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Clay Mineralogy and Diagenesis of Shales: A Case Study from the Mio-Pliocene Tipam and Dupi Tila Shales of Bandarban Anticline, Bandarban Hill District, Bangladesh

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Abstract: A detailed clay mineralogical investigation has been conducted on shale samples from the Mio-Pliocene Tipam Sandstone and Dupi Tila Formations of the Bandarban Anticline, Bandarban Hill District, Bangladesh. The whole shale and the <2.0 μm fractions of the samples were treated for thin section petrography and XRD analysis. The predominant clay minerals identified are kaolinite, illite, mixed-layer illite/smectite, chlorite and muscovite. Mixed-layer illite/smectite forms a major group and there are indications that the diagenesis has taken place involving gradual loss of illite/smectite from younger to older rocks. The gradual decrease of the illite/smectite proportion in the random illite/smectite clay is reflected by a gradual decrease in the 17Å peak intensity which is possibly interpreted as a decrease in the percentages of smectite layers in the illite/smectite clay due to the diagenetic transformation of smectite to illite. This may be a source of additional silica to the interbedded sandstones for the generation of little quartz overgrowth cementation. It is expected that a small volume of water derived from the smectite diagenesis should have been available and migrated from smectite interlayers to the pore water system, contributing to the generation of overpressure encountered in the subsurface of Bandarban Structure. Moreover, kaolinite clay mineral displaying decreasing tendency in older rocks is amongst other diagenetic changes recorded in studied shale.

Key words: Clay mineralogy, diagenesis, Tipam Sandstone Formation, Dupi Tila Formation, Bandarban Anticline

INTRODUCTION

The clay mineral assemblages of shales and their diagenesis has been a subject much studied in recent years. In addition to dissolution and/or cementation processes, involving silica and carbonates, much attention has been given to clay mineral alteration, transformation and precipitation. Neumerous studies have been reported on shale diagenesis based mainly on the examination of clay mineral assemblages (Perry and Hower, 1970; Hower *et al*., 1976; Boles and Franks, 1979; Imam and Shaw, 1985). They noticed the gradual change of the swelling smectite clay mineral to non-swelling illite clay through intermediate mixed-layer illite/smectite clay with increasing depth of burial. More recently, many investigations (Pablo-Galán *et al*., 2001, 2002; Totten *et al*., 2002; Needham, 2004; Gier and Johns, 2005; Grainger, 2006; McCarty *et al*., 2008) have reported that progressive lithification of shales with depth of burial coincides with the progressive loss of swelling clay minerals in many Geological Associations. The decrease and disappearance of kaolinite, appearance and increase in chlorite and loss of k-feldspars are amongst other diagenetic changes recorded in shale with increasing burial depth. Studies on the mineralogy and diagenesis of the Neogene shales in the overall Bengal Basin has been less well studied and only one study on Bandarban Structure (Chowdhury *et al*., 1993) has been reported so far.

The Neogene shales of the Bengal Basin constitute a major lithology of the drilled as well as outcropping sections. The Miocene-Pliocene Surma Group, the most important petroliferous unit in Bangladesh and forming the backbone of the folded belt, is a clastic deltaic sequence with average sandstone:shale ratio of 1:1, although in many sections, shale overwhelmingly predominates and individual shale units are hundreds of meter thick. There have been very little published record on the Bengal shale mineralogy and diagenesis and even less attention has been focused on the possible

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clay-shale dehydration with increasing burial temperature and pressure environments, although the clay dehydration of such large volume of shales would have major implications in overpressure and quartz overgrowth development, in structural shaping and in petroleum migration. This study discusses the clay mineralogy and diagenesis of some shale samples from the Mio-Pliocene Tipam Sandstone and Dupi Tila Formations of Bandarban Anticline, Bandarban Hill District, Bangladesh. The present research is based XRD analysis of outcrop shales of the above anticline and documents: (1) clay mineralogy of separated clay fraction (<2 μm fraction) and (2) diagenesis of shale, particularly of its clay minerals, with special attention to the expandable smectite diagenesis with increasing burial depth and its possible impact on the cementation of the interbedded sandstones. Furthermore, clay dehydration models of well studied areas like Gulf Coast, USA have been reviewed as these are related to the Bengal Mio-Pliocene Tipam and Dupi Tila shales and the possible implications of diagenesis-dehydration on the overpressure development and structural shaping in the Bengal Basin.

MATERIALS AND METHODS

Six oriented shale samples were prepared using standard procedure (Brown and Brindley, 1980) for X-Ray Diffraction (XRD) analysis. X-ray diffraction analysis (XRD) was carried out at the XRD laboratory, Geological Society of Bangladesh, Dhaka, Bangladesh. Detailed clay mineralogy was evaluated by X-ray diffraction patterns of oriented shale that enhance the basal reflections. Clay mineral diffraction patterns are manifested by characteristic peaks position, intensity, shape and breath.

Prior to processing, about 100 g of each sample were oven-dried at 50°C. This 100 g of dried sample was gently crushed and 10-50 g of that crushed sample was further crushed and ground using an agate mortar to make fine powder. To prepare oriented mounting slides, 25 g of previously crushed and powdered (<1 mm size or less) samples were diluted with 100 mL distilled water and stirred for 20 min. The beaker was then placed in an ultra sound bath for 30 min until satisfactory dispersion of clay was achieved. This dispersed clay suspension was stirred and allowed to gravity settle. The <2.0 μm size fraction was obtained by pipette withdrawal of 20 mL of suspended clay from just below the surface and placed in a 50 mL beaker and dried in oven at 60°C overnight to produce a concentrated clay slurry. A concentration of the above clay in 1 mL of distilled water was thus obtained. An oriented mount of the clay was prepared by dropping the 1 mL clay slurry using a dropper onto a glass slide. The oriented samples were dried at room temperature and analyzed by X-Ray Diffractometer (XRD) using CuKα radiation. Following the X-Ray Diffraction analysis of air-dried samples, the oriented clay-aggregate

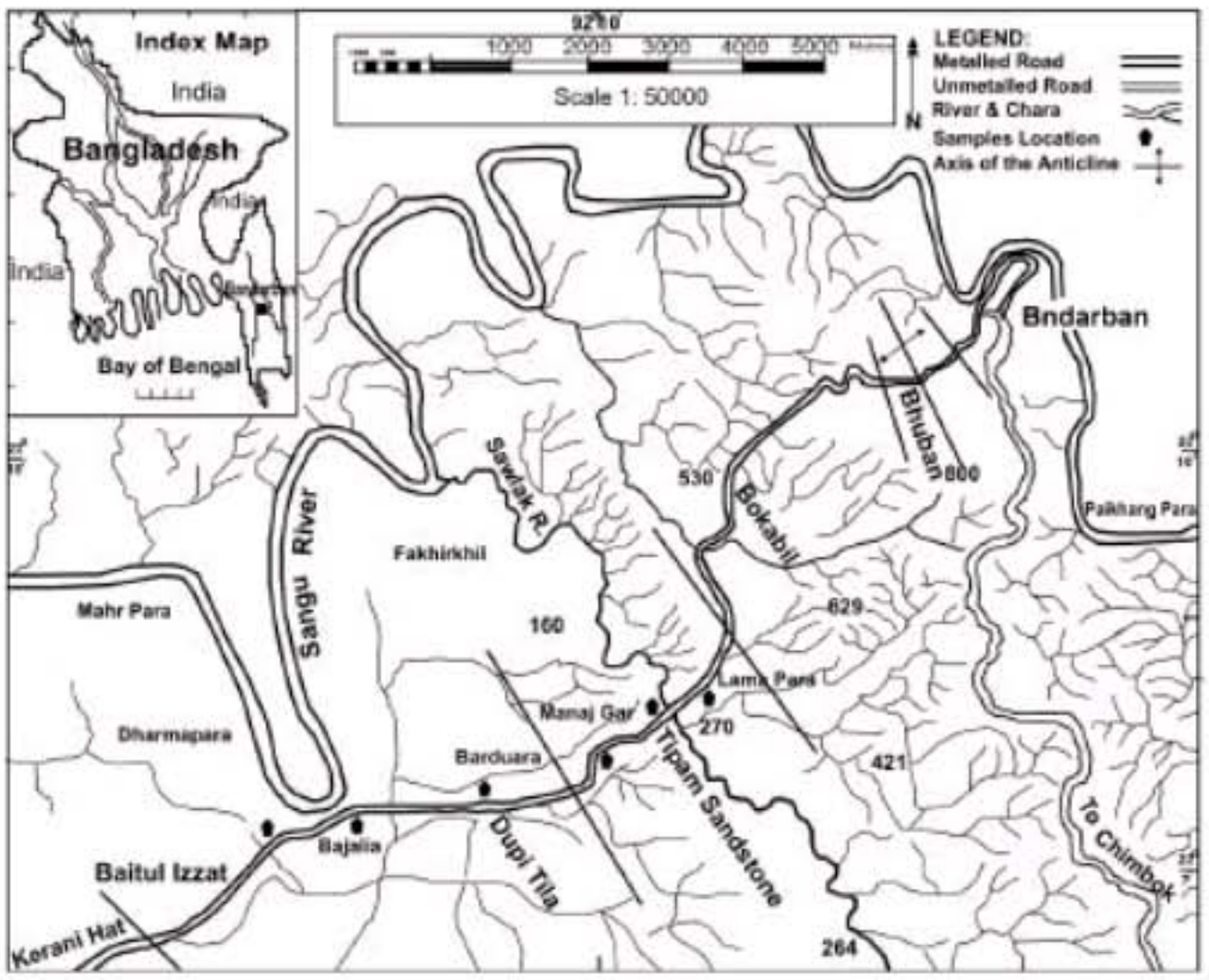

Fig. 1: Map of part of Bandarban Anticline (Western Flank) showing the locations of samples

mounts were placed in an ethylene-glycol atmosphere at 30-40°C overnight. The samples were then re-run under the same diffractometer conditions in order to identify the presence of expandable clay minerals. To determine the existence of certain minerals (like kaolinite), the samples were subsequently heated up to 350°C for 2 h. A semi-quantitative analysis of all the clay mineral species was also performed. Thin sections were made following the method of Carver (1971).

The investigated area lies between Lama Para to Baitul Izzat along Bandarban-Keranihat road cut (Latitude 22°8'40" N-22°6'28" N and Longitude 92°11'4" E-92°6'13" E). Six shale samples, respectively three from the Tipam Sandstone and Dupi Tila Formations were collected and a geological map was prepared (Fig. 1).

RESULTS

Clay minerals: The clay mineral assemblages of the Dupi Tila and Tipam Sandstone Formations of the western flank of the Bandarban Anticline comprise of kaolinite, illite, chlorite, muscovite and mixed-layer illite/smectite. Kaolinite, illite and mixed-layer illite/smectite remain the principal clay mineral constituents of these formations. However, the presence of chlorite is only recorded as trace amounts (Table 1).

Kaolinite: In the present study, most of the kaolinites are found to be poorly crystalline which is reflected in the diffraction patterns by the broadening and weakening of the peaks and a tendency for adjacent reflections to fuse. This is accompanied by an increase in spacing of the basal reflections from 7.02 to 7.14 Å. The basal reflections at 7.02 to 7.14 Å are more intense than those at 3.52 to 3.57 Å (002). The peaks remain unaffected on glycolation (Fig. 2) but are destroyed on heating above 550°C (Millot, 1970). Unfortunately, this basal reflection and temperature of collapse is very similar to that of many chlorites, so, the presence of kaolinite and chlorite together in some of the samples made it difficult to recognize kaolinite on the basis of its major reflections at ~7 and 3.58 Å (001 and 002 reflections, respectively) because of the overlapping with chlorite reflections occurring in this region.

Illite: Illite is identified by the basal spacing at 9.83-10.05 Å (001) and the associated integral spacing at 4.93-4.99 Å (002) and 3.32-3.33 Å (003) (Fig. 2). The 9.83-10.05 Å (001) and 3.32-3.33Å (003) peaks have relatively higher intensity than the 4.93-4.99 Å (002) peak. On glycolation, illite is essentially non-expanding. Small amounts of expandable material will go undetected. On heating to 550°C, the (001) peak of illite may show slight collapse (Chowdhury, 1990). In thin section, detrital illite grains are noticed (Fig. 3a). generally, illite is characterized on the X-ray pattern by somewhat diffuse reflections. Broad, ragged basal peaks differentiate illite from mica. Highly birefringent plates were also noted in thin sections (Fig. 3b), which could be an indicator of relict muscovite or pseudomorphous illite.

Chlorite: The basal reflections of chlorite are recorded at 13.81-14.49 Å (001) and 4.44 Å (003) (Fig. 2). The basal reflections at 7.0 Å (002) and 3.56 Å (004) could not be used directly for identification of chlorite because of the interference with kaolinite reflections (Alam *et al*., 2008). However, iron rich chlorites generally give a very strong reflection at 7.0-7.12 Å. The chlorite peaks are not affected on glycolation and heating up to 550°C (Rahman *et al*., 2005). Clay size chlorite $\{(AlMgFe)_{12}(SiAl)_8O_{20}(OH)_{16}\}$ observed in thin section was pale yellowish green pleochroic with first order interference colors. Greenself coloration and anomalous interference colors are indicative of a detrital origin (Hayes, 1970). Many have been bent or broken during compaction.

Muscovite: The term muscovite is applied to clay-size particles and large flakes. The mineral is identified by 9.99 Å (002), 4.98 Å (004), 4.47 Å (110,111), 4.29 Å (111), 3.87 Å (113), 3.72 Å (023), 3.35 Å (113), 3.48 Å (114), 3.32 Å (024, 006), 3.20 Å (114) and 3.10 Å (115) reflections. The various peaks show weak to moderately strong intensity. The peaks are not affected on glycolation and heating. It seems that 9.99, 4.98 and 3.32 Å peaks of muscovite have been overlapped by the 9.83-10.05 Å (001), 4.93-4.99 Å (002) and 3.32-3.33 Å (003) peaks of illite, respectively (Fig. 2). Relict muscovites were also noted in thin sections (Fig. 3b).

Table 1: The average percentages of each of the clay minerals of the Tipam and Dupi Tila shales, Bandarban Anticline, Bandarban Hill District

	Illite (001)					Kaolinite (001)					Illite/smectite				
Formations	h	½w	h/½w	%	w %	h	½w	h/½w	%	w %	h	½w	h/½w	%	w %
Dupi Tila	3.75	0.20	18.75	51.72	49.67	2.65	0.25	10.60	29.24	30.26	7.60	1.10	6.90	19.03	20.00
	4.00	0.25	16.00	48.48		2.40	0.25	9.60	29.09		11.10	1.50	7.40	22.42	
	3.70	0.20	18.50	48.82		3.70	0.25	12.30	32.46		7.80	1.10	7.09	18.71	
Tipam Sandstone	4.45	0.15	29.60	71.91	70.47	1.55	0.25	6.20	15.06	17.22	5.90	1.10	3.36	13.02	12.29
	4.60	0.15	30.66	70.87		2.00	0.25	8.00	18.49		4.60	1.00	4.60	10.63	
	4.40	0.15	29.33	68.64		1.55	0.20	7.75	18.13		5.65	1.00	5.65	13.22	

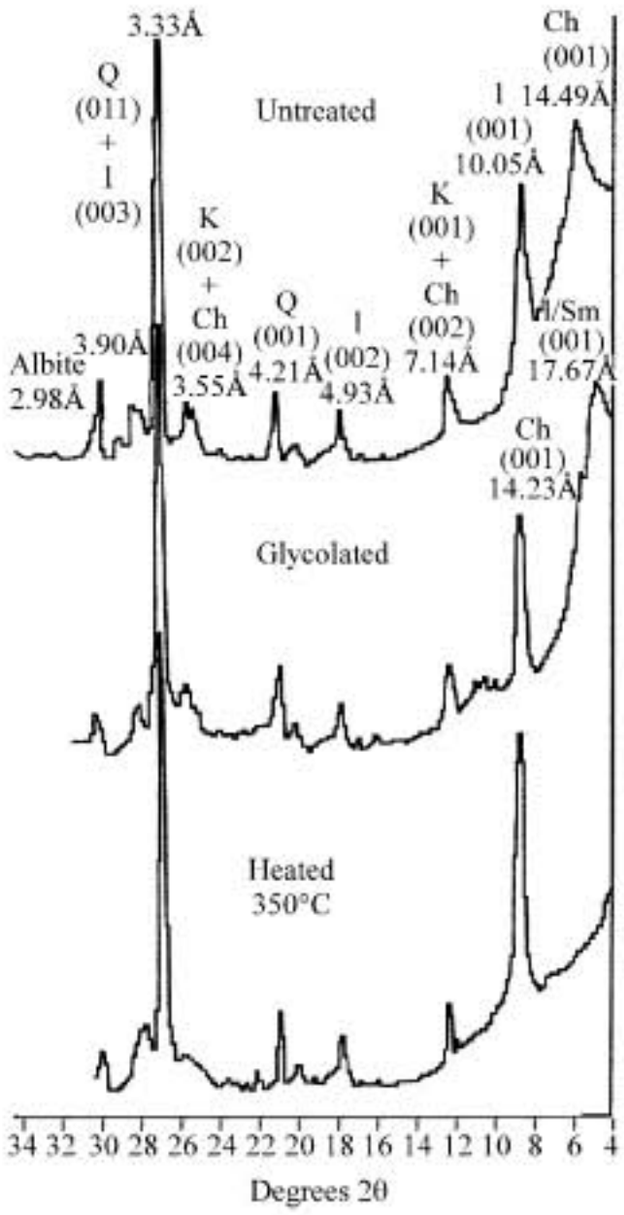

Fig. 2: X-ray diffraction patterns of the clay fractions of the Tipam shales from the Bandarban Anticline (western flank) using CuKα radiation. I/Sm: illite/smectite, Ch: Chlorite, I: Illite, K: Kaolinite and Q: Quartz

Illite/smectite mixed-layer clay: In the present study, the illite/smectite mixed-layer clay is represented by reflections at 16.99-17.67 Å (hereafter denoted as 17 Å) for ethylene glycol treated samples. These reflections disappear with simultaneous increase in the 10 Å reflections when the sample is heated to 350°C. Burst (1969) reported that the 17 Å for an ethylene glycol treated sample could be considered as discrete smectite and the mixed-layer illite/smectite to have basal reflections between 10 and 17 Å depending on their composition. Later, Reynolds and Hower (1970) concluded that in the case of ethylene glycol solvated samples, the 17 Å reflection can also be given by randomly interstratified illite/smectite clays (with 40% or more smectite layers). This means that the first basal reflection at 17 Å for a random mixed-layer illite/smectite clay slightly differ from that of a discrete smectite (i.e., 100% smectite); but according to Reynolds and Hower (1970), the higher order reflections do differ. The reflection designated as $(002)_{10}/(003)_{17}$, resulting from the 2nd and 3rd basal orders of the illite and smectite layers, respectively, is particularly

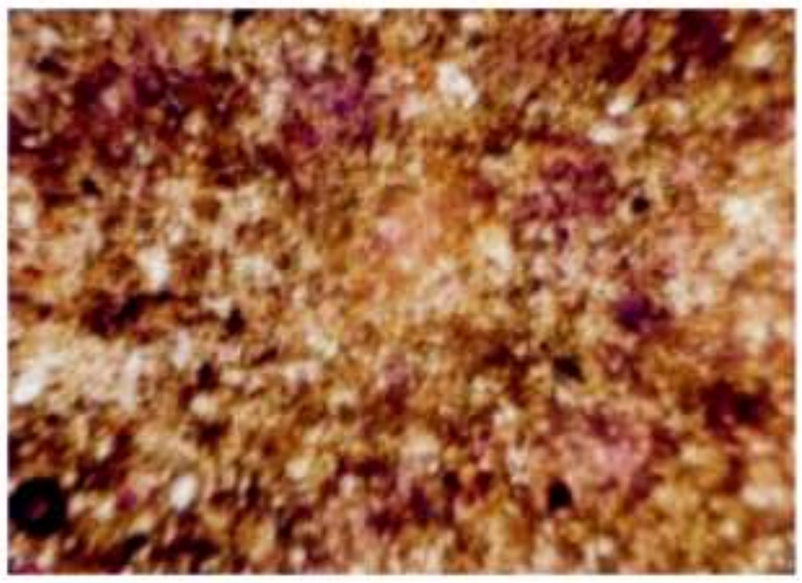

Fig. 3a: Highly birefringent clay sized particles could be pseudomorphous illite under plane polarized light. Scale: 2 mm = 80 µm. Dupi Tila Formation

Fig. 3b: Highly birefringent plates showing relict of muscovite or pseudomorphous illite under cross-nicol (XN). Scale: 2 mm = 100 µm. Tipam Sandstone Formation

important. For a discrete smectite, this peak occurs at 5.62 Å but as the non-expanding illite type is incorporated or its proportion increases, the peak position is shifted towards 5 Å, the 002 of discrete illite (Hower, 1981a). For most of the samples under consideration, the illite/smectite mixed-layer clay phase can be recognized as an important constituent and the 17 Å reflections with significant intensities are noted. This $(002)_{10}/(003)_{17}$ reflection is also identified and measured. It ranges between 5.54 and 5.57 Å (Fig. 4, 5). For this reason, by analogy with the relationships between composition of illite/smectite mixed-layer clay and the $(002)_{10}/(003)_{17}$ as proposed by Reynolds (1980) and compositional dependence of manner of interstratification of illite/smectite as proposed by Hower (1981b), the 17 Å peaks for ethylene glycol treated samples are interpreted in this study as representing random illite/smectite mixed-layer clay mineral.

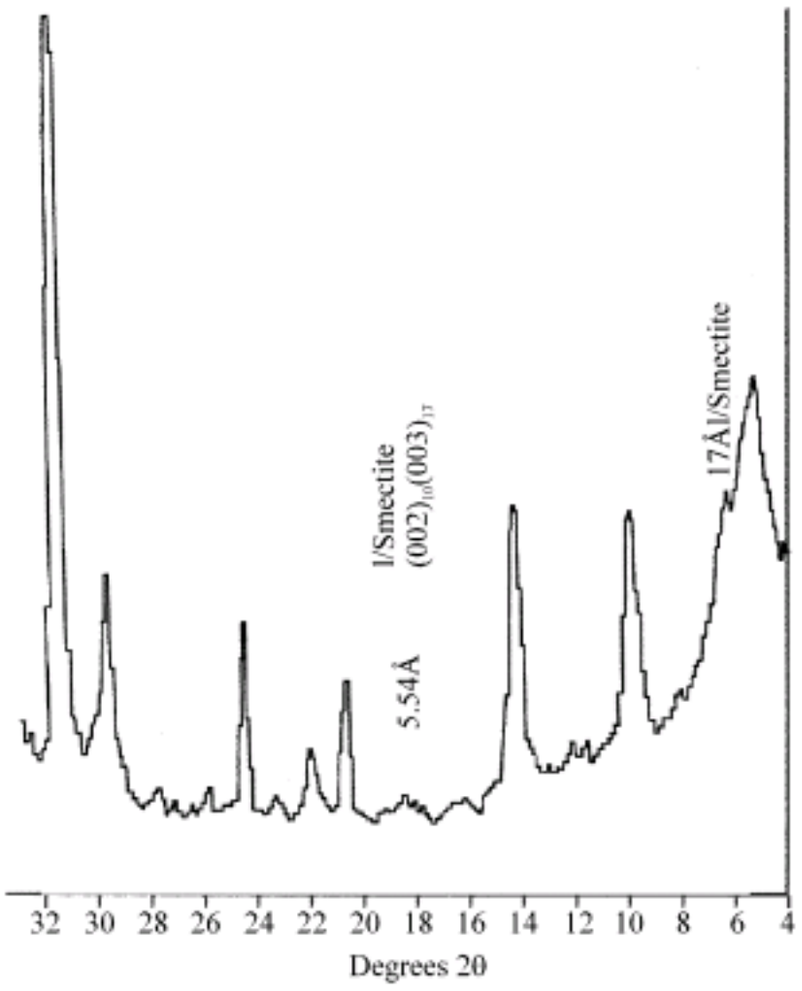

Fig. 4: Identification of illite/smectite mixed-layer clay, X-ray diffraction profile of clay fraction of Tipam shale, western flank of the Bandarban Anticline. Sample solvated with ethylene glycol

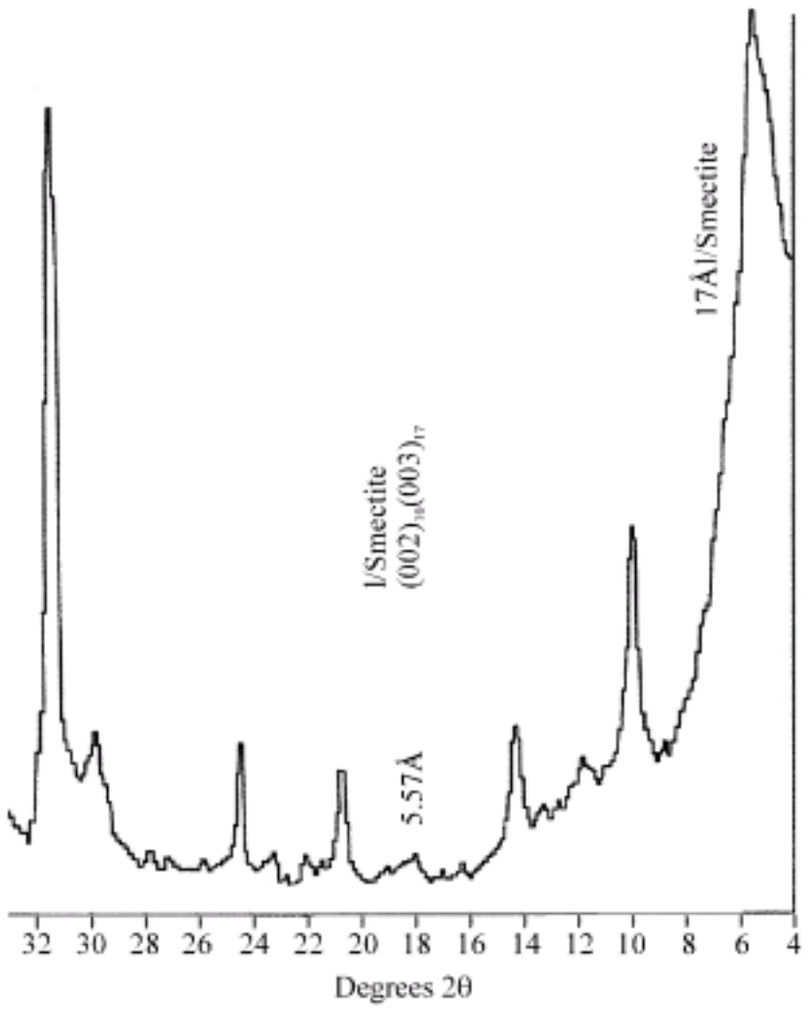

Fig. 5: Identification of illite/smectite mixed-layer clay, X-ray diffraction profile of clay fraction of Dupi Tila shale, western flank of the Bandarban Anticline. Sample solvated with ethylene glycol

Table 2: Relationships between composition of illite/smectite mixed-layer clay and the $(002)_{10}/(003)_{17}$ peak position

Illite (%)	Smectite (%)	D (Å)
0	100	5.62
20	80	5.57
40	60	5.50
60	40	5.37
80	20	5.16
100	0	5.01

Source: Reynolds (1980)

Semi-quantitative XRD analysis: The mineralogical composition of clay fraction were estimated based on the relative peak intensities (peak height/½ by width ratio) of the respective minerals in the XRD charts following Moslehuddin and Egashira (1996). These ratios were obtained by measuring the heights of basal peaks characteristic of the common clay minerals. For kaolinite the 7 Å peak, for illite the 10 Å peak, for chlorite the 14 Å peak and for illite/smectite the 17 Å peak was used. The average percentages of each of the clay minerals of the Bandarban shales are given in Table 1.

The nature of interlayering in mixed-layer illite/smectites: The diagenetic transformation of illite/smectite involves gradual loss of smectite layer due to conversion to illite with increasing temperature. Perry and Hower (1970) observed that the change in the nature of interstratifications of illite and smectite layers take place from a random to an ordered fashion. Reynolds and Hower (1970) indicated that the illite/smectite minerals with smectite of above 35 to 40% are almost always randomly interstratified and those of lower smectite percentage have ordered interstratifications (i.e., lower expandabilities).

The following three methods have been used for determining the proportions of illite and smectite in the randomly interstratified illite/smectite mixed-layer clay.

~5 Å peak migration method: This method of Reynolds and Hower (1970) was used to determine the composition of illite/smectite clay by measuring the $(002)_{10}/(003)_{17}$ peak position from glycolated samples. An increase in the proportion of illite layers in the illite/smectite clays causes this peak to migrate towards 5 Å. Table 2, which is taken from Reynolds (1980) shows the reflection positions for corresponding illite percentages in the illite/smectite clay. In this Table 2, it is shown that the $(002)_{10}/(003)_{17}$ migrates from 5.62 to 5.01 Å corresponding to an increase of illite layer from 0 to 100% in the illite/smectite mixed-layer clay mineral.

In this study, it was observed that for samples of the Tipam Sandstone and Dupi Tila Formation, these reflections appear at approximately 5.54-5.57 Å (Fig. 4, 5).

Table 3: Estimation of smectite percentages in the illite/smectite mixed-layer clay by the saddle/17 Å ratio method

Formations	Saddle/17 Å	Smectite (%)
Dupi Tila	0.49	71
Dupi Tila	0.48	70
Dupi Tila	0.48	70
Tipam Sandstone	0.59	65
Tipam Sandstone	0.58	65
Tipam Sandstone	0.59	64

When these reflections are compared with Table 2, it can be noted that the mixed-layer illite/smectite clays of the above formations contain about 80% smectite layers and 20% illite layers.

Saddle/17 Å intensity ratio method: This method, which involves the measuring of the intensity ratio of 17 Å peak and the saddle on the low angle side of the peak, was first introduced by Reynolds and Hower (1970). The ratio between the intensity of the saddle or valley on the low angle side of the 17 Å peak and the intensity of the 17 Å peak is a function of the proportion of illite layers in the mixed-layer illite/smectite clay. Later, Rettke (1981) modified the original Reynolds and Hower (1970) method by including mechanical mixture of discrete illite in various proportions with illite/smectite clay.

In this investigation, saddle/17 Å peak intensity ratios were measured for the samples of the Tipam Sandstone and Dupi Tila Formations and the results are shown in Table 3.

From Table 3, it can be observed that saddle/17 Å ratio ranges from 0.48 (Dupi Tila Formation) to 0.58 (Tipam Sandstone Formation). For the samples under consideration, adopting illite/smectite proportion of 1:3 from Rettke's (1981) curve, such an increase in saddle/17 Å ratio would correspond to a decrease from about 70% to about 65% smectite layers in the illite/smectite mixed-layer clay within the formation range.

17:10 Å ratio method: The method involved measuring the intensities of the basal 17 and the 10 Å reflections from glycolated and heated (350°C) samples, respectively. In this investigation smectite percentage in the illite/smectite clay by the 17:10 Å height ratio method was measured and the results are shown in Table 4.

From Table 4, it is clear that 17:10 Å ratio decreased from 2.81 (Dupi Tila Formation) to 1.39 (Tipam Sandstone Formation). Using the calibration curve of 17:10 Å ratio, it is found that such a decrease in the 17:10 Å ratio corresponds to a decrease of expandability in illite/smectite clay from 70% smectite layer in the Dupi Tila Formation to 50% smectite layer in the Tipam Sandstone Formation. The values obtained for individual samples

Table 4: Estimation of smectite percentages in the illite/smectite mixed-layer clay by the 17:10 Å height ratio method

Formations	17:10 Å	Smectite (%)
Dupi Tila	2.81	70
Dupi Tila	2.26	60
Dupi Tila	2.65	65
Tipam Sandstone	1.39	50
Tipam Sandstone	2.22	60
Tipam Sandstone	1.75	55

Table 5: Variation in the smectite contents with age (Bandarban Anticline, Bandarban Hill District)

Formations	~5 Å peak migration method	Saddle/17 Å intensity ratio method	17:10 Å ratio method
Dupi Tila	80	70	65
Tipam Sandstone	80	65	55

using the three methods mentioned above give a slight decrease of smectite layers with increasing depth (age) (Table 5).

DISCUSSION

The most important change in the clay mineral assemblage of the shales from the western flank of the Bandarban Anticline is shown in the diffraction profiles of glycolated samples by a gradual loss of 17 Å peak intensity of the illite/smectite clays from the Dupi Tila to the Tipam Sandstone Formation (Fig. 6). There is substantial evidence in the literature of the diagenetic transformation of expanding clay minerals (Imam and Shaw, 1985; Bloch and Hutcheon, 1992; Sato *et al*., 1996; Srodon, 1999; Pablo-Galán *et al*., 2001; Totten, 2002; Kemp *et al*., 2005; Grainger, 2006). These studies indicate that the gradual loss of smectite due to conversion to illite in smectite or illite/smectite mixed-layer clay minerals is amongst the most important diagenetic changes that take place in shales with such expandable clay minerals. Hower (1981b) and Srodon (1999) postulated that the most distinctive characteristic of the diffraction profile of the randomly interstratified illite/smectite (smectite layer more than 40%) is that the 17 Å peak intensity decreases progressively until it completely disappears. Gradual decrease and disappearance of kaolinite is another commonly recorded diagenetic change in shales (Imam and Shaw, 1985).

In this study, randomly interstratified illite/smectite clays are represented by 17 Å glycolated peaks. Gradual loss of 17 Å peak intensity in the diffraction profiles of the sediments under consideration (Fig. 6) are also noticed may be interpreted as a decrease in the percentages of smectite layers in the illite/smectite clay due to the diagenetic transformation of smectite to illite. The three methods namely; (a) ~5 Å peak migration method; (b) Saddle/17 Å intensity ratio method and © 17:10 Å ratio

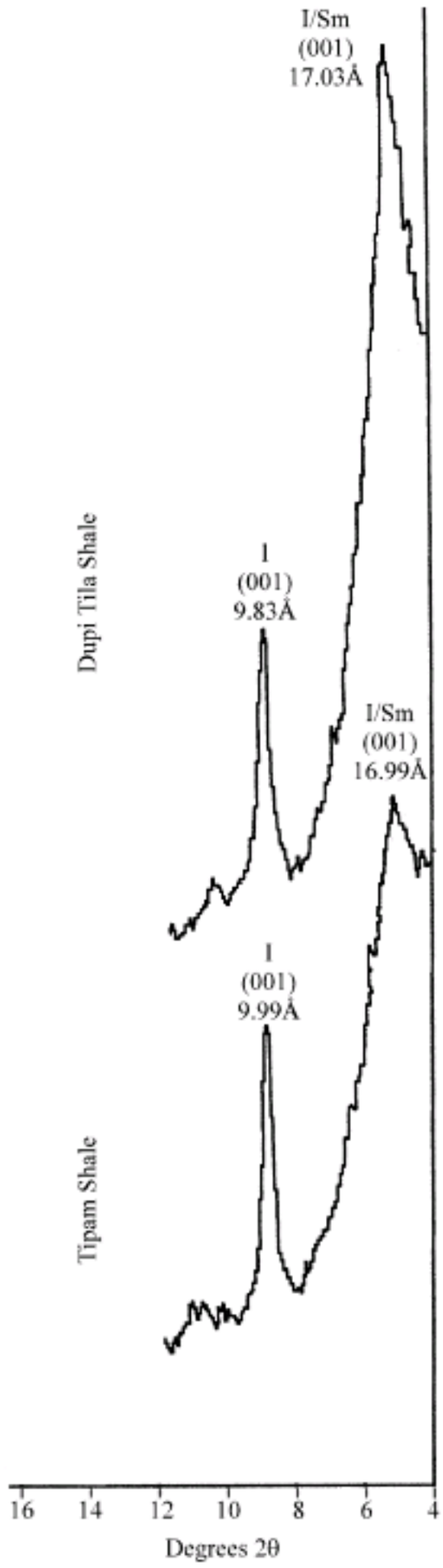

Fig. 6: XRD profiles (glycolated) of clay fractions of the Tipam and Dupi Tila shales, Bandarban Anticline showing gradual loss of 17 Å peak intensity in older rocks

method used to determine the composition of the illite/smectite clays also give a slight decrease of smectite layers with increasing depth (age) as estimated by quantitative method from XRD profiles (Table 5). It would be wise to mention here that different values of smectite percentages in the illite/smectite clay obtained by these different methods as found in this study was also found by Perry and Hower (1970) in the US Gulf Coast Tertiary sequence and Imam and Shaw (1985) in the Neogene clastic sediments, Bengal Basin. It is believed that smectite to illite transformation may be a source of additional silica to the interbedded sandstones for the generation of little quartz overgrowth cementation. That might suggest that shale derived Si^{4+} ions may play a minor role in the secondary quartz cementation. It is also believed that a small volume of water derived from the smectite diagenesis should have been available and migrated from smectite interlayers to the pore water system, contributing to the generation of overpressure encountered in the subsurface of Bandarban structure. The influence of such overpressured shale masses in structural developments i.e., shale diapirs etc. in the Bandarban structure should be properly evaluated.

CONCLUSION

In these exposed Tipam Sandstone and Dupi Tila Formation of the Bandarban Anticline, illite/smectite mixed-layer clays form a major group of clay mineral and there are distinct indications that the diagenesis has taken place involving gradual loss of the smectite from younger to older rocks. There is no discrete smectite in the studied shales but all expandable components are identified as mixed-layer illite/smectite clays. Randomly interstratified illite/smectite clays are represented by 17 Å glycolated peaks. The gradual decrease of the smectite proportion in the random illite/smectite clay is reflected by a gradual decrease in the 17 Å peak intensity which is possibly interpreted as a decrease in the percentages of smectite layers in the illite/smectite clay due to the diagenetic transformation of smectite to illite. Furthermore, kaolinite clay mineral displaying decreasing tendency in older rocks is amongst other diagenetic changes recorded in shale with increasing burial depth (age) while chlorite clays do not show any systematic variation in concentration with increasing age.

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