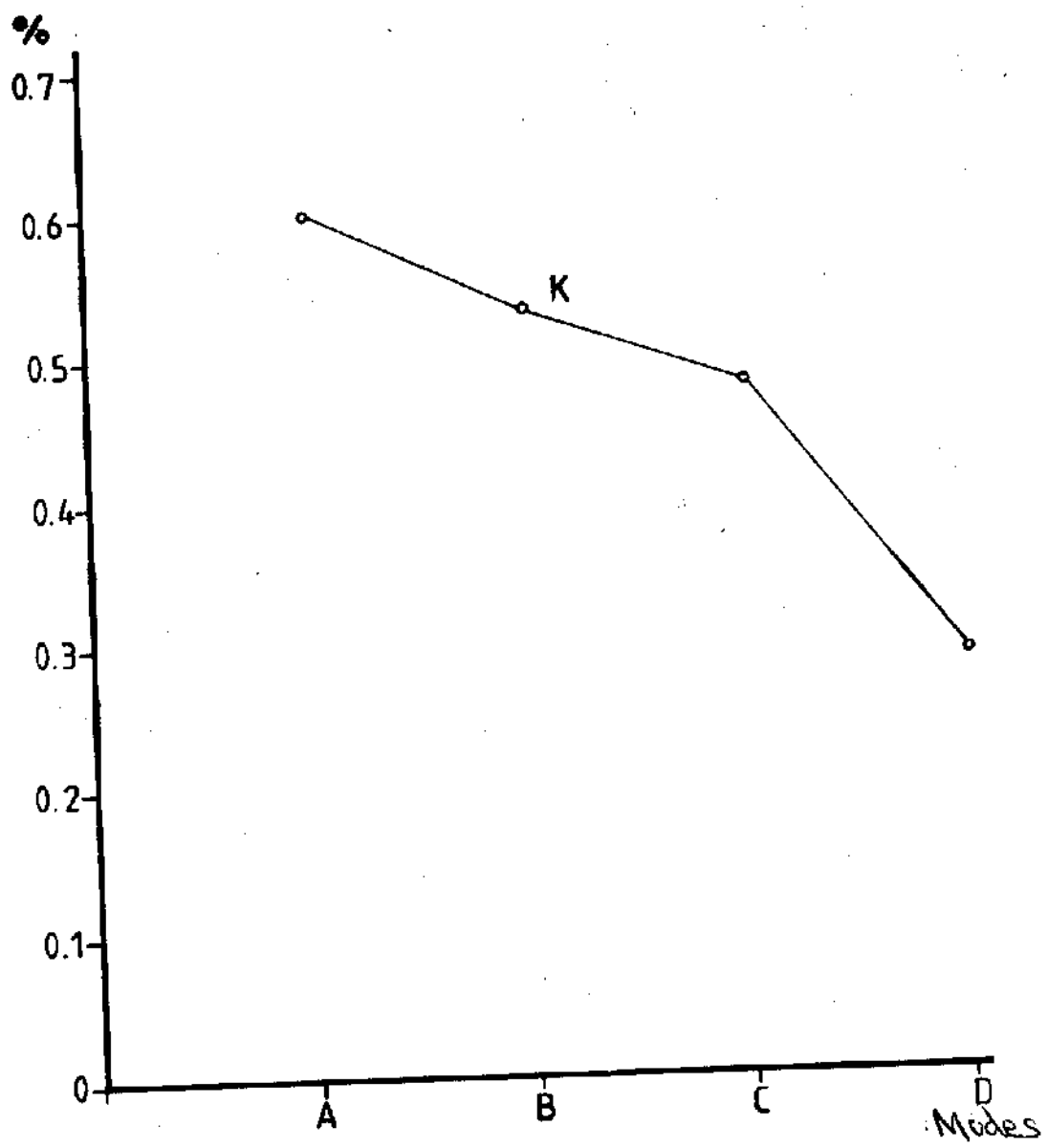


Fig. 5.9 The distribution of average K in the gypsum modes.

A = The Dange discontinous thin beds
B = The Dukamaje discontinous thin beds
C = The Selenite
D = The Septaria

5.3.3.9 Sulphate (SO_4^{2-}) - The trend of SO_4^{2-} from the Dange discontinous thin bedded mode through the Dukamaje discontinous thin bedded, the selenite to the septaria modes is shown in figure 5.10. There is a decrease in the value of SO_4^{2-} from 47 percent in the Dange discontinous thin bed mode through 45.18 percent in the Dukamaje discontinuous thin bed mode and 43.5 percent in the septarian mode. The Dange discontinous thin bed mode has the highest sulphate content of 47 percent. The sulphate values for the Dukamaje discontinous thin beds and the selenite modes vary slightly.

The sulphate is inversely related to Fe, Ti, Al and Si. The sulphate concentration appears to be influenced by marine paleoenvironment.

5.3.4 Discussion

The low concentrations of the elements Na, K, Mg, Si, Al, Ti and Fe and the higher concentrations of (Ca^{2+} and SO_4^{2-}) in the samples probably have paleoenvironmental and mineralogical control.

A close average value is observed in the amount of Mg, Na, K, Al, Ti, Fe, Ca and SO_4^{2-} in the Dukamaje discontinous thin bed and the selenite modes. This shows that the conditions relating to the enrichment of these elements in the Dukamaje discontinous thin bed and the selenite modes remain essentially constant during this type of mineralization.

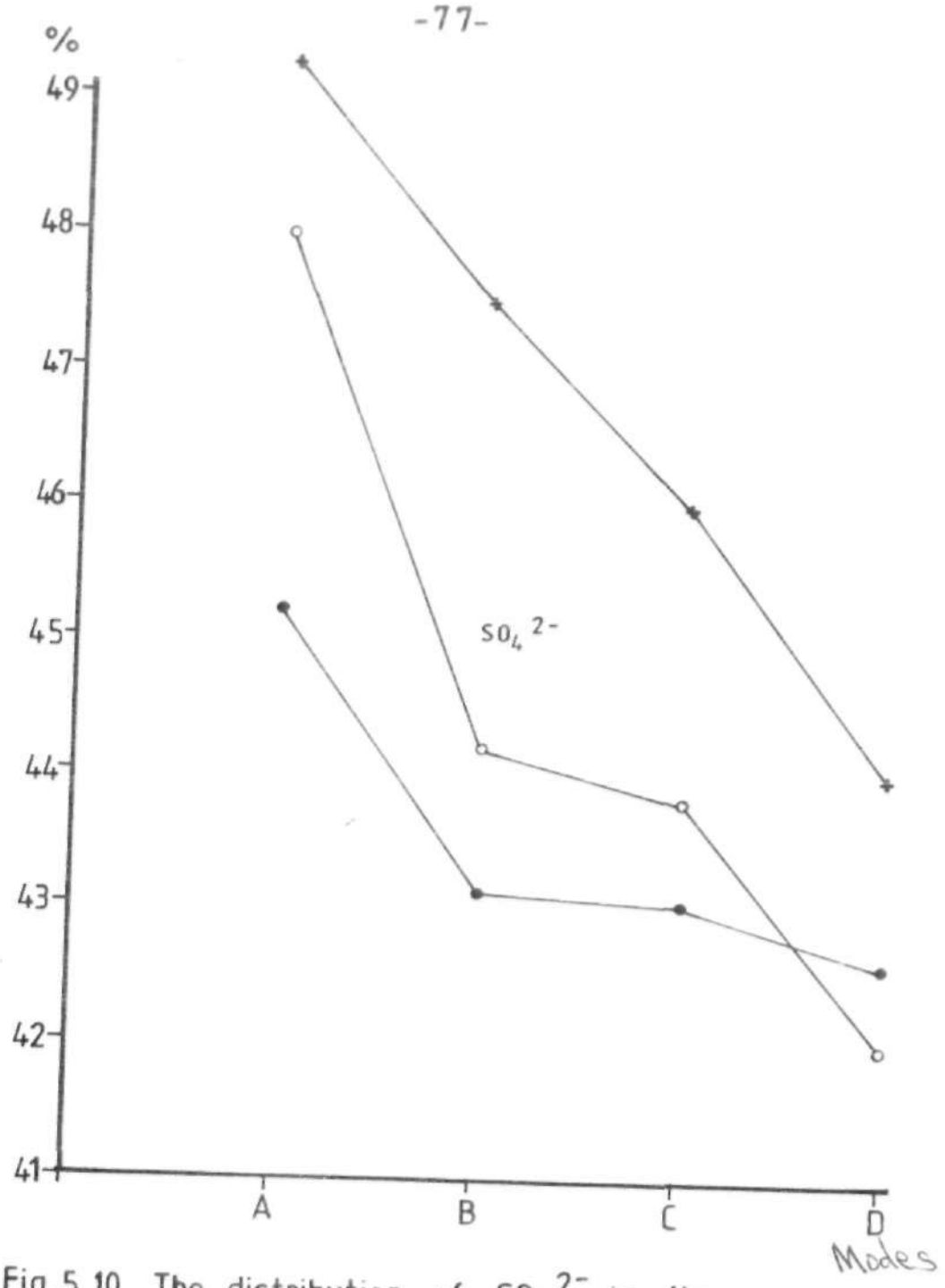


Fig. 5.10 The distribution of SO_4^{2-} in the gypsum modes.

A = The Dange discontinous thin beds
B = The Dukamaje discontinous thin beds
C = The Selenite
D = The Septaria

Highest values
Average values
Lowest values

The values of K, Na, Mg, Ca, SO_4^{2-} generally show a similar trend and they show an inverse relationship to those of Si, Al, Ti and Fe.

The sum total of the value of soda and potash exceeds that of silica. This indicates an alkaline environment of gypsum formation.

Magnesium value is generally greater than that of sodium (see Table 5.1 to 5.5). This could be due to large ionic charge and radii for magnesium over sodium.

Pure gypsum has 32.6% CaO, 46.5% SO_3 and 20.9% H_2O. A triangular plot for the system CaO - H_2O - SO_3 (Fig. 5.11) was carried out using the percentage values of CaO - H_2O - SO_3 of the modes of gypsum. Observation shows that the field of plotted points fall within the field for pure gypsum.

■ (Z) Field of plot for pure gypsum
A = Dange discontinous thin bed
B = Dukamaje discontinous thin bed
C = The Selenite
D = The Septaria

NOTE : Average values for the modes of Gypsum are used in calculating the percentage CaO, H_2O and SO_3.

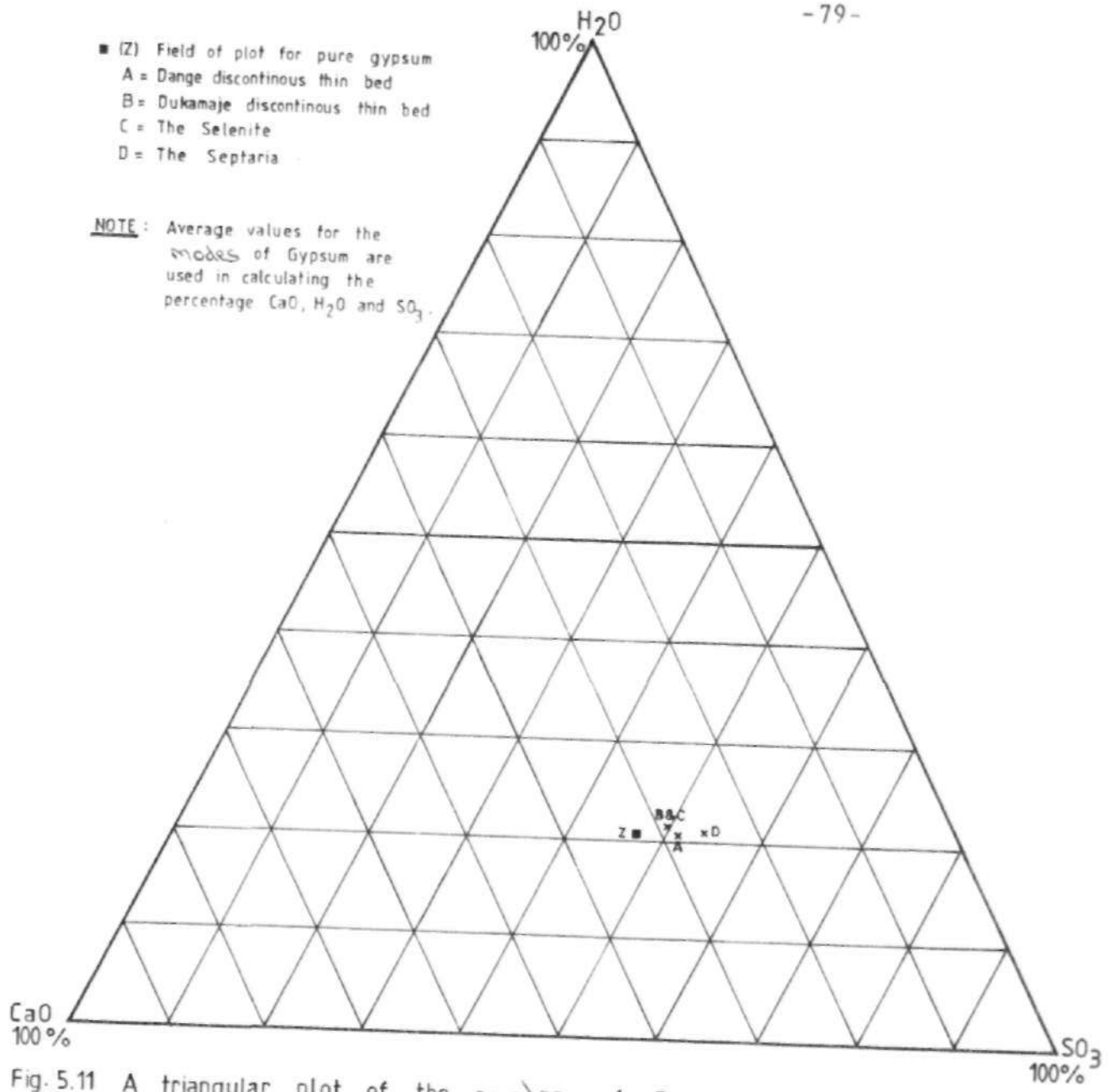


Fig. 5.11 A triangular plot of the modes of Gypsum for the system $CaO - H_2O - SO_3$.

Chapter 6

6.0 ORIGIN OF GYPSUM IN THE PROJECT AREA

6.1 Evidence of Palaeoenvironment

6.1.1 Sedimentology

Clay mineral determinations of the shales of the Dukamaje and Dange Formations by X-ray diffractometry (section 5.1) indicates an alkaline reducing depositional environment. This is evidenced by the lack of dominance of kaolinite which suggests a non-oxidizing acid depositional environment.

6.1.2 Palaeontology

Gypsum shows an intimate association with the fauna relics of the Dukamaje and Dange Formations (Section 3.3). According to Patnode (1941), black, grey or green colouration signify reducing conditions with at least partial preservation of organic matter. The greyish nature of the gypsiferous shales of the Dukamaje and Dange Formations and abundant vertebrate fossils associated imply that gypsum was emplaced under reducing conditions which supports the preservation of the fossils.

6.1.3 Geochemistry

Chemical analysis of the gypsum from Dukamaje and Dange Formations (Section 5.3) shows that the ratio of Ca: Mg is 12:1 & 14:1 respectively. These ratios indicate a marine origin for the fluids that formed gypsum (Davies and Dewiest, 1966). Chemical analysis also reveals an alkaline environment of gypsum formation based on the fact

that the sum total of the value of soda and potash exceeds that of silica, thus indicating an alkaline environment of gypsum formation.

6.1.4 Mode of gypsum occurence

Gypsum in the Dukamaje and Dange Formations of the study area (Section 4.3) are intercalated within shales and may fill up fractures, bedding planes, joints and cavities as well as veins and veinlets. The gypsum exhibits the following modes of occurence

a) Discontinous thin beds and veins of gypsum.

b) Septarian concretion.

c) Selenite crystals.

d) Minute crystals of gypsum.

The lack of a widely-traceable gypsum bed indicates that gypsum was not chemically precipitated from a standing body of brine (i.e. an evaporite). These features of the gypsum occurences indicate a non evaporitic origin for the gypsum and that the gypsum formed by post-depositional processes.

6.2 Evidence of Post-depositional Processes

6.2.1 Fluid inclusions

Fluid inclusion studies of the gypsum (Section 5.2) shows primary fluid inclusions filled with only liquid. Roedder (1979) indicated that fluid inclusions filled with only liquid, probably grew at a temperature less than 40^{o}C.

This suggests that the processes leading to gypsum formation in the Dukamaje and Dange Formations might be a shallow phenomena.

6.2.2 Compaction and Overpressuring

Gravitational compaction due to the load of overlying sediments is evidenced by the presence of lignite in the Taloka Formation. The early transformation of vegetable matter into peat and then to lignite is accompanied by a tremendous reduction in bed thickness. Falini (1965) estimated that the reduction in bed thickness when peat is transformed into lignite would be about ten fold. Thus gravitational compaction, due to the load of overlying sediments, aids in enhancing the concentrations of Ca^{2+} and SO_4^{2-} ions in the interstitial fluid by filter sieving to exceed the solubility product at which gypsum precipitates.

The compression of the fluids as a result of gravitational compaction and the resistance of the shale to fluid expulsion causes over pressuring of the shales whereby the shale serve as a "salt sieving" device.

On the basis of palaeoenvironment and lithology, the Gamba Formation is very similar to the Dange Formation but it is not gypsiferous. This could be due to inadequate load of the overlying sediments and/or the depletion of a reasonable concentration of Ca^{2+} and SO_4^{2-} ions at the time the Gamba Formation was laid down.

6.3 Origin of Formation of Gypsum

There are two different opinions as to the origin of gypsum in the Sokoto Basin. According to Petters (1979) gypsum in the Sokoto area precipitated out of the Mediterranean sea which spilled into the region in the past. That is, the gypsum is an evaporite.

Following the idea of Jones (1948), Kogbe (1979) is of the opinion that the gypsum was formed by the action of sulphuric acid (derived from marcasite) on calcium carbonate of limestone to form a solution containing calcium sulphate from which gypsum crystallized out as follows:

$$2FeS_2 + 2H_2O + 7O_2 = FeSO_4 + H_2SO_4.$$

$$CaCo_3 + H_2SO_4 = CaSO_4 + H_2CO_3$$

In this case the gypsum is formed by secondary processes.

Petters (1986) supports a diagenetic growth within coastal sediments shortly after deposition and direct precipitation from drying bodies of water.

This present study essentially favours an early diagenetic model for the origin of the gypsum in the Dukamaje and Dange Formations. That gypsum forms by this process is not surprising.

Robert and Chamley (1974) reported tabular and acicular authigenic gypsum crystals in eastern mediterranean sapropelic sediments. The crystals were at times found associated with unaltered pyrite. These sediments are rich in sulphur.

Siesser and Rogers (1976) reported authigenic gypsum associated with pyrite in West African slope sediments; again these are organic - rich clays characteristically anaerobic in which the sulphate is reduced to sulphide by the action of bacteria (Zobell and Anderson, 1936). Briskin and Schreiber (1978) noted the occurence of authigenic tabular gypsum crystals in marine sediments.

During the Maastrichtian and Paleocene transgressions that laid down the gypsum bearing brackish water Dukamaje (Maastrichtian) and marine Dange (Paleocene) shales, the salts present in the waters were trapped in these sediments. The entrapment of the fluids could be syngenetic (formed at the same time as the sediments) or epigenetic (owe their origin to subsequent infilteration of saline and other waters into already formed rocks)

The oceanic regime that occupied the Sokoto Basin during the Maastrichtian and Upper Paleocene age appears to provide the physical - chemical framework necessary to produce the Ca^{2+} and SO_4^{2-} ions in sufficient concentration to precipitate gypsum in localized sediments.

Calcium and sulphate are major constituents in the gypsum varieties (tables 5.1 - 5.4) and show a similar trend in the gypsum varieties (Figs. 5.6 and 5.9). Their values are highest in the Dange discontinous thin bed variety and lowest in the Dukamaje septarian variety. The concentrations appear to be influenced by paleoenvironment and mineralogy.

The Ca:Mg ratio (table 5.1 - 5.4) is 12 and 14 of calcium to magnesium. This ratio indicates that the fluids that formed gypsum are marine in origin (Davies and Dewiest, 1966). The relative high content of sodium (>1%) in some of the gypsum samples indicates a more marine environment in line with the affinity sodium has for the oceanic environment. The Dange Formation discontinous thin beds of gypsum have high content of sodium (Table 5.1) compared with the brackish water Dukamaje gypsum varieties.

That gypsum is precipitated in the Dukamaje and Dange sediments supposes that the microenvironment favoured the enrichment of the interstitial fluids with respect to Ca^{2+} and SO_4^{2-} ions. Calcium enrichment could be attributed to the episodic influx of the Sokoto Basin bottom water, originating in the Tethys sea, sweeping over the area dissolving calcium carbonate on the sea floor (Discovery Reports, 1936; El-Sayed, 1966; Sverdrup et al., 1967) The dissolution of Planktonic foramineral tests, carbonate rocks and fossils bones in the Sokoto Basin produces an available source of calcium. This could be the reason for the intimate association of gypsum with the vertebrate fossils in the Sokoto Basin.

Berner (1970) strongly supports a continuous diffusion of sulphate into sediments from the overlying sea water. One possible model proposes that the organic - rich sediment has free sulphide produced by the proteolytic attack on organic debris and that the sulphate is produced authigenically

in the interstitial water by sulfide-oxidizing bacteria such as Thiobacillus thiopus.

Arrhenius (1962) first proposed that high organic content in sediments was thought to be responsible for the origin of deep-sea gypsum. Anaerobic conditions will occur when there is an abundance of organic matter deposited on the sea floor. The decomposition of the organic matter removes all available oxygen creating an anaerobic reducing environment of variable thickness in the bottom sediments.

Anaerobic bacteria, which are normally poisoned by oxygen, can then flourish. These bacteria reduce dissolved sulphate consuming the oxygen by metabolism. The sulphur that they liberate combines with, hydrogen from the water to form hydrogen sulphide (H_2S). Dissolved hydrogen sulphide reacts immediately with reactive forms of iron present forming finely disseminated iron monosulphides (Fes). Further transformation of a monosulphide to the disulphide requires the addition of elemental sulphur (Berner, 1970).

Berner (1964a, 1969, 1970) and Goldhaber and Kaplan (1974) have made a thorough analysis of the source of sulphur and other conditions necessary to form pyrite. Iron is supplied by a variety of clastic iron - bearing minerals (Berner, 1964b; Goldhaber and Kaplan, 1974) and clay minerals (Carroll, 1958). The most abundant and readily available source of iron is adsorbed oxide coatings on clay minerals (Gddhaber and Kaplan, 1974).

Obviously not all of the sulphate was reduced to sulphide in the Dukamaje shales or no gypsum would have formed as septarian concretions having ferruginous cores surrounded with gypsum.

As soon as there is introduction of excess dissolved calcium, it combines with the remaining unreduced sulphate ion to form gypsum.

The intimate association of pyrite and gypsum in the septaria variety is unusual. Gypsum is a sulphate and pyrite is a sulphide; however, both appear to have formed in similar anoxic conditions in the shales.

Within this anoxic, organic-rich environment it is probable that organophosphates will form (Summerhayes, 1973; Emsley and Hall 1976) and that these organophosphates will bind with and eventually replace the adjacent shales/clays (Antia and Whitaker, 1978) and form soft phosphatic nodules which can be concentrated in the sediment (Baturin, 1969, 1971). This provides an alternative explanation for the genesis of some phosphorites and phosphatic bonebeds to the traditional explanation of formation in regions of upwelling currents (McKelvey, 1967).

Gravitational compaction due to load of overlying sediments aids in enhancing the concentrations of Ca^{2+} and SO_4^{2-} ions of the interstitial fluid to exceed the solubility product at which gypsum will precipitate. This is supported by fluid inclusion studies of some of the gypsum samples

and the presence of lignite in the Taloka Formation. That the fluid could have been trapped as a result of pressure buildup due to the load of overlying sediments (Sediments load thickness on Dukamaje and Dange Formations are about 457.2 meters and 368.8 meters respectively Parker, 1961) is evidenced by the presence of dominantly primary type of fluid inclusion.

The interstitial fluids occupy the porespace of most of the buried sediments; upon compaction of the sediments, the interstitial fluids are expelled into the associated rocks. In as much as the argillaceous sediment have high porosity and are permeable in their initial state, the expelled fluids will flow in the direction of least resistance, usually into a more porous layer. As long as the fluid can escape, under normal loading conditions and porosity is intercommunicating, hydrostatic pressures will be encountered.

Excessive fluid pressures within sediments are according to Dickinson (1953), dominated by two factors: (a) the compression of the fluids owing to compaction and (b) the resistance to fluid expulsion.
Abnormal pressures are most common in young, rapidly deposited sediments although they are found in rocks of practically all ages. These conditions give rise to "overpressured" reservoirs, whereas the associated shales are termed "undercompacted" or "overpressured."

Interstitial water in under compacted shales, however, should be more saline than that in well-compacted shales, because in the former case a smaller portion of the more saline fluid present in the center of capillaries is squeezed into adjacent sandstones.

Over-pressured shales have high porosity, and their overpressuring is commonly caused by a lack of permeable silt and sand bodies as channels of migration. The combination of high porosity and lack of permeable sands results in geothermal gradient in the overpressured shale, usually higher than that of normally pressured sections above or below. Consequently, overpressured shales frequently show a dogleg in the geothermal gradient.

Lewis and Rose (1970) developed the concept that overpressured shales with very low thermal conductivities act as heat insulators, causing abnormal temperature gradients to exist across them.

The concentration of the inorganic salts is further increased as a result of compaction and ion filtration by charged-net clay membranes. De Sitter (1947) was the first to suggest that some brines may have been concentrated by the "salt sieving" action of clayey sediments which permitted water to escape retaining some of the ions of dissolved salts. The mobility of the hydrated ions depends essentially on their valence and ionic radius (Posokhov, 1966).

The experimental data obtained by Larsen (1967) tend to indicate that the size of hydrated ions influences the rate of ion rejection by a reverse osmosis process and the order should be $Mg^{2+} \geq Ca^{2+} \geq SO_4^{2-} \geq Na^{+} \geq Cl^{-}$.

White (1965) noted that fine-grained sediments are not equally permeable to all ions, and it seems reasonable to expect less mobility for calcium (radius of hydrated ion = 9.6A°) than for sodium (radius of hydrated ion = 5.6A°).

Strontium's ionic radius, however, lies between those of calcium and potassium and Sr^{2+} might be expected to substitute in the clays for both Ca^{2+} and K^{+}.

Both sulphate and bicarbonate exceed chloride in the shale pore waters of high-pressure zones (Weaver and Beck, 1969). Sulphate concentration may double or tripple in shale pore waters when entering a high-pressure zone. The effectiveness of argillaceous rocks as a semipermeable membrane-filtering device has been demonstrated by several investigators (Teorell, 1935; Davies, 1955; Hanshaw, 1962; McKelvey and Milne, 1962; and Young and Low, 1965).

Katz and Ibrahim (1971) proposed a model, based on the compaction of an argillaceous layer between two permeable sand layers. The model indicated a higher fluid pressure to exist in the middle rather than in the upper and lower portion of the model. This means that it takes more time for the water in the middle of the model (shale) to escape

than at the outer boundaries. Katz and Ibrahim's model illustrates the observed increase in porosity associated with under-compacted shales, extreme drop in permeability with confining lithostatic pressure, and entrapment of high interstitial fluid pressure in the shales. As a result of this pressure build up and temperature rise due to overpressuring, there is a corresponding increase in solubility of the dissolved salts in the high interstitial fluid pressure shales.

On releasing the excess pressure buildup as a result of fracturing and pore fluid leakage, the solubility of the interstitial fluid decreases and consequently, the inorganic precipitation of calcium sulphate (gypsum) when the salinity of brine exceeds 10% occurs and are deposited in zones of weaknesses.

Graf et al (1966) indicated that there should be a 1:1 Loss of Ca^{2+} and SO_4^{2-} ions from solutions when SO_4^{2-} is precipitated in the sediments as anhydrite or gypsum.

Lack of widely traceable gypsum beds indicate that gypsum was not chemically precipiated from a standing body of brine. The gypsum crystals probably grew within the sediment. The septarian concretions, the thin discontinous beds and veins and the minute crystals of gypsum in the study area are characterized by fibrous crystals.

The gypsum of the septarian concretions may have nucleated around the iron core while the selenite crystals occur where the conditions favoured such crystal growth.
The form in which the gypsum now occurs is a result of the particular conditions (structural, chemical etc.) that prevailed at the time of the formation.

Chapter 7

7.0 ECONOMIC IMPORTANCE OF GYPSUM

7.1 Introduction

The largest use for gypsum is based upon the unique property which calcium sulphate has of readily giving up or taking on, water of crystallization. With the application of a moderate amount of heat in a process known as calcination, gypsum is converted to plaster of Paris (the hemihydrate of calcium sulphate, $CaSO_4.\frac{1}{2} H_2O$) which when mixed with water will set as the calcium sulphate returns to the dihydrate form. This semi finished product, usually called stucco, is then manufactured into a large variety of plasters, wallboard and block for construction use, or into plasters for industrial applications. About 70% of the gypsum used in the United States is calcined for these purposes.

Uncalcined uses of gypsum are principally as a retarder for Portland Cement, as a soil conditioner, as a mineral filler, and other industrial applications. At present, the largest users of gypsum in Nigeria as in the rest of the third world are manufacturers of cement.

The gypsum required for cement manufacture can be relatively impure and mixed with clay, so that mineral beneficiation poses only minor problems, if at all. However the lower the quality, the higher the tonnage of imput gypsum material to be handled and vice-versa.

Gypsum for chalk, ceramic moulds and medical casts in the form of plaster of paris, is required in much smaller quantity than for cement but must be of much higher purity and of course costs more.

Impurities in gypsum can be considered of two main types, physical and chemical. Physical impurities consist of such minerals as clay and quartz which occur as discrete grains and which were either deposited with the gypsum or were incorporated during mining operations. Chemical impurities are mainly evaporite minerals that formed at the same time and by the same processes as the gypsum and include halite, sylvite and epsomite etc.

Most commercially used gypsum contain 10 - 15% impurities but the amount of impurity that can be tolerated depends on:

(a) the nature of the impurity
(b) the product to be manufactured

Cement producers can tolerate relatively large amounts of clay impurities (since clay is a component of raw material mix). Perhaps even up to 30%, but even 2% of halite would have serious deleterious effects. Conversely, a ceramic manufacturer requires gypsum virtually free of all impurities.

7.2 Economic Importance

The largest use of gypsum in Nigeria and in the rest of the third world is in the manufacture of cement where it is used to retard the setting time of cement. In North America and other developed countries of the world, gypsum is used mainly as wallboards for building construction, landplaster/fertilizer for agriculture. Other industrial applications include use as a mineral filler in the manufacture of paint, rubber, paper, cotton and food, (Bates, 1969; Dineley, Hawkes, Hancock and Williams, 1976). These industrial applications consume 60,000 - 65,000 tonnes of gypsum annually in Nigeria (Ayodeji, 1986, Oral communication).

With available potential for gypsum production and a policy geared towards industrial diversification, gypsum in Nigeria could be used as follows:

(This information is derived from "Gypsum and Anhydrite" by Frank, C. Appleyard in Industrial Minerals and Rocks).

Three major groups of gypsum end-uses are generally recognised:

(a) construction - all those products used directly as construction or building material;
(b) industrial - all those products used in the manufacture or

processing of other materials; and (c) agricultural.

(a) Construction Group: Gypsum products are used in the building and construction industry as covering and finishing materials. It is in the covering of a structural frame, or in the finishing of a masonry wall, that gypsum products find their greatest utilization.

The basic characteristics of gypsum which give it value in the manufacture of construction materials are its fireproofing qualities, the versatility which comes from being able to use it in many forms, and its economy of application.

It is used entirely in its calcined form, with stucco being formulated and/or manufactured to make plasters, block or tile, and wall boards.

Gypsum is non combustible and is one of the most effective fireproofing materials known, a property which has its basis in the combined water content of the product. When the hemihydrate form of gypsum (stucco) is rehydrated to make a covering material it reverts back to the dihydrate, so that when heat from fire hits a plaster wall (or gypsum wall board) it again begins to lose its combined water in the form of steam which dampens the transmission of heat.

The versatility of gypsum is also a major advantage. Plaster can be used in many forms as can wall board and cast block, or tile, and by the use of different calcining methods and/or additives, physical properties can be varied

to meet a wide range of requirements. These characteristics give the architect and builder several choices of design, and enable maximum flexibility in producing a structure to meet the desired specifications.

Plastered walls and ceilings, which in the early 1900's were widely used in the United States, have to a considerable extent given way to "dry wall" construction employing gypsum wall board. However, one coat "Veneer-type" Plasters have been developed for application over 4-ft-wide plaster base.

The use of gypsum partition and roof tile has declined in recent years, although they are still preferred for some applications. Poured gypsum roof decks are often specially formulated and plaster slurry is pumped in place over a wallboard base to provide a completely fireproof roof. Other construction uses include water-resistant sheathing, plaster base board, and fireproofing building steel.

(b) Industrial Group: Industrial gypsum can be divided into 3 broad categories:, Calcined, anhydrons and uncalcined, each of which utilizes distinct properties of the mineral.

Calcined industrial Gypsum: The largest use of calcined gypsum by industry is for mold making in the manufacture of sanitary ware, pottery, metal casting and decorative objects. Molding plaster is made from high purity gypsum (95%) at selected locations and is marketed on the basis of its water demand, strength, white colour, setting time, and closely controlled expansion/shrinkage qualities. It may be made from either beta or alpha hemihydrate, or a

combination of the two.

The qualities which make gypsum an excellent material for mold making also contribute to its use as a casting plaster, and gypsum casts are widely used by many industries. Included in this category is dental plaster, a highly specialized material made for dental and orthopedic work which is accelerated to produce a setting time of only 3 to 4 minutes.

Another important industrial use is as a cementing agent in well drilling, particularly in the oil and gas industries. This usually is formulated from low consistency alpha hemihydrate (Pressure calcined) and is very effective for sealing off porous or cavernous rock strata often encountered in the drilling of oil, gas or water wells.

A small but growing use of high density, high strength plasters is in the maintenance of floors, and for quick patching high use surfaces (such as highways or airport runways) that cannot be closed down for more conventional repairs. In both this and the well drilling application, gypsum plaster may be mixed with other cementitous materials to obtain optimum results.

Anhydrous Gypsum: There are several products made from calcium sulphate with no water of crystallization, i.e. from "second settle" stucco made when the calcining temperature has been carried up to about 400°F. This material, often

referred to as "Soluble anhydrite," has a high affinity for water, making it, an efficient drying agent, and is sold in various particle size gradations for use as a desiccant in laboratory and commercial applications.

Also, a finely ground soluble anhydrite is used as a carrier for certain types of insecticides which must remain absolutely dry to maintain their toxicity.

When the calcination process is carried to a temperature of 900°F, a dead-burned or insoluble anhydrite product is formed which is used for industrial filler appplications or for the manufacture of Keenes cement. Dead-burned gypsum is preferred for some filler uses over uncalcined gypsum, particularly where the temperature of the product in which the filler is used may go over 120°F, the point at which natural gypsum begins to release water of crystallization. Also, dead-burning usually produces a white product, and is preferred where colour (i.e. lack of colour) is important. The principal uses for dead-burned fillers are as a source of calcium in food products, and for yeast and beer processing. They also serve as a diluent or extender in such compositions as pills, rubber, artificial wood, plastics, paper, and pigments.

Uncalcined industrial gypsum: This category sometimes referred to as "raw" gypsum, includes the largest volume use for the mineral outside that for wallboard, i.e. as a retarder for portland cement. Calcium sulphate compounds

control the setting time of portland cement. They also will control the rate at which the cement paste develops strength, and the shrinkage of cement products during drying.

The amount of gypsum (or gypsum/anhydrite blend) used depends upon: (1) the SO_3 content of the gypsum, (2) the type of cement, and (3) the mix of raw materials used formake the cement clinker. In practice, the amount of gypsum used will very from 4 to 6%, averaging approximately 5% by weight of finished cement. It is mixed with the clinker as it is fed into the grinding mill, and the two materials are then ground together.

Terra alba is a term applied to high purity, finely ground raw gypsum. Usual specifications call for a 95% - 325 mesh, very white colour material whose exact specifications as to colour and particle size vary with the end use. When used in bread and bakery products, or in pharmaceuticals, terra alba must meet food chemicals codex specifications, and generaly is made from +97% pure gypsum. In one of its more interesting applications, the presence of calcium sulphate fillers in water for the brewing of beer has been found to be very desirable because it assist in the development of proper yeast fermentation, helps the settling out of the yeast, and aids in the clarification of the beer.

Glass batch gypsum is the uncalcined mineral ground and sized to be comparable to the fineness of sand, and is used in the manufacture of container glass. The gypsum, which must be fairly dust free, is thoroughly mixed with

the other ingredients and performs three functions: as an oxidizing agent, as a fining agent; and it aids in removal of scum.

A minor use for gypsum and anhydrite is for its sulphur values. The most common approach has been to bring about the thermal dissociation of calcium sulphate to produce calcium oxide and sulphurdioxide, converting the sulphurdioxide into sulphuric acid and the calcium oxide into cement clinker (Bedwell, 1952; Hull, 1957). Another use of gypsum for its sulphur value is in the production of ammonium sulphate.

(c) Agricultural Group: The oldest use of gypsum in the United States, dating back to pre-revolutionary wartimes, is as a soil conditioner. Generally called "Land Plaster," this material can be produced from either gypsum or anhydrite, and is usually ground in an air-swept mill to a fineness ranging between 75 and 90% - 100 mesh. The principal purposes for applying gypsum on land are to:

i) improve the structure of physical conditions of soil by breaking up compacted clays; and by increasing porosity, aid in drainage.

ii) supply neutral, soluble calcium for peanuts and other crops.

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iii) Supply quickly available sulphate sulphur.

iv) Correct high sodium irrigation waters.

v) Conserve nitrogen in manure

vi) Neutralize sodium compounds in alkali soils.

vii) Clear up muddy farm ponds.

viii) Stimulate soil micro-organisms.

In other agricultural related uses, ground calcium sulphate, either in the dihydrate or anhydrous form, is often added as an ingredient in formulating feeds and feed premixes for beef cattle, diary cows, and sheep. In this use, it supplies total sulphur requirements in safe, easy to mix form, increases the efficiency of non protein nitrogen in urea feeds, is an ideal supplement for ensilage enhancers, and is an effective regulator for self-feeding on the range.

Based on the result of the chemical analysis of the gypsum from Dukamaje and Dange Formations, beneficiation is the watchword if it is to be used for chalk, ceramic moulds and medical casts. For cement manufacturing, increased tonnage gypsum is required.

7.3 Potential for future production

Presently, Nigeria's gypsum requirement for use in the cement industry and other applications is in short supply. Table 7.1 shows a breakdown of Nigeria's gypsum requirement for local manufacture of cement. It shows that possible local sourcing of gypsum for cement has a shortfall of about 90%, consistent to 1990. From Table 7.2, it becomes clear that Nigeria's total gypsum needs up to the year 2,000 would be 4 - 6 million tonnes depending on quality. These figures could serve as a target or guide to both policy makers and those burdened with the search for local sourses of gypsum.

Current efforts of the Raw materials Research and Development Council (RMRDC) of Nigeria with respect to

Table 7.1 Nigeria's Gypsum Requirement for Cement Production (Million tonnes)

Year	Limestone for Cement	Gypsum for Cement	Other Gypsum Uses	National Total Gypsum Needed	Possibly Locally Available	Shortfall
1988	4.043	0.202	0.065	0.267	0.03	0.237
1989	4.9	0.245	0.065	0.310	0.03	0.280
1990	5.56	0.278	0.065	0.343	0.03	0.313
Total 1988-90	14.50	0.725	0.195	0.920	0.09	0.83

(Courtesy of the Cement Manufacturers Association of Nigeria in Ike, Woakes and Abaa, 1989).

Table 7.2 Nigeria's Projected Gypsum Requirement to the Year 2000 (million tonnes) with Bias for Quality

Year	Gypsum for Cement	Other Gypsum Uses	Total	Total if 80% Pure	Total if 66% Pure
1988-1990	0.725	0.195	0.920	1.150	1,380
1991-2000	2.420	0.650	3.070	3.838	4.605
1988-2000	3.145	0.845	3.990	4.988	5.985

(Courtesy of the Cement Manufacturers Association of Nigeria in Ike, Woakes and Abaa, 1989)

local sourcing of gypsum for cement and other industries can be said to be in the right direction with the proper execution of the programme. This will go along way in preserving our foreign reserve and increasing employment opportunities by boosting our local industries.

Chapter 8

8.0 SUMMARY AND CONCLUSION

Gypsum occurs in the Sokoto Basin in the brackish water Dukamaje and the marine Dange Formations of Maastrichtian and Upper Paleocene age respectively.

Four modes of gypsum occurence recognised in this study are the discontinous thin beds and veins, the selenite, the septaria and the minute crystals of gypsum.

Clay mineral determinations for the Dukamaje and Dange shales indicate lack of dominance of kaolinite over chlorite and illite. The lack of dominance of kaolinite, intimate association of gypsum with organic relics and the fact that the sum of the values of soda and potash exceeds that of silica (see Table 5.1 - 5.4) indicates alkaline reducing environment for the deposition of gypsum in the Dukamaje and Dange Formations.

Fluid inclusion studies of some samples of gypsum show primary inclusions filled with only liquid, indicating that they probably grew at a temperature less than 40^{o}C (Roedder 1979). This suggests a shallow phenomena for the formation of gypsum in the study area.

Chemical analysis of the gypsum from the Dukamaje and Dange Formations shows that the Ca: Mg ratio is 12 and 14 to one. This ratio indicates that the fluid that formed the gypsum is marine in origin (Davies and Dewiest, 1966).

Lack of a widely-traceable gypsum bed indicates that the gypsum was not chemically precipitated from a standing body of brine. The gypsum crystals are probably emplaced diagenetically within the sediments. This is brought about by continous diffusion of SO_4^{2-} ions into sediments from the overlying seawater and the dissolution of carbonate particles which releases Ca^{2+} ions, thus increasing the concentrations of Ca^{2+} and SO_4^{2-} ions in the sediments. Gravitational compaction, evidenced by the presence of two lignite seams in the Taloka Formation at Taloka village, further increases the Ca^{2+} and SO_4^{2-} ions concentrations by ion filteration by charged - net clay membranes. Both the Ca^{2+} and SO_4^{2-} ions combined to form gypsum once the solubility product of this mineral was exceeded. The fact that only gypsum forms indicates that the porewaters were concentrated with respect to Ca^{2+} and SO_4^{2-} ions.

On the basis of the proposed origin for the gypsum in the study area of the Sokoto Basin, it is unlikely to find a large and extensive gypsum deposit of the evaporite type in the area. However, the Dange Formation looks much more promising than the Dukamaje Formation for the occurence of gypsum because of its depositional environment and the chemical composition of the gypsum which both indicate a more marine environment than that of the Dukamaje.

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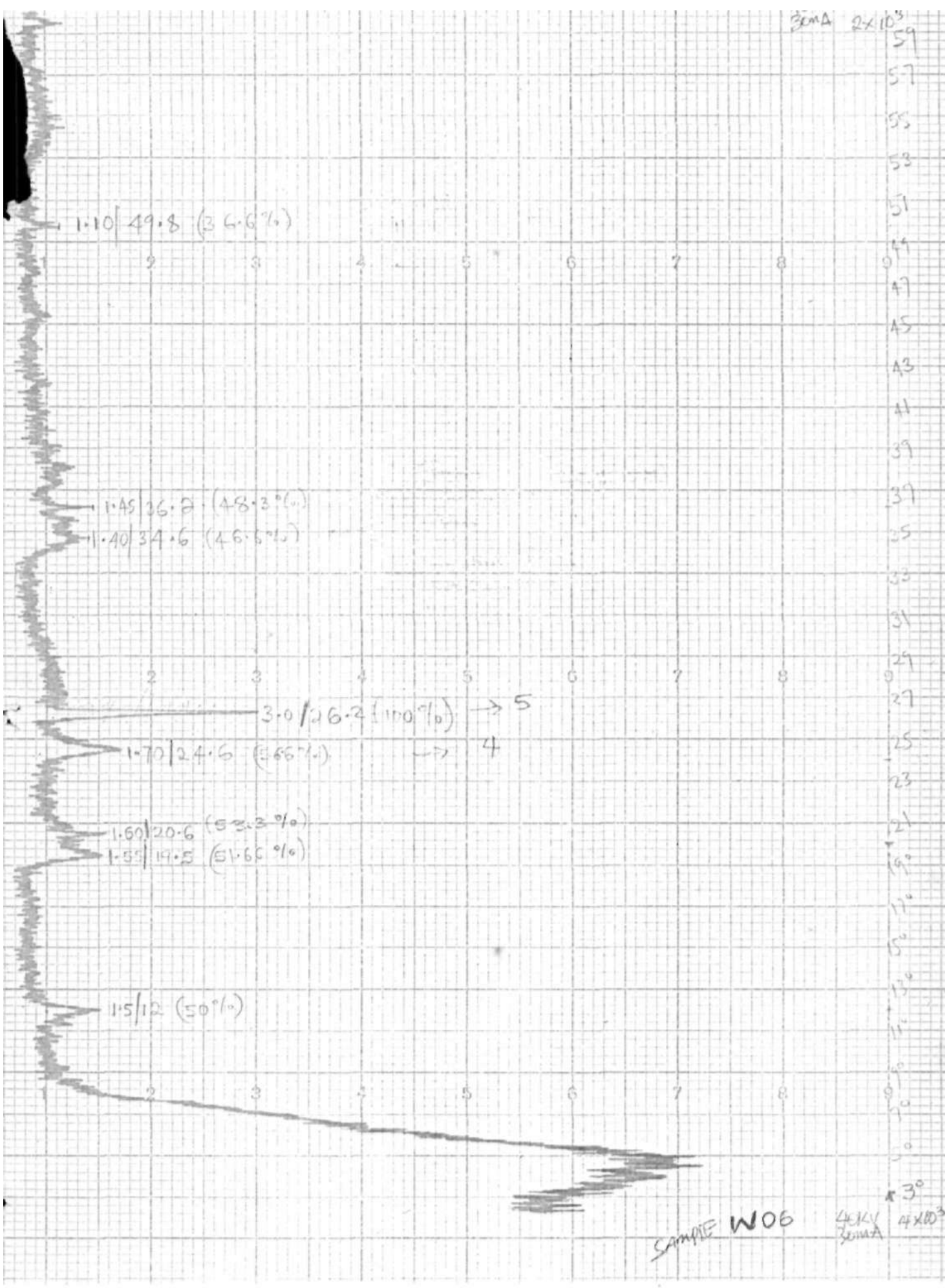
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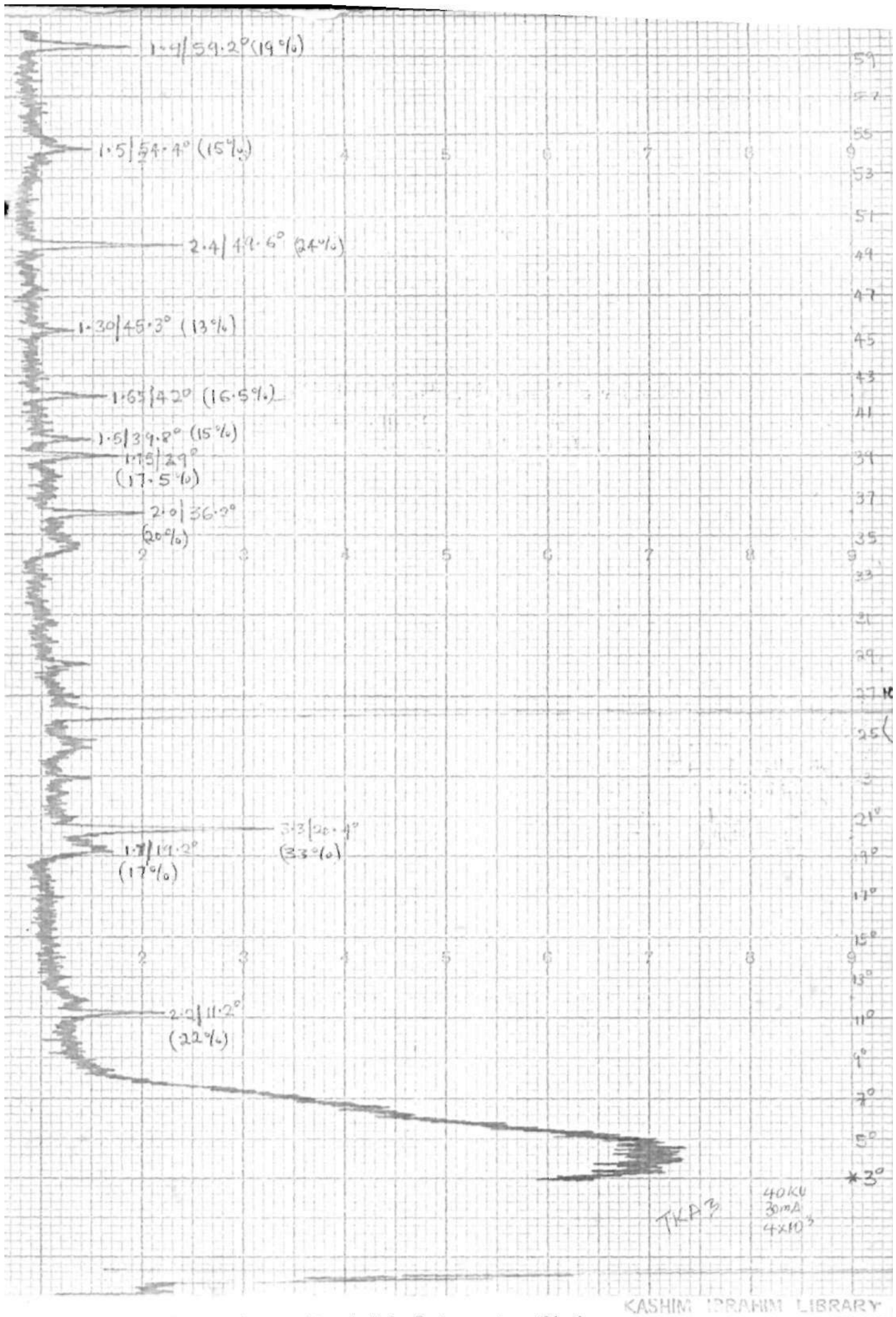
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Appendix 1 (a) Dange Shale



Appendix 1 (b) Dukamaje Shale

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