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RESEARCH ARTICLE

HYDROGENATION OF SILICON, ALUMINUM AND CHLORINE AS CONSTITUENTS OF TARFAYA OIL SHALE (MOROCCO) AT 550°C AND 750°C

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Abstract

The formation of hydrogen chloride as well as silicon hydrides and aluminum hydrides called silanes and alanes respectively are formed when oil shale are thermally treated with hydrogen. These elements are part of the constituents of this oil shale. This work was carried out by a thermobalance (Red Croft) in the dynamic regime for thermal hydrogenation at 550°C and 750°C. The quantitative identification of the elements was done by X-ray diffraction using a dispersive method, a technique that is coupled to the scanning microscope. This work being the continuation of the one already carried out at a temperature of 550°C (A. Attaoui and M. Hafid: 2022), we have extended this to higher temperatures where we have decompositions in addition to kerogen as well as mineral matter. Studies comparing the thermal decomposition of oil shale and kerogen have shown that the mineral matter in oil shale provides surfaces capable of cracking high molecular weight products by thermal or catalytic mechanisms (A.K. Burnham, A. K and J.A.Happe: 1984). These studies indicate that reactions involving the cleavage of carbon-carbon bonds and carbon-hydrogen bonds on mineral surfaces (R.A.Regtop et al: 1985). These authors demonstrated that the carbonized residue in spent shale can increase the magnitude of secondary reactions.

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Introduction:-

Studies on oil shale processing that have examined the catalytic effects of minerals have reported on reactions that result in heteroatom displacement. The motivation for investigating the possibility of removing sulphur, nitrogen and oxygen during oil shale decomposition includes firstly the production of oil acceptable for conventional oil refining operations and secondly the ability to store the oil without insoluble sediments of heteroatom compounds. This research project investigates the effects of eastern and western US shale minerals on thiophene hydrodesulphurization reactions (HDS). The first part of this study shows that shale minerals are capable of desulphureting thiophene to hydrogen sulphide and hydrocarbons and that they promote desulphurization, isomerization and polymerization reactions under autoclave conditions. Current work focuses on understanding the catalytic effect of mineral and burnt waste shale for the removal of sulphur from thiophene. In this context we are monitoring the effects of hydrogen on the elements that are part of the mineral matter of oil shale such as silicon, aluminum and chlorine.

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1/ Bibliographical study:

We are going to look at the mineral compounds (silicon, aluminium and chlorine) and their action with respect to hydrogen at the high temperatures of 550°C and 750°C. These compounds have catalytic effects apart from the hydrides they form in the presence of hydrogen at high temperatures. The effects of eastern and western shale minerals on the hydrodesulphurization reactions (HDS) of thiophene. The first part of this study shows that shale minerals are capable of de-sulphureting thiophene to hydrogen sulphide and hydrocarbons and that they promote desulphurization, isomerization and polymerization reactions under autoclave conditions (A. Shamsi et al: 1987). Current work is focused on understanding the catalytic effect of mineral and burnt waste shale for the removal of sulphur from thiophene. Both eastern and western LTA shale were active in HDS and hydro-denitrogenation (HDN) reactions. This study is a continuation of previous work and aims to identify the mineral(s) responsible for the catalytic behavior and to determine how time and temperature influence the selectivity and conversion of the product in order to improve sulphur removal and minimize side reactions that may contribute to the cracking of higher hydrocarbons into gas. The HDS reaction of thiophene on Colorado LTA shale and shale minerals, such as dolomite, illite, calcite and siderite, was studied. The elemental compositions of high ash shale (HAVE) of these minerals were determined using X-ray fluorescence spectrometry. The elemental analyses are reported in Table 1 as oxide in weight percent.

Table 1:- X-ray fluorescence analysis of HTA of shale minerals (as oxides).

	Siderite	Illite	Colorado dolomite	calcite	MIXM	Colorado Mahogany LTA
CaO	0,70	0,60	34,22	51,41	26,00	26,90
K ₂ O		5,10			1,40	1,80
P ₂ O ₅	0,21	0,31	0,06	0,20	0,72	0,88
SiO ₂	8,80	52,80	6,01		48,60	42,50
Al ₂ O ₃	7,70	22,00			11,60	12,70
Fe ₂ O ₃	60,40	8,90	0,69	0,05	2,50	1,80
TiO ₂		0,71	0,05	0,03	0,14	0,21
MgO	5,50	1,70	1,54	0,54	3,20	8,60
S		0,69	0,08	0,06	0,24	0,05
MnO	1,25	0,02	0,14	0,01	0,08	0,04
ZnO	0,03	0,01	0,01		0,02	0,02
SrO		0,04		0,12	0,07	0,11

A blank reactor test was carried out to investigate the effects of the reactor walls (Inconel) and temperatures on the HDS reactions. A comparison of the results of a blank test and the shale test at LTA indicated that the shale minerals contained active surfaces that favored hydrogen hydrolysis, isomerization and polymerization reactions. The thermal decomposition of thiophene at temperatures below 550°C in a blank reactor was negligible (2% conversion). In addition, hydrogen as a carrier gas appears to be very important for both hydrogenolysis and for maintaining the activity of the shale minerals. The catalytic activity of the minerals pretreated under helium was very low (about 2% conversions) compared to those pretreated under hydrogen. Furthermore, no significant catalytic reaction was observed when the hydrogen carrier gas was replaced by helium. Each of the shale minerals, a mixture of minerals (MIXM), Colorado LTA shale and Colorado spent shale showed HDS activity under the reaction conditions (450-600°C). The time dependence of thiophene conversion for these minerals is shown in Figure 1.

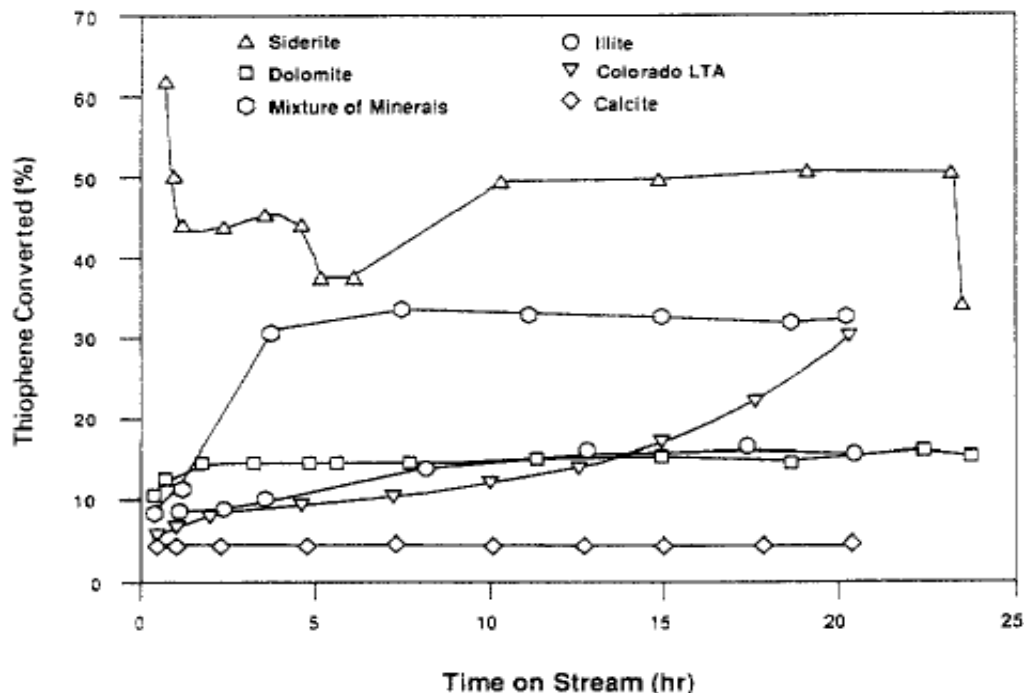


Fig 1:- Time dependence of thiophene conversion on shale minerals at 500°C, 3 atm, $W / F = 2.0 \text{ g s} / \text{cm}^3$, pulse size = 2.2 μmol .

The burning of oils in recyclable solids is the most important cause of yield loss during the degradation process (**P. Udaja et al: 1990**) of oil shale. The kinetics of coke formation is measured by the kinetics of oils and ash (Australia). The reactivity of these ashes decreases with increasing temperatures above that of their formation.

Preparation of the samples in a steam atmosphere reduces their reactivity. It should be noted that charcoal pyrolysis under hydrogen has a rapid reaction compared to that pyrolysis under H_2O and for the same heating rate (**T. Sugawara et al: 1990**). The carbenes and asphaltenes are separated by thermal conversion of oil, maltene in the presence of hydrogen (**J. Blazek and G. Sebor: 1993**) according to the mechanism:

oils \rightarrow resins \rightarrow asphaltene \rightarrow carbenes \rightarrow carboids

The primary asphaltenes formed are due to cracking and hydrogenation. For oil shale abrasion, Laurence Livermore National Laboratory used two empirical model processes derived from the HRS process (**D.F. Aldis: 1992**). The oils produced from the two varieties of Australian oil shale (**J. Korth et al: 1988**) are similar; however it was found that the mineral matrix is more active when the secondary pyrolytic reactions are also active. The Allakhverdov-Nirsha method (**V.Y. Zakharov and I.d.Shchuchkim: 1986**) is used to determine the kinetic parameters of the evolution of C_nH_m , CH_4 , CO and H_2 during degradation. The kinetic parameters in non-isothermal conditions are determined for different gases; the experimental results are in agreement with those calculated.

Generally hydrogen adsorption is done and measured by proton magnetic resonance (**X. Wu et al: 1989**), for example adsorption by quartz reveals two distinct states. The PMR and the volumetric method are in good agreement when the hydrogen adsorption is irreversible.

The decomposition of carbonates, sulphates, pyrite and minerals in soils and rocks can be followed by TG, DTG and DTA (**F. Paulik et al: 1984**), as well as for kaolinite, it takes place at high temperatures (**C.H. Horte et al: 1988**) and depends on the conditions applied. Identification is done by X-ray diffraction. TG and DTA can also quickly determine the characteristics of compounds and residues. It should also be noted that the thermal decomposition of ammonium feldspar in the Green River oil shale has been studied to determine its contribution to the total nitrogen in the shale (**M.S. Oh et al 1993**); ammonium in ammonium feldspar evolves between 400 and 900°C as N_2 and H_2 .

2/Experimental

2.1/Thermogravimetric analysis:

Thermogravimetric analysis (TGA) purpose to characterize materials by directly measuring mass loss as a function of temperature and/or time. It measures the change in mass of a sample when it is subjected to a temperature program, under a controlled atmosphere. This change can be a mass loss (vapor emission) or a gain (gas fixation, oxidation). Experiments can be performed in an oxidizing or inert atmosphere.

The thermo-microbalance is the Red-croft which is a setaram used for thermogravimetric (TG) measurements related to degradation in dynamic regime. It has a resolution of 0.1 μg . In our conditions, it operates between room temperature and 750°C, with different heating rate: 21°C/min, 15°C/min and 09°C/min.

This top-loading thermobalance is easy and safe to use, having no hanging wires or exposed fragile parts. The following picture shows a thermobalance from the Setaram series called Red-croft, which is a flail thermobalance with a compensation system based on a photoelectric source to keep the sample at the same position in the furnace, thus avoiding the temperature gradient due to sample movement in the furnace.

2.2/ Morphology layers (Z₀) before and after hydrotreating (550°C and 750°C)

The photos shown below have been made by the scanning electron microscope (SEM) in the raw state and after having carried out the hydrogen treatment, i.e. hydrolysis in a dynamic regime (21°C/min) under a pressure of one atmosphere.

Scanning electron microscopy (SEM) is a technique based on the analysis of electron-matter interactions. The surface of the sample is subjected to a bombardment of electrons. Some radiation (backscattered electrons, secondary electrons and X-rays) is emitted as a result of the multiple collisions between the electrons that scan the surface and the atoms of the material under test. This radiation is analyzed by different detectors to build an image of the surface and to observe the presence of elements in the analyzed area.

Both samples were examined under the scanning electron microscope. Figures 2, 3 and 4 show the photos of the Z₀ layer before and after hydrotreating(550°C and 750°C)

The experimental results of dispersive X-ray identification are:

2.3/ Ambient temperature range at 550°C

The results of the dispersive X-ray spectra for the five layers of the Tarfaya deposit were carried out in a previous work (M. Hafid and A. Attaoui: 2022). These results lead to an elemental analysis of different elements constituting the shale compounds such as: sulphur, calcium, aluminum, potassium, chlorine, magnesium and iron.

These analyses were carried out for both the raw and hydrotreated (H₂) material up to 550°C (see following figures)

2.4/ Ambient temperature range at 750°C

X-ray spectra for this temperature. The following XRD spectra represent the different layers of the Tarfaya deposit, namely Z₀, Z₁, Z₂, Z₃ and Z₄, raw, hydrogen (H₂) treated (hydrogen flux = 20 cm³/mm and PH₂ = 1 atm) up to a temperature of 550°C and hydrogen treated under the same conditions up to a temperature of 750°C.



Fig 2: Raw Z0 layer

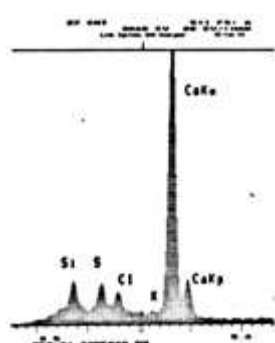


Fig 3: Raw Z1 layer

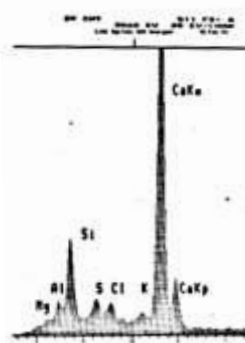


Fig 4: Raw Z2 layer

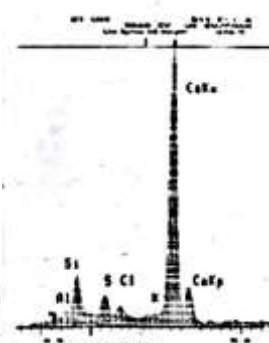


Fig 5: Raw Z3 layer

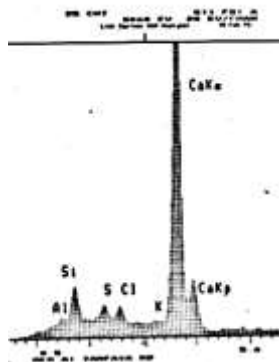


Fig 6: Raw Z4 layer

For the hydrogen treated samples, their XRD analysis can be found in the following spectra at 550°C in temperature

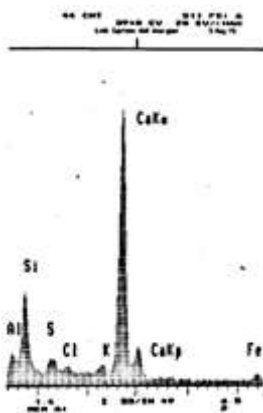


Fig 7: Z0 hydrotreated at 550°C

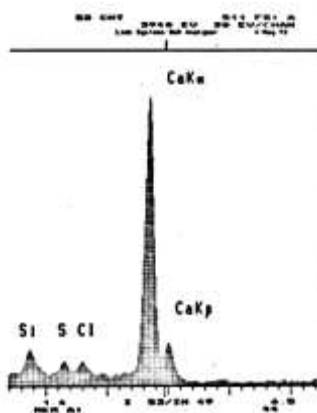


Fig 8: Z1 hydrotreated at 550°C

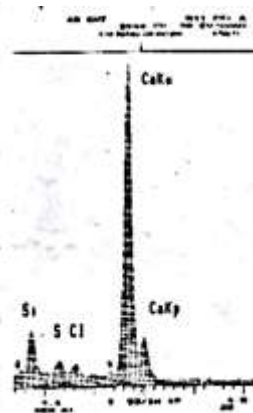


Fig 9: Z2 hydrotreated

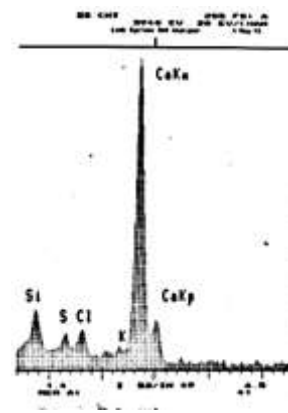


Fig 10: Z3 hydrotreated

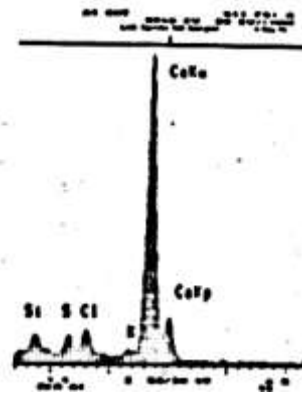


Fig 11:- Z4 hydrotreated at 550°C

Similarly, the spectra were carried out for hydrogen treatments at 750°C

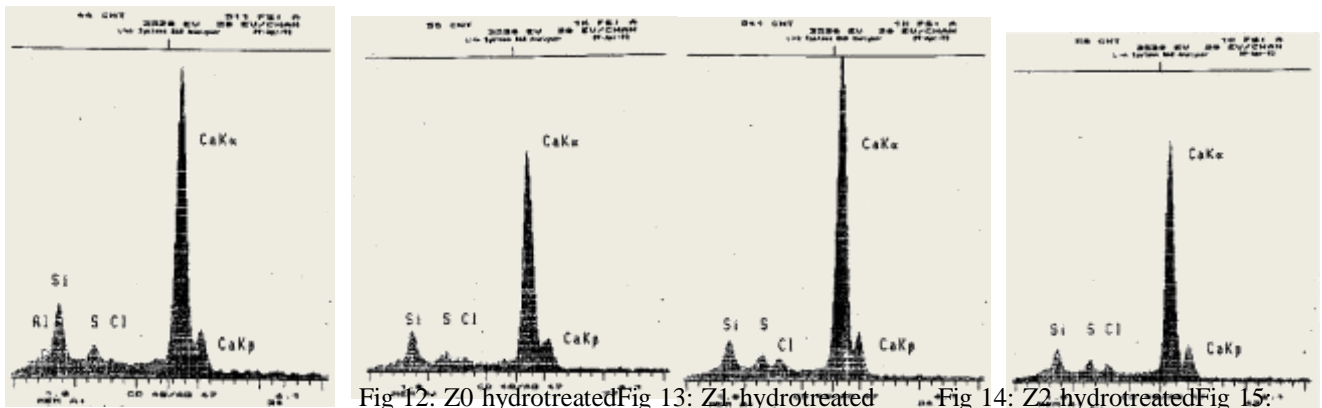


Fig 12: Z3 hydrotreated at 750°C

Fig 13: Z0 hydrotreated at 750°C

Fig 14: Z2 hydrotreated at 750°C

Fig 15: Z1 hydrotreated at 750°C

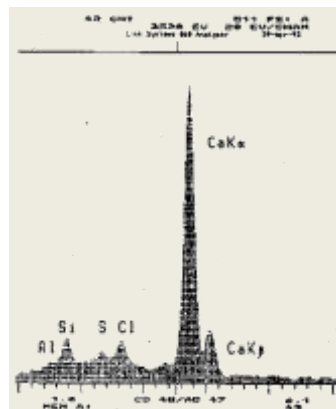


Fig 16: Z4 hydrotreated at 750°C

3/ Table of Results:-

The following table shows the different measurements of the peaks of the previous spectra as well as the percentages of the elements constituting the oil shale. These values are attributed to the temperatures, firstly ambient, then hydrotreating at 550°C and finally hydrotreating at 750°C.

These spectra have led to the results shown in the following table 1. It should be remembered that the spectra with which the measurements and calculations were made have A4 shapes, the spectra in the text are reduced shapes. The same spectra were used for hydrodesulphurization in a previous works(M.Hafid and A.Attaoui: 2022, M.Nadif and A.Attaoui: 2022)

Table 2:- Percentage of chemical elements in the Tarfaya oil shale.at different states.

Composition	Type of oil shale	Z ₀ (cm)	Z ₁ (cm)	Z ₂ (cm)	Z ₃ (cm)	Z ₄ (cm)	%Z ₀	%Z ₁	%Z ₂	%Z ₃	%Z ₄
Magnesium	Raw	1,15	0,60	0,80	0,70	0,60	2,67	1,80	2,12	1,93	2,01
	Traited by H ₂ at 550°C	1,15	0,60	0,80	0,70	0,60	3,71	1,99	2,98	2,06	1,99
	Traited by H ₂ at 750°C	0,85	0,45	0,40	0,35	0,60	3,15	2,46	1,57	1,78	2,39
Aluminum	Raw	2,35	1,20	1,40	1,15	0,80	5,46	3,69	3,72	3,18	2,67
	Traited by H ₂ at 550°C	1,90	0,95	0,80	1,35	0,80	6,13	3,15	2,98	3,97	2,66
	Traited by H ₂ at 750°C	1,45	0,60	0,65	0,55	1,05	5,38	3,29	2,56	2,81	4,19
Silicon	Raw	6,50	3,60	3,40	3,10	1,95	15,11	10,97	9,57	8,58	6,52
	Traited by H ₂ at 550°C	5,4	3,25	2,10	3,65	1,80	17,42	10,78	7,83	10,75	5,99
	Traited by H ₂ at 750°C	3,75	2,10	2,05	1,75	2,20	13,91	11,51	8,07	8,93	8,78
Sulphur	Raw	2,40	2,15	2,15	2,90	2,10	5,58	6,55	5,72	8,03	7,02
	Traited by H ₂ at 550°C	1,60	1,50	1,45	2,20	1,75	5,16	4,97	5,41	6,48	5,82
	Traited by H ₂ at 750°C	1,65	1,05	1,35	1,20	1,50	6,12	5,75	5,31	6,12	5,99
chlorine	Raw	2,20	1,50	2,05	2,30	2,30	5,11	4,57	5,45	6,37	7,69
	Traited by H ₂ at 550°C	1,10	1,30	1,45	2,40	2,10	3,54	4,31	5,41	7,07	6,98
	Traited by H ₂ at 750°C	1,00	0,75	1,10	0,90	2,10	3,71	4,11	4,33	4,59	8,38
Calcium α	Raw	22,30	19,60	22,0	21,20	18,50	51,86	59,75	60,10	58,72	61,87
	Traited by H ₂ at 550°C	15,80	18,60	16,0	18,60	18,80	50,97	61,69	61,94	54,78	62,56
	Traited by H ₂ at 750°C	15,80	11,60	17,0	12,90	15,00	58,63	63,56	68,50	65,82	59,88
Calcium β	Raw	3,85	2,80	3,75	3,20	2,50	8,90	8,53	9,97	8,86	8,36
	Traited by H ₂ at 550°C	2,20	2,80	2,45	3,00	2,85	7,09	9,28	9,14	8,83	9,48
	Traited by H ₂ at 750°C	2,45	1,70	2,45	1,95	2,60	9,09	9,32	9,65	9,95	10,38

We discuss the results for the three elements concerned, which are silicon, aluminum and chlorine

4 / Element silicon and the element aluminum

This time we mention aluminum and silicon, which are the main elements of clays and quartz in the case of silicon,

- Silicon.

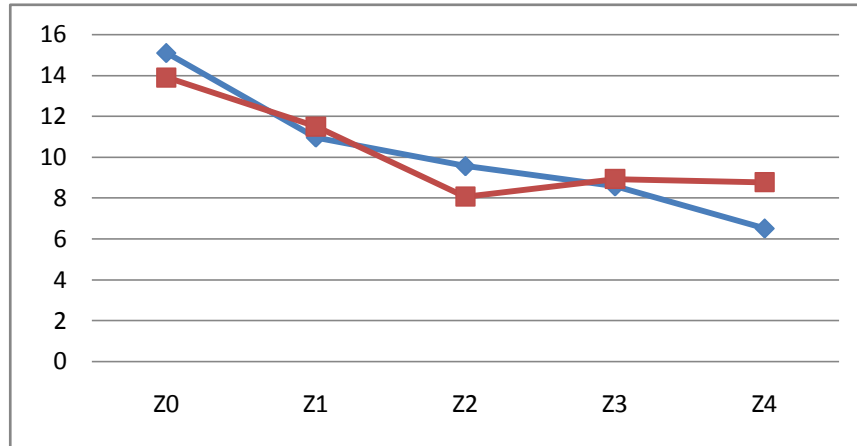


Fig 17: Percentage of raw silicon ■■■■ and after hydrotreated at par à 750 °C ■■■■

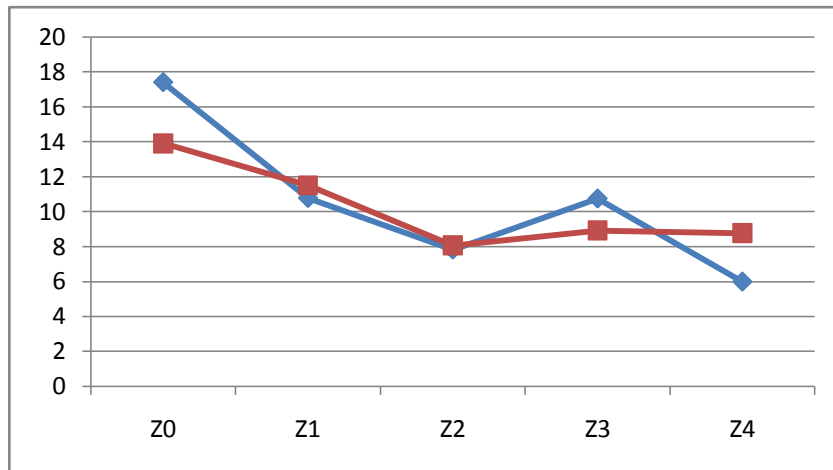


Fig 18: Siliconhydrotreated at 550°C ■■■■ andhydrotreated at 750°C ■■■■

The Z0 layer is the most concentrated in silicon and is the surface layer, then we observe a decrease in silicon with depth, showing that the clays are on the surface and that silicon has the same variation in the Z0, Z1, Z2, Z3 layers, unlike the Z4 layer where we have an agglomeration of this element with the heavy organic matter in this layer (A. Malal, D. Lahmadi and A. Attaoui : 2022). It should be noted that there is some thermal activation in the Z0 and Z3 layers when the temperature varies from 550°C to 750°C.

- Aluminum

Aluminum varies in the same way by decreasing with depth, we observe reaction activation in hydrolysis at 550°C for the Z1, Z2 and Z3 layers and we note the same behavior as previously for silicon.

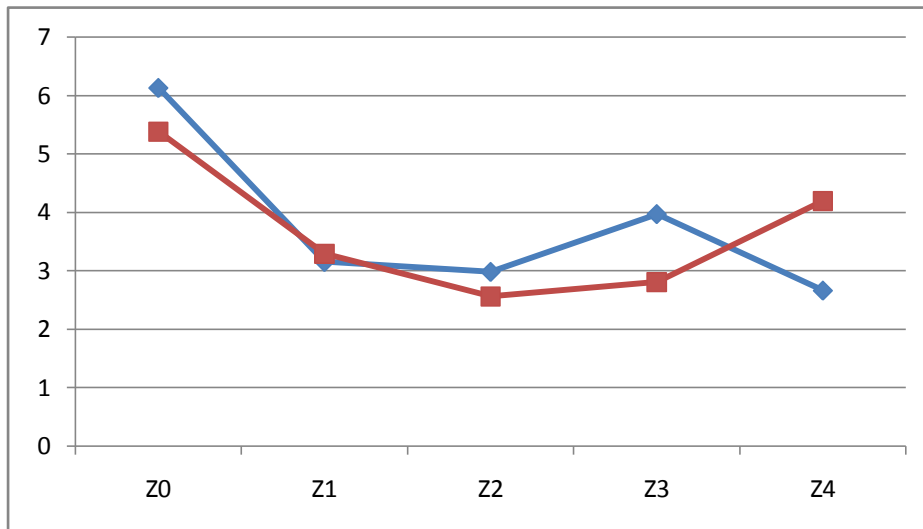


Fig 19: Percentage of raw aluminum ■■■ and after hydrotreated at par à 750 °C ■■■

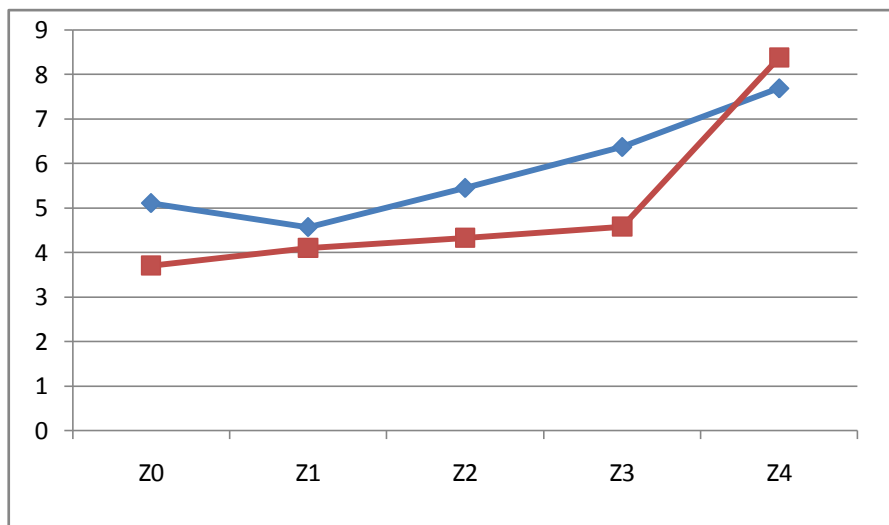


Fig 20: Aluminum hydrotreated at 550°C ■■■ and hydrotreated at 750 °C ■■■

The same thermal reactivity is felt between 550°C and 750°C.

We count a concentration three times higher for silicon than for aluminum, which shows us that silicon is not only part of clays, but is doubly concentrated as a constituent of quartz.

5/ Chlorine

The same evolution is noticed for the five layers considered, namely an increase in concentration with depth, this is consequent of the infiltration of the chlorine solubilized by the rainwater which fell on the site several years before. The location of the curves before and after treatment shows that the thermal effect causes the chlorine level to decrease with increasing temperature, by comparing the curve containing chlorine at a temperature of 550°C with that at 750°C. Thus the temperature range for sensitization of the chlorine-hydrogen reaction is between 550°C and 750°C.

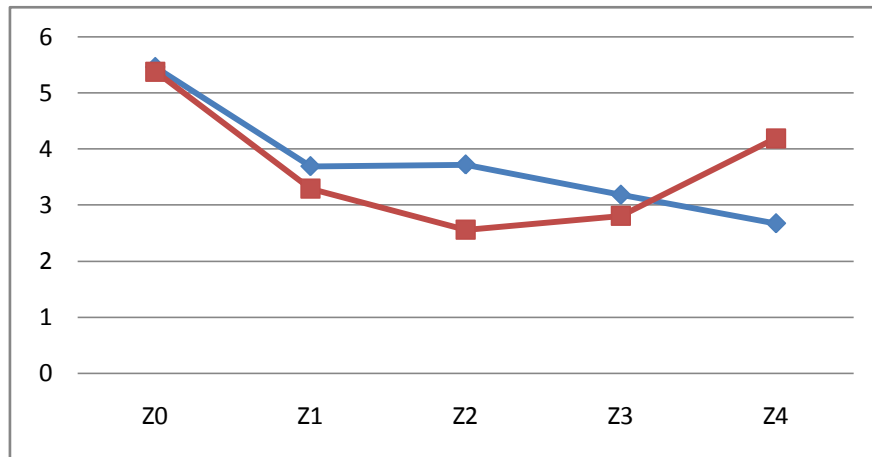


Fig 21: Percentage of raw chlorine ■■■ and after hydrotreated at 750 °C ■■■

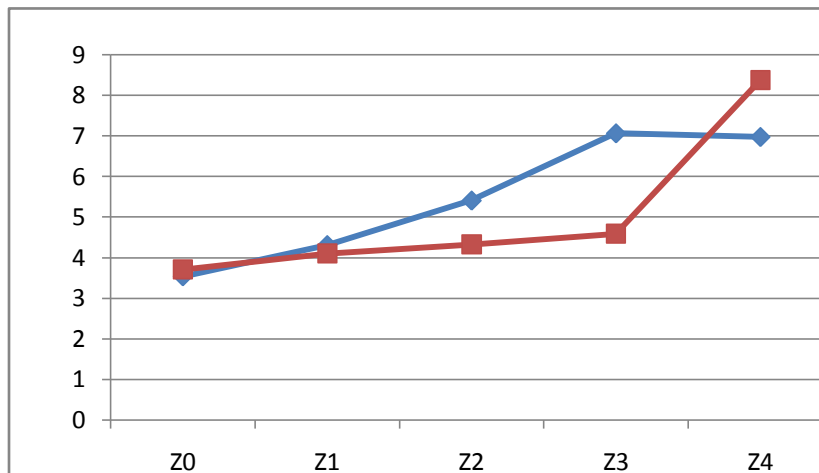


Fig 22: Chlorine hydrotreated at 550°C ■■■ and hydrotreated at 750 °C ■■■

6/ Morphology of Z0 before and after hydrotreatment.

The photos below were taken with a scanning electron microscope (SEM) for the Z0 layer in its raw state and after treatment with hydrogen, i.e. hydrolysis in a dynamic regime (21°C/min) under a pressure of one atmosphere and at temperatures of 550°C and 750°C

Scanning electron microscopy (SEM) is a technique based on the analysis of electron-matter interactions. The surface of the sample is subjected to electron bombardment. Radiation (backscattered electrons, secondary electrons and X-rays) is emitted as a result of the multiple collisions between the electrons that scan the surface and the atoms of the material under test. This radiation is analyzed by different detectors in order to construct an image of the surface and to observe the presence of elements in the analyzed area.

Both samples were examined by scanning electron microscope. Figures 23, 24 and 25 show pictures of the Z0 layer before and after hydrotreating at 550°C and 750°C.

We note the volume contraction for the hydrotreated Z0 sample which becomes increasingly apparent as the temperature increases from 550°C to 750°C.

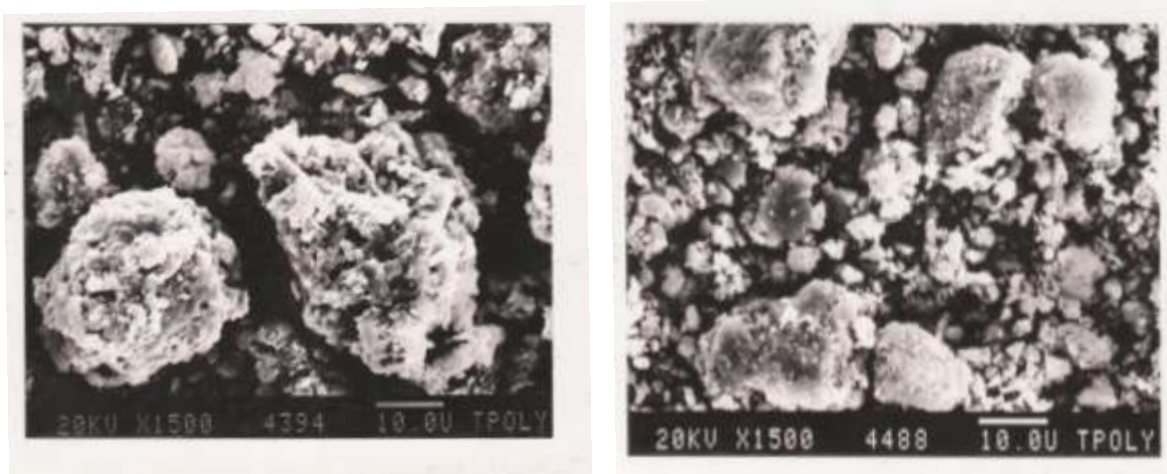


Fig 23:- Raw Z_0 layer. **Fig 24:-** Z_0 layer after hydrotreating at 550°C.

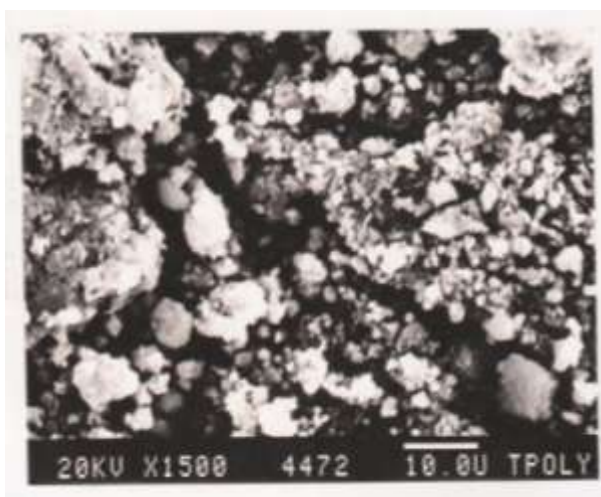


Fig 25:- Z_0 layer after hydrotreating at 750°C.

Conclusion:-

The hydrogenation of the metal aluminum, the metalloid silicon and chlorine as a non-metal was the subject of this study. These elements are constituents of oil shale, the deposit chosen being that of Tarfaya (Morocco). Five layers of this deposit were thermally treated under hydrogen in a Setaram-type balance (Red-Croft) in a dynamic regime (21°C/min) at a hydrogen pressure of one atmosphere. The progressive temperature varied from ambient to 750°C. The deposit was divided into five layers, Z0, Z1, Z2, Z3 and Z4. Reserves in the Z layer descend to a depth of 125m over its entire extent with a total of 795 million m³. The overall estimated reserves of this deposit are around 80 billion tons of rock at most, containing 22 billion barrels of oil at most at an average grade of 60 liters/ton. Some layers can contain up to 100 L/t. In this work, we are interested in using dispersive X-ray diffraction to analyse the different layers of the Tarfaya deposit. The elements monitored this time are silicon, aluminum and chlorine, their reaction with hydrogen leads to the formation of silanes, alanes and hydrogen chlorides respectively.

Several results were revealed: The Z0 layer is the most concentrated in silicon and it is the surface layer, then we observe a decrease in silicon with depth, showing that the clays are on the surface and that silicon has the same variation in the Z0, Z1, Z2, Z3 layers, unlike the Z4 layer where we have an agglomeration of this element with the heavy organic matter in this layer. It is worth noting that there is some thermal activation in the Z0 and Z3 layers when the temperature varies from 550°C to 750°C.

Aluminum also varies with decreasing depth, we observe hydrolysis reaction activation at 550°C for the Z1, Z2 and Z3 layers, we note the same behavior as previously for silicon. The same thermal reactivity is felt between 550°C and 750°C.

We note a threefold higher concentration of silicon compared to aluminum, which shows us that silicon is not only part of the clays, but is also doubly concentrated as a constituent of quartz.

The same evolution can be seen for the five layers considered, i.e. an increase in concentration with depth, which is due to the infiltration of the chlorine solubilized by the rainwater that fell on the site several years before. The location of the curves before and after treatment shows that the thermal effect causes the chlorine level to decrease as the temperature increases, by comparing the curve containing chlorine at a temperature of 550°C with that at 750°C. Thus the temperature range for awareness of the chlorine-hydrogen reaction is between 550°C and 750°C.

As for the morphology, we took the example of Z0 before and after hydrotreatment. The photos were taken with a scanning electron microscope (SEM) for the Z0 layer in its raw state and after the hydrogen treatment. The previous figures show the pictures of Z0 layer before and after hydrotreating at 550°C and 750°C. We note the volume contraction for the hydrotreated Z0 sample which becomes increasingly apparent as the temperature increases from 550°C to 750°C.

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