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Analysis on geochemistry of oil shales of the Sargelu formation in central Zagros

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Abstract

To investigate the geochemical features of oil shales located in the vicinity of Zardkuh Bakhtiyari (Central Zagros) a section was selected where the sediments have cropped out. The precise location is at the Asal Keshan (AK) in the west of Chaharmahal and Bakhtiyari Province (CMBP). The sediments belong to the Jurassic Sargelu Formation and are composed of black to dark brown shales. The lower boundary is continuous and interfingering with the Neyriz Formation although with the upper formation where lies the Fahliyan Formation the boundary is discontinuous and unconformable. The oil shales in the area have formed at deep sea and in reduction conditions. Notwithstanding, they have never been able to develop oil due to a lack of continuous deposition and high temperature, which the latter is greatly required for oil maturing and development. The sole method to transform kerogen embedded in these shales to oil is Rock-Eval Pyrolysis. Some 10 samples taken from the shales were analyzed by the Rock-Eval Pyrolysis. The result was 9 variables by which diagrams of Tmax, TOC, kerogen type and hydrocarbon generation were constructed. The kerogen is predominantly of II variety. Hydrocarbon generation is restricted to crude oil of good to great oil-generating potential. the data obtained from XRF and ICP geochemical analyses showed that the number of heavy metal indices (NI, Pb, Rb, Sr, V, W, Zr, Zn) and trace elements (Ba, Ce, Co, Cr, Cu, Th, Nb, Ni, Rb, Sr, Mo, U), as well as the percentage of metallic and non-metallic oxides of Sio2, Al2o3, Na2o, Mgo, K2o, Tio2, Mno, Cao, P205, Fe203 inside the oil shales, were high. Therefore, the above shales have a dual economic value, which with the increasing reduction of oil and gas energy resources, and minerals can be of special importance in the future in terms of energy and minerals.

Keywords: Oil shales, Asal Keshan section, analysis, central Zagros, Rock Eval Pyrolysis 2020 MSC: 74G65

1 Introduction

The term oil shale addresses a group of organic-rich fine-grained sedimentary rocks, black to brown. Such rocks incorporate solid bituminous organic and inorganic materials that can be solved in most organic solutions [16]. Oil shales are pondered as the unconventional resources of oil that hold perceptible volumes of organic material from which huge volumes of oil could be extracted using the distillation process [2, 10]. Kerogen in oil shales is more of the first

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and second type [1] that has not reached maturity due to lack of sufficient temperature. Notwithstanding, this issue can be handled using temperature (artificial maturation), transforming their organic material into oil [4]. During this process, oil is heated by $450 - 500^{\circ}C$ under anaerobic conditions. Kerogen bonds are broken and transformed into oil, as a result. At some shale formations e.g., the Kening Basin in Western Australia, the rate of shale maturity is to the extent that organic material can generate dry gas, therefore, the thermal process is introduced into the gas window. Such a shale is called gas shale [16]. Oil shales develop through the deposition of fine-grained and mainly argillaceous along with marine micro-organisms at deep sea. They then are introduced into the petrification stage by controls such as pressure, having their organic material transformed into kerogen by the temperature generated over the burial [4].

Oil shale formations have extensively cropped out in the west of CMBP somewhere around Zardkuh. They have not been studied appropriately due to, perhaps, the ruggedness of the area or have not been taken for granted as prospective energy sources. With the world's oil and gas reserves at the onset of depletion, most countries are floundering to seek a reliable replacement for their conventional energy-producing resources. One of such replacements could be oil shale [9]. Many countries have conducted perusal geochemical studies on the global oil shale deposits amongst which the Canadian Green River suggested that oil shales could consist of anomalies of several metallic and non-metallic elements, despite their wonderful strength in generating hydrocarbon [17]. Speaking of the significance of the issue, the type section at the AK with an extensive outcrop of oil shale was chosen. Some 10 samples were taken to perform geochemical analysis.

2 Section location and regional geology

The study section is located in the west of Kuhrang at CMBP and coordinates N32° 34' 32.73", E49° 55' 03.80". It is 135 km from Shahrekurd the capital city of the province (Fig. 1). The 1:250000 geology map of Shahrekurd [18] incorporates the area (Fig. 2), and in terms of the geological division of Zagros [14], it lies over the High Zagros at the Central Zagros.



Figure 1: Location of CMBP and study section on Iran's map.

The most prominent faults that have played a role in the area's tectonism are the two reverse faults of Zardkuh and Zagros, forming the highest altitudes of the Zagros basin. The Sargelu oil shale has cropped out along these faults. Besides, the entire anticline and synclines have the same NW-SE trend as the Zagros Mountain does [2]. Palaeontologically, the oldest sediments are composed of evaporate deposits of the Hormuz Series aged Infra-Cambrian which have uplifted along the main faults' trends forming salt domes, and uplifted younger deposits of the Palaeozoic while ascending. The youngest deposits consist of present-day alluvial deposits and quaternary terraces. Microscopic study of thin sections suggests that the oil shale deposits all belong to the Jurassic Sargelu Formation and have been deposited at a relatively deep sea. They are black to brown and intensely fetid together with pyrite (Fig. 3). Field investigations show that the lower boundary of the Sargelu Formation with the carbonate-clastic deposits of the Neyriz Formation is continuous and interfingering. The Sargelu Formation due to the lack of Upper Jurassic deposits was discontinuously overlain by the carbonate deposits of Lower Cretaceous Fahliyan Formation.



Figure 2: Location of study section on 1:250000 Shahre kurd Geology Map.

3 Methodology

Nowadays, chemical analyses are used in studying oil shales around the world. Hence, the main objectives of this work are to geochemically analyse samples of oil shale to identify oil-generating potential, kerogen variety, organic carbon level, organic material variety and thermal maturity. The methodology entails the determination of different stages of oil-generating potential, kerogen variety, organic carbon, organic material, thermal maturity and other variables pertaining to organic material in oil shales, by the Rock-Eval pyrolysis. Some 10 samples were taken and analyzed by means of the aforesaid Rock-Eval pyrolysis, the obtained data were assessed and unfathomable factors became fathomable and complete.

4 Discussion

4.1 Characteristics of oil shales

In ideal conditions when organic material in reduction marine environment is buried by fine-grained sediments, it becomes denser and transforms into hard rock, having their organic material also transformed into solid kerogen. Such processes all are triggered by the pressure of overlying strata and by diagenesis. The pressure and temperature keep rising over millions of years due to the bulk and weight overhead, resulting in the breaking down of kerogen molecules in source rock, transforming into crude oil under sophisticated microbial-chemical processes. The generated oil will later migrate upward due to pressure exerted by the source rock (from high-pressure to low-pressure spots) and enter the reservoir rock. Oil shales, however, do not experience such stages/processes, which means their kerogen remains untapped after diagenesis and the development of sedimentary rock. The reason why is that kerogen is not exposed to sufficient heat to break down due to discontinuous or even lack of deposition. The same is true in the oil shale of the Sargelu Formation. This shale has not found the leisure to mature due to the particular tectonism governing the Tethys Basin. At this time and region, on one hand, the activity of Zagros, Zardkuh and Bazoft faults and uplift of the basin through the Jurassic orogeny, on the other, have prevented deposition of Upper Jurassic sediments over the shales as the basin has been active. Lack of deposition and insufficient temperature at depth, as a result, is expected to have prevented the oil shale from maturity. As temperature causes kerogen in source rock to shift into oil and gas, through maturing the rock, breaking the kerogen molecules, conducting chemical activities and generating oil and gas, these shales have remained immature as they were, being disqualified to generate oil. Therefore, the sole way to generate oil is the synthetic method through which shale receives heat. Most oil shales, on a global scale not only have hydrocarbon potential but also inorganic material of economic value [5].



Figure 3: Close view of oil shale at AK section in Central Zagros (west of CMBP).

4.2 Rock-Eval Pyrolysis

This is a very quick, inexpensive and standard method to assess hydrocarbon generation in source rocks e.g., in shales and in organic facies which are widely applied in the oil industry [8]. Using Rock-Eval pyrolysis, genetic potential, production index, hydrogen index and oxygen index are obtained. The method also produces invaluable data on total organic carbon (TOC), variety and volume of organic matter, potential, thermal maturity of organic matter and the type/variety of maturity [3, 7]. There are two methods to measure the TOC in source rock:

The first and least common method in which the sample is directly burnt. The sample is ground and washed by NaCl and then carbonate is burnt by oxygen and under atmospheric pressure at a temperature of ~ 1000°C. The volume of organic carbon is identified by measuring the CO2 produced over the burning of organic matter.

During the second and newer method the sample is analyzed by the Rock-Eval pyrolysis and the TOC is determined in wt and based on pyrograph peaks and, as a result, the pyrolyzed and remaining carbon can be told apart. Using the obtained factors the TOC can be calculated:

$$TOC = 0.82(S1 + S2)/10 + S3c^{\circ}/10$$

In brief, the unconfined hydrocarbons that once existed during deposition at the source rock (shale) or have formed during burial are vaporized at ~ 300°C. The process known as Thermal Vaporization would create an S1 peak in solely 3 min (free hydrocarbons in rock). The process will then continue at 300-600°C and unconfined and heavy kerogen and bitumen which have been vaporized by 300°C, are broken and form S2 peak (the hydrocarbons that are released during pyrolysis) which shows the potential of the rest of the rock. The highest temperature at which the S2 peak can develop the greatest volume of hydrocarbons through the thermal breakdown of kerogen molecules is called Tmax. At 300-390°C carboxyl groups and other oxygenated compounds and CO₂ are generated. The released CO₂ is measured by a thermal conductor and registered as an S3 peak. The hydrogen index (HI) represents the level of hydrogen and the capability of generating hydrocarbons in one hydrocarbon source rock is immature or of low maturity, the HI shows the kind of generated hydrocarbons in one hydrocarbon source rock. As the level of S2 declines, positive anomalies occur in the oxygen index (OI). Having increased the conservation level of organic matter and decreased oxidizing factors, the OI has risen. This is because the further the oxidant the setting becomes, the further the organic matter is oxidized and its hydrogen level and, as a result, its HI is declined. The S3 peak displays the organic source of CO₂ and represents oxygenated compounds in carboxyl groups which are disintegrated by 390°C, releasing CO₂ (mgCo₂/g rock).

4.3 Analysis of Rock-Eval Pyrolysis data

The results obtained from the study of 10 samples are shown in Table 1. During the tests, the numerical values of 9 variables were measured, tables and diagrams pertaining to the study objectives were constructed and discussions and analyses were made regarding the entire process.

	Table 1. Data obtained by pytolysis of Tix section samples									
NO.AS	ΟΙ	HI	$T_{\rm max}$	$S_3 co(mg/g)$	$S_3, (mg/g)$	$S_3(mg/g)$	$S_2(mg/g)$	$S_1(mg/g)$	TOC	
AS1	14	1167	446	0.36	13.51	0.75	61.03	0.80	5.23	
AS2	4	476	445	0.4	12.66	0.70	78.08	1.65	16.40	
AS3	5	444	445	0.22	10.33	0.68	66.68	0.98	15.01	
AS4	7	501	443	0.44	11.03	0.87	65.02	1.54	12.99	
AS5	4	493	444	0.25	10.89	0.49	58.59	1.29	11.88	
AS6	15	458	439	1.35	15.44	3.5	105.52	1.55	23.03	

Table 1: Data obtained by pyrolysis of AK section samples

AS7	12	472	438	0.98	12.04	2.54	103.99	1.36	22.99
AS8	11	488	440	0.96	13.88	2.04	87.66	0.96	17.95
AS9	14	478	442	0.74	11.3	1.52	50.36	1.58	10.54
AS10	29	1125	444	0.5	10.6	1.62	63.35	0.84	5.63

4.3.1 Oil-generating quality

Based on Table 1, the most level of TOC amongst the shales is 22.99 and the lowest is 14.068. Therefore, to determine the frequency of TOC, the frequency diagram was constructed for TOC (Fig. 4). Since TOC < 2wt% (Table 2) alludes to a very good source rock for oil shales [11]. Hence, the AK oil shale samples represent a great source rock and a high oil-generating quality.

Table 2: Quantity of source rocks based on different rock types and associated TOC [11].

Quantity	TOC in shales $(wt\%)$	TOC in carbonates (wt%)
Poor	0 - 0.5	0-0.2
Fair	0.5 - 1	0.2 - 0.5
Good	1 - 2	0.5 - 1
Very good	2–5	1-2
Excellent	> 5	>1



Figure 4: TOC diagram shows frequency of organic carbon in AK section.

4.3.2 Kerogen variety determination

The kerogen variety in samples is determined using an S2/TOC diagram. The samples that fall within the kerogen I realm, have lacustrine-marine (algae in fresh to brackish waters) origin, hence, they are otherwise known as algal kerogen [6]. Such kerogens live in lacustrine and marine environments. Lipids are among their principal components. Kerogen I tends to generate crude oils that are rich in saturated hydrocarbons. The samples that fall within the kerogen II realm have a marine origin and are known as lipstick kerogen. Particles in them are chiefly composed of disintegrated phytoplanktons and zooplanktons and are amorphous. This variety of kerogen tends to generate crude oils rich in aromatic and nephtic compounds. The samples whose realm are within kerogen III, have the onshore origin and are rich in lipidic organic material e.g., herbal resin, spores, roots and twigs of vascular plants. Figure 5 shows the kerogen variety in the AK section measure based on TOC/S2. In this section, ~ 80% of the samples are of kerogen II and ~ 20% are of kerogen I type. A large number of samples at the AK section on the kerogen II realm have marine origin and can generate oil. Comparing them with the standards of Peters and Cassa [12], kerogen II ranks very good to great in terms of oil-generating criteria. Speaking of origin, the organic materials are marine and have been developed by the disintegration of phytoplankton, zooplankton and marine algae bodies that have settled in an intensely reductive environment.

4.3.3 Thermal maturity determination

Thermal maturity is one of the factors that are assessed by the Rock-Eval pyrolysis. Formation of oil initiates at 60°C, although the maximum rate of oil formation occurs at ~ $100^{\circ}C$ and declines with the increase in temperature. The window of oil formation halts at 175°C and the principal gas generation stage initiates. This is not, however,

Kerogen type	HI (mg HC/g TOC)	S2/S3
Ι	< 800	< 15
II	300-600	10-15
II/III	200-300	5 - 10
III	50-200	1-5
IV	> 50	> 1





Figure 5: Standard parameters to determine kerogen type in AK section samples based on RockEval Pyrolysis analysis.

commensurate with laboratory and Rock-Eval pyrolysis experiments as the temperature under which oil is generated at the source rock differs from that of the laboratory setting. For instance, the beginning of the oil window is highlighted by the time the T_{max} variable is obtained from the Rock-Eval pyrolysis and the temperature varies between 435-460°C. The T_{max} diagram, pertaining to the AK section, was constructed using Figure 6 and the Rock-Eval pyrolysis data. As displayed on the T_{max} and compared with Peters and Cassa [12] standard, the T_{max} variable initiates at ~ 440°C and culminates at 450°C which means that the samples are at the last stage of diagenesis and at the starting point of oil window. The realm of the samples is represented as a dark halo on the diagram. Besides, a guide to pyrolysis for quality and quantity of oil shale thermal maturity by Peters and Cassa [12] is incorporated in Figure 6. On this table, which is both based on T_{max} and vitrinite reflexing coefficient percentage (R_0 %), oil shales rank in the realm of immature to post-mature. In this work, the R_0 % has not been measured, although the T_{max} average has been measured at 442.60 using the Rock-Eval pyrolysis. Comparing the table data with each other, the oil shales of the AK section fall within the realm of early maturity.

Stage of the	ermal maturity for oil	Maturation			
Vitrinite	reflectance $(R_0\%)$	Rock-Eval $T_{max}(^{\circ}C)$			
Immature	0.8 – 0.2	< 435			
Mature					
Early	0.6 - 0.85	435-445			
Peak	0.65 - 0.9	445-450			
Late	0.9 - 1.35	450-470			
Post mature	> 1.35	> 470			

Table 4: Standard parameters to evaluate hydrocarbon potential based on Rock-Eval pyrolysis [12].

4.3.4 Determination of type of hydrocarbon generated

The type of the produced hydrocarbon from the shale samples is solely crude oil with no gas generation. This was found regarding the HI variables (Table 1) using Peters [11] standard table (Table 5). Therefore, the ultimate product after heating the oil shales, is crude oil.

5 Chemical analysis by XRF, ICP method

5.1 Geochemical analysis by XRF method for Asal Keshan section

There are two main methods for measuring the amount of elements in a rock:



Figure 6: T_{max} diagram and rank of hydrocarbon-generation potential in AK section samples.

Table 5: Geochemical parameters	describing type c	of hydrocarbon	generated	[11]	
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Type	$HI(mg HC/g C_{org})$	S2/S3
Gas	0 - 150	0–3
Gas and oil	150-300	3 - 5
Oil	+300	+5

Chemistry of decomposition, which mainly uses titration, and non-destructive X-ray decomposition, which is called X-ray Fluorescence (XRF). XRF can analyze many elements qualitatively and quantitatively with a very high operating speed. Due to its high speed and absence of chemicals, it is cheaper than other analytical methods and does not pollute the environment.

Geochemical analysis data of Asal Keshan samples as aggregates for heavy metals includes an average of ten samples of the Asal Keshan section, from one to ten. The concentration and average of laboratory data are recorded in the table below, and also the frequency chart is drawn separately.



Table 6: The concentration and average of laboratory data

Figure 7: The frequency chart

5.2 Geochemical analysis by ICP method for Asal Keshan section

Inductively Coupled Plasma spectroscopy is an atomic emission spectroscopy that uses an electric current to change a time-varying magnetic field to create an electromagnetic induction and create a warm plasma environment. The ICP device is used to analyze metal elements, especially heavy metals and some non-metals. In general, the Inductively Coupled Plasma spectroscopy is a type of emission spectrometer. In other words, the ICP spectrometer is an element analysis technique that uses the sample emission spectrum to identify and quantify the elements in the sample and the intensity of this emission will indicate the concentration of the element in the sample.

Geochemical analysis data of Asal Keshan neck samples as aggregates for trace elements includes an average of ten samples of the Asal Keshan section, from one to ten. The concentration and average of laboratory data are recorded in the table below, and also the frequency chart is drawn separately.

Table 7: The concentration and average of laboratory data									
Ba	\mathbf{Ce}	Co	\mathbf{Ce}	\mathbf{Cu}	$\mathbf{N}\mathbf{b}$	Mo	\mathbf{U}	\mathbf{Th}	\mathbf{CL}
ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm
130.3	40.1	12.3	13.5	46.5	14	13.33	3.9	4.1	13.2



Figure 8: The frequency chart

Table 8: Results	of chemical analysis	of Asal Keshan	section samples	by XRF	` and ICP	methods
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Sample	AS-1	AS-2	AS-3	AS-4	AS-5	AS-6	AS-7	AS-8	AS-9	AS-10	Average	\mathbf{EF}
No.												
Ba	136	128	118	126	131	129	132	168	117	118	130.3	0.58
Ce	40	41	37	39	33	42	33	49	43	44	40.1	1.37
Со	11	11	13	10	16	12	13	14	14	9	12.3	1.43
Cr	115	160	115	125	145	127	130	132	149	153	135.1	3.60
Cu	50	42	47	49	46	52	46	57	35	41	46.5	2.40
Nb	13	11	15	14	12	12	17	11	17	18	14.0	2.00
Mo	10	14	12	11	13	14	14	15	15	15	13.3	_
U	5	5	6	4	4	3	4	3	3	2	3.9	3.33
Th	4	4	5	3	4	5	5	5	1	5	4.1	0.78
Cd	14	11	12	13	15	16	11	10	16	14	13.2	_
Ni	118	121	116	114	121	118	119	134	119	121	120.1	5.81
Pb	4	3	4	3	5	5	3	6	5	5	4.3	0.55
Rb	36	37	36	39	36	38	32	43	44	30	37.1	0.62
Sr	168	168	151	158	154	148	163	134	148	146	153.8	2.00
V	106	98	97	98	92	91	90	103	87	101	96.1	1.73
W	3	4	4	5	5	3	5	5	4	6	4.4	4.07
Y	15	13	15	14	13	16	16	12	17	17	14.8	1.48
Zr	112	109	112	102	112	109	110	111	122	113	111.2	1.50
Zn	26	29	31	27	29	25	26	23	30	21	26.7	0.84

5.3 Description of the results of geochemical analysis of oil shales

5.3.1 Enrichment of elements

Post-Archean Australian Shale (PAAS); Taylor and McLennan [15] was used as a reference to investigate trace element enrichment in the Sargelu Formation. The distribution patterns of PAAS-normalized trace elements in the Sargelu Formation in the Asal Keshan and Abkaseh sections are shown in Figure 9, respectively. Although the element patterns in this figure differ in detail, their general trends are similar.

The elements Zr, Y, V, Rb, Pb, Th, Nb, Co, Ce and Ba in this section show depletion in the Sargloo Formation. Elements W, Ni, U and Cr in this section are enriched in Sargelu Formation. Elements Zn and Sr in the Asal Keshan section show depletion, Cu element is slightly enriched in a small number of samples of Sargelu Formation in the Asal Keshan section and in other samples of this section show depletion.

Enrichment factor (EF) was also calculated for the average concentration of major oxides and trace elements using

the following equation.

 $EF = (element/Al)_{sample}/(element/Al)_{PAAS}$

According to the values of EF, the enrichment of the main oxides in the Sargelu Formation in the Asal Keshan section is CaO > MgO > P2O5 > Fe2O3 > MnO > TiO2, so the highest enrichment belongs to CaO and the lowest to TiO2.

EF values show that the order of enrichment of trace elements in the Asal Keshan section is Ni > W > Cr > U > Cu > Nb = Sr > V > Zr > Y > Co > Ce, so Ni is the most enriched and Ce have the least enrichment. The four elements Ni, U, W and Cr in this section have the highest enrichment. Enrichment is mainly seen in elements sensitive to oxidation and reduction [13].



Figure 9: Distribution pattern of trace elements normalized by PAAS [15] in Asal Keshan section.

6 Conclusions

- 1. It was revealed that the immaturity of oil shales of the Sargelu Formation is due to the tectonism governing the basin. The activity of faults and uplift of the basin triggered by Jurassic orogeny has prevented any deposition of Upper Jurassic sediments over the shales. Subsequently, due to the lack of sufficient heat, the maturity of oil shales has not occurred and the source rock has remained immature, being disqualified from generating oil.
- 2. RockEval geochemical analyses revealed that the hydrocarbon indices in samples are very high and optimum; hence, the newly discovered oil shales at the AK section could be viewed as a prospective replacement to the globally depleting energy resources.
- 3. The wt% of TOC, as a result, is very high for the AK section samples (14.068), therefore the samples are ranked as very good source rock.
- Based on the TOC/S2 diagram it can be concluded that ~ 80% of samples are of kerogen II and ~ 20% are of kerogen I type which shows their marine source.
- 5. T_{max} average of the samples that have been obtained by the RockEval analysis is 442.60 which falls within the low maturity realm.
- 6. Using data obtained by the RockEval pyrolysis and Tmax diagram, it was revealed that the Tmax variable begins at $\sim 400^{\circ}$ and culminates at $450^{\circ}C$ which shows that the AK samples have appeared at the end of diagenesis and beginning of thermal maturity or actually when the oil window began to form.
- 7. Considering the HI values, it was found that crude oil is the sole hydrocarbon produced by oil shales of the Sargelu Formation and no gas is generated.
- 8. The elements Zr, Y, V, Rb, Pb, Th, Nb, Co, Ce and Ba in this section show depletion in the Sargelu Formation. Elements W, Ni, U and Cr in this section are enriched in Sargelu Formation

- 9. Given the values of EF, the enrichment of the main oxides in the Sargelu Formation in the Asal Keshan section is CaO > MgO > P2O5 > Fe2O3 > MnO > TiO2, so the highest enrichment belongs to CaO and the lowest belongs to TiO2.
- 10. EF values show that the order of enrichment of trace elements in the Asal Keshan section is Ni > W > Cr > U > Cu > Nb = Sr > V > Zr > Y > Co > Ce, so Ni is the richest and Ce have the least enrichment. The four elements Ni, U, W and Cr in this section have the highest enrichment.

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