CHAPTER-VI
CLAY MINERALOGY

6.1. INTRODUCTION

Clay mineral study is considered as one the most important tools for the provenance analysis, and also to draw inference on burial history of the sedimentary formation. Clay minerals are hydrous aluminium phyllosilicates occasionally with variable amount of iron, magnesium, alkali metals, alkaline earth and cations. Clay minerals are common weathering products (including weathering of feldspars), and low temperature hydrothermal alteration products. Clay minerals are very common in fine grained sedimentary rock such as shale, mudstone and siltstone.

Clay minerals are mainly detrital inorganic which suggest the source rock character, and alteration in fluvial and sub-aerial environment. The origin of clay minerals is through three processes:-

1. Detrital
2. Inheritance

The alteration of clay minerals commonly occurs during weathering, sedimentation, and diagenesis. The variation in clay mineralogy may also result from the source rock modification related to tectonism (Chamley, 1989), but can also occur due to the intensity of weathering of the source rock or change in depositional conditions coupled with climatic conditions (Schieber, 1986).
6.2 STUDY OF CLAY MINERALS

The clay minerals are very important constituents, and form 25-30 % of terrigenous fraction of sedimentary rocks. They represent the stable secondary products of decomposition of other alumino-silicates. Their formation is dependent on the chemical character of the parent material, and the physico-chemical environment in which alteration of the mineral takes place.

The clay minerals are hydrous aluminous silicate belonging to kaolinite, montmorillonite or illite groups. Fine grained chlorite and vermiculites are also generally included in clay minerals.

A particular type of clay mineral is directly related to macro to micro climate factors, organic decomposition and type of concentration of inorganic ions in the genetic environment. Clay minerals indicate source rock character, and alteration in fluvial and subaerial environments (Weaver, 1960). Earlier, all clay minerals present in sandstones had been regarded as detrital. Clays may also filter into sandstones, move downwards by pore waters from muddy interbeds. Extensive infiltration drastically alters the texture of the sediment, and decreases the original texture, and compositional maturity. Tucker (1988) and Blatt (1992) state that clay minerals can also enter sandstones by mechanical infiltration from overlying soils. Such clays adhere to detrital sand surfaces and are oriented parallel to those surfaces.

In most of the sandstones the volume of authigenic clay minerals is minor, and not more than 1- 2 % of the rock. Illite and kaolinite are the most common authigenic clays in sandstones, but montmorillonite-chlorite also occur.
The clay mineral suites provide reliable indication of palaeogeographic evolution of exposed land masses, where tectonic changes are noticeable, and diagenetic changes occur (Chamley, 1989). The alteration of clay minerals commonly occurs during weathering, sedimentation, diagenesis and metamorphism (Choudhri and Kalitha, 1985). The paleoclimatic and palaeogeographic conditions are also deduced from clay mineral variations (Diester – Haass et al., 1993). They are also useful in determining nature of ancient environments. Clay minerals are potentially promising materials for documenting and resolving wide spectrum of different genetic environments and reactions.

Clay minerals are commonly detrital in nature, and clay mineralogy is a reliable tool for identification of palaeoenvironmental conditions (Chamley, 1989). In recent years much attention has been given to study clay minerals because they are excellent indicators of depositional environments, diagenesis of sediments and their burial history. They are also reliable indicators of palaeogeographic evolution of the exposed land masses where tectonic changes are noticeable. Palaeoclimatic conditions may also be deduced from clay mineral variations.

6.3 X-RAY DIFFRACTION TECHNIQUES FOR MINERALOGICAL STUDIES

Clays are fine grained, less than 2 micrometer in size; and so require special analytical techniques- standard X-ray diffraction, electron diffraction method, Differential Thermal Analysis (DTA), and various spectroscopic methods such as infrared spectroscopy and EDS. In the present study, X-ray diffraction technique is used to study the clay mineralogy.
X-ray is an electromagnetic radiation of extremely short wavelength that the properties of ionizing a gas when passing through it and of penetrating various thickness of solids. The range of wavelength extends from the extreme ultraviolet into the gamma ray region that is from $10^{-7}$ to $10^{-9}$ centimetres. X-ray arises wherever cathode rays encounter solids.

X-ray diffraction is versatile, non-destructive method that reveals detailed information about chemical composition, crystallographic and microstructure of all type of natural and manufactured products. X-ray diffraction methods are commonly used for routine identification of mineral components in soil, sediments and rocks, but particularly for clay minerals in sediments and soils. X-ray diffraction technique is the most sought after in view of its versatility and veracity.

### 6.4 QUALITATIVE MINERALOGY

The qualitative mineralogy of the clay samples was determined with the standard interpretation procedures of XRD data (Biscay, 1965; Grim, 1968; and Brindley and Brown, 1980). Minerals identified and studied in DNR/58, 60 & 61 cores samples include Illite, Kaolinite, Chlorite and Montmorillonite (Tables 6.1, 6.2 & 6.3 and Figs. 6.1-6.52).

#### 6.4.1 Illite

A major clay mineral illite is present in all samples, and found to be a dominant mineral in these studies. Illite may be formed by the alteration of mica minerals. The presence of illite is confirmed by XRD analysis.
The X-ray diffraction response to illite often indicates the diagenetic, and low-grade metamorphic history of sedimentary rocks (Weaver, 1960; Kubler, 1968; Dunoyer de Segonzac, 1970; Frey, 1970; Gill et al., 1977; and Stalder, 1979). Kaolinite may be changed into illite by diagenesis after burial. The main requirement for the diagenetic alteration of kaolinite to illite is the availability of adequate potassium (Keller, 1970).

Illite has formed mainly from three major processes i.e. i) by leaching degradation of illite material to formation of expandable layer (opening of illite), ii) crystallisation of illite in the bottom part of the weathering zone and iii) by wet and drying transformation of smectite into illite layers (Sarodon and Eberl, 1984).

During weathering, granitic rocks tend to produce more kaolinite than other types. Due to subsequent diagenesis the kaolinite may be altered to illite under alkaline conditions, especially in a K-rich, tropical chemical weathering environment (Boles and Franks, 1979; Hower, 1981; and Khoshroo and Devaraju, 1990). In addition, granitic source rocks also produce minor quantities of illitic clays (Barshad, 1966; Barnhisel and Rich, 1967; Chesworth, 1979; Fritz, 1988; and Cox and Lowe, 1995a). Illite is one of the early products of weathering of feldspathic and micaceous rocks, and is stable under moderate palaeoclimatic conditions (Chaudhri and Kalitha, 1985).

The maximum intensity shown in d-spacing values reflected at 3.3 Å, 5 Å and 10 Å for both glycolated and untreated samples of DNR-58 core (Figs. 6.1-6.12), DNR-60 core (Figs. 6.13-6.32) and DNR-61 core (Figs. 6.33-6.52).
6.4.2 Kaolinite

Kaolinite is considered to be abundant in regions of intense weathering under tropical conditions (Biscay, 1965). According to Whitehouse and McCarter (1958), the low salinity of water is favourable for settling of Kaolinite compared to Smectite, and the estuarine conditions favour the deposition of Kaolinite (Narayana and Suresh Kumar, 1994). Thus, in a straight approach, the presence of low amount of kaolinite may not favour intense weathering, and estuarine condition of deposition of clay minerals. However, the burial diagenesis may drastically alter the kaolinite occurrence in the sedimentary rocks.

Kaolinite is another component in all the samples. It is represented by d-spacing values at 3.57 Å and 7.15 Å for both glycolated and untreated samples. Its presence is confirmed in the basal reflections at 7.18 Å, 3.58 Å and 7.17 Å in DNR-58 core (Figs. 6.3-6.5 & 6.9-6.10), DNR-60 core (Figs. 6.13-6.23 & 6.28-6.30) and DNR-61 core (Figs. 6.33-6.35, 6.39-6.40, 6.42-6.46 & 6.51-6.52).

6.4.3 Chlorite

Chlorite is commonly found in igneous rocks as an alteration product of mafic minerals such as pyroxene, amphibole and biotite. In this environment chlorite may be a retrograde metamorphic alteration mineral of existing ferromagnesian minerals or it may be present as a metasomatic product via addition of Fe, Mg or other compounds into the rock mass. Chlorite is a common mineral associated with hydrothermal ore deposits, and commonly occurs with epidote, sericite, adularia and sulphide minerals. Chlorite is also a common metamorphic mineral, usually indicative of low-grade metamorphism. It is the diagnostic species of the zeolite facies, and of lower greenschist facies. It occurs in the quartz, albite, sericite,
chlorite garnet assemblage of pelitic schist. Within ultramafic rocks, metamorphism can also produce predominantly clinochlore chlorite in association with talc.

The d-spacing values of chlorite are shown in both untreated and glycolated samples at 3.5Å, 4.7Å, 7Å & 14Å. It is also reflected at the values of 3.7 Å, 4.07 Å, 4.1 Å, 4.2 Å & 4.3 Å in DNR-58 core (Figs. 6.1-6.3, 6.5-6.9 & 6.11-6.12), DNR-60 core (Figs. 6.13-6.15, 6.19-6.22 & 6.25-6.27) and DNR-61 core (Figs. 6.33, 6.35-6.37, 6.39, 6.41 & 6.43-6.45, 6.47, 6.49-6.50 & 6.52).

### 6.4.4 Montmorillonite

Montmorillonite is a very soft phyllosilicate group of minerals that typically form in microscopic crystals, forming clay. Montmorillonite is the main constituent of the volcanic ash weathering product, bentonite. Chemically it is hydrated sodium, calcium aluminium magnesium silicate hydroxide (Na, Ca)\(_{0.33}\) (Al, Mg)\(_2\) (Si\(_4\)O\(_{10}\)) (OH)\(_2\) \(n\)H\(_2\)O. Potassium, iron, and other cations are common substitutes, and the exact ratio of cations varies with source rocks. It often occurs intermixed with chlorite, muscovite, illite, cookeite and kaolinite.

Many ethylene glycol treated samples show the presence of montmorillonite clay. The reflected d-spacing values of montmorillonite are at 4.3Å, 5.7Å, 8.5Å, 13Å, 15Å & 17Å in DNR-58 core (Figs. 6.2, 6.4, 6.10), DNR-60 core (Figs. 6.16, 6.18, 6.20, 6.23, 6.26 & 6.28) and DNR-61 core (Figs. 6.34, 6.36, 6.40, 6.42 & 6.52).
6.5 DISCUSSION AND INTERPRETATION

The clay minerals are reactive response of minerals to energies characterising certain environments during the time of deposition. But in some cases, they may not be in-situ in occurrence. They may be transported (detrital) or may undergo diagenetic alteration. Detrital inheritances, transportation of existing clay minerals and neoformation or authigenesis are the three main processes depending on space and time, to account for the genesis of the clay minerals in sedimentary rocks.

Illite is by far the most dominant species of clay minerals in argillaceous sedimentary rocks (Grim, 1968). Illite is favoured by an alkaline environment, and low K-aluminous mica like mineral can form during burial metamorphism (Velde and Hower, 1963) or as early product under immediate conditions (Meunier, 1980). A non-acidic condition appears favourable and possibly essential for the genesis of illite (Keller, 1956). Weaver (1960) states that this mineral is commonly found in fluviatile and near shore environments although he agrees that it can occur in abundance in all other environments.

A typical chemical reaction in near neutral pH conditions that can result in the precipitation of authigenic clay is –

\[
2\text{Al} (\text{OH})_3 + 2\text{H}_4\text{SiO}_4 \rightarrow \text{Al}_2\text{SiO}_2 (\text{OH})_4 + 5\text{H}_2\text{O}
\]

Aluminium Silica in Solution Kaolinite Water

in solution

Addition of appropriate amounts of metallic cations can result in the precipitation of the other clay minerals and chlorite: K\(^+\) for illite, Na\(^+\) and / or Ca\(^{2+}\)
for montmorillonite, and $\text{Mg}^{2+}$ and / or $\text{Fe}^{2+}$ for chlorite. It is assumed that adequate amounts of alumina and silica are available. All reactions in the sedimentary environments are solution-precipitation reactions; solid-solid transformations can occur only at metamorphic temperatures and pressures (Blatt, 1992).

In marine influence environment, kaolinite is either transformed into illite or chlorite during diagenesis (Mankin, 1970). Presence of kaolinite indicates an environment of strong leaching and removal of $\text{Ca}^{2+}$, $\text{Na}^{2+}$, $\text{Mg}^{2+}$, K and Fe, possibly SiO$_2$ and addition of $\text{H}^+$ (Kushwaha and Singh, 1996). Kaolinite is possibly derived from pre-existing rocks as a product of weathering in acidic environment where drainage system was good.

The presence of illite, chlorite and kaolinite reflects variations in source materials. Kaolinite generally forms in warm and humid climatic zones with good drainage, and is normally deposited as a weathering product of granites and schistose rocks. Chlorite is possibly derived from metamorphic rocks of green schist facies and or is a weathering product of igneous rocks (Das and Bora, 1992).

Chlorite forms on marine diagenesis, particularly in lagoon or near-shore marine environments where river sediments bearing iron, derived from weathering zone enter the sea. It is also common in marine sands that have received contributions from the basic igneous rocks, and forms on diagenesis, deep burial, and metamorphism. Much of it is simply detrital derived from older schist, phyllites etc. It possibly formed to a small extent in soils by weathering of micas and montmorillonite (Folk, 1980).

The presence of illite, and kaolinite suggest a possibility of admixture of marine water in the continental or near shore environment which ultimately resulted
in the transformation of some kaolinite into illite. The occurrence of kaolinite along with illite and the chlorite is probably due to diagenetic changes that take places slowly and continuously.

According to Millot (1970), illite and chlorite are constituents of near shore marine environment. The high concentration of K, and Mg in sea water is a favourable factor for the formation of illite and chlorite.

Bhattacharya (1984) on the basis of clay mineral study of oil field areas of Assam, states that the Barail sediments mainly contain kaolinite, illite, and chlorite and were probably deposited in a transitional environment preferably under deltaic and or estuarine conditions. The Badami sediments deposited at the margin of the basin near Deshnur contain illite, kaolinite and chlorite, and are of dominantly fluviatile, in which weathering effects are very well impressed upon the sediments.

The abundance of illite in Badami sandstones and shale suggests that the detrital kaolinite clay has been transformed into illite in burial diageneis in alkaline conditions.

A warm to moderately warm climate, and a continental area with sufficient drainage where precipitation favours the weathering of the source rocks that produce more kaolinite (Millot, 1970).

Grim (1968) provides definite evidence that some illite, and chlorite form quickly when fresh water sediments enters the marine environment. The high concentration of K and Mg in sea water is a favourable factor for the formation of some illite. The abundant component of illite with kaolinite indicates a near shore marine environment in a localized area. Chamely (1989) also reported kaolinite,
illite, chlorite and montmorillonite minerals in the clay from other river sediments. Occurrence of kaolinite along with illite and smectite indicate that diagenetic changes are very slow, and seem to be a continuous process (Choudhri and Grewal, 1988).

6.6 CONCLUSION

X-ray diffraction analyses are carried on selected representative samples of 7 from DNR/58, 10 samples from DNR/60 and 10 samples from DNR/61 (on a set of untreated, and glycolated). The study focuses on the evolution of climate variability by using the clay mineralogy. Clay minerals identified in the core samples of Deshnur area are illite, kaolinite, chlorite and montmorillonite as shown in the figures (Figs. 6.1-6.52). The X-ray diffraction studies of glycolated samples reveal the presence of expanding clay mineral (Montmorillonite) in all the treated samples of DNR-58, 60 and 61 cores.
Fig. 6.1 X-Ray Diffraction patterns of clay minerals DNR-58, Depth 171.45m

Fig. 6.2 X-Ray Diffraction patterns of clay minerals, glycolated of DNR-58, Depth 171.45m

i.e., I-Illite, Ch-Chlorite, Mon-Montmorillonite
Fig. 6.3 X-Ray Diffraction patterns of clay minerals DNR-58, Depth 177.10m

Fig. 6.4 X-Ray Diffraction patterns of clay minerals, glycolated of DNR-58, Depth 177.10m i.e., I-Illite, Ch-Chlorite, Ka-Kaolinite, Mon-Montmorillonite
Fig. 6.5 X-Ray Diffraction patterns of clay minerals DNR-58, Depth 181.40m

i.e., I-Illite, Ch-Chlorite, Ka-Kaolinite,

Fig. 6.6 X-Ray Diffraction patterns of clay minerals, glycolated of DNR-58, Depth 181.40m
Fig. 6.7 X-Ray Diffraction patterns of clay minerals DNR-58, Depth 181.70m

Fig. 6.8 X-Ray Diffraction patterns of clay minerals, glycolated of DNR-58, Depth 181.70m

i.e., I-Illite, Ch-Chlorite
Fig. 6.9 X-Ray Diffraction patterns of clay minerals DNR-58, Depth 181.70m

Fig. 6.10 X-Ray Diffraction patterns of clay minerals, glycolated of DNR-58, Depth 181.70m

i.e., I-Illite, Ch-Chlorite, Mon-Montmorillonite, Ka-Kaolinite
Fig. 6.11 X-Ray Diffraction patterns of clay minerals DNR-58, Depth 181.40m

Fig. 6.12 X-Ray Diffraction patterns of clay minerals, glycolated of DNR-58, Depth 187m

i.e., I-Illite, Ch-Chlorite
Fig. 6.13 X-Ray Diffraction patterns of clay minerals DNR-60, Depth 66.10m

Fig. 6.14 X-Ray Diffraction patterns of clay minerals, glycolated DNR-60, Depth 66.10m

i.e., I-Illite, Ch-Chlorite, Ka-Kaolinite
Fig. 6.15 X-Ray Diffraction patterns of clay minerals DNR-60, Depth 75.40m

Fig. 6.16 X-Ray Diffraction patterns of clay minerals, glycolated of DNR-60, Depth 75.40m

i.e., I-Illite, Ch-Chlorite, Ka-Kaolinite, Mon-Montmorillonite
Fig. 6.17 X-Ray Diffraction patterns of clay minerals DNR-60, Depth 83.50m

Fig. 6.18 X-Ray Diffraction patterns of clay minerals, glycolated of DNR-60, Depth 83.50m

i.e., I-Illite, Ka-Kaolinite, Mon-Montmorillonite
Fig. 6.19 X-Ray Diffraction patterns of clay minerals DNR-60, Depth 92.00m

Fig. 6.20 X-Ray Diffraction patterns of clay minerals, glycolated of DNR-60, Depth 92.00m
i.e., I-Illite, Ka-Kaolinite, Mon-Montmorillonite, Ch-Chlorite
Fig. 6.21 X-Ray Diffraction patterns of clay minerals DNR-60 core Depth 140.10m

Fig. 6.22 X-Ray Diffraction patterns of clay minerals, glycolated of DNR-60, Depth 140.10m

i.e., I-Illite, Ka-Kaolinite, Ch-Chlorite,
Fig. 6.23 X-Ray Diffraction patterns of clay minerals DNR-60, Depth 212.40m

i.e., I-Illite, Ka-Kaolinite, Mon-Montmorillonite

Fig. 6.24 X-Ray Diffraction patterns of clay minerals, glycolated of DNR-60, Depth 212.40m

i.e., I-Illite, Ka-Kaolinite, Mon-Montmorillonite
Fig. 6.25 X-Ray Diffraction patterns of clay minerals DNR-60, Depth 217.75m

Fig. 6.26 X-Ray Diffraction pattern of clay minerals, glycolated of DNR-60, Depth 217.75m

i.e., I-Illite, Mon-Montmorillonite, Ch-Chlorite
Fig. 6.27 X-Ray Diffraction patterns of clay minerals DNR-60, Depth 221.20m

Fig. 6.28 X-Ray Diffraction pattern of clay minerals, glycolated of DNR-60, Depth 221.20m

i.e., I-Illite, Mon-Montmorillonite, Ch-Chlorite, Ka-Kaolinite
Fig. 6.29 X-Ray Diffraction patterns of clay minerals DNR-60, Depth 224.30m

Fig. 6.30 X-Ray Diffraction patterns of clay minerals, glycolated of DNR-60, Depth 224.30m

i.e., I-Illite, Ka-Kaolinite
Fig.6.31 X-Ray Diffraction patterns of clay minerals DNR-60, Depth 237.35m

Fig.6.32 X-Ray Diffraction patterns of clay minerals, glycolated of DNR-60, Depth 237.35m

i.e., I-Illite
Fig. 6.33 X-Ray Diffraction patterns of clay minerals DNR-61, Depth 5.50m

Fig. 6.34 X-Ray Diffraction pattern of clay minerals, glycolated of DNR-61, Depth 5.50m

i.e., I-Illite, Mon-Montmorillonite, Ch-Chlorite, Ka-Kaolinite
Fig. 6.35 X-Ray Diffraction patterns of clay minerals DNR-61, Depth 29.70m

Fig. 6.36 X-Ray Diffraction patterns of clay minerals, glycolated of DNR-61, Depth 29.70m
i.e., I-Illite, Mon-Montmorillonite, Ch-Chlorite, Ka-Kaolinite
Fig. 6.37 X-Ray Diffraction patterns of clay minerals DNR-61, Depth 41.10m

Fig. 6.38 X-Ray Diffraction patterns of clay minerals, glycolated of DNR-61, Depth 41.10m

i.e., I-Illite, Ch-Chlorite
Fig. 6.39 X-Ray Diffraction patterns of clay minerals DNR-61, Depth 98.50m

Fig. 6.40 X-Ray Diffraction patterns of clay minerals, glycolated of DNR-61, Depth 98.50m
i.e., I-Illite, Mon-Montmorillonite, Ch-Chlorite, Ka-Kaolinite
Fig. 6.41 X-Ray Diffraction patterns of clay minerals DNR-61, Depth 108.50m

Fig. 6.42 X-Ray Diffraction pattern of clay minerals, glycolated of DNR-61, Depth 108.50m

i.e., I-Illite, Mon-Montmorillonite, Ch-Chlorite, Ka-Kaolinite
Fig. 6.43 X-Ray Diffraction patterns of clay minerals DNR-61, Depth 111.05m

Fig. 6.44 X-Ray Diffraction patterns of clay minerals, glycolated of DNR-61, Depth 111.05m

i.e., I-Illite, Ch-Chlorite, Ka-Kaolinite
Fig. 6.45 X-Ray Diffraction patterns of clay minerals DNR-61, Depth 128.00m

i.e., I-Illite, Ch-Chlorite, Ka-Kaolinite

Fig. 6.46 X-Ray Diffraction pattern of clay minerals, glycolated of DNR-61, Depth 128.00m

i.e., I-Illite, Ch-Chlorite, Ka-Kaolinite
Fig. 6.47 X-Ray Diffraction patterns of clay minerals DNR-61, Depth 171.20m

Fig. 6.48 X-Ray Diffraction patterns of clay minerals, glycolated of DNR-61, Depth 171.20m

i.e., I-Illite, Ch-Chlorite, Ka-Kaolinite
Fig. 6.49 X-Ray Diffraction patterns of clay minerals DNR-61, Depth 210.50m

Fig. 6.50 X-Ray Diffraction patterns of clay minerals, glycolated of DNR-61, Depth 210.50m

i.e., I-Illite, Ch-Chlorite
Fig. 6.51 X-Ray Diffraction patterns of clay minerals DNR-61, Depth 237.35m

i.e., I-Illite, Mon-Montmorillonite, Ch-Chlorite, Ka-Kaolinite

Fig. 6.52 X-Ray Diffraction patterns of clay minerals, glycolated of DNR-61, Depth 237.35m

i.e., I-Illite, Mon-Montmorillonite, Ch-Chlorite, Ka-Kaolinite
Table 6.1 Minerals identified from XRD Analysis in DNR/58 core

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Table 6.2 Minerals identified from XRD Analysis in DNR/60 core

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