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Clay minerals study of the Kolosh Formation at selected sections from northern Iraq: Implications for provenance history

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Abstract

The mineralogical study using X-ray diffraction (XRD) supported by scanning electron microscopic (SEM) examination and energy-dispersive spectroscopy (EDS) on the claystone of the Kolosh Formation from northern Iraq was conducted to Shows the provenance history of rocks. Chlorite, montmorillonite, illite, palygorskite, and kaolinite were recorded in different amounts in the study area. The association of montmorillonite and chlorite in the claystone of the Kolosh Formation (Paleocene) refers to the marine environment. Chlorite and montmorillonite are the common minerals in the Kolosh Formation with less common of illite, kaolinite and palygorskite. These clay minerals are of authigenic, detrital and diagenetically origin, which are controlled mainly by the source rocks, paleoclimatic conditions and the burial diagenesis. The clay minerals assemblages refer to be derived mainly from Fe-Mg rich with minor Si-Al rich silicate minerals, which are very common in the ophiolites associated with the basic igneous rocks. These rocks composed the major lithological units in the Zagros Thrust Belt of NE Iraq.

Keywords: Clay minerals; Sem; Xrd; Eds; Kolosh Formation; Northern Iraq.

دراسة معدنية لتكوين كلوش في مقاطع مختارة من شمال العراق: تداعيات حول تاريخ المصدر

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الخلاصه

أجريت الدراسة المعدنية باستخدام حيود الأشعة السينية (XRD) المدعومة بالمجهر الماسح الإلكتروني (SEM) وفحص مطيافية تشتت الطاقة بالأشعة السينية (EDS) للحجر الطيني ضمن تكوين كولوش في شمال العراق لإظهار مصدر المعادن الطينية. تم تسجيل معادن الكلوريت والمونتموريلونايت والإيلايت والباليجورسكايت والكاؤولين بنسب مختلفة داخل صخور الدراسة. تشير ترافق الكلوريت والسميكتايت في الحجر الطيني لتكوين كولوش (باليوسين) إلى البيئة البحرية لهذا التكوين، حيث تظهر الدراسة وفرة الكلوريت والسميكتايت في تكوين كولوش، كما تظهر نسبة أقل من معادن الإيلايت والكاولين والباليجورسكيت. هذه

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المعادن الطينية هي من أصل متبقي وتشكل جديد وناتج عن العمليات التحويرية، والتي يتم التحكم فيها بشكل أساسي عن طريق صخور المصدر وظروف المناخ القديم وتطور الدفن. تشير تجمعات معادن الطين إلى أنها مشتقة أساسًا من معادن غنية بالحديد المغنسيوم مع معادن سيليكات ثانوية غنية بالسيليكون والالمنيوم، وهي شائعة جدًا في الأفيولايت والصخور البركانية القاعدية المرتبطة بها. تتكون هذه الصخور من الوحدات الصخرية الرئيسية في حزام زاكَروس الزاحف شمال–شرق العراق.

1. Introduction

The Kolosh Formation is considered a flysch-type sediments deposited in a Middle Paleocene trough trending NW-SE relatively, a narrow area in northeastern Iraq [1]. It is composed of interbedded sandstones and mudstones/shales successions arranged in graded turbidity cycles [2]. The Kolosh Formation is widely distributed in the high folded zone in NE Iraq, representing rock outcrops marked on the tectonic map (Figure 1).

Some previous studies [3] Indicated the dominance of chlorite in claystone of the Kolosh formation. The [2] indicated montmorillonite is an abundant mineral in the claystone units. The origin of most of these minerals and the source of their predominant composition are igneous and metamorphic rocks rich in ferromagnetic minerals [4].

The study aims to clarify the origin and provenance of the Kolosh Formation from north Iraq based on studies deduced from clay mineral study.

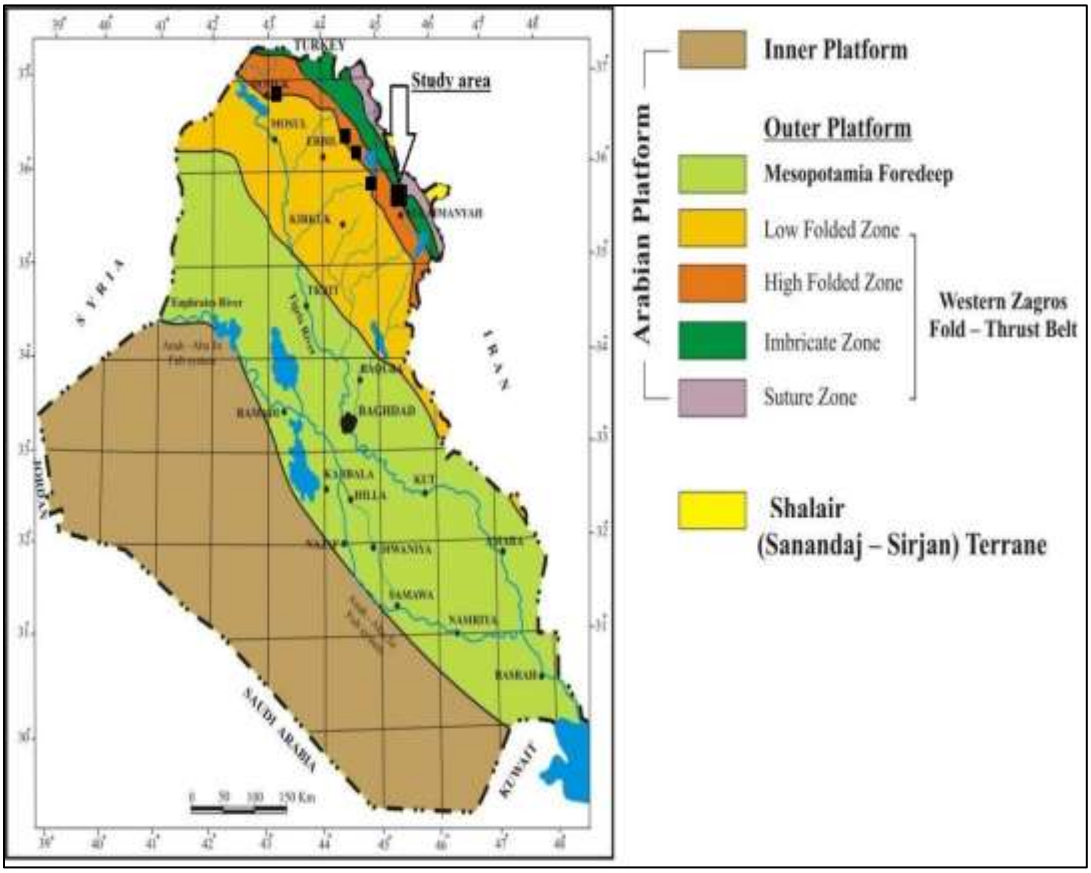


Figure 1: Tectonic map shows the selected sections north of Iraq after [5].

2. Materials and Methods

Clay mineral analysis was performed by x-ray diffraction (XRD), scanning electron microscope (SEM) and energy-dispersive spectroscopy (EDS) of selected samples from the studied formation at both localities in northern Iraq. Five clay stone samples with their locations are presented in (Table 1). The distribution of clay minerals in the claystone samples is almost similar. Therefore, representative scans for x-ray diffraction and energy-dispersive spectroscopy were conducted at the Ministry of Industry and Minerals / Construction Department (Figures 2 and 5). These selected scans were chosen to represent the clay fractions. The clay fraction was then concentrated before x-ray diffraction techniques. Calcium carbonate was removed from the samples using 1/5 N hydrochloric acid. According to the Stokes law, a fraction smaller than 2mm was decanted to make an orientated slide. [6].

The clay minerals were identified using the information provided by [7], [8], [9].
SEM analysis was carried out using the smart scan strategy by Inspect F 50 Device from the US FEI company at Al-Khoura Office, Baghdad.

Table 1: Location of the selected sections

	Selected sections	Position	Longitude (E)	Latitude (N)
1	Tigran	Azmar	45°31'27.2"	35°40'24.4"
2	Kalksmaq	Kalksmaq	44°55'10.2"	35°56'03.4"
3	Jelly	Jelly town	44°37'32.5"	36°10'22.9"
4	Hiran	Hiran	44°31'55.8"	36°16'44.0"
5	Pady	Duhok	43°05'40.7"	36°54'18.3"

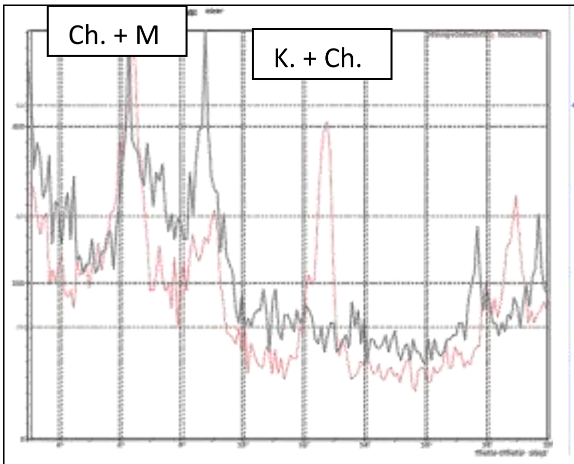
3. Results

X-ray diffraction (XRD) analysis revealed the presence of chlorite, montmorillonite, kaolinite, palygorskite and illite are distributed in study areas (Figure 2). It was found that the chlorite and kaolinite as dominant minerals, as indicated by [8], while the proportion of montmorillonite, palygorskite, illite and vermiculite minerals were found in less quantity.
The EDS results of claystone showed are listed in Table 1. The EDS examination was carried out by point-counting, i.e. a comprehensive sample survey, as shown in Figure 3. High iron and magnesium and less than oxygen and silica support the dominance of Fe-Mg minerals such as chlorite in all sections.

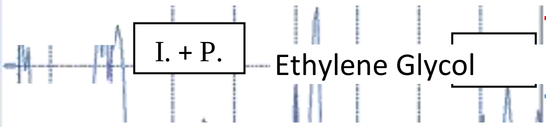
SEM shows the presence of montmorillonite as framboidal forms and palygorskite as long fibers as delicate filamentous outgrowths from montmorillonite (Figures 3 and 4). Illite was found as fine flakes or as a crust, and chlorite was found in small disc shapes. Kaolinite was found in a degraded state with pitted surfaces, while illite was found as fine flakes or as a crust. The following is a full explanation of the clay minerals that have been identified:

3.1 Chlorite

Chlorite is one of the widely distributed clay minerals in rocks. Chlorite has been distinguished depending on the base reflections, in the first order at 14 A^{o}, in the second order at 7A^{o} and the third reflection at 4.7 A^{o}, respectively. Chlorite is characterized by no effect when treated with ethylene glycol; when heated to 550 degrees, the peak intensity of the level (002) decreases as a result of removing kaolinite, which has the same peak value and leads to an increase in the reflection intensity to the 001 and it decreases to the other levels [7].



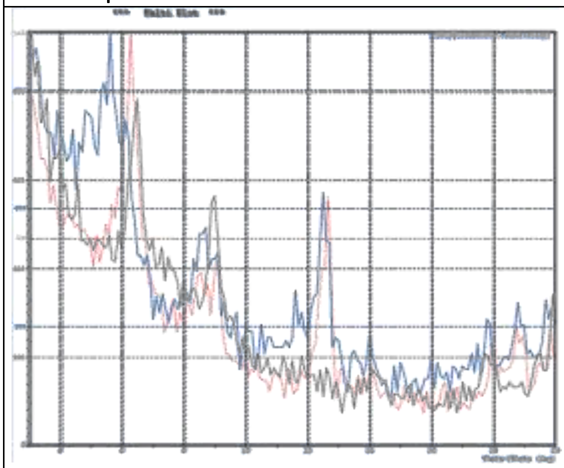
Normal



Ethylene Glycol

Heating 550°

1- sample from first section

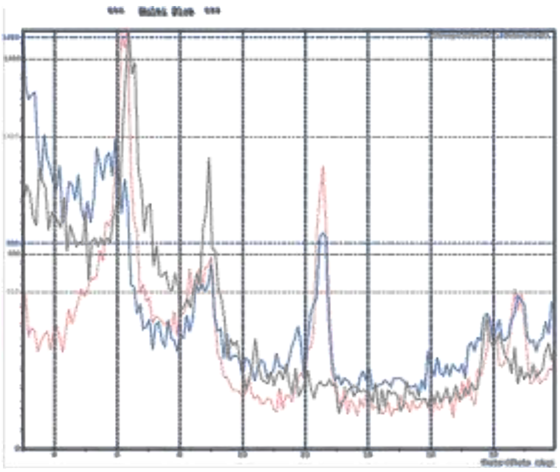


Normal

Ethylene Glycol

Heating 550°

2- sample from second section

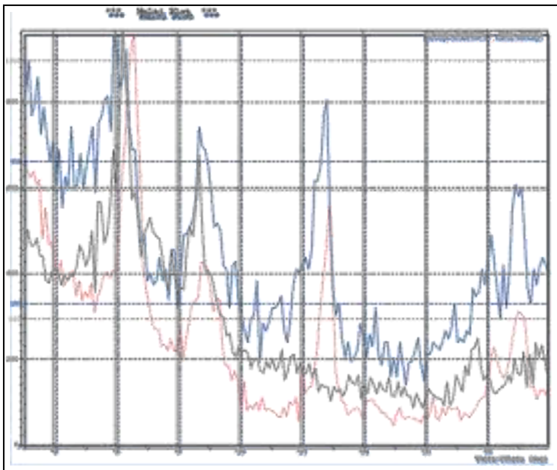


Normal

Ethylene

Heating 550°

3- sample from third section

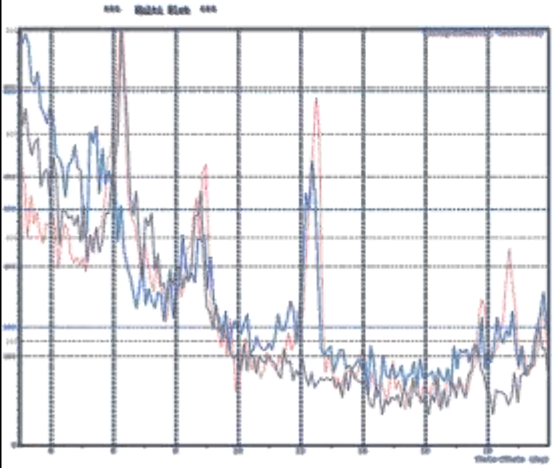


Normal

Ethylene Glycol

Heating 550°

4- sample from fourth section



Normal

Ethylene Glycol

Heating 550°

5- sample from fifth section

Figure 2: X-ray diffraction shows various clay minerals identified in the sections.

Chlorite is the most common mineral found in the Kolosh Formation. It is formed by weathering ferromagnesian minerals with high Mg, Fe, and Ca content, which are abundant in the basic igneous and metamorphic rocks [10]. SEM images reveal tiny disc-shaped chlorite (Figure 4 E and 4 F), which may be inherited from previous chlorite-rich igneous rocks or derived as detrital clays from older volcanic rocks [10].

3.2 Montmorillonite

Montmorillonite is one of the group smectite minerals and is less common than chlorite but more common than kaolinite and illite. Distinguish this mineral by the (001) basal reflection at an angle ($2\theta = 6.25^{o}$) with a distance (d = 14A°). When treated with ethylene glycol, the reflection value changes as it shifts to the right because ethylene glycol leads to an expansion in the crystalline structure of the mineral, and montmorillonite changes to illite upon heating to a temperature of (550°C) [11], [12]. SEM shows montmorillonite as framboidal and platy forms, indicating an authigenic origin (Figure 3 B).

3.3 Palygorskite

Palygorskite was diagnosed by the basal reflection of the plane (001) at an angle (2θ = 8.20°) and with a distance (d = 10.5 A°) [8]. When treated with ethylene glycol, it is noticed that its peak does not expand. After heating at 550°C, its intensity decreases due to water loss without changing its mineral composition, which distinguishes it from illite [7]. It is formed from the weathering of ultrabasic igneous rocks [13]. This mineral is made up of either naturally occurring long flexuous fibers (Figure 3 A) or detrital fibers that are short and fragmented (Figure 3 F). Because of the subsiding shallow marine deposition with typical turbidity currents, which favors the detrital nature of clay minerals, the chemical precipitation of these clay minerals is not conceivable [3].

3.4 Illite

The 001 basic reflection distinguished illite at an angle (2θ = 8.5) and a distance (d = 9.9A^o). The value does not change upon exposure to ethylene glycol vapour, and the intensity of the reflection increases upon heating to (550°C) [11]. Illite is formed diagenetically or in weathering zones due to alteration of muscovite, biotite, and k-feldspar [14]. The different proportions of illite in the Kolosh formation [15] indicate the mineral was formed in non-acidic environments rich in potassium as a result of weathering of potassium feldspar within igneous and metamorphic rocks. The SEM shows illite as flakes or as crusts (Figure 3 F) indicating an alteration form of illite from older feldspars or other silicate minerals.

3.5 Kaolinite

Kaolinite mineral has been distinguished depending on the basal reflections of the level (001) at (7.1) A^o. Both chlorite and kaolinite at 7.1 for the 001 level of kaolinite and 002 for chlorite show that their survival with the same intensity upon heating indicates the presence of chlorite mineral. In contrast, its disappearance suggests the presence of kaolinite [10]. Scanning electron images show kaolinite as hexagonal plates with pitted surfaces (Figure 4 A), degraded plates (Figure 3 C), and plates aligned face-to-face (Figure 3 D) might all be regarded as removed and redeposited detrital kaolinite [16]. The illatization was noticed in kaolinite (Figure 4E).

3.6 Vermiculite

Vermiculite appears at ~14 A^o, showing an increase in the peak intensity when treated with ethylene glycol and collapsing at 550 C^o. Vermiculite deposits are formed from the alteration of igneous rocks [17]. Polygonal sheets with flaked borders are observed in vermiculite formed by altering parental materials [18]. Vermiculite is shown in scanning (Figure 4 D).

4. Discussions

The clay minerals in the terrigenous Paleocene sequences (Kolosh Formation) from northeastern Iraq are either of detrital, authigenic or diagenetic origin. The clay may reflect changes in the source rocks and drainage conditions within the weathering zone. This may result in increased leaching effectiveness processes and hence the conversion of specific minerals to others, such as montmorillonite and kaolinite to illite or palygorskite [19].

Palygorskite may be formed from the precursor smectite (Figures 3 A and C). These delicate filamentous outgrowths from a platy nucleolus resemble the transition of precursor clays depicted by [19]. Palygorskite formed as an authigenic mineral in lagoons and evaporitic basins due to chemical sedimentation [20], [10]. It also can be developed due to the diagenesis processes of precursor clays during early diagenesis by direct crystallization in calcareous soils or hydrothermal alteration of basaltic glass in the open oceans in association with fore-arc basins [21]. Palygorskite fibrous clay mineral was recorded in mudstones of the Kolosh formation as a scarce component. [22], [23], [24] have explained that the presence of palygorskite in oceanic basins adjacent to the continental margins is strong evidence for the detrital origin of palygorskite. The alteration of muscovite, biotite and k-feldspar produces illite diagenetically or in weathering zones [25], [14]. As smectite is transported, it converts to illite in seawater, which has an entirely different chemical environment from the weathering and transportation conditions [26]. Illite is found as flakes or crusts (Figure 3 F) in this study, which could indicate that it is an altered form of illite derived from older feldspars or other silicate minerals. Detrital kaolinite is generated mainly from volcanic rocks rich in potash feldspars or older sedimentary rocks reworking [10]. Illite is derived from the alteration of feldspars and degradation of muscovite under alkaline conditions with high concentrations of Al and K ions. Many authors, e.g. [27], [11], [10], suggested that illite and chlorite together are derived from continental weathering of shale and metamorphic source rocks [28] or feldspar-bearing rocks. They explained that the existence of illite is also related to the digenetic effect. The [29] in [10] suggested that increases of illite relative to other clays indicate a marine environment. The presence of detrital kaolinite in the source location further indicates that there is little leaching and chemical weathering [19]. Plates having pitted surfaces (Figure 4 A), degraded plates (Figure 3 C), and plates that are significantly orientated face-to-face (Figure 3 D) could all be regarded as transported and redeposited (detrital) kaolinite [30]. [10] suggested that the kaolinite results mainly from weathering and maybe form in the soil. Since the presence of kaolinite in the Kolosh formation is considered minor constitutes. [28] pointed out that it indicates relatively little leaching effect accompanied by effective chemical weathering in the source area. The digenetic origin of kaolinite results from the presence of carbonate ooze that supplies Ca^{++} ions within confined solutions [12], [31]. Chlorite is formed by weathering ferromagnesian minerals with high Mg, Fe, and Ca content and is used extensively in basic igneous and metamorphic rocks [10]. These rocks are prevalent in Iraqi and Turkish ophiolitic complexes and could be the source of the Kolosh Formation's sediments [32], [33]. SEM images reveal tiny disc-shaped chlorite (Figure 4 F), inherited from previous chlorite-rich igneous rocks or derived as detrital clays from older igneous rocks. It is most probably of detrital origin, which may have formed during alteration of the oceanic crust or by alteration of deep marine sediments by the flow of basaltic lava, e.g. contact metamorphism [19]. On the other hand, the chlorite formation in deep-sea sediments required special conditions and was mostly of detrital origin [22]. Most chlorite is related to increases in the igneous and metamorphic rock fragments. Thereby, the chlorite of the Kolosh Formation should be of detrital origin derived from metamorphic and chlorite-bearing igneous rocks. Montmorillonite is one of the smectite mineral groups derived from different origins. The distinction between the authigenic and detrital origin of smectite may be difficult. However, such clays are derived mainly from basic and intermediate volcanic rocks, e.g. basalt and andesite, hydrothermal activity or digenetic processes. Moreover, submarine alteration of basaltic igneous glass and igneous rock fragments is one of the most important authigenic smectite [19]. Special conditions for montmorillonite formation include a high concentration of basic cations, high silica activity, high pH and base-rich parent rocks. The vermiculite may have derived from weathering of mica in addition to the decomposition of the interlayer hydroxide sheet in chlorite, which is most probably a weathering product of other minerals e.g. illite, chlorite. It could be digenetic in origin derived from basic and volcanic rocks. It was not identified by electron microscopy but was diagnosed on an X-ray.

5. Conclusions

The dominant clay minerals in the kolosh Formation are chlorite, montmorillonite, kaolinite, palygorskite, illite and vermiculite. The mineralogy of clay fraction shows various varieties of range of Fe and Mg-rich minerals essentially reflecting ophiolites source rocks and almost of origin detrital. The presence of illite and chlorite clay minerals indicates a cold and arid climate. In addition to the influx of terrigenous materials eroded from previous Cretaceous successions in the area, kaolinite may demonstrate the prevalence of warm temperatures that help preserve these minerals. Illite is derived from the alteration of feldspars and degradation of muscovite under alkaline conditions with high Al and K ions concentrations. Montmorillonite is derived mainly from basic and intermediate volcanic rocks, e.g. basalt and andesite, hydrothermal activity or diagenetic processes, which reflect the formation rocks' origin. Palygorskite in an environment of oceanic basins adjacent to the continental margins is strong evidence for detrital origin. The vermiculite may have derived from weathering of mica in addition to the decomposition of the interlayer hydroxide sheet in chlorite, which is most probably a weathering product of other minerals, e.g. illite, chlorite. It could be digenetic in origin derived from basic and volcanic rocks. Various clay minerals indicate different source rocks, provenance and tectonic settings. The variation in clay mineral assemblage in the Kolosh Formation is attributed to the shift in climatic conditions from humid to arid. This may be related to factors such as sea-level change and tectonic activity. The subsiding shallow marine nature favors the detrital nature of clay minerals with common turbidity currents, which precipitated detrital rather than chemical clay minerals. The authigenic deep-marine clays are mainly Fe and/or Mg-rich minerals, including chlorite.

Table 2: Results of elements (%) by EDS analysis

Element	Range of Atomic %	Range of Weight %
C	12.2 – 17.6	7.7 – 11.5
O	60.8 – 63.2	51.2 – 54.9
Na	0 – 0.9	0 – 1.1
Mg	4.3 – 9.4	5.7 – 12.0
Al	1.6 – 3.1	2.4 – 4.6
Si	6.2 – 10.9	9.5 – 16.1
K	0 – 0.2	0 – 0.4
Ca	0.9 – 6.1	1.9 – 13.2
Ti	0 – 0.1	0 – 0.2
Mn	0 – 0.3	0 – 0.8
Fe	0.8 – 2.1	2.4 – 6.3
Cr	0	0 – 0.1
Ni	0 – 0.1	0 – 0.2
Br	0	0

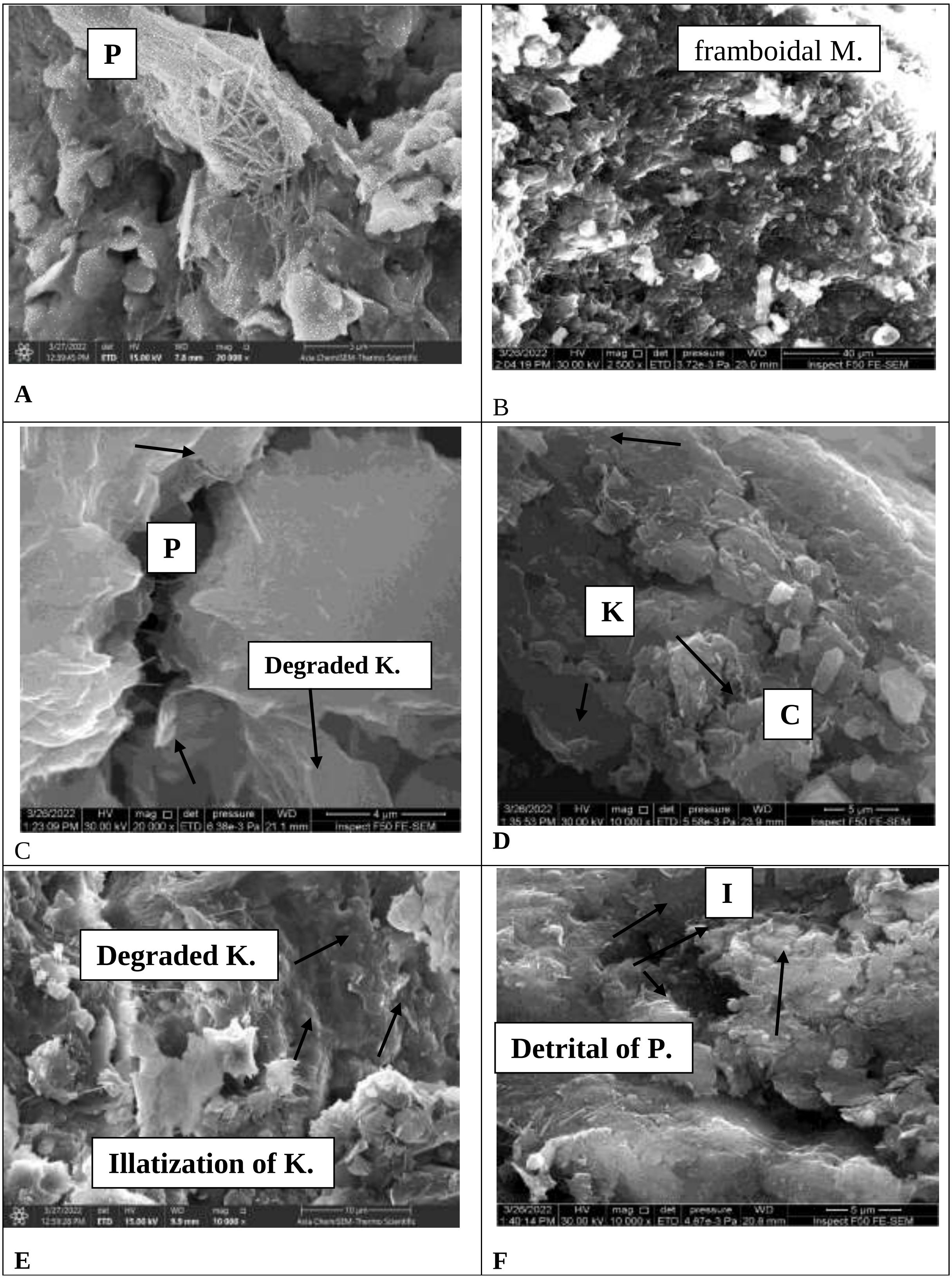


Figure 3: SEM displays various morphologies of the clay minerals: A- Authigenic palygorskite (P), B- framboidal montmorillonite (M), C- Degraded Kaolinite plates (K) and short palygorskite fibers (P), D- Kaolinite plates aligned face-to-face (K) and carbonate crystalline (C), E- degraded kaolinite (K) and illatization of kaolinite, F- Illite fibers (I) and detrital palygorskite.

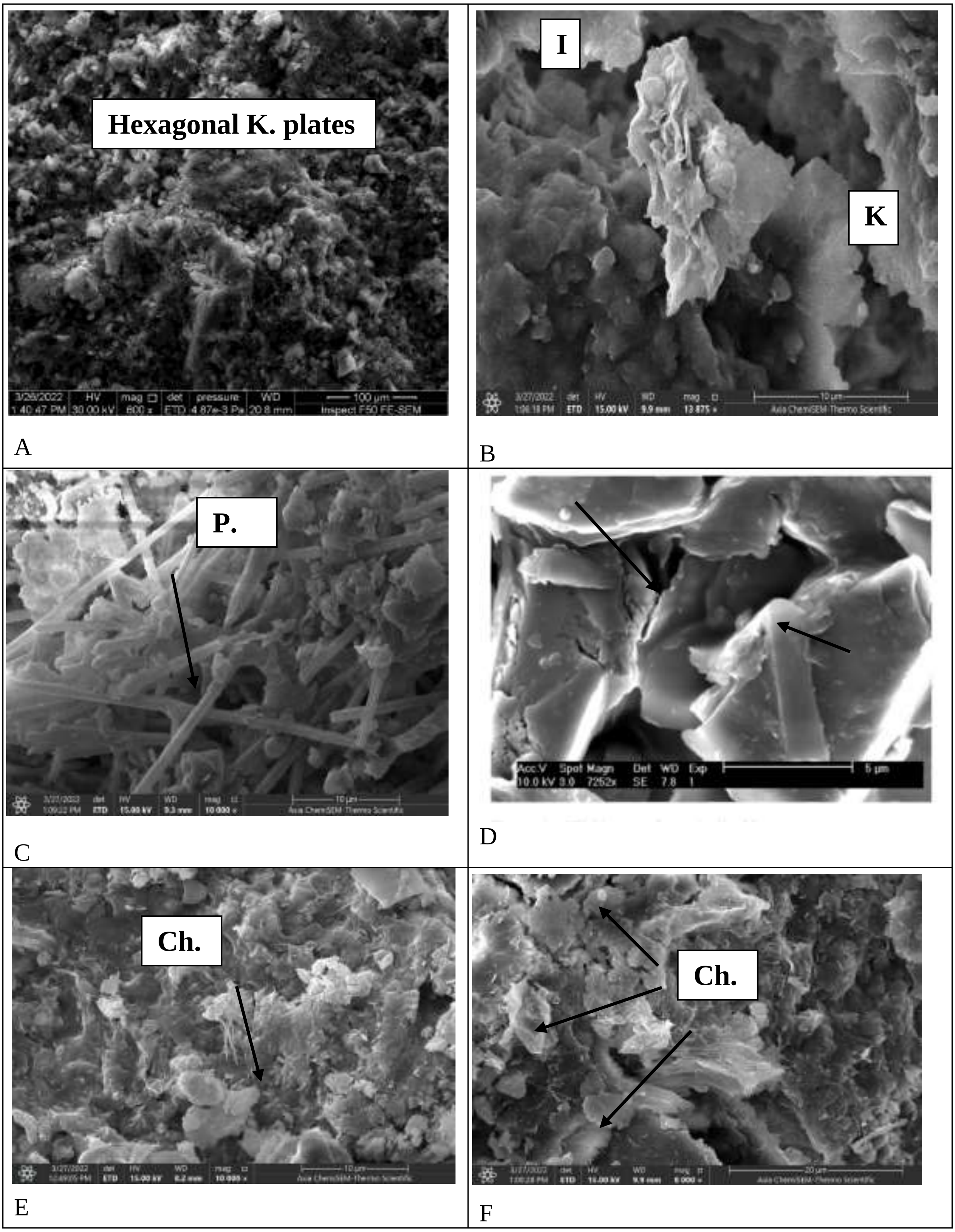
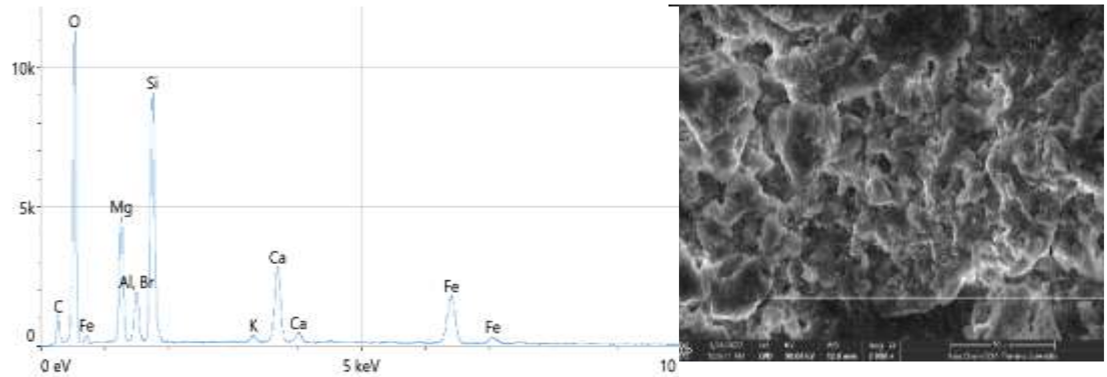
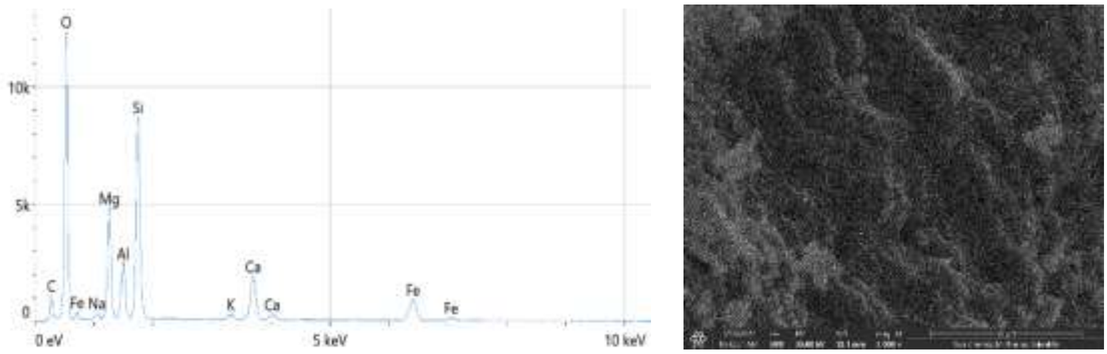


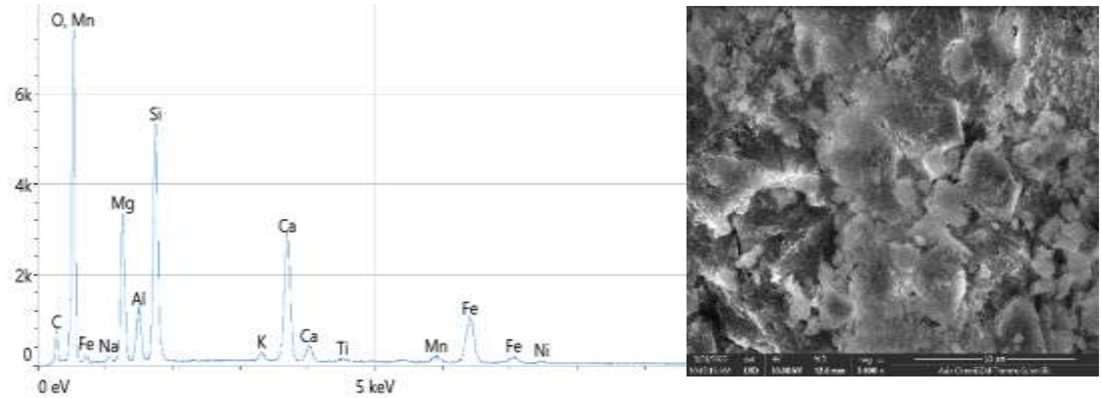
Figure 4: SEM displays various morphologies of the clay minerals: A- Hexagonal kaolinite plates (K), B- Illite (I) and kaolinite (K), C- Detrital palygorskite (P), D- Vermiculite (V), E- Chlorite (Ch), F- Chlorite disc-shape (Ch).



EDS of sample from the first section. Total number of counts: 387 767.

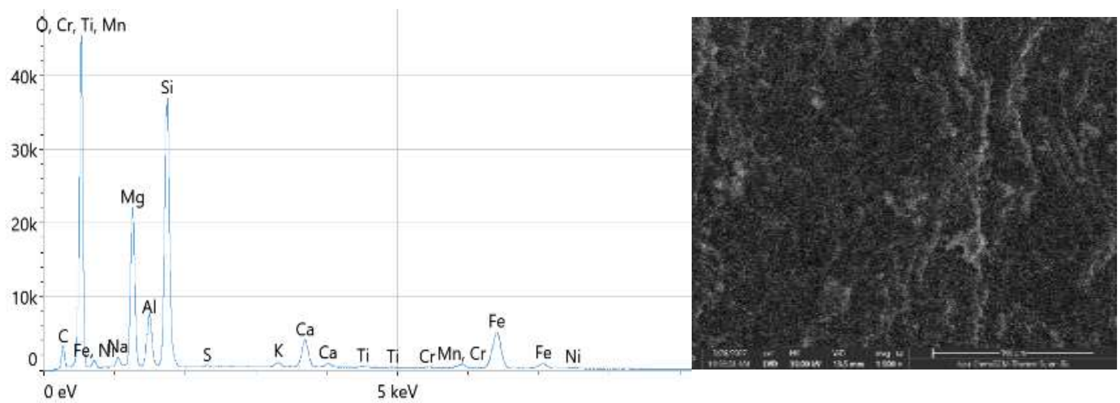


EDS of sample from the second section. Total number of counts: 339 637.

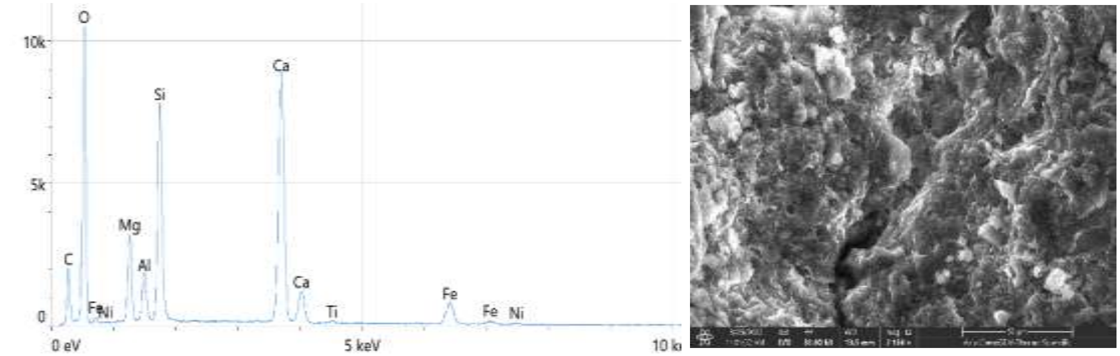


EDS of sample from three sections. Total number of counts: 268 013.

Figure 5 a: Represents a graph of the presence of elements in a sample tested with Eds.



EDS of sample from the fourth section. Total number of counts: 1 362 138



EDS of sample from the fifth section. Total number of counts: 424 082

Figure 5 b: Represents a graph of the presence of elements in a sample tested with Eds.

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