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Mineralogical and Geochemical Investigation of the Tiyon Formation from Surjan Anticline, Thano Bula Khan, District Dadu, Sindh

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ABSTRACT: *Twenty samples of the Tiyon Formation from Thano Bula Khan, District, Dadu Sindh, have been investigated with the aim to know the mineralogical composition of the whole-rock and clay fraction (<2μ) samples and also to have some insight regarding the origin of minerals and the environments of the deposition of the studied sediments.*

The whole-rock samples are composed of detrital components e.g. quartz, kaolinite, vermiculite, mica-montmorillonite. Calcite is the only non-detrital mineral. The clay fraction is composed of Mg-vermiculite, mica-montmorillonite and kaolinite. The Mg-vermiculite is the major constituent in association with kaolinite, illite and mica-montmorillonite in the clay fraction samples.

Vermiculite seems to have been produced by the degradation of sheet silicates e.g. illite, cholorite, biotite, mica-montmorillonite, either in the locality of the provinance or in the basin of the deposition at Thano Bula Khan.

INTRODUCTION

The province of Sindh has widespread exposures of the sedimentary rocks. These, rocks have not been investigated for their mineral composition, particularly for the species of clay minerals.

After realizing importance of the species of clay minerals in the exploration of hydrocarbons, their application in agriculture and other geological investigations, we initiated a series of research projects dealing with the mineralogical and geochemical investigations of the sedimentary rocks of the Province of Sindh. The mineralogical and geochemical results of samples of the "TIYON FORMATION", from Thano Bula Khan, presented in this paper.

NOMENCLATURE

The name "Tiyon Formation" was given to this Formation by the M/S Hunting Survey Corporation (1960), .This name was derived from the "Tiyon Nai", a stream draining the west flank of the Laki Range in the north, where this Formation is well exposed. Other exposures of the Tiyon Formation, have been reported from the Thano Bula Khan, Kalu Kuhar and west of the Bara Nala.

LITHOLOGY

The lithology of the Tiyon Formation, from Surjan anticline, mainly is composed of shale, marl, limestone. The shale is calcareous, gypsiferous, mostly bluish green, greenish brown and yellowish grey in colour. Tiyon Formation is highly fossiliferous and mostly contains foraminifera.

The Tiyon Formation, is conformable with the underlying Laki Formation and the overlying Kirthar Formation. Fig.3, shows the columnar section of the said Formation. There is a major fault known as "Sumbak Fault" which is dipping 60-75 degrees west, and it's strike is 15 degrees to the north. Hunting Survey Corporation (1960), has reported the presence of many faults in this area.

AGE

The Hunting Survey Corporation (1960), on the basis of fossils assemblage assigned, Late-Middle Eocene age to this Formation. Usmani (1985), also on the basis of the presence of planktonic foraminifera in this Formation assigned the same age, mentioned above.

MATERIALS

Twenty samples were collected at an interval of ten feet from the western flank of the Surjan anticline, located N-E of the Thano Bula Khan, to cover the total thickness of 200 feet of the said Formation. Fig. 1, shows the locality from where the samples were collected.

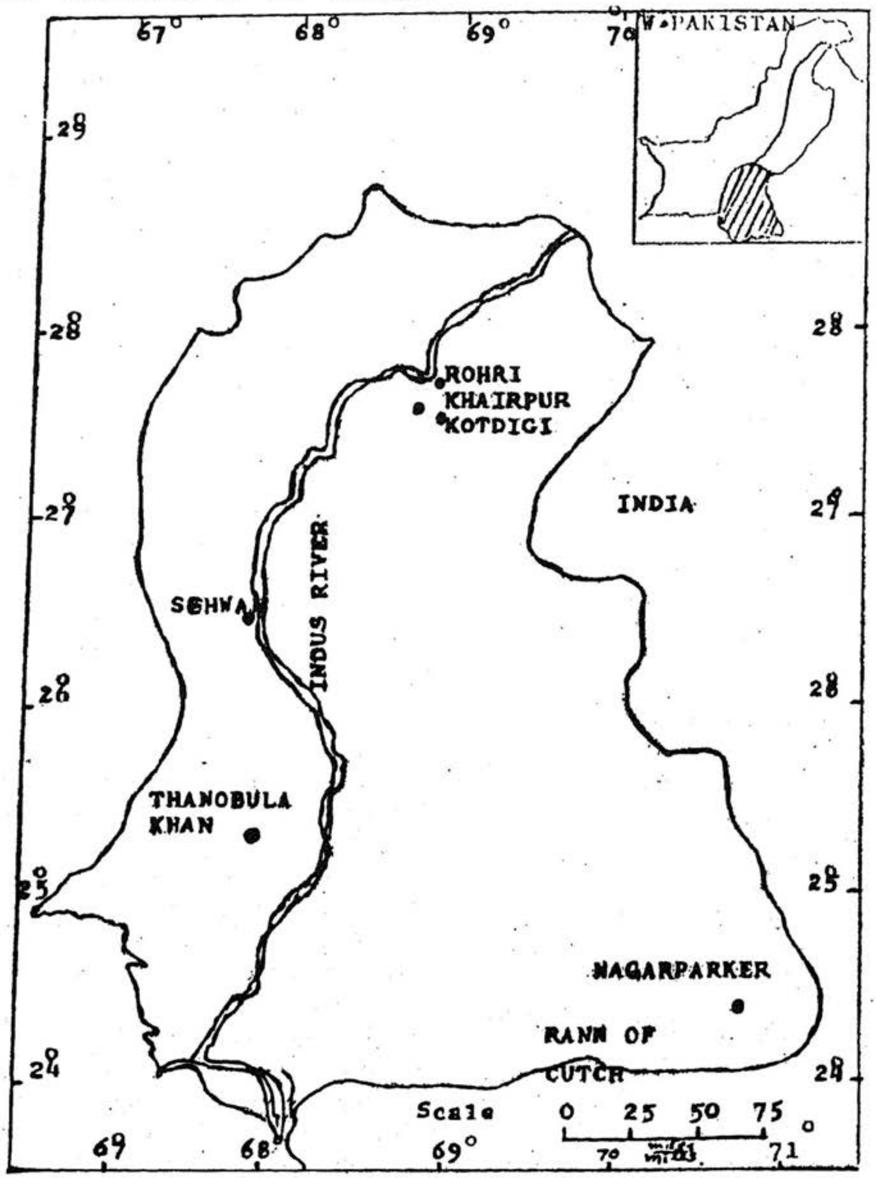

Fig. 1. The location map showing sample localities.

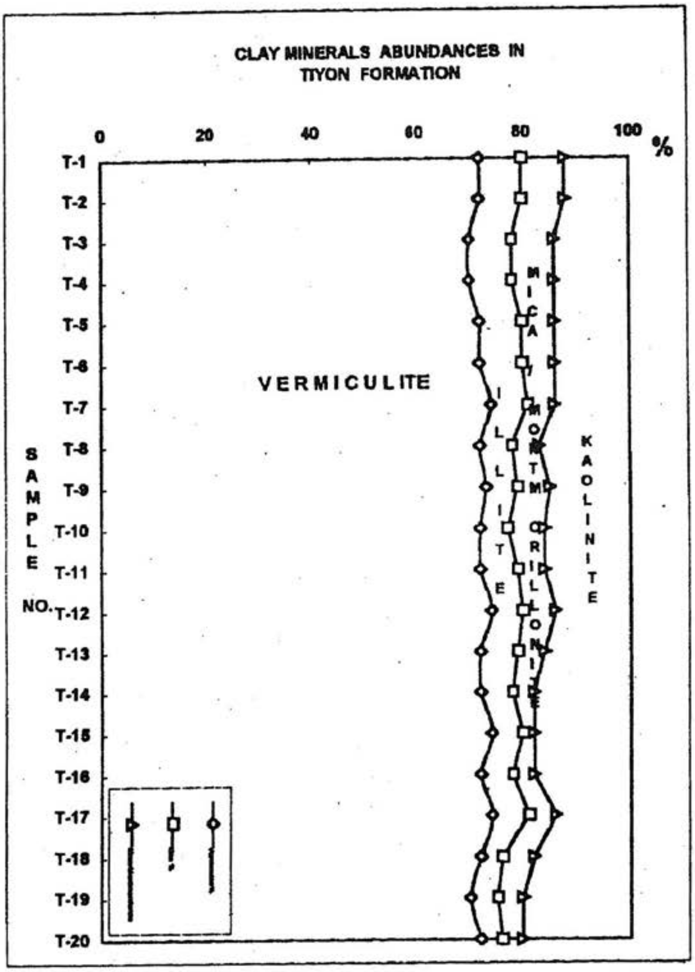

Fig. 2. Shows the vertical distribution of clay minerals in the sediments of the Tiyon Formation from Thanobula Khan, District Dadu, Sindh.

ANALYTICAL TECHNIQUES

The collected samples were given many treatments for achieving the desired results. Different preparations of samples were made for analysing the non-clay and clay mineral species, present in the studied samples, by the X-ray diffraction technique described by Klug and Allexander (1974).

The samples were given the following treatments:

1) The samples were dried at 110°C temperature for removing the moisture.
2) The dried samples were pulvirised for obtaining powder of the desired grain size.
3) Carbonates were removed by heating the samples with 10% acetic acid.
4) The clay fraction (<2μ), grain size, was separated by following the conventional methods of sedimentation and centrifugation, described by the Brindley and Brown (1980), and Moore (1984).
5) Four oriented slides of the clay fraction of each sample were prepared. Treatments, such as glycolation, acid digestion and heat treatment, were given to these slides as

described by Brindley and Brown (1980), and Moore (1984).

X-RAY DIFFRACTOMETERY

(A) Random Powder Mineral Analysis
The whole-rock Powder of each sample, randomly loaded in a powder-holder, was scanned on the Siemen's D-5000 X-RAY Diffractometer from 2 degrees 2 θ- 65 degrees 2 θ, according to the settings shown in Table 2 Klug and Allexander (1974) and Schultz (1964) have mentioned that all minerals which are present in sediments in appreciable amounts, indicates their strong peaks on the X-ray diffractogram whithin the above mentioned range.

Minerals, in the whole-rock and in clay fraction (<2μ) samples, were identified on the basis of the recognition of diagnostic d-spacings mentioned by Pei-Yuan Chen (1977), Brindley and Brown (1980), Cosgrove (1974) and Baig (1984). The whole-rock samples on X-ray diffraction indicated the presence of quartz (weak refractions), calcite (pure), vermiculite (macroscopic). Diagnostic X.R.D peaks shown by the minerals are shown in Table 5 and also in Fig. 3. Description of each non-clay minerals is given below:

a) Quartz: Mineral quartz showed, it's occurrence in the studied samples by it's weak and difused d-spacings at 3.34 Å (26.6, 2θ) and 4.24 Å (20.85, 2θ). Baqri (1980), also reported weak reflections of quartz from Thano Bula Khan area. The difused peaks, shown by quartz, may be attributed to the nature of provinance of the studied sediments.

b) Calcite: Mineral calcite is the only non-detrital mineral in the studied sediments. It indicated it's presence by it's diagnostic d-spacings at 3.03 Å (29.44, 2θ), 2.09 Å (49.24, 2θ) and 2.28 Å (40.46, 2θ), confirming that it is a pure calcite.

c) Vermiculite: Mineral vermiculite, occurs in macroscopic and microscopic grain size in sediments. Vermiculite showed it's peaks in the whole-rock samples due to it's macroscopic grain size at 14.4 Å d-spacings.

(B) Clay Fraction Mineral Analysis
The Clay fraction (<2μ), grain size, was separated from each sample and the same was used for the preparation of oriented slides as described by Brindley and Brown (1980) and Moore (1984). Removal of carbonate from the Clay fraction is essential for the satisfactory identification of the species of clay minerals. Therefore, carbonate free slury was used for the preparation of the 4th oriented slide as described by Brindley and Brown (1980), Moore (1985), and Baig (1984). One oriented slide marked (G) was given glycolation treatment for one hour at 60 degrees C, the second slide marked (H) was given heat treatment for two hours in a Muffle furnace at 550 degrees C, the third slide marked (N) is untreated. Vermiculite and chlorite are the very common clay minerals in soils and marine sediments. In order to differentiate between vermiculite and chlorite, clay fraction was given acid digestion treatment, during this treatment sample becomes free from chlorite leaving vermiculite unattacked.

All the four slides, of each sample, mentioned above, were scanned on the Siemen's D-5000 X-ray diffractometer from 2 degrees 2θ to 36 degrees 2θ.

c) Semi-quantitative Estimation of Clay Minerals
The abundances of clay minerals, tabulated in Table 1 were calculated, following the method described by Matter (1974). Baig (1982), found this method satisfactory for the estimation of the abundances of clay minerals in the sediments of the Oxford Clay and Kellaways Formations from southern England.

The clay minerals which showed their presence by their diagnostic d-spacings, which also are illustrated in Fig. 4. are listed below:

1. Kaolinite
2. Vermiculite
3. Illite
4. Mica-Montmorillonite.

COLUMNAR SECTION OF TIYON FORMATION.

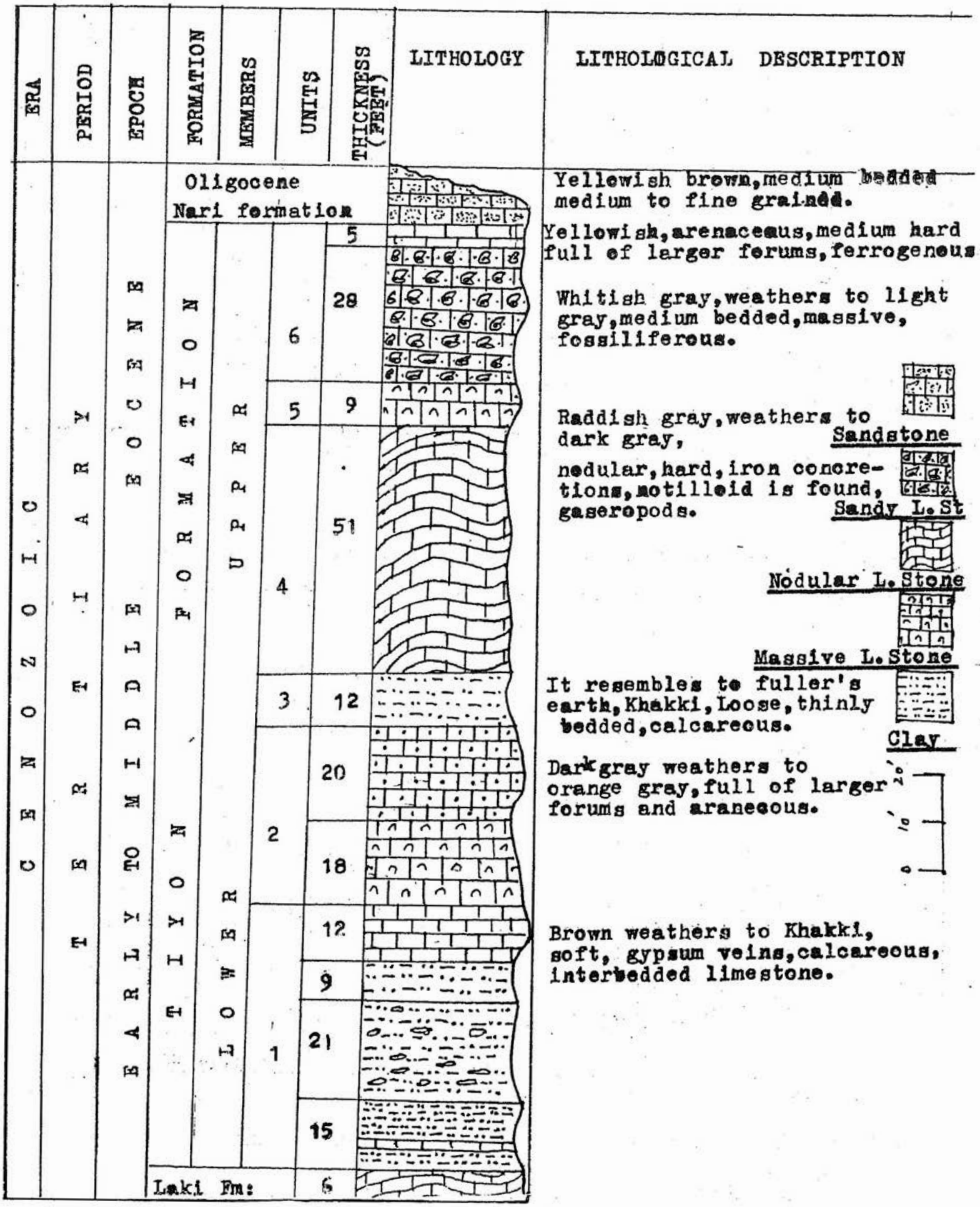

Fig. 3. Columnar Section of Tiyon Formation.

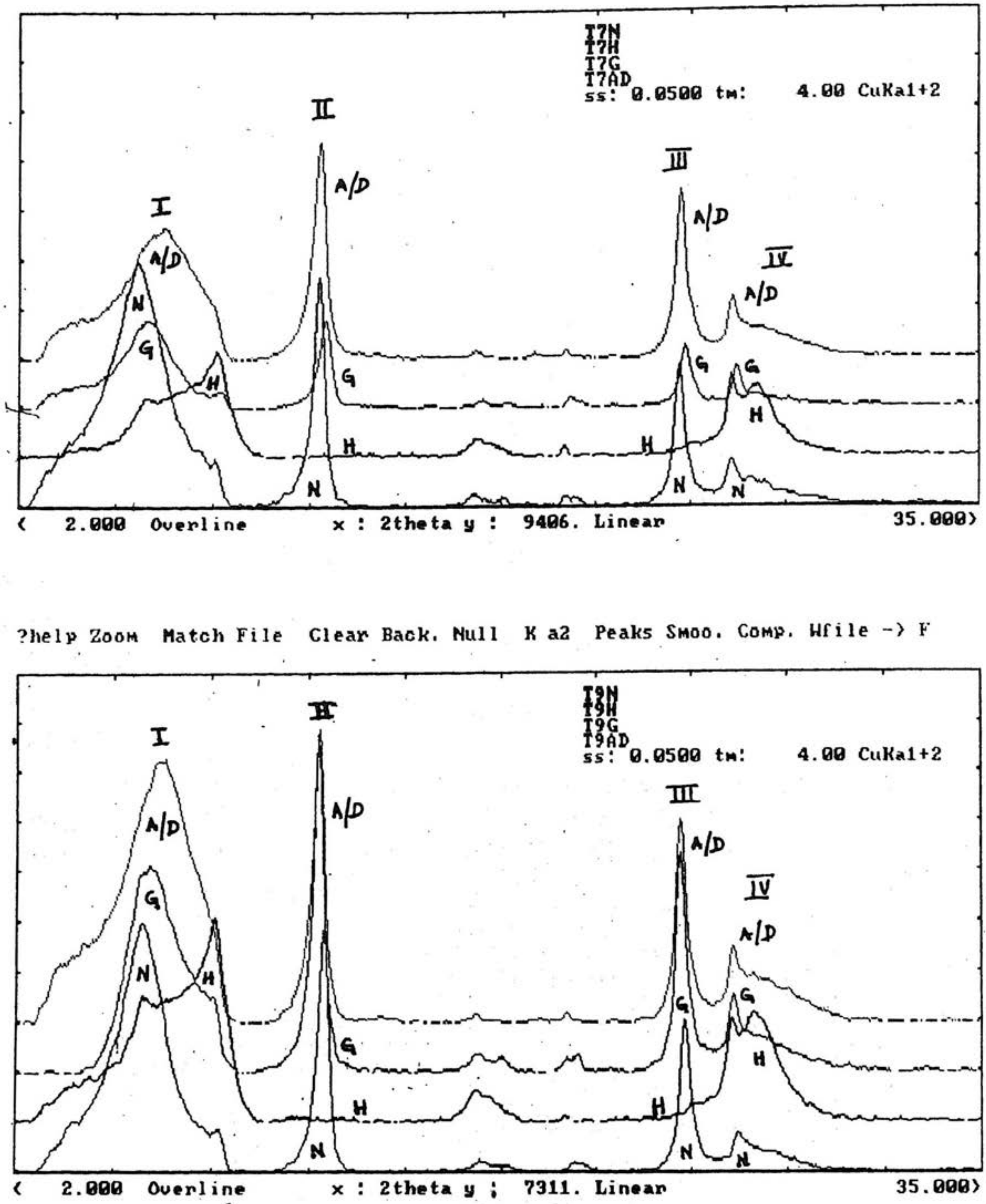

Fig. 4. Showing the behaviour of the reflections of the clay minerals during X-ray diffraction, after different treatments.

I. Vermiculite.Mg. (14.4 Å) II. Kaolinite. (7.1 Å) III. Kaolinite (3.6 Å) IV. Mica/Illite (3.35 Å)

1. Kaolinite: Mineral Kaolinite, showed it's presence by diagnostic d-spacing at 7.13 Å (12.4 degrees 2θ) on normal, glycolated and acid digested slides. Heated slide did not show any peak for Kaolinite at this d-spacing, confirming the presence of Kaolinite.

2. Vermiculite: Mineral Vermiculite, showed it's peak in the whole-rock samples due to it's macroscopic nature. Vermiculite and Chlorite are the very common clay minerals in marine sediments and soils.

   Vermiculite showed it's presence by scanning the normal, glycolated and acid digested oriented slides, respectively at 14.4 Å and 10.0 Å d-spacings.These d-spacings, indicated that Mg & Ca cations are present in the inter-layers of the vermiculite, (Weaver & Pollard,1975) present in the studied samples.

3. Mica/Illite: Mica/Illite, showed their presence by weak X.R.D peaks at 10.1-9.96 Å (8.75-8.7 degrees, 2θ), 5.00 Å - 4.99 Å (17.73 degrees, 2θ - 17.81 degrees, 2θ) and 3.35 Å 3.32 Å (26.6 degrees, 2θ - 26.8 degrees 2θ).

4. Mixed-layers Clays (Illite-Montmorillonite): Illite-Montmorillonite, mixed-layers Clays (1:1), indicated their presence by the X.R.D peaks at 25.8 Å (3.21 degrees 2θ), 12.7 Å (6.96 degrees, 2θ) and 3.23 Å (25.22 degrees, 2θ).

The presence of mica- illite and illite-montmorillonite is of significant importance in the present study. It suggests that the vermiculite, present in the studied samples, is the alteration product of mica, illite and illite-montmorillonite, (Weaver & Pollard, 1975).

TABLE 1. CLAY MINERAL ABUNDANCES IN TIYON FORMATION

Sample No.	Vermiculite%	Kaolinite%	Illite %	Mica/ Montmorillonite%
T-1	72	12	08	08
T-2	72	12	08	08
T-3	70	.14	08	08
T-4	70	14	08	08
T-5	72	14	08	06
T-6	72	14	08	06
T-7	74	14	07	05
T-8	72	17	06	05
T-9	73	15	06	06
T-10	72	16	05	07
T-11	72	16	07	05
T-12	74	14	06	06
T-13	72	16	07	05
T-14	72	18	06	04
T-15	74	18	06	02
T-16	72	18	06	04
T-17	74	14	07	05
T-18	72	18	04	06
T-19	70	20	05	05
T-20	72	18	04	04

TABLE 2. CHEMICAL ANALYSIS OF TIYON FORMATION

	T-1	T-3	T-7	T-15	T-17
SiO_2	50.88	50.50	51.98	50.50	49.50
Al_2O_3	15.35	16.00	15.50	15.50	16.00
CaO	17.00	18.25	16.00	16.50	16.50
MgO	2.50	2.60	3.00	3.00	3.00
NaO_2	0.52	0.54	0.52	0 .52	0.52
K_2O	2.45	2.35	2.50	2.00	2.00
Fe_2O_3	2.50	2.00	2.50	2.00	2.00
H_2O	8.35	7.76	8.00	10.96	10.43
Total	100.0	100.0	100.0	100.0	100.0

Mineral Composition of Whole-Rock Samples

1. Quartz
2. Calcite (Pure)
3. Vermiculite
4. Clays

X-R.D Settings	Random Powder Sample	Clay fraction Sample
Scanning range	2 - 65, 2θ	2 - 36, 2θ
Time Constant	1 Second	4 Second
Step Size	0.050 Degrees	0.050 Degrees

TABLE 3. SHOWS THE AVERAGE OF CHEMICAL ANALYSIS OF TIYON FORMATION FROM THANO BULA KHAN AND IT'S COMPARISON WITH AVERAGE VALUES OF BENTONITIC CLAYS FROM SINDH

	1	2	3
SiO_2	50.66	56.42	57.50
Al_2O_3	15.66	14.90	17.15
Fe_2O_3	02.26	05.92	09.15
MgO	02.82	05.01	03.82
CaO	17.25	00.19	00.24
Na_2O	00.52	00.39	00.71
K_2O	02.26	01.99	03.00
H_2O	09.50	07.42	07.80

1. Average of 5 samples from Tiyon Formation Thano Bula Khan, (Present work).
2. Average of 3 samples of Bentonitic Clays from Thano Bula Khan, (Reported by Baqri 1980).
3. Average of 8 samples of Bentonitic Clays from Khairpur, (Reported by Baqri 1980).

TABLE 4. BASAL d-SPACINGS OF VERMICULITE AFTER HEATING & GLYCOLATION TREATMENTS

Cations	d- Spacing(OO2) Normal Slide	d-Spacing Glycolated Slide	After Heat Treatment 170 °C	250 °C	610 °C
Mg++	14.39Å	14.3 Å	11.8 Å	10.0 Å	09.4 Å
Ca++	15.00 Å	17.6 Å	11.8 Å	10.1 Å	09.6 Å
Sr++	15.00 Å	--	--	--	--
Li++	12.20 Å	--	--	--	--
Na++	12.6 Å	14.8 Å	10.3 Å	10.4 Å	10.4 Å
K+	10.4 Å	--	10.4 Å	10.4 Å	10.4 Å

BASAL d-SPACINGS (Å) OF VERMICULITE SATURATED WITH VARIOUS CATIONS AT DIFFERENT RELATIVE HUMIDITY

Cation	32% R.H	52% R.H	79% R.H	In water
Ca	14.8	14.9	14.9	14.9
Mg	14.3	14.4	14.4	14.6
Na	12.3	12.6	14.6	14.9
K	10.4*	10.4*	10.5*	10.5-14.3

*Broad or asymetrical reflection.

Reproduced from G.Brown (1961), X-ray identification and crystal structure of Clay Minerals, Min. Soc-London.

TABLE 5. SHOWS THE d-SPACINGS, USED FOR THE IDENTIFICATION OF NON-CLAY AND CLAY MINERALS, PRESENT IN THE TIYON FORMATION

1	Quartz -	3.34 Å 4.26 Å 1.82 Å	2	Calcite -	3.03 Å 2.28 Å 2.09 Å
3	Vermiculite (Macroscopic)-	14.4 Å	4	Kaolinite -	(N) 7.13 Å (12.4 , 2θ) (G) 7.13 Å (12.4 , 2θ) (A/D)7.13Å (12.4, 2θ) (H) - No Peak
5	Vermiculite - (Clay Fraction)	(N)14.4 Å (G) - No Change (A/D)-No Change (H) 9.8 - 10 Å	6	Illite -	(N) 10.5 - 3.3 Å (G) - No Change (H) - No Change
7	*Mica/Illite	10.1 - 9.96 Å (8.75-8.87, 2θ) 4.9 Å (17.77,2θ) 3.35 - 3.32 Å (26.60-26.85,2θ)	8	*Mixed-Layer Clays (Illite/ Montmorillonite)	25.8 Å (3.42 , 2θ) 12.7 Å (6.96 , 2θ)

* d-Spacings mentioned in a "Table of key lines in X-ray Powder Diffraction Patterns of Minerals in Clays and Associated Rocks", by PEI-YUAN CHEN (1977).

TABLE 6. SHOWING THE BEHAVIOR OF THE REFLECTIONS CORRESPONDING TO BASAL SPACING DURING X-RAY DIFFRACTION AFTER DIFFERENT TREATMENTS. (REPRODUCED FROM MILLOT, G.1959)

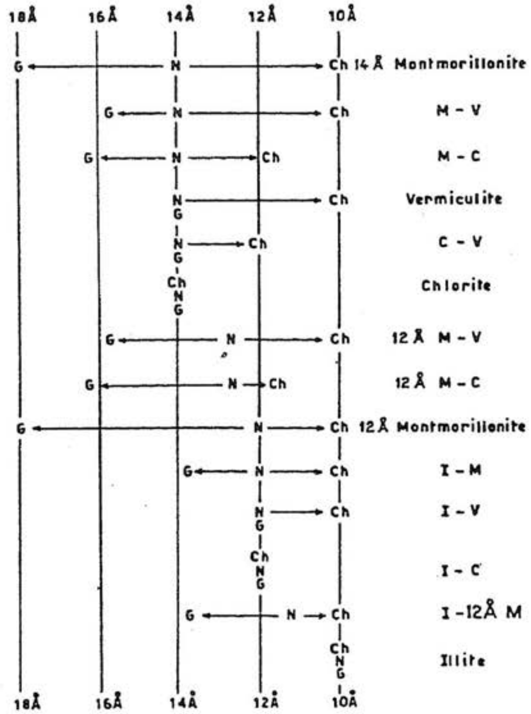

FIG. 2. — *Schematic representation of the behavior of the reflections corresponding to basal spacing during X-ray diffraction after different treatments* (after LUCAS, CAMEZ and MILLOT, 1959).
G: Treatment with glycerol; N: Natural sample; Ch: Heating at 490°.

RESULTS AND DISCUSSION

Table 1, 2 and 3 respectievly, shows the results of the calculated abundances of species of the clay minerals encountered, data of the chemical analysis of the studied sediments and the comparison of the results of chemical composition of Tiyon Formation, with the results of bentonitic clays, reported by Baqri (1980), from Thano Bula Khan.

It is evident from Table 2 that the bulk-rock samples are mainly composed of detrital components e.g quartz, vermiculite (macroscopic), kaolinite, illite and mixed-layers clays. Mineral quartz showed it's weak reflections on the X-ray difraction Baqri (1980), also reported weak reflections of quartz fromThano Bula Khan, area. It may be attributed to the nature of the provinance of the studied sediments.

Mineral calcite is the only non-detrital mineral which indicated it's presence by it's diagonistic d-spacings at 3.03 Å (29.44,2θ) and 2.09 Å (49.24, 2θ) and 2.28 Å (40.46, 2θ), confirming that it is pure calcite.

The high concentration of CaO, shown in Table 2, is due to the presence of calcite. Some amount of CaO is also associated with the clay minerals e.g illite, vermiculite and mixed-layer mica-montmorillonite. These minerals contain CaO in their inter-layers position. The same table also shows appreciable amounts of MgO, Na_2, K_2O and Fe_2O_3. Vermiculite and mixed-layers chlorite-vermiculite, contains Mg and Na in their inter-layers position (Weaver & Pollard, 1975).

Table 1, indicates that in the investigated sediments vermiculite occurs as the most abundant clay mineral in the clay fraction in association with illite, illite-montmorillonite and kaolinite.

In the present study the vermiculite showed it's presencce by diagonistic d-spacings at 14.4Å, 3.6Å and 2.8Å on the normal slide and at 10.1Å on the heated slide, exhibiting that this vermiculite is Mg-vermiculite. The vermiculite, chlorite, and vermiculite layers, interstratified with mica and chlorite layers, commonly occur in marine sediments and also in the weathered detritus. Brown, 1953; Hathaway, 1955; Droste, 1956; Weaver, 1958; Gjems, 1963; Millet and Camez, 1963; Barshad and Kishk, 1969, have mentioned the occurrence of vermiculite in soils and marine sediments. The vermiculite is produced by the degradation of chlorite, biotite, mica and montmorillonite, by the process of replacement of K which is replaced by Mg, Na and Ca cations in the inter-layers position of the above mentioned minerals (Weaver & Pollard, 1975). Biotite and Chlorite, both contain iron in their lattice, due to this reason both minerals are easily weathered and thus the removal of K and the addition of Mg, Na and Ca cations, in the inter-layers of degraded clay minerals becomes easily possible, threrefore, the vermiculite is produced by the degradation of biotite and chlorite etc. (Weaver and Pollard 1975).

Mineral Vermiculite, has not been reported by the early workers from the sedimentary rocks of Sindh. Baqri (1980) studied clay mineralogy of the bentonitic clays from Sindh including Thano Bula Khan area. He reported the presence of Kaolinite, illite, montmorillonite, chlorite and palygorskite from the Thano Bula Khan and did not mention about the occurrence of vermiculite.

In the studied sediments vermiculite showed very good relationship with illite and illit-montmorillonite, as illustrated in Fig. 2. This relationship appears to be genetic and indicates that clay minerals i.e. illite and illite-montmorillonite/mica, most probbably were the source minerals for the formation of Mg-vermiculite, in the studied sediments.

Table 3 shows the depleted amounts of Fe_2O_3, MgO, SiO_2 and high amounts of CaO, K_2O, Na_2O and H_2O in the samples of the

Tiyon Formation from Thano Bula Khan as compared to the average values of these elements reported by Baqri (1980), for the bentonitic clays from Thano Bula Khan. The reason of depletion is the presence of abundant amounts of Mg-vermiculite, illite, mica-montmorillonite and calcite in the samples of the Tiyon Formation. The higher values of Fe_2O_3, MgO reported by Baqri (1980), in the bentonitic clays from Thano Bula Khan are due to the presence of (43%) illite + palygorskite,(12%) montmorillonite and (6%) chlorite. The values of SiO_2 and Al_2O_3, determined in the present study, showed good agreement with the values reported by Baqri (1980) in the bentonitic clays from Thano Bula Khan.

It is obvious that the major part of SiO_2, belongs to quartz and some amount of SiO_2 and Al_2O_3 resides in the lattice of the clay minerals, present in the studied sediments. Tables 4 and 6 show the basal d-spacings of vermiculite, after heat and Glycolation treatments. Table 4 also shows the basal d-spacings of vermiculite, saturated with various cations at different relative humidity.

The clay minerals and non-clay minerals assemblage of the studied sediments, tabulated in Tables 1 and 2 indicated that investigated samples are mainly composed of detriatal components such as quartz, kaolinite, vermiculite (macroscopic), illite, and mica, indicating that the provinance of these sediments was in the weathered igneous or meta-sedimentary rocks. Weaver and Pollard (1957), mentioned that vermiculite can be produced by the weathering of fine-grained schists, sandstones, shales, chlorite and basalt. Baqri (1980), stated that weathered debris of erroded basaltic rocks to the east, was transported westwards by the rivers, into shallow evaporating sea extending from Kotdigi to Thano Bula Khan. Baqri's views regarding the provinance of the bentonitic clays of Thano Bula Khan, provided confidence to the present authors, to conclude that the provinance of the Tiyon Formation of the Thano Bula Khan, was the same as mentioned by Baqri (1980).

Taking into consideration the mineral composition of the samples of bulk-rock and the clay fraction, which are dominantly composed of detrital components, e.g quartz, kaolinite, vermiculite etc., it can be concluded that these sediments were deposited under shallow marine enviornments. Usmani (1985), on the basis of the presence of planktonic foraminifera fossils in the Tiyon Formation from Thano Bula Khan, also concluded shallow-marine condition of the deposition for the studied sediments. Farshori (1972), also mentioned that the enviornments of deposition, of the Tiyon Formation of Thano Bula Khan area were shallow marine.

CONCLUSIONS

The above study may be concluded as under:

1. The whole-rock samples are mainly composed of detrital components e.g. quartz, kaolinite, vermiculite, mica, etc.
2. Quartz, showed very weak and difused peaks on the X-ray diffractograms. The poor degree of crystalinity, shown by quartz, may be due to it's possible provinance of volcanic rocks, derived from the east.
3. Pure Calcite is the only non-detrital mineral in the studied sediments.
4. The clay fraction ($<2\mu$) samples are composed of Mg-vermiculite, illite, kaolinite, mica-montmorillonite.
5. Mineral vermiculite, showed it's strong relationship with illite, mica-montmorillonite, suggesting that vermiculite was produced by the deggradation of these clay minerals.
6. The detritus for the studied sediments was contributed by the igneous rocks, most

probbably the volcanic rocks exposed in the east.

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