

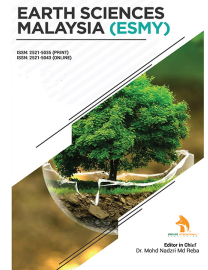


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## RESEARCH ARTICLE

# GEOCHEMICAL INVESTIGATION OF THE EARLY EOCENE LAKI FORMATION SHALES IN THE SELECTED REGIONS OF SOUTHERN INDUS BASIN PAKISTAN

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## ABSTRACT

A systematic geochemical investigation of the Laki Formation from the selected regions of Southern Indus Basin Pakistan Basin has been carried out. This paper presents the characterization of hydrocarbon potential, type of kerogen, thermal maturity, the origin of organic matter, depositional environment, and bulk mineralogy of the Laki Formation shales. The total organic carbon (TOC) content of the Laki shale ranges from 0.53 to 2.66 wt. %. The values indicate that Laki shales have fair to very good hydrocarbon potential. Type III kerogen is identified in the Laki shale based on TOC and residual potential ( $S_2$ ) data. Maturity parameters (Tmax & Methyl phenanthrene index) suggest that Laki shales are thermally immature for the generation of hydrocarbons. Normal alkane data and stable carbon ( $\delta^{13}C$ ) isotopic value (-9.67 ‰ PDB) of the Laki shale indicates the predominantly marine origin of the organic matter. Dibenzothiophene/phenanthrene (DBT/P) ratio (0.08), Phytane (Ph)/n-C<sub>18</sub> versus Pristane (Pr)/n-C<sub>17</sub>, and Pr/Ph versus DBT/P data suggest a marine environment of the Laki shale. The marine setting of the Laki Formation shale is further evident by *coralline algae* and *Alveolina oblonga*. X-ray diffraction (XRD) data reflect that Laki shale is brittle since it is dominated by high quartz and carbonates.

## KEYWORDS

Geochemical investigation, Laki Formation shale, Source rock, Southern Indus Basin.

## 1. INTRODUCTION

Geochemical assessment of a particular region shows the source rock quality, age, paleoenvironment, and maturity. The source rock potential is closely related to total organic carbon (TOC), volume, and maturity (Tissot and Welte, 1984; Peters and Cassa, 1994). Geochemical investigations of shale gas are rapidly increasing across the world due to its huge potential and the substantial exploration achievement in North America (Curtis et al., 2012; Hao et al., 2013). Paleocene, Jurassic, and Cretaceous successions of the Lower Indus Basin in Pakistan are studied by previous researchers as a source rock aspect (Robinson et al., 1999; Energy Information Administration, 2015; Shah and Ahmed, 2018). The Eocene Laki Formation shales have not been evaluated as a source rock perspective in Southern Indus Basin Pakistan. Keeping in view the current economic significance of the shales, this study focuses on the geochemical investigation of Laki Formation shales in the selected wells of the Southern

Indus Basin. The principal goal of the current research is to provide a better understanding of the hydrocarbon source rock potential of the Laki Formation shales. For the geochemical investigation of the Laki Formation shales, Hashim Kher and Khadeji wells are selected from Southern Indus Basin Pakistan (Figure 1).

## 2. GEOLOGICAL SETTING

The Indus Basin of Pakistan is a huge Late Paleozoic depression that occupies along the northwestern edge of the Indian shield (Dolan, 1990). The eastern two-thirds of Pakistan is covered by the Indus Basin. The axis of the Indus Basin is coinciding with the passage of the river Indus (Kingston, 1986). The Southern Indus Basin limits its northern boundary with Mari-Kandkot High, Indian shield to the east and its western edge is culminated by Chaman and Ornachal faults (Figure 1). Quadri and Shuaib

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suggested that the Murray Ridge-Owen fracture zone confines the south-southwest offshore extension of the Basin (Quadri and Shuaib, 1986). The Indian plate was drifted towards the north during Late Mesozoic in the Southern Indus Basin (Shuaib, 1982; Quadri and Shuaib, 1986; Quadri and Shuaib, 1987). The present study is restricted to the geochemical evaluation of the Laki Formation shales in selected regions of Southern Indus Basin Pakistan, hence its lithology is briefly described. The generalized stratigraphy of the Southern Indus Basin is represented in figure 2. Noetling proposed "Laki series" for the lower part of Blanford's "Kirthar series" (Noetling, 1903).

Hunting Survey Corporation introduced the name Laki group for Laki Formation (Hunting Survey Corporation, 1961). At the type locality Laki Formation has a thickness of about 240 meters. The lithology of the Laki Formation is cream to grey limestone, marl, calcareous shale, sandstone, and lateritic clay. In the Kirthar province, Laki Formation is categorized into two members (Sohnari & Chat). Both these members are approved by a stratigraphic committee of Pakistan (Shah, 1977). In the project area (Southern Indus Basin) Laki Formation consists of shale and limestone (Shah, 1977; Raza et al., 1990). The Sohnari Member includes lateritic clay, shale with yellow arenaceous limestone beds, ferruginous, calcareous sandstone, and lignite layers. This member represents the basal part of the Laki Formation and directly overlies the Paleocene Ranikot Group (Shah, 2009). The lower section of Chat Member comprises creamy white nodular limestone, while its upper section has interbedded shale and limestone with minor sandstone. The shale is of variable colors, ferruginous and gypsiferous. The sandstone is generally ferruginous, while limestone is slender bedded, and arenaceous (Shah, 2009). The Laki Formation is typically exposed in the Southern part of the Kirthar province and the surrounding area of the Marri-Bugti hills in the Sulaiman province. The lower contact of the Laki Formation with the Ranikot group is unconformable and it makes conformable contact with the upper Kirthar Formation in Kirthar province. The Formation is fossiliferous and characterized by foraminifera. Echinoids and algae have been reported by Noetling (Noetling, 1905). Based on fossils Early Eocene age is assigned to the Laki Formation.

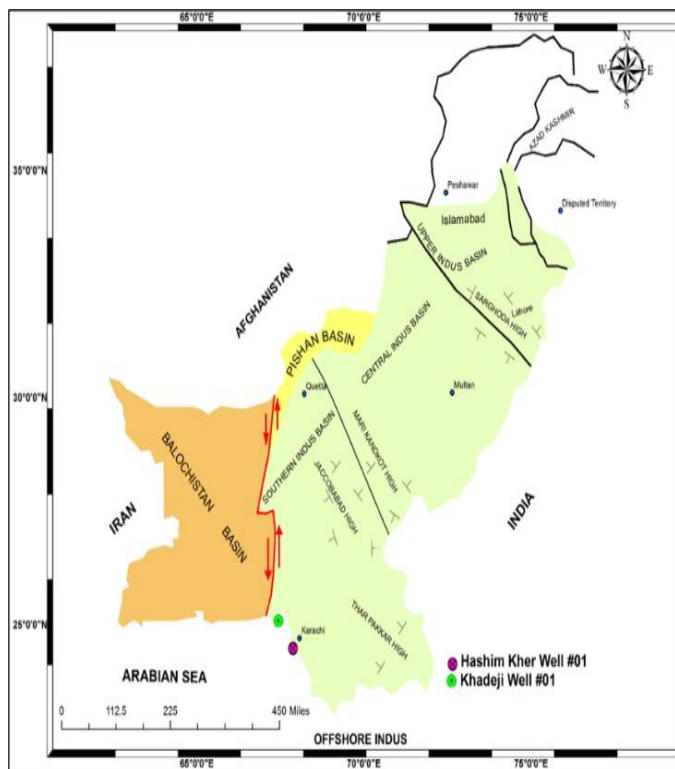


Figure 1: Map showing sedimentary Basins of Pakistan (Farah et al., 1984). The research area is represented by Hashim Kher and Khadeji wells.

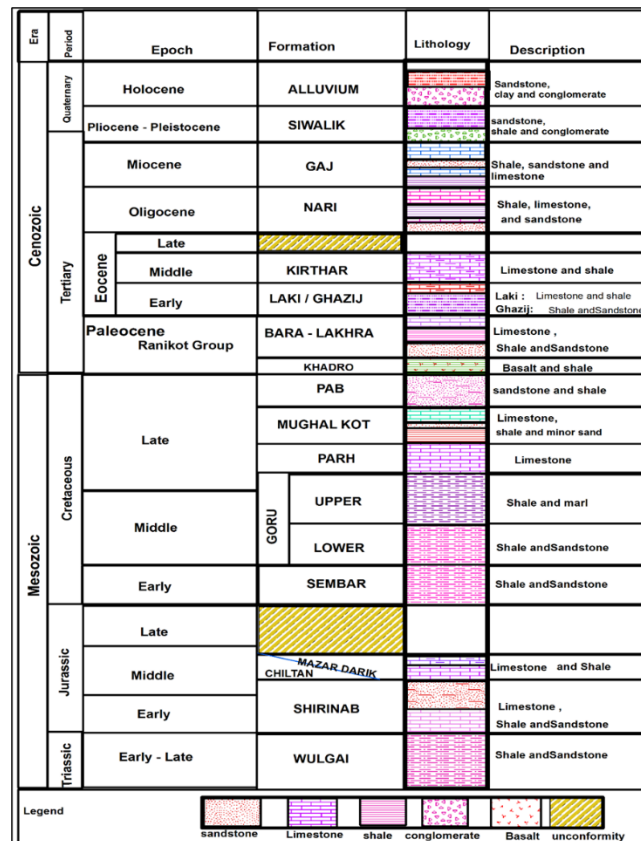


Figure 2: Showing Stratigraphy of Southern Indus Basin Pakistan (Shah, 1977; Raza et al., 1990).

### 3. MATERIAL AND METHODS

For the geochemical investigation of the Early Eocene Laki Formation shales, well-cutting samples (Table 1) were provided by Oil & Gas Development Company Limited (OGDCL). Total organic carbon (TOC) analyses were performed on twenty-three samples and Rock-Eval (RE) pyrolysis analyses were carried out on three samples. The pyrolysis and TOC data have been obtained using Rock-Eval 6. A Soxhlet tool was used for extractable organic matter of a shale sample, with dichloromethane (DCM) and methanol in a volumetric ratio of 97:3. This process was then followed by column chromatography and different solvents were used to segregate the extractable organic matter (EOM) into various portions (aromatic hydrocarbons and saturated hydrocarbons). Gas chromatography-mass spectrometry (GC-MS) analysis was performed with Varian Ion Trap GC-MS 4000, an instrument with Column C98911, VF-IMS, and 30\*0.25.

The temperature of the injector was 300 °C. The initial temperature was set to 50 °C for 1.5 min, then ramped to 300 °C at 3.5°C/min. Helium was used as a carrier and was set to flow at a rate of 1.0 ml/min. Stable carbon isotope ( $\delta^{13}C$ ) analysis was carried out on the powder sample of Laki Formation shale using Pee Dee Belemnite (PDB) standard. X-ray diffraction (XRD) analysis was performed on one sample. One gram shale sample was pulverized by hand before analyzing in an x-ray diffractometer (model X Pert PRO). The x-ray instrument was functioned at the condition of an x-ray with wavelength Cu K alpha radiations 1.540598 nm, generating voltage 40 kilovolts, Tube current 30 milliamperes, Divergent slit fix, receiving slit 0.1, Scan step size continuous, Scan steps size 0.01, and Time per scan is 0.25.

### 4. RESULTS AND DISCUSSION

#### 4.1 Organic matter richness and hydrocarbon generation potential

The organic richness of source rock is typically represented as the total organic carbon (TOC) content in weight percent (Peters and Cassa, 1994; Hunt, 1996). The hydrocarbon potential of source rock depends on the quantity of total organic carbon (TOC) as well as the thermal maturity of

organic matter (Hunt, 1996). The TOC values of the Laki shales are presented in Table 1. The results of the Laki Formation shales are correlated with Jarvie to assess their hydrocarbon potential (Jarvie, 1991). The TOC values of the Laki shale in the Hashim Kher well are varying from 0.53 to 2.66 wt. % (Table 1). These values suggest that Laki shale has fair to very good hydrocarbon potential. The TOC values of Laki shales in Khadeji well are ranging from 0.55 to 1.53 wt. % (Table 1), and indicates that hydrocarbon potential is fair to good. The hydrocarbon potential of the shale is also deduced from the cross plot between TOC versus depth (Figure 3).

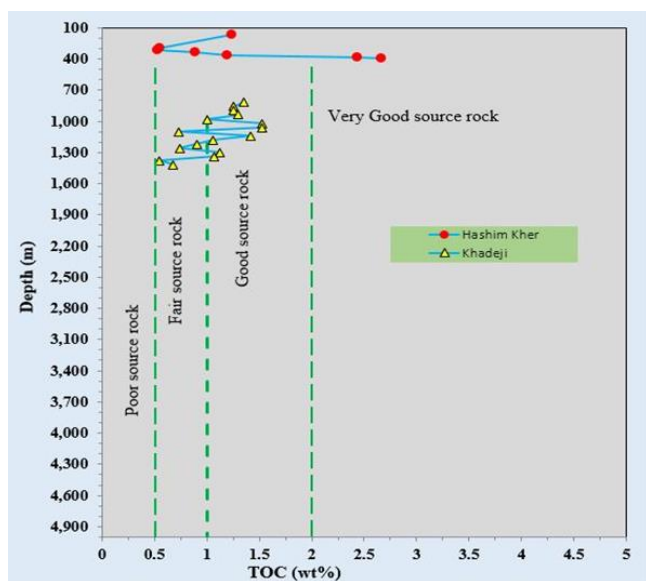
The diagram designates predominantly good hydrocarbon source rock

potential. Laki shales are also evaluated as a hydrocarbon generation potential based on S<sub>1</sub> and S<sub>2</sub> values. The S<sub>1</sub> (Table 1) values of Laki shales are ranging from 0.00 to 0.08 mg HC/g rock, whereas the S<sub>2</sub> varies from 1.39 to 9.97 mg HC/g rock. The values indicate poor hydrocarbon generation potential. The hydrocarbon generative potential of the Laki shale is also represented by generation potential (GP) values. GP values of the Laki shales (Table 1) indicate poor hydrocarbon generation potential. GP values of the Laki shale are compared with Ghori and Haines; Tissot and Welte (Ghori and Haines, 2007; Tissot and Welte, 1984). The hydrogen index (HI) and oxygen index (OI) values of the Laki shales (Table 1) suggested that type of hydrocarbon is gas. The observed values of S<sub>1</sub>, S<sub>2</sub>, HI, and OI are correlated with Peters (Peters, 1986).

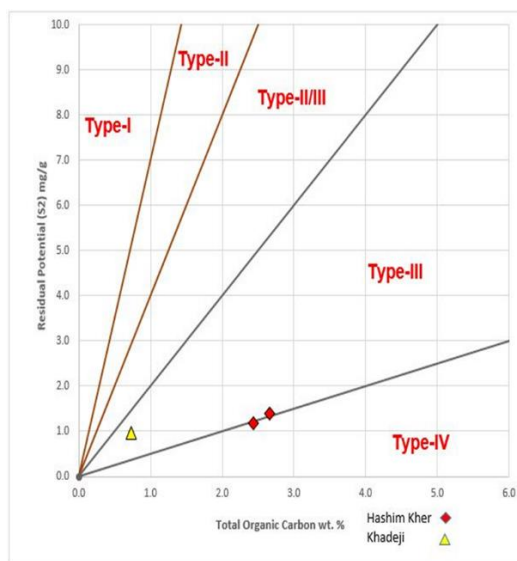
**Table 1:** Showing TOC and Rock-Eval pyrolysis parameters of Laki Formation shales

Well	Depth (m)	TOC (Wt. %)	T <sub>max</sub> (°C)	S <sub>1</sub> mg HC/g Rock	S <sub>2</sub> mg HC/g Rock	HI Mg HC/g TOC	OI Mg CO <sub>2</sub> /g TOC	GP mg/g
Hashim Kher	170-75	1.24						
	300-5	0.55						
	320-25	0.53						
	340-45	0.89						
	360-65	1.19						
	380-85	2.44	432	0.01	1.18	48	61	1.19
	390-95	2.66	434	0.00	1.39	52	68	1.39
Khadeji	820-25	1.35						
	860-65	1.25						
	900-05	1.25						
	940-45	1.30						
	980-85	1.00						
	1020-25	1.53						
	1060-65	1.53						
	1100-05	0.73	434	0.08	0.97	133	82.2	1.05
	1140-45	1.42						
	1180-8	1.06						
	1220-25	0.90						
	1260-65	0.74						
	1300-05	1.12						
	1340-45	1.07						
1380-85	0.55							
1420-22	0.68							

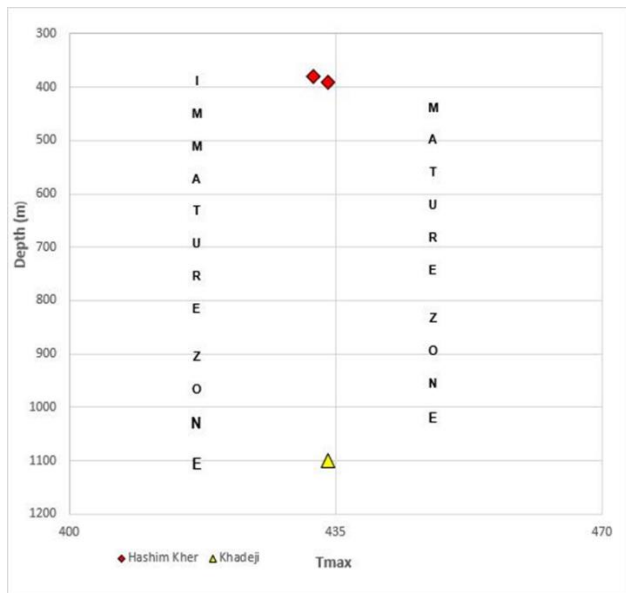
TOC: total organic carbon; Tmax: maximum temperature; S<sub>1</sub>: free hydrocarbons; S<sub>2</sub>: remaining potential; HI: hydrogen index; OI: oxygen index; (GP): generation potential.



**Figure 3:** Showing cross plot relation between TOC and depth of the Laki shales (diagram boundaries are after Tissot and Welte, 1984; Peters and Cassa, 1994).



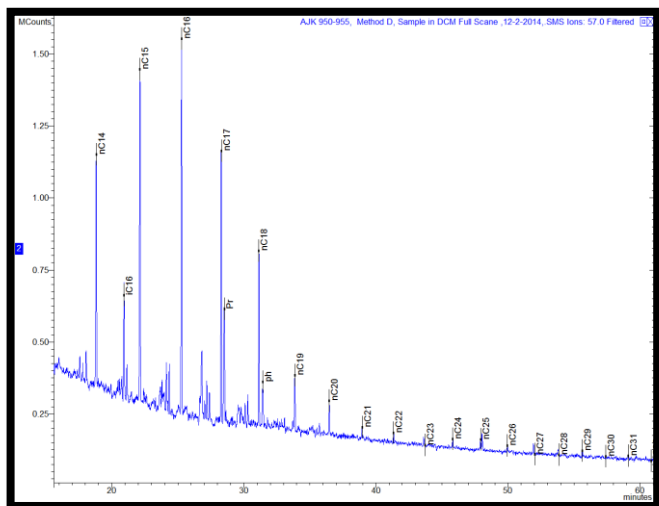
**Figure 4:** Showing cross plot relation between TOC and depth of the Laki shales (diagram boundaries are after Tissot and Welte, 1984; Peters and Cassa, 1994).



**Figure 5:** Plot of Tmax versus depth for Laki shales (diagram boundaries are after Tissot and Welte, 1984; Peters and Cassa, 1994).

**4.2 Origin of Organic Matter**

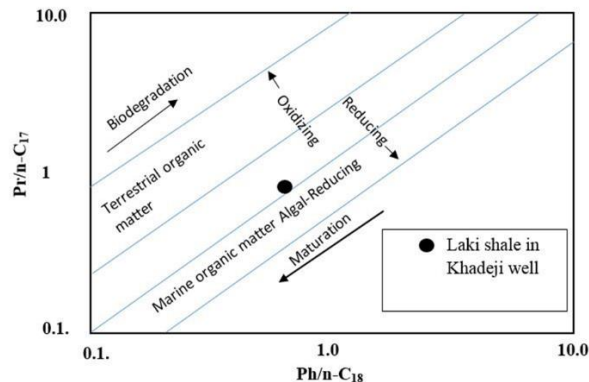
Marine and terrestrial organic matter sources of the Laki shales were discriminated based on  $\delta^{13}C$  isotopic value and normal alkane data. The  $\delta^{13}C$  values for land-derived organic matter vary from -20 to -30‰ (Decker and de Wit, 2006). Whereas  $\delta^{13}C$  values for an organic matter of marine origin ranges between -5 to -25 ‰ (Schoeninger and DeNiro, 1983). The  $\delta^{13}C$  value for the shale of the Laki Formation is -9.67 ‰ (PDB). The value of stable carbon isotope for Laki shale indicates predominantly that the organic matter was a marine source. Based on saturated hydrocarbons organic matter provenance is evaluated for the Laki Formation shale in Khadeji well. The source of low molecular weight normal alkane (< n-C<sub>20</sub>) are marine algae and microorganisms (Cranwell, 1977). The normal alkane ranges from n-C<sub>21</sub> to n-C<sub>25</sub> are originated from aquatic macrophytes (Ficken et al., 2000). The long straight-chain alkanes (> n-C<sub>27</sub>) are then derived from higher land plants (Eglinton and Hamilton, 1967). The Laki Formation shale is dominated by n-C<sub>16</sub> followed by n-C<sub>15</sub> and n-C<sub>17</sub> (Figure 6). The alkane data suggest that algae and microorganisms were contributed to the organic matter of Laki Formation shales. The lesser amount of n-C<sub>20</sub> to n-C<sub>31</sub> is also observed in the Laki shale (Figure 6). The presence of n-C<sub>20</sub> to n-C<sub>31</sub> indicates minor aquatic macrophytes and higher plant input in the shale of the Laki Formation. Based on saturated hydrocarbons the organic matter of the Laki Formation is mixed. Therefore, the most probable type of organic matter is mixed (land plants & marine algae).



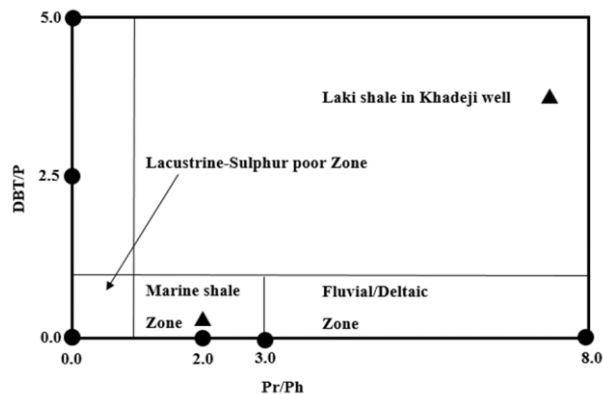
**Figure 6:** Chromatogram of Laki Formation shale extracts showing distribution of alkanes (m/z 57).

**4.3 Depositional Environment**

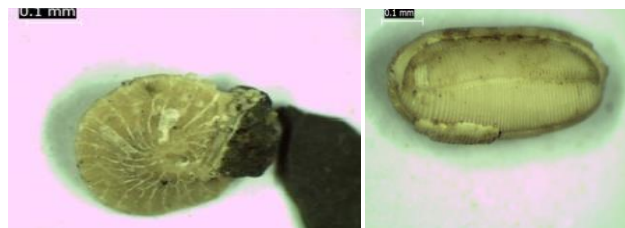
According to a study, dibenzothiophene/phenanthrene (DBT/P) ratio is used to discriminate the marine shale and carbonates (Hughes et al., 1995). The DBT/P ratio <1 indicates marine shale, while >1 suggests marine carbonates. The DBT/P value is 0.08 indicating that Laki shale was deposited in a marine setting. This marine environment is evident by cross plots between Ph/n-C<sub>18</sub> versus Pr/n-C<sub>17</sub> (Figure 7) and Pr/Ph versus DBT/P (Figure 8). The diagrams suggest a marine environment. The marine condition of the Laki Formation shale is further supported with *coralline algae* and *Alveolina oblonga* (Figure 9).



**Figure 7:** Showing Cross correlation of pristane/n-C<sub>17</sub> versus phytane/n-C<sub>18</sub> ratios of Laki Formation shales boundaries are after Connan and Cassou (Connan and Cassou, 1980).



**Figure 8:** Cross plot of DBT/P vs. Pr/Ph showing depositional environment of Laki shale (boundaries after Hughes et al., 1995).

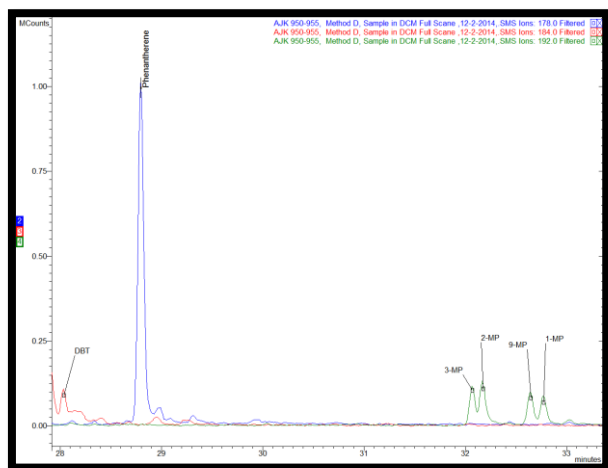


**Figure 9:** Photomicrographs of Laki Formation shale in Khadeji well showing *Alveolina oblonga* and *coralline algae*.

**4.4 Maturity**

The Laki Formation has Rock-Eval pyrolysis Tmax values are below 435°C (Table 1) suggesting that shales are immature for hydrocarbon generation. Tmax values of the Laki Formation shales are interrelated with (Bacon et al., 2000; Peters and Cassa, 1994). Tmax data from the Laki shales are plotted (Figure 5) versus depth. The diagram designating that shales are immature for the generation of hydrocarbons. The vitrinite reflectance for shale and coal-based on methylphenanthrene indices are 0.5% for immature source rock and the values of 0.65 to 1.35% indicate oil window. The values of 1.35 to 2.0% reflect higher maturity (Radke and Welte, 1983; Boreham et al., 1988). Methyl phenanthrene index (MP-1) is

used for the Laki shale for the assessment of maturity (Figure 10). The calculated MP-1 is 0.16% suggesting that the Laki shale in this well is immature.



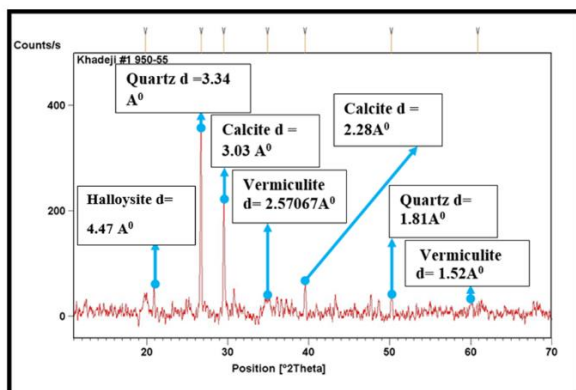
**Figure 10:** Chromatogram of Laki Formation shale extracts showing distribution of phenanthrene (m/z 178), alkyl phenanthrene (m/z 192) and dibenzofuran (m/z 184).

**4.5 X-ray diffraction (XRD)**

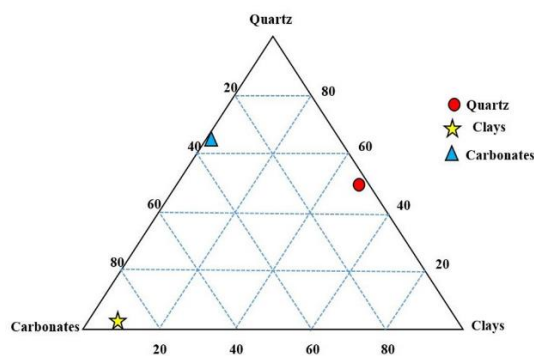
The XRD analysis of the shale of the Laki Formation is carried out to study their mineralogy. The mineralogical composition of Laki shales is recognized by correlating the latticed-spacing values acquired from x-ray diffractogram (Figure 11) with International Centre for Diffraction Data (International Centre for Diffraction Data, 2001). The mineralogical composition of the Laki shale in the Khadeji well as quartz, calcite, vermiculite, and halloysite (Table 2; Figure 11). The total quartz of the Laki shale is 51.8 wt. %, whereas total carbonates and total clays are 36.63 wt. % and 11.56 wt. % (Table 2). Total quartz, clays, and carbonates (Table 2) contents of the Laki shale are plotted in the ternary diagram (Figure 12). The diagram suggested that Laki shale is brittle since it is dominated by high quartz and carbonates.

**Table 2:** Showing mineralogical composition of Laki shale in Khadeji # 01.

Observed d-spacing A <sup>0</sup>	Standard d-spacing A <sup>0</sup>	Mineral	Wt. (%)	Total Quartz Wt. (%)	Total Clays Wt. (%)	Total Carbonates Wt. (%)
4.47	4.445	Halloysite	10.54	51.8	11.56	36.63
3.34	3.34	Quartz	100			
3.03	3.036	Calcite	60.07			
2.570	2.570	vermiculite	9.36			
2.28	2.285	Calcite	16.6			
1.81	1.818	Quartz	8.42			
1.52	1.528	vermiculite	4.3			



**Figure 11:** Showing powder x-ray diffraction pattern of Laki Formation shale in Khadeji well.



**Figure 12:** Ternary diagram showing the mineralogical distribution of quartz, carbonates, and clays of Laki Formation shales.

**5. CONCLUSIONS**

Based on the present geochemical investigation of Laki Formation shales, the following conclusions can be drawn.

1. TOC data of the Laki Formation shales show fair to very good hydrocarbon potential.
2. The Rock-Eval (RE) pyrolysis data indicate that the organic matter in the Laki shale is characterized by Type III kerogen.
3. The organic matter in the Laki shale is predominantly of marine origin.
4. Dibenzothiophene/phenanthrene ratio and microfossil data suggest that Laki shales were deposited in a marine environment.
5. Maturity parameters reflect that Laki shales are thermally immature for the generation of hydrocarbons.
6. Laki shales are characterized as brittle based on XRD data.
7. The present geochemical analyses of the Laki shales indicate good hydrocarbon potential, concerning the generative potential hydrocarbon source rock (based on S<sub>1</sub> & S<sub>2</sub> values) the Formation may encounter a high risk in terms of the financial side. This fact is reflected in maturity data that the shales are thermally immature.

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