Deciphering Mineralogical Composition and Clay Diagenesis in Exposed Shales of Jaintiapur, Surma Basin: Unveiling Insights into Burial Diagenesis

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ABSTRACT: Mineralogical analysis of exposed shale samples in the Jaintiapur region was conducted utilizing X-ray diffraction (XRD) techniques. The studied shales are characterized by illite, chlorite, illite/smectite mixed layer clay, and kaolinite as the predominant clay minerals, with a relatively lower proportion of smectite. Additionally, the non-clay fraction consists mainly of quartz, feldspar, dolomite, and trace amounts of pyrite. Notably, illite and chlorite exhibit prominent abundance within the clay portion, while quartz is the prevailing non-clay mineral. Furthermore, the investigation reveals a notable enrichment of illite and chlorite with the advancement from younger to older stratigraphic formations in the studied shale samples, indicating greater intensity in clay mineral diagenesis in deeper stratigraphic formations. The transition from smectite to illite minerals can release Si, Ca, Fe, and Mg ions, potentially entering nearby sandstones and leading to cementation with quartz, chlorite, and calcite. This process aids in retaining pore water in shales. In this context, overpressure development influenced by clay diagenesis is not primarily due to cementation while undergoing smectite to illite conversion but due to illite packet formation within smectite, reducing shale permeability. During this conversion, interlayer water shifts to free water, causing volume reduction compounded by the low permeability of shale, trapping fluids in the pores. This expansion of pore water, reduced velocity, and increased pore pressure suggest that mineral transformation with burial depth might contribute to overpressure development. However, the observed decline in smectite clays in sediments transitioning from younger to older, culminating in their absence in older shale-dominated formations, explains the presence of diagenetic illite and chlorite. This study underscores the progressive diagenetic transformation in clay mineralogical composition with progressive burial across the sedimentary succession.

Keywords: Outcrop Shales; Mineralogy; Clay Diagenesis; Surma Basin

INTRODUCTION

Situated within an actively dynamic tectonic region where the Indian, Burmese, and Eurasian plates converge, the Bengal Basin is a vast repository of sediments (Hossain et al., 2020). The intricate river network coursing through the basin is pivotal in the substantial sediment deposition, resulting in a stratigraphic thickness of approximately 22 km (Alam et al., 2003). The Bengal Basin's Surma Group has a sedimentary sequence of about 2.3-3.1km, and its deposition is attributed to a deltaic to the shallow marine transitional environment (Rahman et al., 2022). Located north of Chittagong-Tripura Fold Beld, the Sylhet Basin is an active subsiding basin accommodating thick Tertiary sediments (16 - 18 km). The Neogene shale constitutes a primary lithology of the Surma Basin, forming the "backbone" of the folded belt consisting of alternating sandstone, shale, and siltstone units and has an overall shale: sand ratio of 1:1, though shale is prevalent in the lower sections of Middle Bhuban unit having shale: sand ratio up to 10:1. In addition, Kopili Shale deposited during the Eocene has a sedimentary thickness of about 450m whereas, the Jenum Shale is reported to be ~2000m thick within the Surma Basin area (Holtrop and Keizer, 1970).

The transformation of clay minerals through an intermediate mixed clay layer represents a diagenetic reaction widely recognized and established in shale-dominated lithologies during progressive burial. Previous studies have documented this transformation, highlighting its role in releasing hydrocarbons from mudrocks (Burst, 1969; Bruce, 1984), catalyzing HC accumulation (Johns and Shimoyama, 1972), generating excessive subsurface hydraulic pressure (Powers, 1967), as well as contributing to cementing processes in clastic rocks (Lahann, 1980).

Burst (1957) conducted one of the initial comprehensive studies of clay mineral assemblages and reported that

swelling smectite transforms into non-swelling illite as the depth of burial increases. Since then, several studies have confirmed that the ongoing clay lithification with greater stratigraphic depths is related to the gradual reduction in the presence of expandable clay minerals across different geological formations. Other diagenetic changes include a reduction and diminishing of kaolinite, simultaneous augmentation of chlorite, and a decrease of feldspars spotted accompanied by increased T-P with increased stratigraphic depth. Hower et al. (1976) examined the mineral composition and chemical properties of Cenozoic sediments in the Gulf Coast region of the United States. They found that the dominant illite/smectite mixed clay layer transforms from being primarily composed of ≤ 20% illite layers at 1250 meters to approximately 80% illite layers at 5500 meters.

Lanson et al. (1998) investigated the diagenesis of clay minerals in several distinct sedimentary basins worldwide. He discovered that almost identical physicochemical attributes associated with the I-S mixed clay layer are found in sedimentary basins with lower geothermal gradients. Nevertheless, the sediment's age correlates with the greater prevalence and improved crystalline quality of pure illite. Yang and Hesse, (1991) characterize the diagenetic transformation of shale-dominated lithologies in the overthrust belt of the southern Canadian Appalachians based on an X-ray diffraction study.

The lower part of the Bhuban Formation, the Jenum, and the Kopili formations of the Surma Basin have a simple tectonic and depositional history with continuous down warping in foredeep and significant uplift in the fold belt. This tectonic setting with a large volume of shales has substantial implications for clay diagenesis and structural shaping concerning stratigraphic depth (Powers, 1967; Bruce,1984). A comprehensive study of outcrop shales in the Jaintiapur region can reveal the potential dehydration of clay-rich shales due to rising burial temperatures and pressure conditions with stratigraphic depth within the Surma Basin. The present study aims to understand the mineralogy and clay diagenesis with progressive burial from outcrop shale samples of the Jaintiapur area within the Surma Basin.

GEOLOGIC SETTING

The Surma Basin is a prominent and tectonically active sedimentary basin in north-eastern Bangladesh. As part of the Bengal Basin, it started subsiding in the Oligocene and continued until its peak during the Pliocene. The Surma Basin, covering approximately 2.5 million acres, is flanked by the Tripura High to the south, the Barail-Imphal Ridges extending eastward to Assam, and the Shillong Massif along with the surrounding Barail mountains in a northerly direction. It gradually inclines upward as it approaches the Eocene Hinge Zone.

The Shillong Massif's uplift and movable IBR coincided, resulting in the Surma Basin, which produced a high-relief landscape of anticlines and troughs (Hiller and Elachi, 1984). The Burmese Plate's westward movement caused the southeast portion of the Surma Basin to fold. The basin's northern and eastern reaches are significantly more intricate than its southern and western reaches (Haque and Alam, 1982). The synclines of this basin have served as important late Neogene and Quaternary depocenters, with up to 7km of structural relief (Hiller and Elchi, 1984).
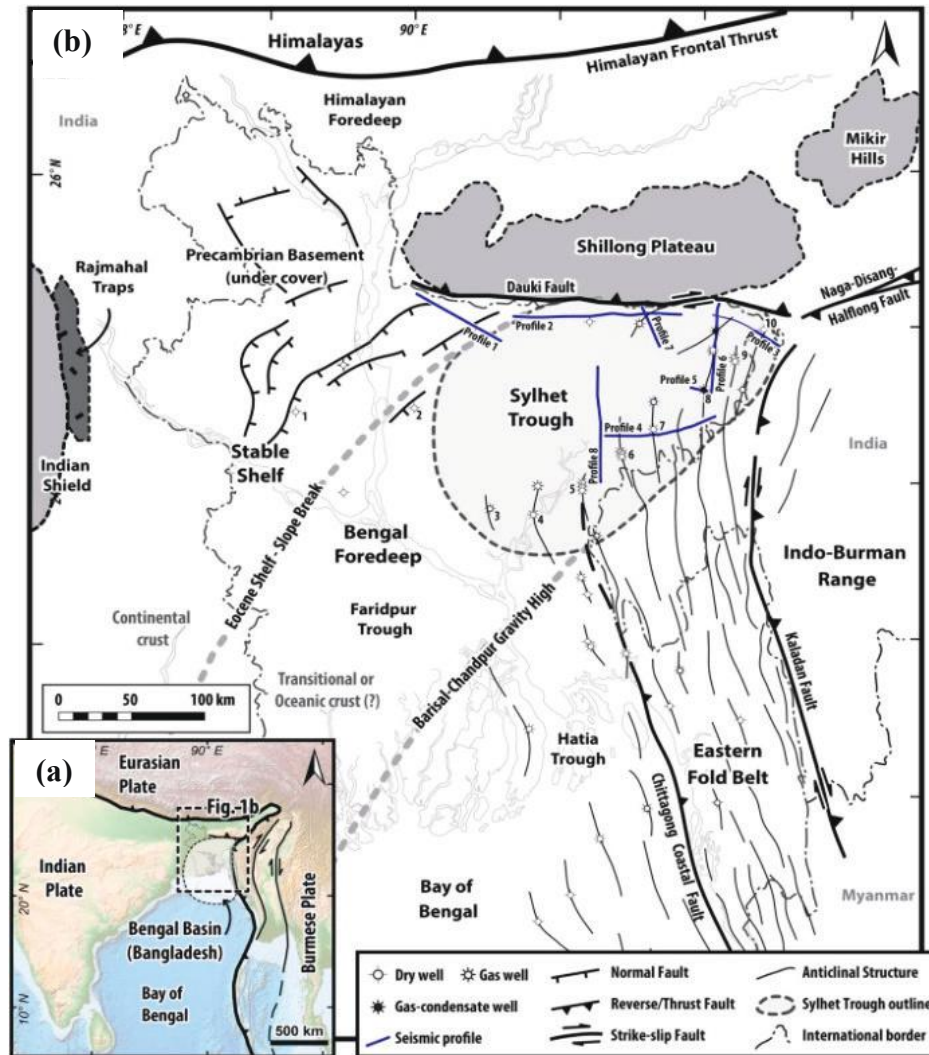
Figure 1: Structural Development and Tectonostratigraphic Representation Depicting the Sylhet Trough, a) Geo-graphical Position of the Sylhet Trough, b) Geological Dynamics Within the Sylhet Trough (Modified after Alam et al., 2003; Abdullah et al., 2013)

Miocene and Pliocene sediments are exposed in the Jaintiapur-Jaflong outcrop area of the Sylhet region. The late Eocene marine Kopili Shale and Oligocene Jenum Shale have also been found to be exposed in the Sylhet region (Reimann and Hiller, 1993; Shamsuddin et al., 2001). The Miocene Surma Group is a substantial succession of clastic sediments that filled the expansive area within the Surma Basin throughout the Miocene-Pliocene era. The Surma Group unconformably overlies the Oligocene-aged Barail Group and is overlain by the Pliocene-aged Tipam Group (Holtrop and Keizer,

1970). Based on gross lithology, the Bhuban and Bokabil formations are the two subunits that make up the Miocene Surma Group (Hiller and Elchi, 1984). The Surma Group contained progradational sequences accumulated in delta-front settings during the transition phase (Gani and Alam, 1999). Conversely, the Tipam Group comprises the Girujan Clay and Tipam Sandstone units formed in environments characterized by braided river systems and lacustrine habitats, respectively (Johnson and Nur Alam, 1991).

`**Table 1:** Stratigraphic Succession of Surma Basin in Bangladesh (Slightly modified after Reimann and Hiller, 1993) (∿∿∿∿ Unconformity)

Age	Group	Formation	Lithology	Thickness (m)
Recent		Alluvium	Sand, silt, clay	Variable
Pleistocene	Dihing	Dihing	Siltstone and pebbly sandstones	3350
Pliocene	Dupi Tila	Upper Dupi Tila	Clay/Shale with occasional sandstones in channels	>175
		Lower Dupi Tila	Coarse ferruginous sandstone with layers of quartz pebbles, a few clays intercalation, and large pieces of petrified wood. Cross-bedding and cross-lamination are common.	
	Tipam	Girujan Clay	Alteration of Clays, sandy clays, and sandstones, which are missing in central Surma valley. Shales are progressively more common towards the top, widely eroded or non-deposited.	3500 (Gowain Trough)
		Tipam Sandstone	Mainly coarse-grained to pebbly sandstone with up to 10-20% argillaceous beds. The pebbles consist of Upper Marine Shales. Shillong granite, quartzite, shale, hornblend, quartz and muscovite.	
Miocene	Surma	Boka Bil	The upper part consists of dark grey shales with pyrite, and the lower part is mainly shales and sandy shales, followed by dominant sand.	3350
		Bhuban	In the upper part, shale predominates, with increasing fine sand at the top. The middle unit alternates shale, sandy shale, and fine-grained sandstone. But lower is mainly sandstone with pebbly sandstone, sandy shale, shale, and silty shale.	
Oligocene	Barail	Renji	Predominantly moderately compacted fine to medium-grained sandstones with few intercalated shales.	860
		Jenum	Firmly compacted, dark shales with interbedded quartzitic sand layers; at the top (Atgram IX), conglomerates; thin conglomeratic intercalations.	>261
		Laisong	Predominantly sandstones.	2,400 (Assam)
Eocene	Jaintia	Kopili Shale	Dark grey shales with sandstone layers at the top and Limestone bands at the bottom.	Several Hundred meters
		Sylhet Limestone	Micritic to bioclastic, partially dolomitized flysch clastics.	Up to 250 m
Paleocene		Tura Sandstone	Alternation of sandstone and fossiliferous Limestone, gray to dark gray shale common. Sandstone contains coal.	—

DATA AND METHODS

A subset of eight surface outcropping shale samples from the Jaintiapur area under the Surma Basin have been chosen for XRD analysis.
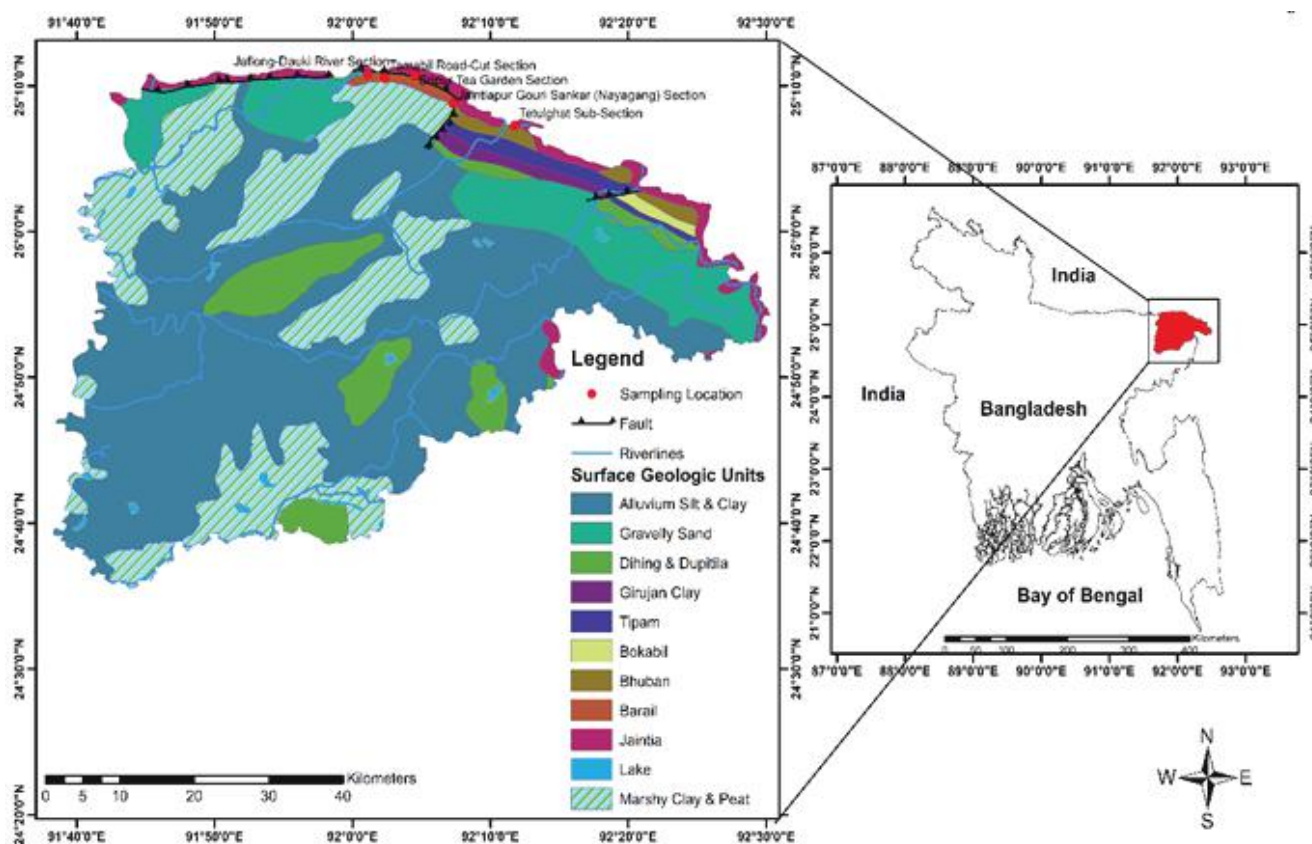

Figure 2: Geological Map Shows the Sampling Location under the Study Area

An un-oriented mount for clay fraction analysis sample weighing approximately 5 grams underwent cleaning, drying, and crushing into small fragments. Whole-rock powder was mounted on the Rigaku X-ray powder diffractometer sample holder facilitated by the CARS authority at Dhaka University.

The unoriented mounts of powdered entire rock specimens were analyzed in an X-ray diffractometer between $5°2\theta$ to $40°2\theta$ using CuKα radiation with a scanning rate of $0.2°2\theta$ /minute and a chert speed of 20 mm per minute providing $0.2°2\theta$ per centimeter print out.

Due to its simplicity, the glass slide method was used to analyze the clay fractions (<2 microns) to create orientated mounts for clay mineralogical analysis. A glass rod was used to thoroughly mix 1 g of dry, sieved sample with 5 ml of distilled water in a 10 ml cylinder. After 10 to 15 minutes, the top solution was carefully drawn using a pipette and gradually spread across the entire surface of a clean glass slide, after which it was left to air-dry under normal environmental conditions. Three different conditions were used to examine the oriented mount of the clay samples: (i) air dried, (ii) following one hour of heating at 550°C, and (iii) after ethylene glycol treatment.

The oriented mounts of the samples were also analyzed utilizing CuKα radiation at 30 Kilovolt and 20 milliamperes with a scanning rate of $3°2\theta$ per minute. The diffractograms were analyzed via "X–ray powder diffraction analysis software." The relative percentages of clay samples were determined by Biscaye's Method. The relative ratios of clay minerals of the studied clay fractions were determined automatically by matching the curve with the reference patterns database from the American Mineralogists Crystal Structure Database (AMCSD).
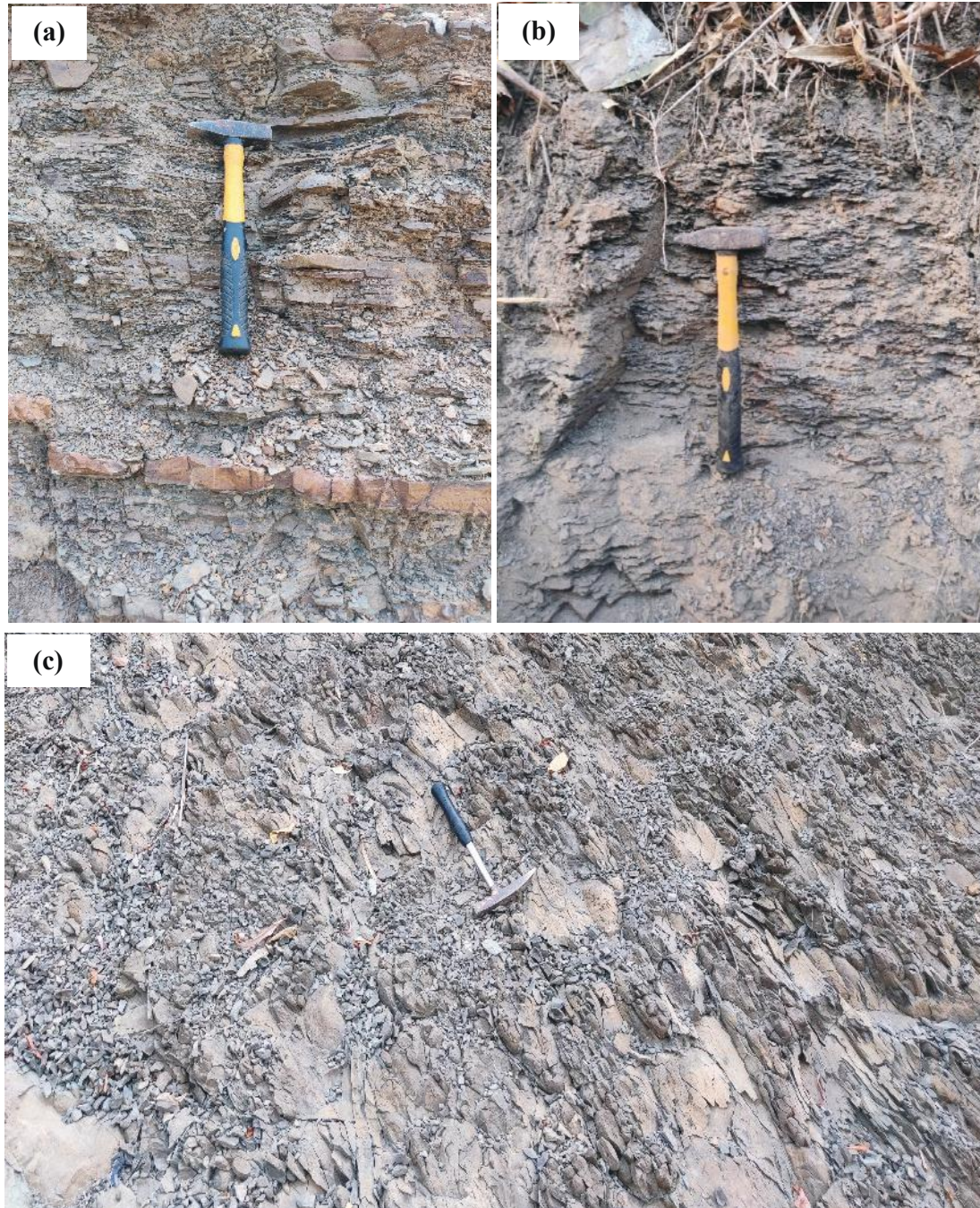
Figure 3: Outcrop Photographs of Investigated Shale Dominated Formations and Outcrop Samples of Three Different Lithologies have been Collected During Field Investigation. a) Kopili Shale of Jaintia Group from the Jaflong- Dauki River Section, b) Jenum Shale of Barail Group from the Sripur Tea Garden Section, c) Bhuban Shale of Surma Group from Tetulghat Section

The X-ray diffraction technique is the most efficient method for determining clay minerals in shale because it provides more accurate results to determine the nature and properties of clay minerals. Qualitative mineralogy of the outcrop shales under investigation is determined with standard XRD interpretation procedures (Moore and Reynold, 1989). The initial step in the qualitative identification process involves identifying a mineral that can account for the most prominent peaks. This selection is subsequently validated by determining the positions of less pronounced peaks associated with the same mineral.

RESULTS AND DISCUSSION

The X-ray diffractograms of selected representative samples under three distinct geological formations are shown in Figure 3. It is required to precisely identify and quantify the different layers within the mixed-layer clay to understand how smectite is converted into illite through interstratified clay minerals (Mosser-Ruck et al., 2005). X-ray diffraction patterns of clay samples after particular treatments are frequently examined in this analysis. The ethylene glycol solvation method typically identifies smectite and vermiculite with high charges. The XRD patterns varied significantly depending on the type of glycation used (Ethylene Glycol liquid or vapor). Since a material exhibits a higher d-value when saturated with liquid ethylene glycol, the liquid EG solvation method was adopted in this investigation. Using liquid ethylene glycol, for instance, caused highly charged vermiculite and smectite (d values of 14Å to 15Å) to expand to 17 Å.

Clay Mineralogy

Illite: Illite is identified as the most prevalent clay constituent determined by the basal peaks at 10.04Å to 10.3Å, 9.91Å, 4.98Å, 4.45Å, and 1.89 –1.92Å slightly larger than the normal d-spacing (Figs. 4 and 5). It does not mean the presence of smectite with illite in the illite layer. Values over 10Å are probably due to structural disorder caused by the presence of hydrated cations in the interlayer sites. The profiles of illite are unaffected by ethylene glycol solvation or exhibit a minor contraction that occurred as the samples were subjected to a temperature of 550°C due to the presence of expandable illite layers. No significant variation in illite peak intensities depends on their position in stratigraphy or geography.

Kaolinite: Distinguishing Kaolinite from other group members is challenging until it is predominantly composed of a single mineral. In this context, "Kaolinite" pertains to the Kaolinite clay mineral group, a notable sample component. Reflections at 7.14Å and 3.50Å indicate the presence of Kaolinite. Coexistence with chlorite complicates identifying Kaolinite using distinct reflections at 7Å and 3.58Å. Kaolinite loses X-ray crystalline structure at 550°C for an hour, sometimes erasing the diffraction pattern (Fig. 6). Notably, peak intensities remain consistent regardless of sample stratigraphy.

Chlorite: Chlorite and Kaolinite have distinct structures and origins. Chlorite's diffraction peaks at 14.2 Å differ from Kaolinite's at 7.1Å. Chlorite's even-order peaks often overlap with Kaolinite's (001) series, making differentiation challenging, especially with certain Chlorites. It is delineated by the peaks at 14.5Å to 14.3 Å, 7.13Å to 7.20 Å, 4.7Å to 4.79 Å, and 2.86Å (Figs. 4 and 5). Nevertheless, the 7.20 Å and 3.56 Å peaks were inappropriate for directly identifying chlorite due to potential overlap with kaolinite peaks. Therefore, reflections at 14.25Å and 4.70Å are most readily used for chlorite identification. In the glycol-treated profiles, chlorite reflection at 14.25Å has some intensity, indicating mixed layering.

This research could not quantify the prevalence of smectite because there were no 17Å reflections present. All the samples lack distinct or pure smectite; instead, they contain an identified illite/smectite mixed-layer phase at 14.5 Å in the representative clay samples. It could be attributed to the conversion of smectite into illite by forming mixed clay layers composed of illite and smectite with increasing stratigraphic depth of burial.

Non-Clay Mineralogy

Quartz: In the outcrop samples under investigation, ranging from older to younger formations, quartz emerges as one of the predominant minerals across the spectrum. In the analyzed samples, the presence of quartz is determined by the peaks at 4.26 Å, 3.34Å, 2.46 Å, 2.27Å, 2.24 Å, 1.81Å and 1.54 Å (Fig. 4). However, it also corresponds to a prominent illite reflection of 3.3Å, making 3.34Å quartz peak with the highest intensity. However, it can't be used as a benchmark for quartz abundance.

Feldspars: Identification of feldspars are often be limited to recognizing feldspar's presence and distinguishing alkali-feldspars from plagioclase. Feldspar is found to be the second most abundant non-clay mineral during the investigation, and it is denoted by a pair of peak reflections at 3.18Å to 3.30Å and other particular reflections in the d-spacing range of 3.66 Å to 4.03 Å. K-feldspar is present as a trace amount, which is identified by weak peak reflections at 4.35 Å, 3.74 Å, 3.24Å, and 2.91 Å (Fig. 4). Prominent and distinct peaks at 3.21Å and secondary peaks at 4.03 Å, 3.66-3.68 Å, and 3.75-3.76Å show the presence of plagioclase feldspar in all the samples (Fig. 4). Plagioclase feldspar

is present in greater abundance compared to potash feldspar.

Dolomite: Peaks at 4.03Å, 3.69Å, 2.89Å, 2.67Å, and 2.54Å are typically used to identify dolomite. The presence of dolomite as a trace amount is indicated by a faint signal at 2.88Å in the analyzed samples (Fig. 4).

Pyrite: 3.59Å, 2.79Å, and 2.56Å are significant d-spacings for identifying pyrite. All the examined samples contain trace levels of pyrite, which are detected by a very faint signal at 2.79Å (Fig. 4).

Mica is also found in trace amounts in all the studied samples.
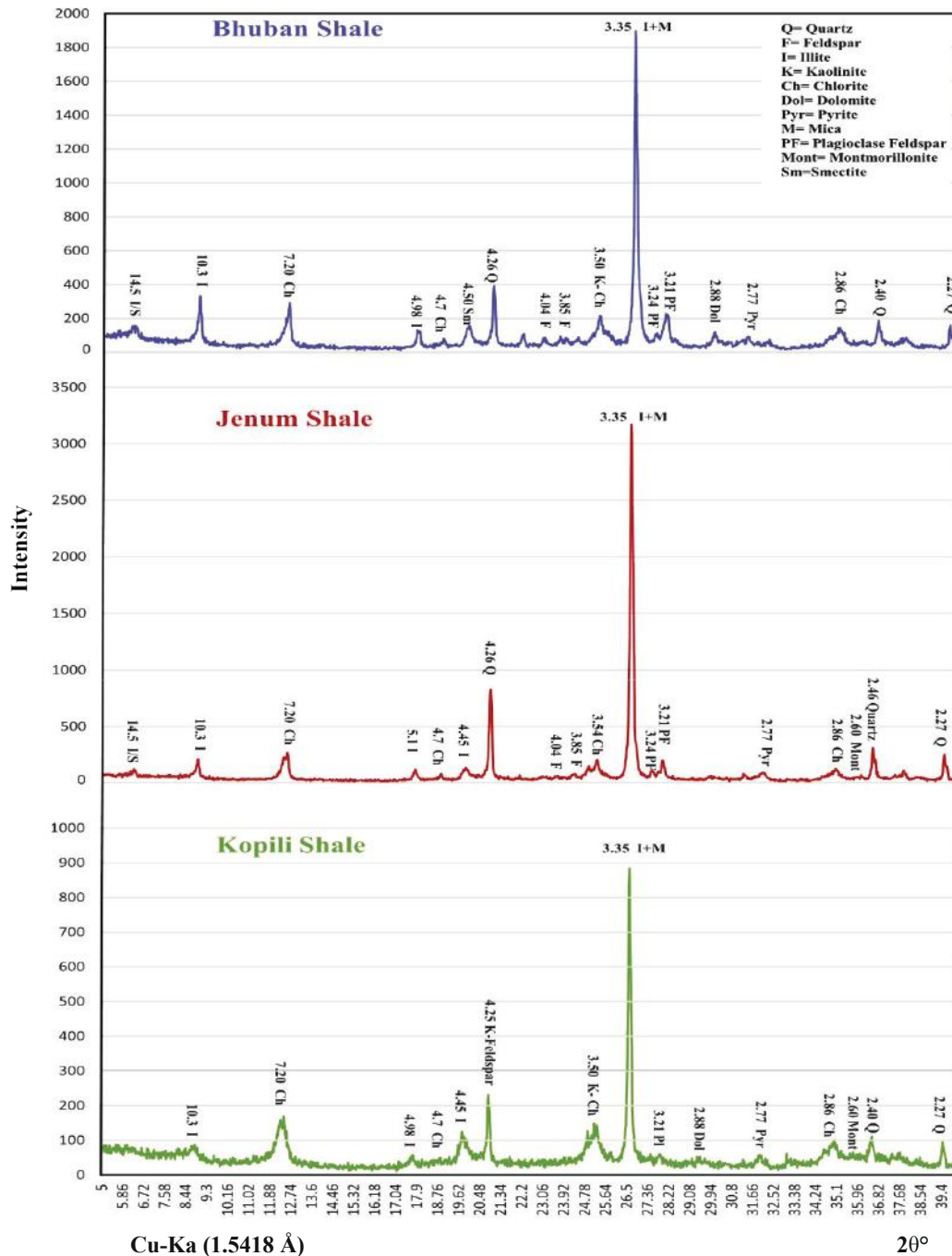
Figure 4: X-ray Diffractograms Display the Overall Mineral Composition of the Whole Shale Samples from Three Different Formations in the Jaintiapur Outcrop Area. Smectite is Absent Whereas, Illite is Found to be Abundant by Several Prominent Reflections (10.3 Å, 4.98Å, 4.45Å). However, Presence of (I/S) Mixed Layer Clay May Indicate Incomplete Conversion of Smectite into Ilite
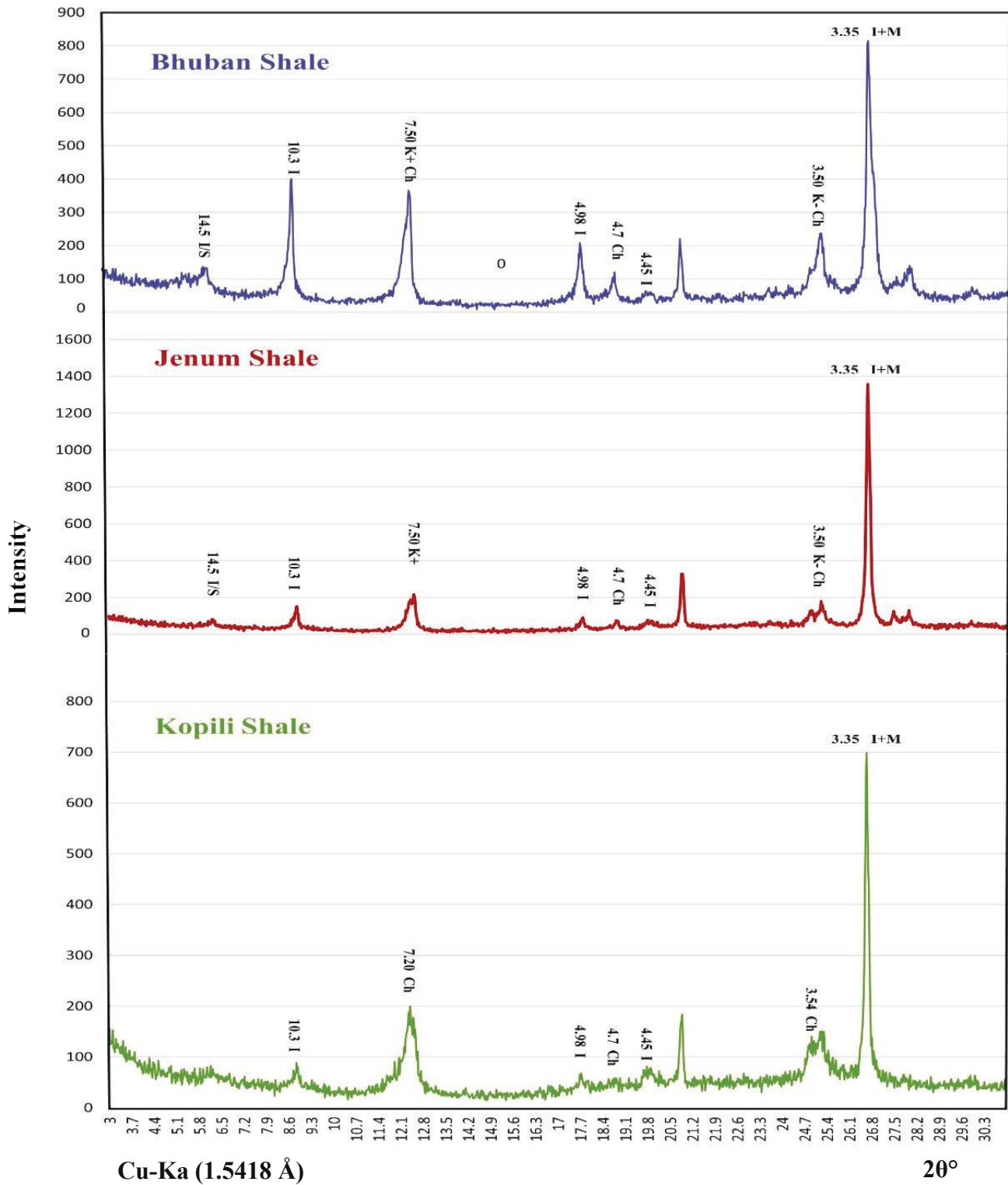
Figure 5: X-Ray Diffractograms of Shale Samples (After Ethylene-Glycol Treatment) of the Jaintiapur Area Show Decrease and Disappearance of 17A° Peak Associated with (I/S) Mixed Clay Layer from Younger to Older Formations. Presence of (I/S) Mixed Layer Clay May Indicate Incomplete Conversion of Smectite into Illite. However, in the Kopili Shale Sample, Absence of Both Smectite and (I/S) Mixed Layer Clay, Coupled with Existence of Illite, May Suggest Potential Illitization of Smectite

Table 2: Relative Percentages of Clay Minerals within the Analyzed Clay Fractions of the Studied Shale Samples Determined Automatically by Matching the Curve with the Reference Patterns Database from American Mineralogists Crystal Structure Database (AMCSD)

Relative abundance of Clay fractions of the collected field samples			
Minerals	**Kopili Shale**	**Jenum Shale**	**Bhuban Shale**
Illite/Smectite	Nil	3.79	14.75
Illite	45.28	37.55	58.32
Chlorite	12.43	10.34	5.48
Illite+Mica	40.00	48.32	18.45

Diagenesis of Clay Minerals

Smectite abundance remains undetermined owing to the lack of a 17Å peak, yet most samples indicated substantial mixed layer Illite/Smectite presence at 14.2 – 14.5 Å (Figs. 4 to 6) (Table 2).

The primary alteration observed in the clay fraction of the shale samples as they undergo burial is the progressive reduction in the reflection at 17 Å (or 18 Å reflection after ethylene glycol treatment) associated with the I/S mixed clay layer. Imam (1983) suggested two potential explanations for the observed alteration in the 17 Å peak: (i) a steady reduction in the quantity of illite/smectite as depth increases, and (ii) progressive diagenetic transformation of the illite/smectite mixed-layer clay in conjunction with stratigraphic depth. In 1994, he observed the nonexistence of 17 Å peak in Neogene rocks retrieved from increased stratigraphic depth. This disappearance is considered diagenetic and attributed to the orderly interlaying of illite with illite/smectite.

Earlier research (Mineralogy and clay diagenesis of Neogene mudrocks in the Sitakund anticline, Bengal basin: An approach to infer burial diagenesis from surface outcropping samples by Gazi et al., 2017; The diagenesis of Neogene clastic sediments from the Bengal Basin, Bangladesh by Imam et al. 1985; X-ray diffraction study on Neogene shales from the Patharia Anticline, eastern Bangladesh, with emphasis on smectite clay dehydration and implications by Imam, 1994) reveals that increasing burial depth will gradually reduce the proportion of smectite within the illite/smectite mixed-layer clay. Hence, the gradual disappearance of the 14.2-14.5 Å peak in the clay fractions diffractograms (Fig. 5) is accompanied by the diagenetic alteration of smectite into illite, which causes a steady decline in the percentage of smectite in the illite/smectite mixed layer clay from younger to older in the outcrop shale samples.

The studied samples had a substantial amount of the I/S mixed clay layer (Fig. 5) (Table 2), implying the occurrence of clay diagenesis and resulting in a reduced smectite content.

The X-ray diffraction profiles of the Sylhet region surface samples show a decreasing trend of expandability. However, ordered illite-smectites are not found (Figs. 4, 5, and 6) that would be in the stratigraphic deeper sediments than the presently studied samples. Imam (1993) showed similar evidence of diagenetic illitization of random illite-smectite by studying the X-ray diffraction profiles of the shale samples of the different wells of the Bengal Basin. He observed the decreasing 17 Å glycolate peak intensity trend until its disappearance, supposedly at about 60% illite, 40% smectite composition, and beginning of ordered interstratification.
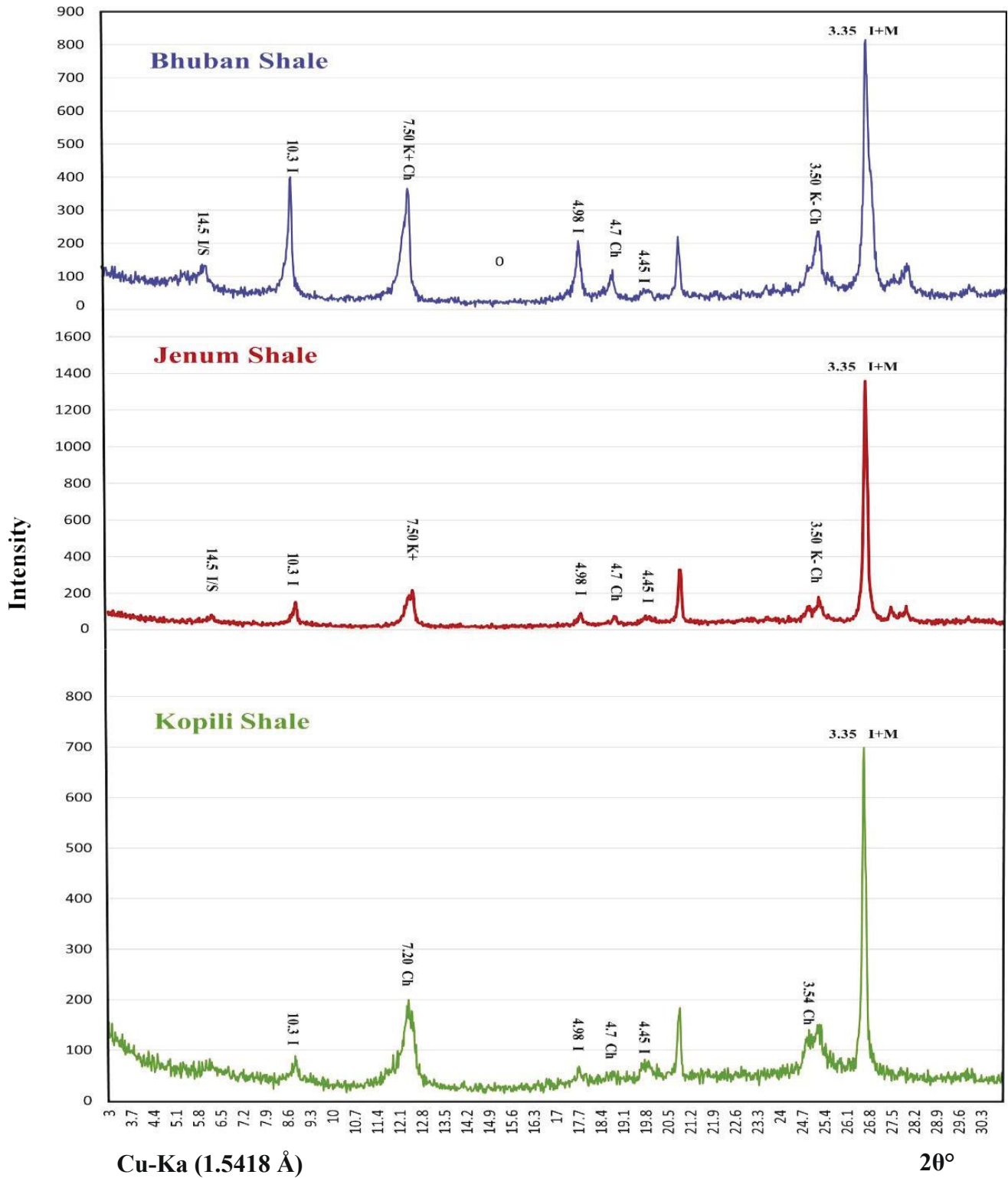
Figure 6: X-ray Diffractograms of Clay Fractions of the Tetulghat Bhuban Shale Sample from the Jaintiapur Area after Air Drying, Heating to 550°C, and Reatment with Ethylene Glycol

CONCLUSION

The qualitative mineral composition of the outcrop shales was ascertained using conventional XRD analysis techniques. The predominant clay minerals identified in the samples from three distinct formations encompass illite, chlorite, illite+mica, illite/smectite mixed layer, and kaolinite. Concurrently, prevailing non-clay minerals comprised quartz, K-feldspar, plagioclase feldspar, dolomite, and pyrite. Progressive disappearance of the 14.2-14.5 Å peak in the diffractograms of the clay fractions of the studied shale samples occurred through the loss of smectite, which led to a decline in the proportion of smectite within the mixed-layer illite/smectite with increasing burial depth from younger to older formations as well as resulting in an organized structure of I/S mixed layers.

The smectite-illite transformation creates a closure impact by releasing Si, Ca, Fe, and Mg ions. These ions can enter nearby clastic rocks and settle Quartz, Chlorite, and Calcite cement. This process can potentially cause cementing and aiding in the retention of pore waters within the shales.

In contrast, overpressure development accompanied by diagenetic transformation of smectite into illite doesnot occur due to cementation rather than the reduction in shale permeability caused by the creation of illite clusters. This formation decreases the accessibility of water pathways through dislocations along with ion transportation, potentially creating an effective hydraulic seal.

The diagenetic conversion of smectite-into-illite and chlorite with progressive burial is evident by significant amounts of Illite, Chlorite, and I/S mixed-layer clay within the examined samples. All these are clear evidence of clay diagenesis with progressive burial, and the intensity of clay diagenesis increases in the deeper formations compared to the younger ones within the Surma Basin.

Furthermore, during the smectite-to-illite conversion, a common diagenetic process in shales, the interlayer water in smectite transfers to free water, causing a volume reduction in the shale. This volume decrease, combined with the limited permeability of shales, results in the trapping of fluids in the pore system. As a result, the pore fluid expands in the nearby sandstones, increasing pore pressure and, thus, overpressure development. Hence, the mineral transformation associated with burial depth could potentially have played a role in the formation of overpressure.

ACKNOWLEDGMENT

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