Low-temperature ashing for Z0, Z1 and Z2 Tarfaya (Morocco) oil shale layers, followed by acid etching to prevent physical and chemical effects

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Abstract :

An approach to the reactivity of an acid attack on oil shale samples from three layers (Z0, Z1, Z2) of the Tarfaya deposit was the subject of this study. The experimental method used consists of low-temperature ashing (LTA) in order to oxidize the organic matter with as little change as possible in the inorganic phases. In other words, acid etching followed by back-assaying were carried out, with the shale samples studied first isothermal combusted at low temperatures ranging from ambient to 300°C. The evolution of acid etching reactivity on the oil shale samples studied shows a competitiveness between material decomposition and thermal effect as a function of the temperature range considered. by ashing at low temperatures (LTH) in combustion of the organic material, the mineral matrix can be kept to a minimum **(A.Attaoui et al: 2022).**

We recall this study of the reactivity of acid attack on a few samples from three layers of the Tarfaya oil shale, in other words we are going to carry out a low-temperature ashing which consists of isothermal combustions from ambient to 300°C on these samples, more precisely on the organic part, with as little change as possible in the inorganic phases, followed by an acid attack (HCl) and a pH-meter (Tacussel) feedback, in order to understand the thermal effects and associated reaction mechanisms.

Introduction:

The formation of oil shale is the result of the same process that generated crude oil millions of years ago, mainly through the deposition of organic debris on the ocean, lake and seabed. Indeed, they formed over long periods of time under conditions of high temperature and pressure, which are also responsible for the formation of crude oil and natural gas; however, temperature and pressure were not as high in the case of oil shale. Oil shale is sometimes called "the rock that burns" because it contains enough oil to consume itself **(M.A.Ahmadi: 2017)**

1) Literature review:

The study of the oxidation of oil shale is important as it is the basis for the "in situ" mining process. Work by Mallon **(R.G. Mallon and W.C. Miller: 1975)**, Tyler **(A.L. Tyler: 1975)** and Huang **(Huang: 1977)** on the air thermal behavior of shale blocks where Mallon et al observed abnormally high velocities near the center of a shale block. The mechanism known so far is not satisfactory to explain this behavior. Indeed, the transport of oxygen by diffusion through the surface of the block towards the interior is weak enough to cause an abrupt rise in temperature. Similarly, Tinen et al **(S.S. Tihen et al:1968),** in calculating the thermal infusibility, showed the opposite phenomenon, i.e. the temperature rise at the surface is faster than in the interior. These authors explained this phenomenon by introducing pressure-sensitive capillaries inside the block to detect sudden combustions of hydrocarbons by the oxygen available inside the block. These combustions cause pressure pulses and of course temperature rises. The same phenomenon was observed by Sohns**(H.W. Sohns et al: 1972)**. Carbon, which is the essential element of hydrocarbons, was oxidized by water vapor **(T. Umehara et al: 1983)** to find out its kinetics at 973K, the result verifying the original Langmuir-Hinshelwood equation. The reaction rate is relatively high. Auxiliary reactions by adding Na_2SO_4 indicate that the non-volatile inorganic residues of DBS (sodium dodecyclobenzenesulphonate) have a catalytic effect. Measurements of oxygen adsorption by the samples show that thermal regeneration alone is 65% of the adsorption capacity, while thermal regeneration followed by steam completely restores the adsorption energy for carbon DBS. The effect of steam and air was also studied on coal **(C. Valenzuela et al: 1987)** which showed a low reactivity, this is explained by the effects of cracks when studying the textural and surface characteristics of coal. The latter, which are more reactive than its char, parent (coconut char) **(S.M.J.O. Hill et al: 1989)**, was the subject of a kinetic study which showed that it is not possible to use the exact formula of the rate equation **(G. Hakvoort et al: 1989)**. For this reason, a step-by-step method was proposed.

Oil shale can be burned directly to produce heat. This method has been used by mankind since ancient times, as oil shale burns directly without the need for treatment. Today, Estonia generates over 90% of its electricity by burning oil shale. Although overall thermal efficiency depends primarily on the amount of oil that can be harvested from a given shale, it can also depend on the particular technology applied to extract the oil **(O.I.Ogunsola: 2010).**

Any technology for producing shale oil generally comprises three general stages: mining, oil extraction and oil upgrading **(L.M.Petkovic: 2017);**

Shale oil can be obtained by extracting and processing shale rock. However, extracting oil from shale is not as straightforward as extracting oil from underground reservoirs, because shale oil occurs naturally as solid particles that cannot be pumped directly from the ground. Extracting shale oil is much more expensive than the conventional methods used to produce crude oil, because the oil shale must first be extracted, then heated to high temperatures (a process known as "Thermal Decomposition"). The oil shale is melted after heating, and the resulting fluids are separated and collected **(S.Zendehboudi et al: 2017).**

Shale oil is an unconventional petroleum, and its extraction is defined as the process of decomposing oil shale and transforming its kerogen into synthetic crude oil. The extraction process involves hydrogenation, pyrolysis and/or thermal dissolution. The efficiency of extraction processes is assessed by comparing their products with those of a Fischer analysis performed on the shale sample **(M.A.Ahmadi: 2017)**

Thermal decomposition or pyrolysis of the kerogen contained in shale to obtain shale oil is the most common method of utilizing oil shale, apart from the direct combustion of oil shale as a power plant fuel to generate electricity **(H. Gua et al: 2013)** In this process, oil shale is heated in the absence of oxygen until its kerogen breaks down into non-condensable flammable oil shale gas and condensable shale oil vapors. The composition of the oil shale can add value to the extraction process through the recovery of by-products, including ammonia, sulfur, aromatics, pitch, waxes and asphalt.

An energy source is required to heat the oil shale to pyrolysis temperature and to carry out the endothermic reactions involved in kerogen decomposition. Two strategies are employed to reduce, or even eliminate, the need for external thermal energy: oil shale gas and carbonization by-products generated by pyrolysis can be burned as an energy source, and the heat contained in hot spent oil shale and oil shale ash can be used to preheat raw oil shale. Figure 1 gives an overview of oil shale extraction techniques **(M.A.Ahmadi: 2017)**.

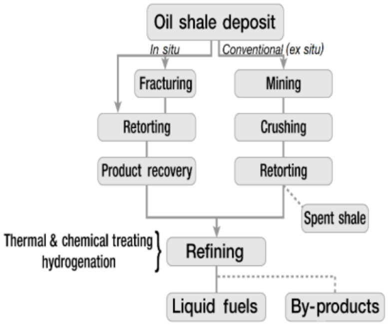

Fig 1: Overview of shale oil extraction techniques (Office of Naval Petroleum and Oil Shale Reserves, U.S. Department of Energy)

There are two types of oil shale processing techniques: ex situ (surface) and in situ (underground). Ex situ processing involves extracting the shale and thermally decomposing it in a dedicated above-ground facility. In situ processing, or conversion underground, involve applying heat to the shale in the geological formation **(H.Gua et al: 2013).**

The residual material after pyrolysis, known as spent shale or coal, consists of most inorganic materials as well as trapped heavy carbon compounds. In some cases, this coal can be used to generate additional heat for the retorting process. Numerous pyrolysis technologies have been patented, and several have been developed at different scales. They differ in the design of the retort and the way heat is applied to the oil shale particles **(Nord energy council: 2013)**

2) Tarfaya oil shale deposit, Morocco, Geological aspects:

- The structure of the deposit consists of a Cretaceous anticline truncated by erosion.
- The known part of the deposit is divided into two flanks on either side of the sabkha known as the Tazrha: the western flank area: over 200 km2, the eastern flank: over 140 km2.
- The deposit covers more than 4,500 km2.

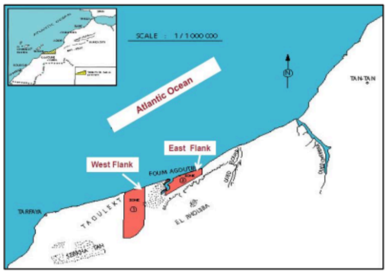

Fig: 2 Tarfaya deposit (ONHYM office national des hydrocarbures et des mines 2009)

-The layer with the highest organic carbon content is called the Z zone.
- The average thickness of the Z zone is 28 meters. Total resources of the Tarfaya field are estimated at 22 billion barrels of oil in place. Reserves on the western flank are estimated at 2.4 billion barrels **(M.Bencherifa: 2009).**

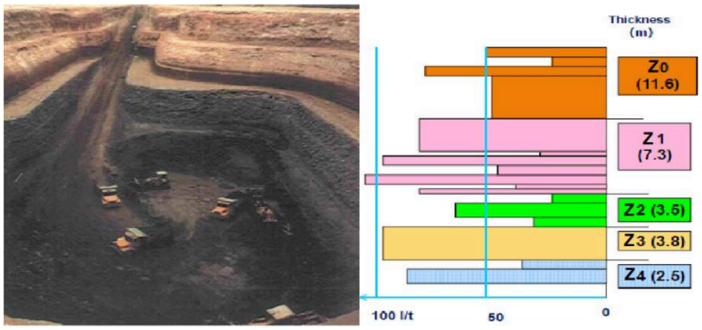

Fig 3: Tarfaya oil shale deposit (Histogram of oil shale units (ONHYM 2009))

3) Low temperature ashing.

Low temperature ashing (LTA) has been used to extract minerals from coal **(A.R.Shirazi et al: 1993)**. The problem is that the calcination temperature could not be measured, and therefore the calcination processes could not be controlled. The calcination temperature in the present study was measured by thermographs, which indicate the highest temperature reached during calcination. Some of the disadvantages of LTA were shown to be due to the calcination temperature being too high (150-300°C). It has also been confirmed that the mineral materials, especially sulphide, generally oxidize and some are lost as SO2. To solve these problems, a new method has been developed, very low temperature ashing (VLTA), to preserve and extract mineral matter from coal without loss or damage to the initial phases. It has been shown that to obtain an in-situ ash, the ashing temperature should not exceed 60-70°C, regardless of the coal quality. To lower the calcination temperature, oxygen was diluted with helium to reduce the reaction rate. To reach this very low temperature, the calcination atmosphere (oxygen/helium ratio) has to be adjusted for the coal type and sulphur composition **(A.R.Shirazi et al: 1993)**

For various applications, it is necessary to study the isolated state of minerals found in coal, e.g. clay, sulphide and carbonate minerals, in a group, which means that most of the coal must be removed. It should be checked that none of the minerals have been lost or changed in composition or phase during sample preparation. Low temperature ashing (LTA) has been used to study the occurrence of different sulphide minerals in coal. It is obviously not possible to use high-temperature calcination in the presence of high concentrations of oxygen, as the sulphide then oxidizes rapidly and there is a significant risk of losing the initial phases. LTA has been a relatively good solution for separating the mineral content from the carbon matrix in coal. However, LTA does not oxidize a coal sample completely, and there is no temperature measurement or control, either in surface area or in number of samples. The temperature of the calcination of a coal sample in this process (LTA) is a function of the sample calorific value, the concentration of oxygen in the calcination atmosphere (gas flow rate) and the intensity of ash reduction of the LTA. The calcination temperature in the LTA cannot be determined by conventional temperature measurement, for example with a thermocouple. As there are reasons to believe that the temperature could be quite high, especially above the surface of the sample, methods to measure the calcination temperature have been investigated **(A.R.Shirazi et al: 1993)**.

The aim of this study was to oxidize coal samples as much as possible, without any oxidation of the sulphide minerals. When a coal sample has been incinerated in this way, the analytical work with the minerals will be simplified and improved, especially for minerals that occur at low concentration. Very low temperature ashing (VLTA) is a process in which the temperature (calcination process) can be controlled for various samples of different calorific value of coal. In summary, this study shows that the type of coal still plays a major and necessary role for low temperature ashing. At 100% oxygen (LTA), high quality coals oxidize faster and more easily than low quality coals. This relationship is reversed in the process under consideration (VLTA), which means that the low-quality coal can be oxidized less quickly than the other. This can be explained by the surface factor.

There is no complete oxidation of the samples in either the LTA or VLTA processes. In VLTA the total oxidation is lower than in LTA, but some carbon atoms are left in the ash in both methods. Selective oxidation in VLTA starts at the beginning of the calcination process at a temperature of 60-70°C. This means that if there is no sulphur lost at the beginning of the ash reduction process, there will be any loss over a long period of ash reduction. However, after a sufficient time the selective oxidation ceases and the sulphur is depleted, so the oxidation process must be stopped **(A.R.Shirazi et al: 1993)**.

4) Experimental part:

4. 1: Materials and methods:

We carried out HCl acid etching on samples of the tree layers (Z0, Z1, Z2) of Tarfaya oil shale in the raw state and after isothermal combustion using a muffle furnace at different temperatures (T =150°C, T = 225°C and T = 300°C), in other words we performed incineration at low temperatures which serves to extract the minerals from the shale by oxidizing the organic matter with as little change as possible in the inorganic phases or mineral part.

Acid etching is often used for CO_2-releasing compounds in gasometry, as well as for the separation of organic and inorganic matter using a soxhlet. Samples burned at different temperatures are subjected to acid attack. Using a precision balance, 0.2 g of the isothermal samples are weighed and each sample is attacked with 50 cm3 of HCl (0.076N), filtered and 10 cm3 of the filtrate is dosed with NaOH (0.033N) to determine the quantity consumed during the attack (back-dosing). The dosing operation is monitored using a Tacussel pH meter with an Orion electrode.

Fig 4: Precision balance

Fig 5; Muffle furnace

5) Relationship between organic matter and geological period:

The Tarfaya oil shale deposit is of considerable importance and ranks among the marine deposits. The organic matter (kerogen) in this type of shale is quantitatively variable in layers ranging from Z0 to Z4 (deep layer). Dynamic thermogravimetry was used to quantify overall organic matter and UV spectroscopy was used to monitor the amount of high molecular weight organic matter (heavy organic matter).

The sedimentation of organic matter in the Tarfaya deposit, generally formed by the debris of algae and zooplankton as well as other forms of marine life, depended on the climate of the geological eras. The geological eras in question are the secondary and tertiary eras, and organic matter is logically abundant when the climate is warm **(A.Attaoui et al: 2022).**

By combining the two techniques, thermogravimetry and UV analysis, Attaoui et al. found that for the first three layers Z0, Z1 and Z2, the order of the two techniques is probably identical.

Table 1: Concentration of total organic matter and high molecular weight organic matter in the different Tarfaya layers

Oil shale layer	Thermogravimetry, organic matter %	UV, high molecular weight organic matter concentration (chloroform)
Z0	6.5%	0,153
Z1	12.13%	0,162
Z2	7.4%	0,1205

Organic matter (kerogen) is formed by sedimentation, which varies according to climate: a warm climate results in abundant sedimentation, and vice versa. The quality of this organic matter was determined by thermogravimetry. Attaoui et al, were able to adapt the stage of formation according to the period considered, depending on the quantity of this organic matter, as shown in Table 7 below **(A.Attaoui et al: 2022)**

Table 2: Stage of formation of the layers studied in the Tarfaya deposit.

Qil shale layers	Organic matter %	Geological pereiod
Z0	6.5%	Early Tertiary (cold climate)
Z1	12.13%	Maestrichtien (secondary) (warm climat)
Z2	7.4%	Senonaen (secondary) (cold climat)

6) Acid etching of Tarfaya oil shale samples (Z0, Z1 and Z2) having combustion at different temperatures:

The assays used to study Tarfaya oil shale samples (Z0, Z1 and Z2) having undergone at different temperatures are shown in the following figures.

a) Layer Z0

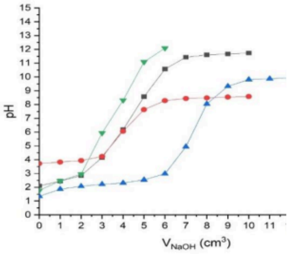

Fig 6: Dosing curve for the filtrate after acid etching with NaOH for the Z0 layer, which is raw and has undergone combustions at different temperatures:
• brut, • at 150 °C, ▲ at 225 °C and ▼ at 300 °C

b) Layer Z1

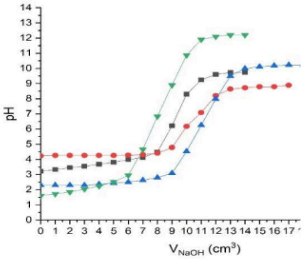

Fig 7 : Dosing curve for the filtrate after acid etching with NaOH for the Z1 layer, which is raw and has undergone combustions at different temperatures:
• brut, • at 150 °C, ▲ at 225 °C and ▼ at 300 °C

c) Layer Z2

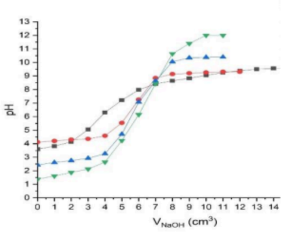

Fig 8: Dosing curve for the filtrate after acid etching with NaOH for the Z2 layer, which is raw and has undergone combustions at different temperatures:
• brut, • at 150 °C, ▲ at 225 °C and ▼ at 300 °C

The following curve (Fig 9) represents the determination of the filtrate after acid (HCl) attack by NaOH of the Z0, Z1 and Z2 layers in the crude state and at temperature ambient.

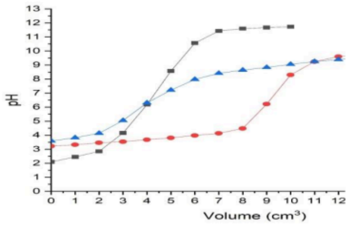

Fig 9: Dosing curve of filtrate with NaOH after acid etching (HCl) of oil shale layers Z1, Z2 and Z3 in the raw state at ambient temperature: • Z0, • Z1 ▾Z2

The following curve (Fig:10) shows the determination of the filtrate after acid (HCl) attack by NaOH of the Z0, Z1 and Z2 layers combusted at 150°C.

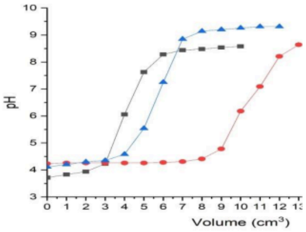

Fig 10: Dosing curve of the filtrate with NaOH after acid etching (HCl) of oil shale layers Z1, Z2 and Z3 after combustion at a temperature of 150°C: • Z0, • Z1 ▾Z2

Similarly, the following curve (Fig 11) shows the determination of the filtrate after acid (HCl) attack by NaOH of the Z0, Z1 and Z2 layers combusted at 225°C.

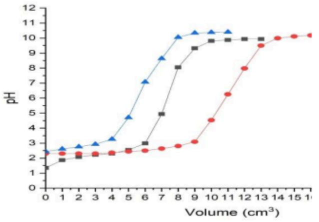

Fig 11: Dosing curve of the filtrate with NaOH after acid etching (HCl) of oil shale layers Z1, Z2 and Z3 after combustion at a temperature of 225°C: • Z0, • Z1 ▼Z2

Finally, the following curve (Fig12) shows the determination of the filtrate after acid (HCl) attack by NaOH of the Z0, Z1 and Z2 layers after combustion at 300°C.

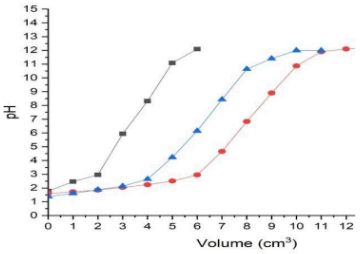

Fig 12: Dosing curve of the filtrate with NaOH after acid etching (HCl) of oil shale layers Z1, Z2 and Z3 after combustion at a temperature of 300°C: • Z0, • Z1 ▼z2

4) Results:

The following table 3 shows the dosing volume of 10 ml of filtrate with NaOH, the pH of the neutralization and the volume of acid used to etch the layer burned at different temperatures:

Table 3: NaOH dosing volumes and attack volumes for different oil shale samples pre-combusted at different temperatures.

Samples	Température (°C)	Dosage volume (ml)	PH	Volume of acid to attack the layer of oil shale considered according to its combustion treatment at different temperatures (ml)
HCl seul	--	23.03	7.75	→
Z_0	Ambient	4.25	6.97	18.78
	150°	4.05	6.2	18.98
	225°	7.4	6.4	15.63
	300°	2.8	6.1	20.23
Z_1	Ambient	9.25	6.8	13.78
	150°	10.3	6.45	12.73
	225°	11	6.3	12.03
	300°	7.85	6.7	15.18
Z_2	Ambient	3.75	5.9	19.28
	150°	5.8	6.8	17.23
	225°	5.8	6.6	17.23
	300°	6.45	6.7	16.58

5) Interpretations:

Three factors influence the reactivity of this acid attack:
- Organic matter composition.
- Mineral composition.
- The sedimentation conditions of the layer in question.

The predominant factor according to the four experiments carried out is the burial conditions to be mentioned, we observe the low quantity of acid that was consumed in the case of sample Z1, yet Z1 was produced in a hot climate **(A.Attaoui et al: 2022)**

The action of temperature for each layer is remarkable, it also follows decomposition stages.
The effect of temperature on each layer is remarkable, as it also follows decomposition stages.

For sample Z0, with its low concentration of organic matter, the thermal effect is not influential. In fact, between ambient and 150°C, we observe the coexistence of decomposition and the thermal effect; in fact, both phenomena occur simultaneously, but in this case, decomposition prevails over the thermal effect. Between 150°C and 225°C, the thermal effect predominates. Whereas for sample Z1, which contains a high concentration of organic matter, we observe a competitiveness between the two phenomena (decomposition and thermal effect), in fact between room temperature and 150°C we observe a predominance of the thermal effect. Between 150°C and 225°C, the two phenomena coexist and are reversed: decomposition deactivates and the thermal effect activates, and the two compensate for each other to produce a superposition of the two curves, but in reality, the thermal effect prevails over decomposition.

The two layers Z0 and Z1 show significant material decomposition between 225°C and 300°C.

For Z2 between ambient temperature and 150°C, the thermal effect predominates. Between 150°C and 225°C, the two phenomena coexist and offset each other. We therefore observe low acid consumption at 300°C, which leads us to say that decomposition is low at this temperature and the thermal effect predominates.

Conclusion:

Oil shales are very different from one another, varying in their chemical composition, mineral content, age, type of kerogen and the way they have been sedimented.

As low-temperature ashing is the main reaction in this study, followed by acid (HCl) attack and pH-meter feedback, this is a quantitative method for devising the quantity that has been consumed during this attack. This study investigates the reactivity of acid etching on samples from 3 layers of Tarfaya oil shale (Z0, Z1, Z2) which, prior to this etching, underwent isothermal combustions from ambient to 300°C. We found that 3 factors influence the reactivity of this attack: organic matter composition, mineral matter composition and burial conditions, the latter being the predominant factor for the different layers studied.

The evolution of acid attack reactivity on the oil shale samples studied was carried out in three distinct ways, depending on the temperature range considered, and different results emerged, summarized in the following table:

Oil shales are very different from one another, varying in their chemical composition, mineral content, age, type of kerogen and the way in which they have been sedimented.

As low-temperature ashing is the main reaction in this study, followed by acid (HCl) attack and pH-meter feedback, this is a quantitative method for designing the quantity that was consumed during this attack. This study investigates the reactivity of acid etching on samples from 3 layers of Tarfaya oil shale (Z0, Z1, Z2) which, prior to this etching, underwent isothermal combustions from ambient to 300°C. We found that 3 factors influence the reactivity of this attack: organic matter composition, mineral matter composition and burial conditions, the latter being the predominant factor for the different layers studied.

The evolution of acid attack reactivity on the oil shale samples studied was carried out in three distinct ways, depending on the temperature range considered. Different results emerged, which are summarized in the following table:

Sample	Temperature range	Reaction evolution
Z_0	Ambient – 150°C	Coexistence of decomposition and thermal effect with predominance of material decomposition.
	150°C – 225°C	Thermal effect.
	225°C – 300°C	Material decomposition.
Z_1	Ambient – 150°C	Thermal effect.
	150°C – 225°C	Coexistence of decomposition and thermal effect with predominance of thermal activity.
	225°C – 300°C	Material decomposition.
Z_2	Ambient – 150°C	Thermal effect
	150°C – 225°C	Coexistence of decomposition and thermal effect
	225°C – 300°C	Thermal effect.

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