ISSN: 2413-5267

The Uses of The Al-Faidiyah Formation Oligocene-Miocene in The Drilling Mud Fluids (Bentonite) at Umm Ar Razam, North-East Libya

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استخدامات تشكيل الفايديه اوليغوسيني – الميوسين في سوائل الحفر (البنتونيت) في أم الرزّم، شمال شرق ليبيا

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قسم علوم الأرض، جامعة بنغازي، بنغازي، ليبيا.

Abstract

The lower member (Faidia Clay) of the Oligocene-Miocene Al Faidiyah Formation at Umm Ar Razam Quarry, northeast Libya has been investigated for its potential as a chemical additive (bentonite) in drilling oil wells. The thickness in outcrop of the examined clay unit is variable but information from the subsurface indicates a maximum thickness of about 25 m. Normally, these clay deposits have a dark green color that becomes brown when weathered. When wet, the clay forms a very plastic sticky mass with soap-like textures. A number of mineralogical, chemical, and physicochemical laboratory analyses using X-ray diffraction (XRD), scanning electron microscopy (SEM-SE & SEM-BSE), X-ray fluorescence (XRF), ion exchange capacity (CEC) and surface area have been made in order to characterize the grade and quality of the examined clays. The overall results show that montmorillonite represents the highest percentage (46 %), minor kaolinite (36 %), and trace chlorite (4 %). Other minerals of heterogeneous mixtures of non-clay minerals such as fine quartz grains, calcite, dolomite, gypsum, K-feldspar, ilmenite, anatase, and hematite are also recognized. The presence of a heterogeneous mixture of non-clay minerals reduces the rheological and physical properties as well as detracts from the commercial performance of the Faidia clay. The unprocessed clay and clay after treatment with the addition of up to 6 % of Na_2CO_3, did not achieve a swelling volume comparable with standard bentonite. The Faidia Limestone Member (the upper part of the Al Faidiyah Formation) is characterized by very high purity in Al Fatayah Quarry. It is extensively used for concrete aggregates, road tiles, blocks, paints, carpets, the cement industry, papers, pharmaceuticals, water treatment, agriculture, and plastics. The equivalent member in Umm Ar Razam Quarry has a lower grade due to the presence of other carbonates (dolomite) and non-carbonates such as quartz, clay, and glauconite minerals. Fossil contents and lithological nature indicate that this is a typical shelf environment.

Keywords: *Umm Ar Razam, Al Faidiyah Formation, Faidia Clay Member, Fatayah, Hematite, Libya*

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1. Introduction

Clay and limestone raw materials of the Al Faidiyah Formation are economic interest. These raw materials were studied to gain a better understanding of other much larger and purer deposits, which are of economic importance. A series of earlier studies have been carried out from many workers on the Faidia Clay Member from the Umm Ar Razam area because of its economic potential (El Ebaidi, 1999; Lat and Zamarsky, 1992; Waston and Arhuma, 1992; El Ebaidi and Bakar, 1991; Sassi, 1991; Alami and Salem, 1981; and PRC, 1987). The descriptions, identification and analyses of clay minerals in this paper are based on the work of many authors (Moore and Renolds, 1997; Velde, 1992; Tucker, 1991 &1988; Deer *et al.*, 1992; Berner, 1971; Carrol, 1970; and Grim, 1968). The suitability of the clay raw material as a component in oil drilling mud fluid was also investigated. Evaluation of the clay need included laboratory tests of mineralogical, geochemical, physical and petrographical rock types for oil industry potential end use. The study area is poor in mineral resources, due to the absence of magmatic rocks. The Al Faidiyah Formation contains mainly limestone and clay deposits; no ore mineralization was found during this study. Iron ore (hematite) is rarely occurred in the Faidia Clay Member of the Al Faidiyah Formation. Faidia Limestone Member is extensively used for various applications such as aggregates in construction with specified size ranges, depending in the end use, road tiles, blocks, paints, carpets, lime production, cement industry, paper, pharmaceutical, water treatment, agriculture and plastics. Suitable limestone and clay deposits in Al Faidiyah Formation are available in sufficient quality, thickness and accessibility and also close to good transportation and to the market (clay in Umm Ar Razam and limestone in Al Fatayah Cement Quarries). A number of mineralogical, geochemical and physical laboratory assessments have been used to identified the grade and quality of the Al Faidiyah Formation in Umm Ar Razam and Al Fatayah quarries and to compare these clays with other commercial clays. This paper focusses on Faidia Clay Member from point of view of its application as a chemical additive (bentonite) in drilling oil wells for deep hole drilling, where high pressure and temperature occur. Smectite clays used for mud drilling fluids must meet the American Petroleum Institute (A.P.I.), or the Oil Companies Materials Association (O.C.M.A.) standards. Only certain natural *Na* and *Na*-exchanged *Ca* smectites have the potential for meeting the A.P.I. and/or O.C.M.A. specifications.

1.1. Stratigraphy of Al Faidiyah Formation

1.1.1. Al Faidiyah Formation (Upper Oligocene-Lower Miocene)

The Al Faidiyah Formation is the youngest unit in the Umm Ar Razam area and stratigraphically ranges from the Upper Oligocene to Lower Miocene. The formation consists of limestone, whitish to yellowish, thick bedded to massive. It contains fossiliferous layers with dominant coralline algae. Pietersz (1968) introduced the name Faidia Formation, derived from the Qaryat (Qaryat in Arabic this means village) Al Faidiyah. It comprises of two

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members; the lower, Faidia Clay Member and an upper Faidia Limestone Member. The Al Faidiyah Formation have been studied at two different localities:

1.1.1.1. Al Faidiyah Formation (Al Fatayah Cement Quarry)

The Al Faidiyah Formation in this location consists mainly of limestone (Faidia Limestone Member), with green clay at the surface in the lower part of the formation. The limestone is mostly white, highly brightness (> 95 %) and according to Harries, 1979 is classified as a very high purity limestone (> 98.5 %), medium to fine grained, medium hard and contains corals of two genera *Cyphastrea* and *Aleveopora* (Figure 1), with abundant burrowing bivalves. It also contains encrusting red algae, echinoderm fragments with syntaxial overgrowth, molluscan shell fragments, large benthonic foraminifers (*Nummulites sp*. and *Discocyclinids*), bryozoans, gypsum crystals. It is dolomitized (1.7%) especially in the lower part, with micron sized euhedral dolomite replacing matrix.

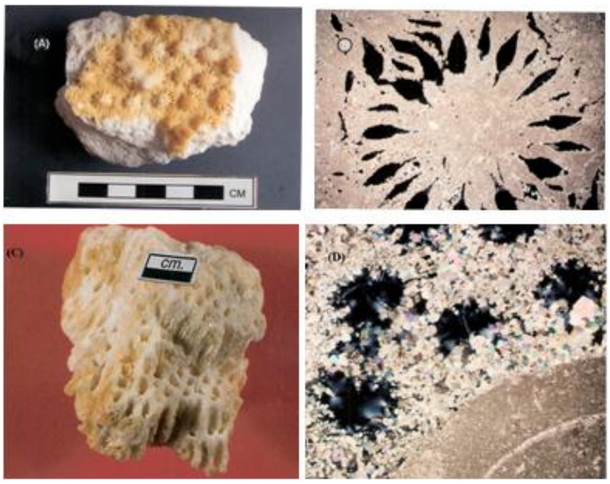

Figure 1. *A*) Typical coral reefs of *Cyphastrea* sp.; *B*) Under the microscope showing mouldic porosity of the same colony (A).; *D*) *Aleveopora* sp. with abundant boring of bivalves (Bu). Field of view= 6 *mm*, (XPL), in Al Fatayah Cement Quarry (Khameiss *et al.*, 2016).

1.1.1.2. Al Faidyah Formation (Umm Ar Razam Quarry)

This section of this quarry is located of about 3 *km* northeast Umm Ar Razam village, of about 45 *km* from Darnah City and 350 *km* from Benghazi City (Figure 2). The lower boundary of the Faidia Clay Member is sharp and unconformable with the underlying Al Abraq Formation (Middle to Upper Oligocene). A sharp contact between the two members was observed (Figures 3 & 4). The Faidia Clay Member is predominantly composed of montmorillonite, with major or minor kaolinite and chlorite. It contains a variable proportion of non-clay minerals including fine quartz, calcite, ilmenite, dolomite, gypsum, K-feldspar, anatase and hematite. It varies in thickness but drilling of twenty boreholes have proved a maximum thickness of 25 *m* (El Ebaidi and Bakar, 1991). The Faidia Limestone Member is 2

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m thick, highly fractured and low grade where it consists of yellowish, medium hard limestone, with a packstone texture that contains glauconitic grains, benthonic foraminifers (*Lepidocyclina*), echinoderm fragments and common bryozoans. It is slightly dolomitized (up to 2% of the rock volume), some of the echinoid spines are replaced totally by micron sized clear dolomite crystals.

Figure 2. Location map of the studied areas at Al Fatayah Cement Quarry and Umm Ar Razam village, NE Darnah City.

Figure 3. The sharp contact between the Faidia Clay and Faidia Limestone Members of Al Faidyah Formation in Umm Ar Razam area.

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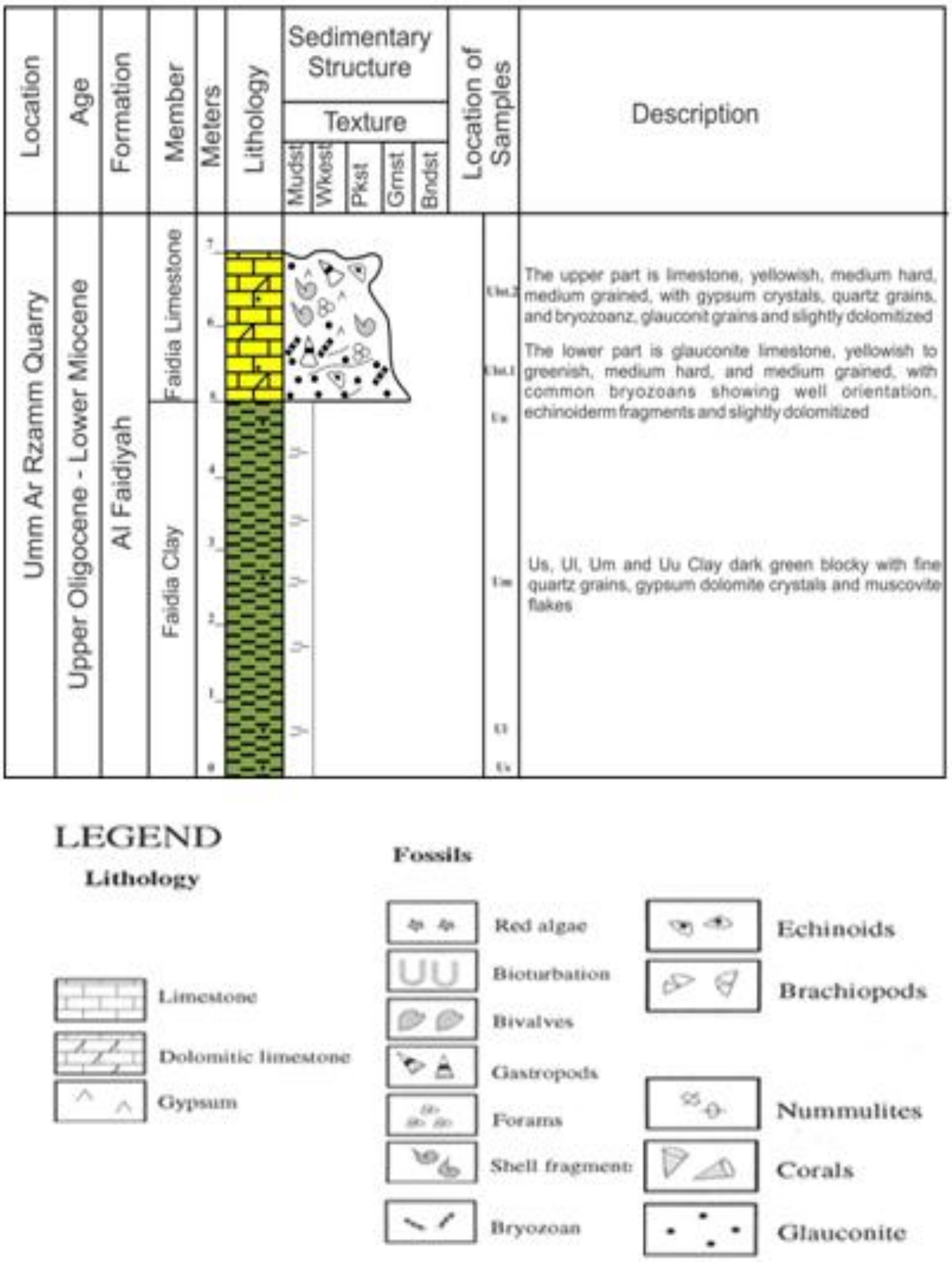


Figure 4. Log of the Al Faidiyah Formation, in Umm Ar Razam Quarry

2. Laboratory Evaluations of the Faidia Clay Member (Grade and Quality)

2.1. Fluid loss

The Umm Ar Razam clay has very high filtrate values compared to other standards (Table 1).

Table 1. A comparison Table illustrate the readings of the fluid losses of the Libyan Clay and other international clay standards.

Type	O.C.M.A.	Wyoming	Bulgarian	Algerian	Libyan Umm Ar Razam clay
Filtrate *ml* / 30 *mm*	15 max.	11.75	12.31	15	> 50

The successive fluid loss of Umm Ar Razam clay will generate problem in oil drilling wells such as increasing the filter cake thickness leading to potentially stuck pipes, poor performance of electric logs, an unstable well borehole with possibility of caving, and total or partial loss of drilling mud fluids with chemicals into formations.

2.2. Mineralogical and Chemical Evaluations

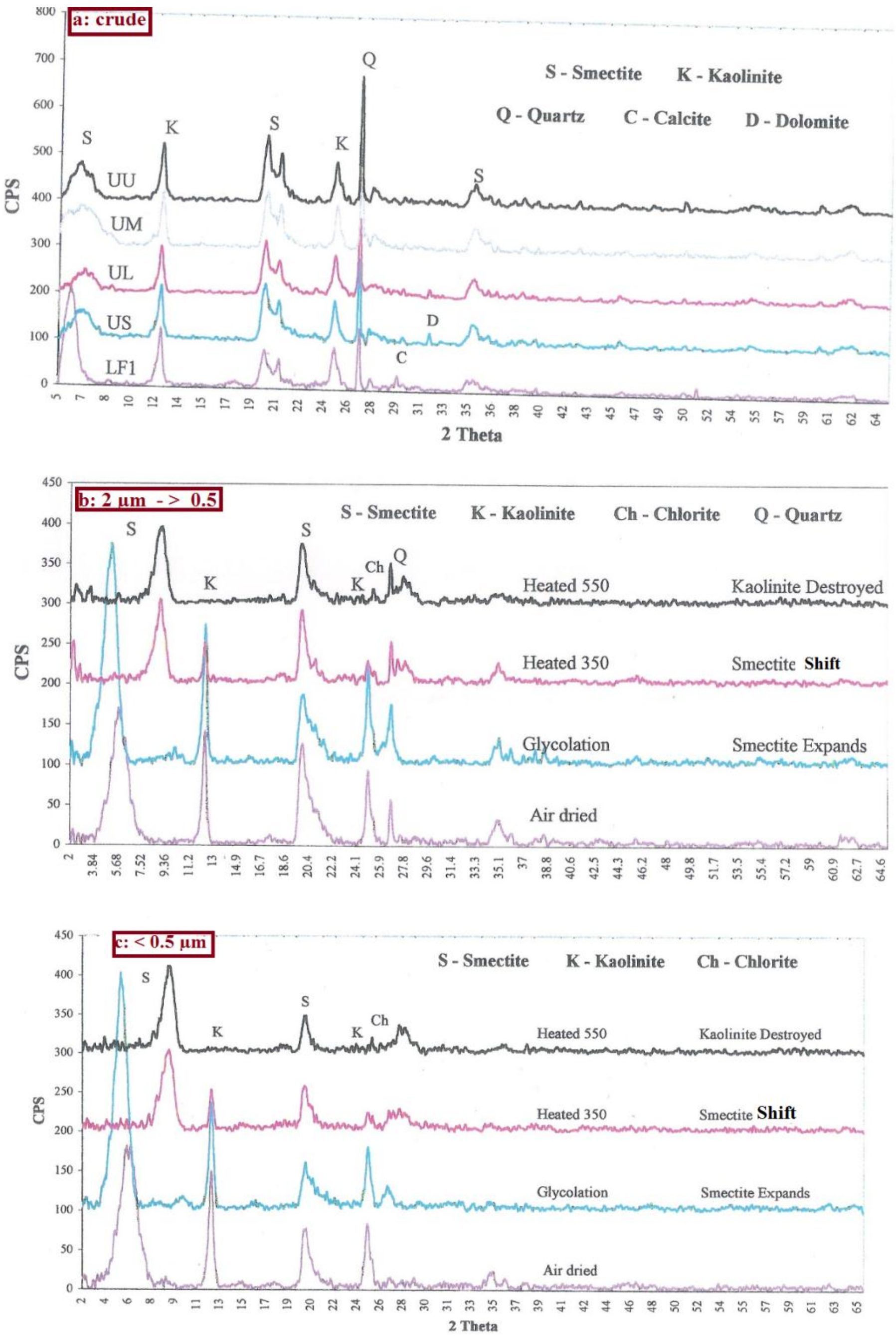
A number of mineralogical identifications and chemical analyses have been done to characterize the Faidia Member using XRD, SEM, optical microscopy and XRF techniques.

2.2.1. X-ray Diffraction of Faidia Clay Minerals

Oriented mounts were produced on glass slides using Wilson's method (Wilson, 1987), clay fractions of < 2.0 μm to > 0.5 μm and < 0.5 μm were clay separation method been used. Clay samples were scanned from 2 - 65 2ϴ after untreated air drying, after glycol solvation and after heating to 350 and 550 °*C* for one hour (Figure 5). The presence of these impurities in crude sample leads to poor rheological and binding properties of the Faidia clay.

2.2.2. Scanning Electron Microscopy

Scanning electron microscopy (SEM) has been used to provide additional useful information about the textural relationships between the smectite and associated minerals. Chemical analyses of clay minerals from the Faidia Clay Member were determined using SEM-EDX technique. The study of SEM also revealed other important petrographic features not apparent from the optical microscope, for example the identification of non-clay minerals (impurities) such as ilmenite, calcite, dolomite, anatase and orthoclase, quartz, gypsum and hematite (Figures 6, 7, and 8) and Table (2).



* Sample numbers; UU, UM, UL, US (from Umm Ar Razam quarry), and
LF1 (clay from Al Fatayah quarry) of Al Faidiyah Formation

Figure 5. XRD pattern of the Faidia Clay Member (**a**: crude, **b**: 2 μm -> 0.5 μm, and **c**: < 0.5 μm) at Umm Ar Razam quarry showing Smectite (S) and Kaolinite (K), Chlorite (Ch) Quartz (Q), Calcite (C) and Dolomite (D) minerals.

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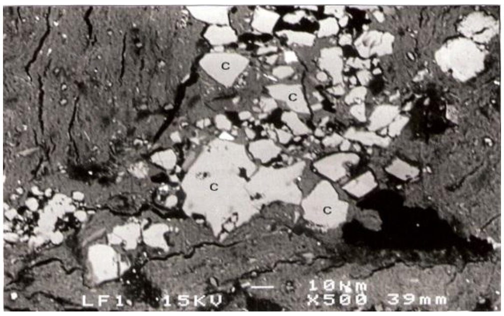

Figure 6. SEM-BSE shows calcite cement (C), filling pore spaces. Sample no. LF1 of the Faidia Clay Member of the Al Faidiyah Formation, in Al Fatayah Cement Quarry.

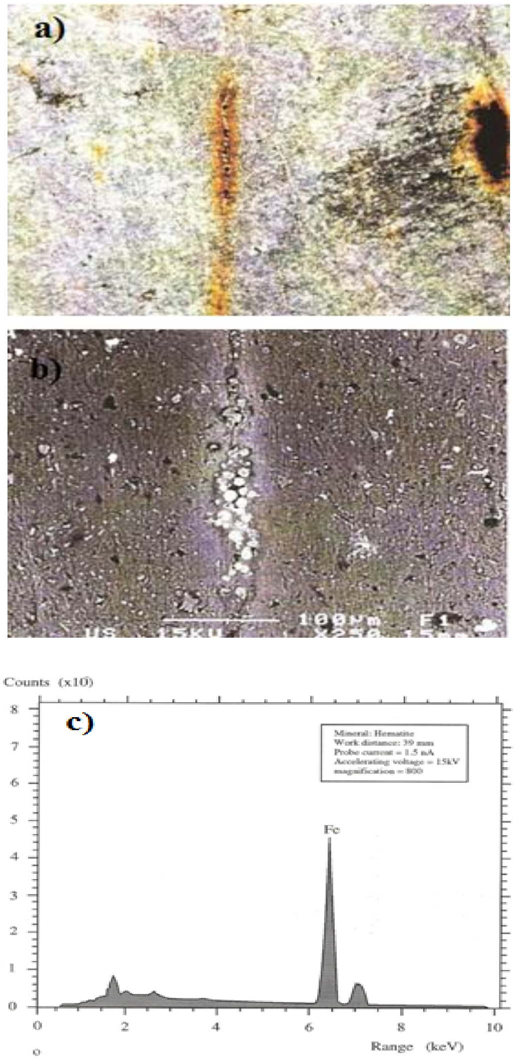

Figure 7. a) Hematite filling fractures (sample no. US), Field of view = 6 *mm*, (XPL);
b) SEM-BSE view of hematite filling pore spaces; **c**) SEM-EDX spectrum for hematite.
Faidia Clay Member in Umm Ar Razam Quarry.

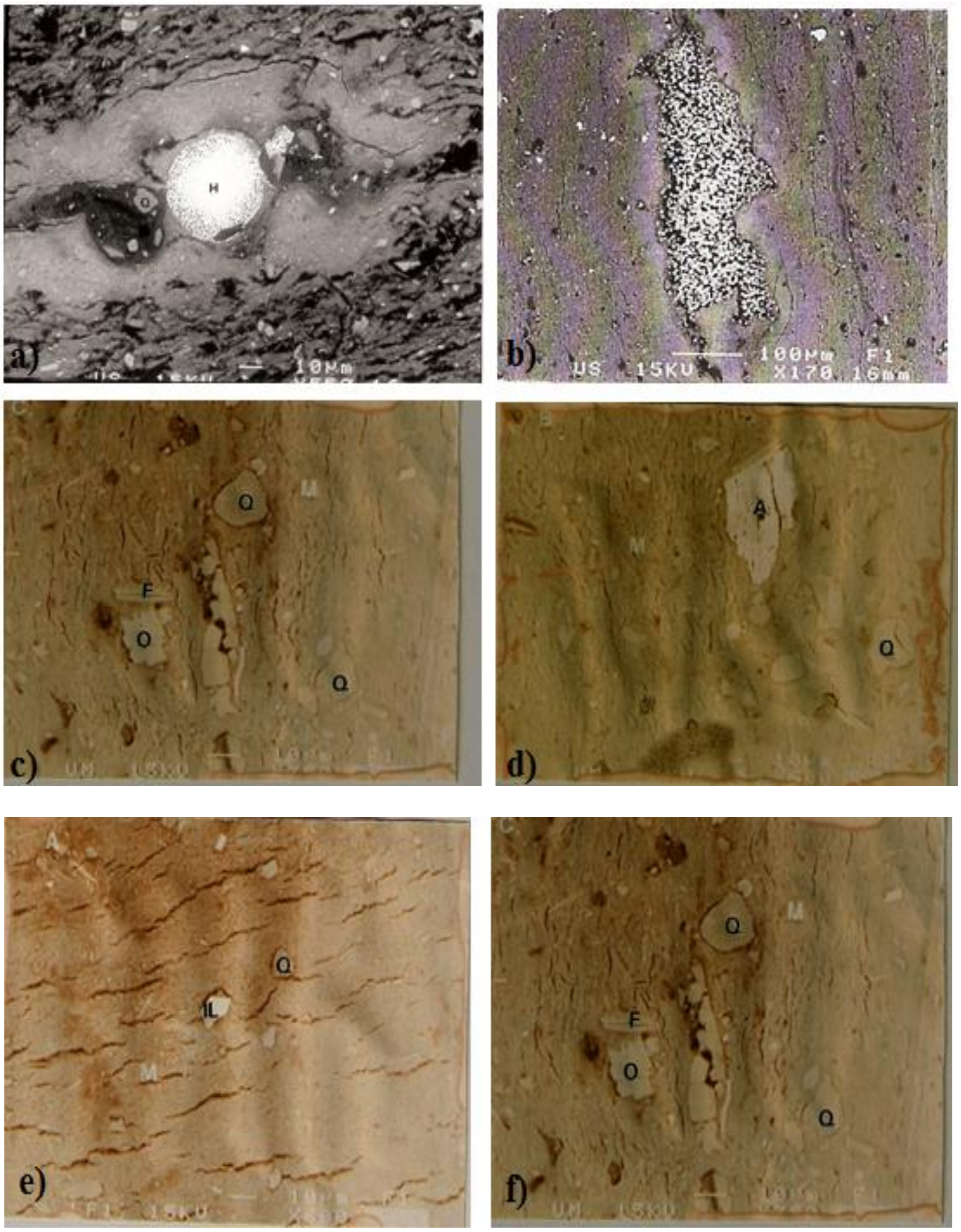

Figure 8. a & **b**) SEM-BSE hematite (H) filling pore spaces (sample no. US);
c, d, e & **f**) SEM-BSE showing Orthoclase (O), Quartz (Q), Feldspar (F), Anatase commonly distributed by parallel bands (A) and Ilmenite (IL). All were embedded in montmorillonite clay (M), in Al Fatayah and Umm Ar Razam Quarries.

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Table 2. SEM-BSE analyses of nonclay minerals found in the Faidia Clay Member (Crude)

Oxides *wt.* %	Quartz	Calcite	Hematite	Ilmenite	Orthoclase	Anatase	Anatase *Standard
FeO	-	-	63.20	35.29	-	1.57	5.0
MnO	-	-	0.31	1.11	-	-	-
MgO	0.15	-	1.03	0.19	-	0.17	-
CaO	-	55.58	0.22	-	-	0.37	-
SiO_2	100.1	-	4.82	0.36	63.31	3.67	0.6
Al_2O_3	0.26	-	0.85	-	17.63	1.76	2.1
K_2O	-	0.31	-	-	15.83	-	-
Na_2O	0.21	-	0.88	0.32	0.40	0.27	-
SO_3	-	-	0.37	-	-	-	-
TiO_2	-	-	-	55.93	-	83.88	88.6
Total	100.72	55.89	71.68	93.20	97.17	91.69	96.30

*Anand and Gilkes (1984)

2.2.3. X - Ray Fluorescence (XRF)

A summary of geochemical analysis from X - ray fluorescence of the Faidia Clay Member in Umm Ar Razam quarry of the Faidiyah Formation is shown in Table (3).

Table 3. Average XRF data (*wt.* %) of five samples of the Faidia Clay Member

Oxides (*wt.*%)	SiO_2	Al_2O_3	Fe_2O_3	*CaO*	*MgO*	SO_3	K_2O	TiO_2	Na_2O	*MnO*	P_2O_5	Total
Faidia clay	44.38	18.53	7.02	5.55	4.38	2.83	1.35	1.38	1.29	0.05	0.02	**86.78**
Wyoming Bentonite	54.63	16.71	3.59	1.36	2.13	-	0.53	-	-	-	-	**78.95**

2.2.4. Differential Scanning Calorimetry (DSC)

Samples of the Faidia Clay Member and other standards were run using DSC technique. The samples were heated continuously at regular rate from 25°*C* to 1200°*C* at 10°*C/min*. Curves for the various clay minerals obtained by DSC (Figure 9) are typically as expected for montmorillonite.

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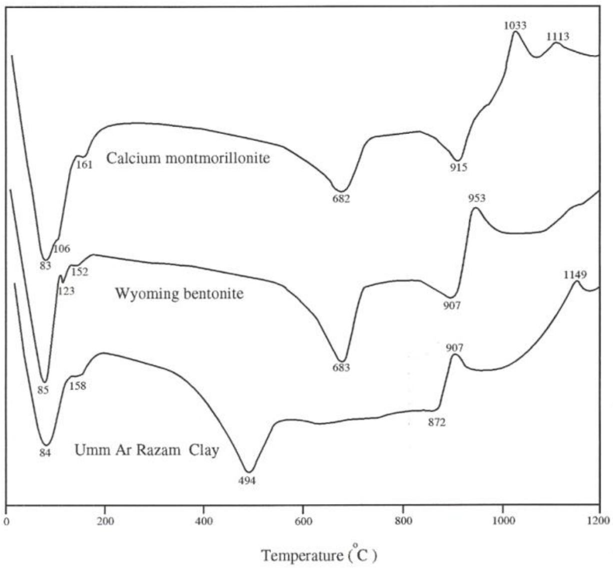

Figure 9. DSC curves of Umm Ar Razam clay showing different dehydroxylation behaviour compared with Wyoming bentonite and Calcium montmorillonite (Standard).
Note the well-defined shoulder < 200 *°C* on the low temperature endothermic peak.

2.2.5. Estimation of organic matter + H_2O content by loss on ignition (LOI)

A standard method with ignition at 1000*°C* was used (Gale and Hoare, 1991), the result of LOI (%) experiments of five samples are shown in Table (4). XRD was also run to examine the sample patterns after heating to 1000*°C*. Heat treatment of the Faidia Clay Member at this temperature caused collapsed and most of the reflections of the clay minerals were destroyed (Figure 10).

Table 4. LOI (%) of various clays from Libya and USA as a standard heated at 1000*°C*

Type	Al Fatayah Cement Quarry	Umm Ar Razam Quarry				USA Bentonite
Loss at 1000*°C* (%)	LF1	US	UL	UM	UU	Wyoming
	20.1	17.1	19.9	19.7	19.2	17.64

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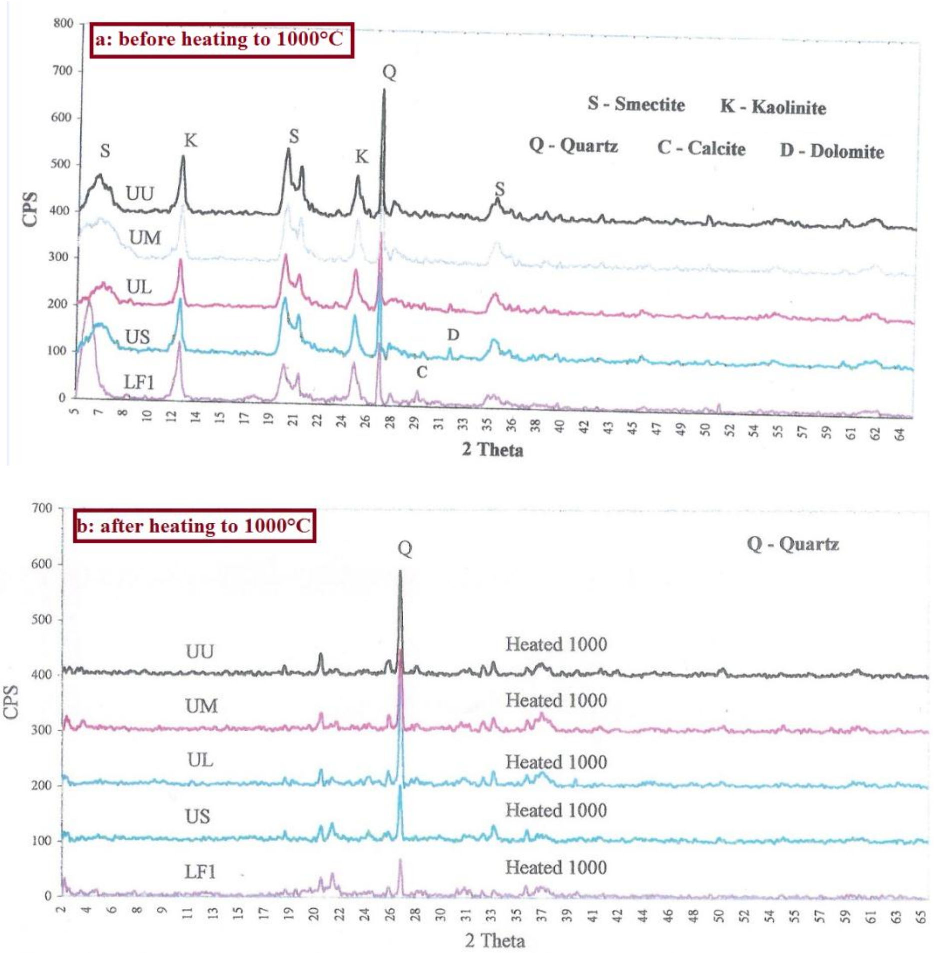

Figure 10. XRD patterns of clay samples (Crude) of the Faidia Clay Member befor (*a*) and after (i) heated to 1000°*C*

2.2.6. 2-Ethoxyethanol Ethylene Glycol Monoethyl Ether (EGME), Surface Area Test

Total surface area is fundamental property of layer silicates and has been used as a criterion for identification. This has been used to determine the total surface area of Faidia clay samples, to determine the retention of EGME the procedure followed Carter *et al.*, (1965). Pure smectites have surface area of 800 m^2/g, other clay minerals such as Kaolin < 40 m^2/g and non-clay mineral (including quartz) < 5 m^2/g (Moorlock and Highly, 1991; and Inglethorpe *et al.*, 1993). The surface area values of the Faidia Clay Member compared other clay standards are illustrated in Table (5).

Table 5. EGME surface area values for Faidia Clay Member and control montmorillonite clay sample (Standard) using 2-ethoxyethanol method

Sample no.	***Surface area (Crude) m^2/g***	***Surface area (< 2 µm - > 0.5 µm) m^2/g***	***Smectite (%) (Crude)***	***Smectite (%) (< 2 µm - > 0.5 µm)***
UU	333	490	42	61
UM	389	490	49	61
UL	389	559	49	70
US	428	559	54	70
LF1	420	524	53	66
Control Sample	661	-	83	-

2.2.7. Swelling test

Swelling efficiency of the clay raw material used the Christidis and Scott (1993) procedure. Natural smectite clay s range from strongly swelling to non-swelling depending on the smectite clay species and on the ratio of exchangeable calcium ions. It is usual in industry to convert the non-swelling to swelling clay by treatment with soda ash (sodium carbonate, Morgan, 1994). An amount of sodium carbonate between 1 to 6 % by weight were mixed with Faidia clay samples (< 125 *µm* fraction size). A moderately swelling bentonite will swell to a volume of 15 - 20 *ml*., and a good bentonite to of about 25 *ml*. An excellent grade will swell to 30 *ml*. or more (Inglethorpe *et al.*,1993). The Faidia clay showed unchanged swelling power. Table 6 shows the results for the Faidia clay in comparison with other clay standards.

Table 6. Swelling test values for the Faidia Clay Member, Wyoming and Mexico clays with addition 1 to 6 % sodium carbonate (Soda ash)

Type	***Before 24 hrs.***	***After 24 hrs.***	***Swelling volume (ml.)***
Umm Ar Razam Quarry (Clay)	4.60	4.60	Nil
Al Fatayah Cement Quarry (Clay)	2.50	2.50	Nil
Wyoming (USA)	2.50	4.00	15
Chihuahua (Mexico)	1.80	2.30	5

2.2.8. Cation Exchange Capacity (CEC)

CEC can be measured by a variety of methods and to some extent the result obtained is dependent on the method used. The cation exchange capacity of relatively pure smectite clays between 70 to 130 *meq*/100*g* (Odom, 1984). The $BaCl_2/MgSO_4$ method; British Geological

Survey for measurement of CEC was used. Table (7) gives the results for Faidia clay compared with Wyoming bentonite.

Table 7. CEC values of Faidia Clay Member of the Al Faidiyah Formation and Wyoming bentonite

Sample no.	LF1	US	UL	UM	UU	Wyoming
CEC values *meq*/100*g*	50	55	563	52	60	70

Note: pH value of 8.1 was obtained

3. Discussion and Conclusion

The source of Hematite and Anatase in the Faidia Clay Member;

- Chemically, the main result of the alteration of ilmenite mineral is an increase in (formation of co-existing different phases) TiO_2, Fe_2O_3 and a decrease in *FeO*. When the ilmenite is oxidized at a certain temperature the structure breaks down into rutile/anatase and hematite. With increasing degrees of alteration ilmenite disappears and the ratio of TiO_2:Fe_2O_3 increases until the altered grain becomes rutile/anatase (Temple, 1966). Hematite in Faidia Clay Member was found filling pore spaces and fractures. A porous anatase mineral was observed with pores filled entirely by clay minerals. The alteration of ilmenite is responsible for the formation of the hematite and anatase minerals in the Faidia Clay Member. The explanation is based on Flinter (1959); Temple (1966); Cambell (1973); Grey and Reid (1975); Anand and Gilkes (1984); Deer *et al*., (1992) and Babu *et al*., (1994). The alteration of ilmenite in the Faidia clay took place in two stage processes; 1) all iron oxidized and diffused from ilmenite leaving pseudo-rutile in which closely packed oxygen layers remain intact and 2) the alteration pseudo-rutile dissolves. Iron is removed by solution and both rutile/anatase (TiO_2) and hematite Fe_2O_3 precipitate, according to the following reaction: There is a perched aquifer in the Faidia clay quarry, which may aid the alteration of ilmenite mineral. The area of Umm Ar Razan is highly fractured effects by faulted and jointed area, which may hydrate iron during hydrothermal alteration and removed it as a mobile hydroxide.
- The nearest volcanic source is at least 500 to 600 *km* away to the southwest and southeast. Therefore, it is likely that the Faidia Clay is of secondary origin. The primary volcanic ash is altered to montmorillonite and then redeposited in marine environment. Alternatively, some of the montmorillonite may be authigenic (formed in situ), it may have derived during the eruption of these volcanoes at that time, it varies in thickness at some parts (lenses like) and provides a suitable high grade montmorillonite. Whilst the majority of the other parts are not suitable for mud drilling fluids (bentonite).

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- The Faidia clay samples appear similar in most parts and in commercial terms are relatively low grade (46% montmorillonite), with calcium as the dominant exchangeable cation.
- The Faidia clay in fraction size < 2 μm may be suitable for use as mud drilling fluids and water drilling wells at certain depth where low pressure and temperature.
- The Faidia clay may use if mixed as a raw material with bentonite (Standard) and treated using chemical additive like sodium carbonate (Soda ash).
- The presence of non-clays (impurities) affects the rheological and physical properties and detracts from the commercial performance of the Faidia clay. The Faidia Clay Member has relatively high amounts of Fe_2O_3 and CaO, whereas the SiO_2, Al_2O_3 and Na_2O are low compared with other commercial bentonites. The Faidia clay is non-swelling clay and did not achieve the necessary specifications compared with standard. Possible future uses are in foundary moulding sand, floor decorations, water and waste water treatment, agriculture, bleaching and animal feed industries. Huge reserves are available for more suitable types of industries, where bentonite enrichment is not critical parameter (use in low value applications).
- The Faidia Limestone Member in Al Fatayah Cement Quarry (25 *m* thick) is characterized by very high purity $CaCO_3$. The equivalent Member in Umm Ar Razam Quarry has lower grade (Impure) Figure (11).

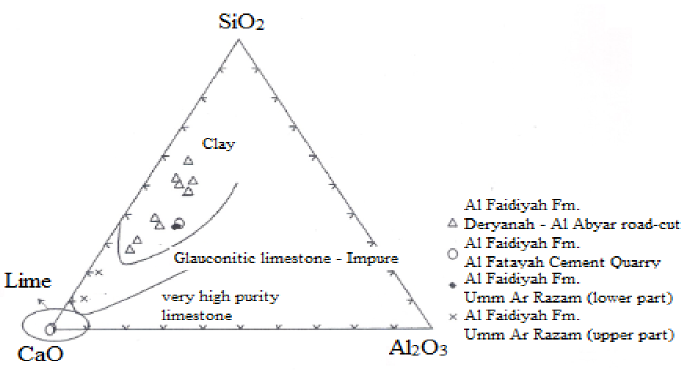

Figure 11. SiO_2-CaO-Al_2O_3 Ternary diagram (XRF analyses) of the Al Faidiyah Formation (*wt.* %), at Deryanah-Al Abyar road cut near Benghazi City, Al Fatayah Cement Quarry and Umm Ar Razam Quarry.

Acknowledgement

The author is very grateful to Dr. Brent Miller from Department of Geology and Geophysics, Texas A & M University for his critical review of the manuscript.

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