A Comparative Study of Two Samples of Khangat Sidi Nadji Biskra Rocks

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Abstract

This study examines two rock samples from Khangat Sidi Nadji in Biskra, employing a range of techniques including X-ray fluorescence, Fourier transform infrared spectroscopy, X-ray diffraction, and thin-slice technology. These methods were utilized to identify the crystal phase, crystal system, space group, and unit cell for the different compositions present in the two samples. The fluorescence analysis clearly indicated that sample R01 comprises 98% quartz, while sample R02 possesses a notably higher proportion of calcite. This distinction was further supported by the Fourier transform infrared technique, revealing the prevalence of quartz in the most absorbent areas of the first sample and a contrasting presence of calcite in the second sample. These findings aligned seamlessly with the X-ray diffraction results processed through the X'pert Highscore program. Sample R01 exhibited a high concentration of quartz with a hexagonal crystal structure under space group $P3_221$, whereas sample R02 predominantly showcased calcite with a rhombohedral crystal structure under space group R-3c. Subsequently, to validate these outcomes comprehensively, the thin blades technique was applied to the rocks. The confirmed results mirrored those obtained through earlier methodologies, solidifying the understanding that despite originating from the same region, these two samples possess distinct appearances and compositions.

Keywords: - XRF. FTIR. XRD. ROCKS. Quartz. Calcite

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Introduction

Algeria enjoys rich and diverse natural resources extending across its vast area. The development and exploitation of these resources must be actively supported as a strategy to promote economic and social development. These resources include rocks, soil, oil, and water, and can be explored and exploited in more than one way.[1] Rocks are an important source of minerals that are used in a wide range of industries such as construction, manufacturing, information technology and agriculture. Classifying rocks into categories such as sedimentary rocks, metamorphic rocks, and igneous rocks can help determine their potential uses.[2] Techniques such as X-ray fluorescence (XRF) contribute to the precise analysis of rock chemistry, which is useful for understanding rock formation and dealing with specific environmental problems. X-ray diffraction (XRD) is also important for studying crystal structure in rocks and other solid materials. They can be used to analyze the crystalline structure of minerals in rocks and precisely characterize their components.[3]

This in-depth research into rocks plays an important role in understanding the environment and its sustainability and providing information that helps in making strategic decisions for sustainable development. This research has many benefits in a wide range of fields from industry to electricity and infrastructure technologies. Investments in this field stimulate the achievement of comprehensive and sustainable development in Algeria.

Materials and methods

Sample collection

In moth of December 2021, rock samples were collected from the Khangat Sidi Nadji area in Biskra, located at coordinates 34°49'36 "N 6°44'05"E. The rocks were found on the surface and varied in size, color, shape, and texture, indicating a diverse geological composition in the area. The samples were carefully collected and handled following proper procedures to ensure their integrity and secure the accuracy of the data obtained from them. This was done to enable researchers to study the characteristics and information related to these rocks, identify their components, and determine their physical and chemical properties.

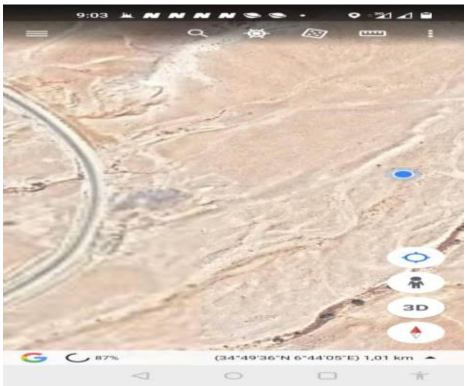


Fig1: Location of the studied area.

Fig2: An image of the studied area

Fig3: An image of sample R01

Fig4: An image of sample R02

The used spectrophotometric techniques

X-ray fluorescence

The XRF spectrometer allows quantitative and qualitative identification of rock samples, where the S2PUMA-BRUKER device was used. X-ray interaction with matter is used to determine its composition and is suitable for solids, liquids and powders.

XRF is widely used for rapid, accurate, and non-destructive characterization

Fourier transform infrared spectroscopy

FTIR spectroscopy is a valuable technique used to analyze the molecular composition of various materials, including organic and inorganic substances, as well as crystalline and non-crystalline materials present in a given sample. In this study, a Thermo Scientific Nicolet spectrometer model 380 was employed, covering a broad spectral range from 400 to 4000 wave numbers.

To prepare the samples for analysis, a mixture was created by combining 1 mg of the target sample with 100 mg of dry potassium bromide. The resulting mixture was then crushed and subjected to a pressure of 6.104 N/cm for approximately five minutes to form a solid pellet. This pellet serves as the sample for FTIR spectroscopic analysis, allowing for accurate molecular characterization of the studied materials.

X-ray diffraction (XRD)

XRD spectroscopy is utilized to determine the crystal structure of the analyzed rock samples. The Proto AXRD Benchtop device was employed, using a radiation wavelength λCukα=1.54 Å, operating at a voltage of 30kv and a current intensity of 20Ma. All samples underwent scanning within a range from 10 to 80, with a step size of 0.1 degrees/s. Prior to analysis, the samples were thoroughly crushed into a glass slurry.

To achieve a flat surface, the XRD diagrams were processed using the X'pert High score program. Subsequently, the data extracted from the aforementioned diagrams were compared with the program's databases, facilitating the identification of the most closely-matching crystal structure for each sample.

Thin blades

Two (02) thin sections were made according to the MO-GEO-T02 operating mode of the two samples taken in the field; R01, R02, to be the subject of detailed petrographic observations, using the polarizing optical microscope according to the MO-GEO-S5 operating mode.

This petrographic description is essentially based on the identification of the different mineralogical constituents and the cements that make up each sample, as well as the characterization of the porous medium. The classification of Dunham, (1962) was adopted for the appreciation of the texture of carbonates.

Results and discussion

X-ray fluorescence

Two rock samples from the study area underwent X-ray analysis, and the results indicated(Table1) that sample R01 had a high percentage of silicon oxide (98.75%), while the rates of aluminum oxide, iron oxide, calcium oxide, potassium oxide, and sulfur oxide were low

(0.12%, 0.11%, 0.45%, 0.23%, respectively). In addition, the ratios of NA2O and MGOs were 0.03% and 0.07%, respectively. Conversely, sample R02 had a low percentage of quartz (7.36%) and a high percentage of calcium oxide (48.24%), with very low rates of aluminum oxide, iron, magnesium, potassium, and sulfur (0.45%, 0.41%, 0.72%, 0.25%, 0.10%, respectively). The effect of sodium oxide was also minimal, with a rate of 0.01%.

	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	K_2O	Na_2O	SO_3	PAF	Total%
R01	98.75	0.12	0.11	0.45	0.07	0.23	0.03	0.19	0.08	100.03
R02	7.36	0.45	0.41	48.24	0.72	0.25	0.01	0.10	43.33	100.87

Table1: Chemical analysis of our samples by X ray fluorescence

Si–O bond[10], 692 cm^{-1} symmetrical bending for the Si-O-Si bond [8, 11, 12]

Analysis by FTIR

Infrared ray is a qualitative analysis that allows us to know the functional groups of the studied samples and thus we can determine their components[4]. and by comparing our results with other studies[2], the obtained results of XRD imply that the peaks are attributed to quartz and calcite in two different samples of the same region.

For sample R01(Table2), the results of FTIR spectroscopy showed that the peak at 3446cm^{-1} and 1655 cm^{-1} indicates the presence of water in this sample[5, 6], also found were the two peaks 2920 cm^{-1} and 2848 cm^{-1} resulting from stretching vibration of the C-H bond due to the presence of organic impurities[4, 7], while at peak 1508 cm^{-1} asymmetrical stretching of$(CO_3)^{-2}$ The evidence for the presence of calcite[5], which is consistent with the results of XRD, which also indicates the presence of silicon found in peaks 1084 cm^{-1} with symmetrical stretching of the Si-O-Si bond[5, 8, 9], and 777 cm^{-1} with symmetrical stretching for the Si–O bond[10], 692 cm^{-1} symmetrical bending for the Si-O-Si bond [8, 11, 12].

As for the sample R02 (Table2), according to the XRD results, which indicate the opposite of what was found in the sample R01, as it was found to contain a higher percentage of calcite and a lower percentage of quartz, which was confirmed by the FTIR technique, so that calcite was found in peaks 2873 cm^{-1} [13], 2511 cm^{-1} [14], 1795 cm^{-1} [14], and 875 cm^{-1} [2], which are attributed to CO_3, and the presence of quartz was observed at peak 713 cm^{-1} [9], corresponding to the symmetrical stretching for Si-O bond[15, 16].

Table2: The main bands of IR absorption and vibration that characterizing in our samples.

Band (cm^{-1})	Bond (Vibration mode)	Compound
3446	H-O-H (stretching vibration)	water
2920	C-H (stretching vibration)	organic carbon
2873	C-H (stretching vibration)	organic carbon
2848	C-H (stretching vibration)	organic carbon
2511	$(CO_3)^{-2}$ (asymmetrical stretch and symmetrical stretching)	Calcite
1795	$(CO_3)^{-2}$ (plane bending and symmetrical stretching combination mode)	organic carbon
1655	H-O-H (stretching)	water
1508	$(CO_3)^{-2}$ (asymmetrical stretching)	Calcite
1084	Si-O-Si (symmetrical stretching)	Quartz
875	$(CO_3)^{-2}$ (out-of-plane bending)	Calcite
777	Si-O (symmetrical stretching)	Quartz
713	Si-O (symmetrical stretching)	Quartz

692	Si-O-Si (symmetrical bending)	Quartz

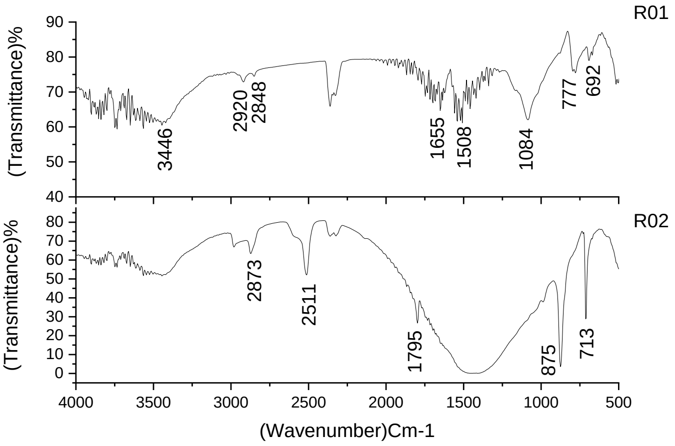
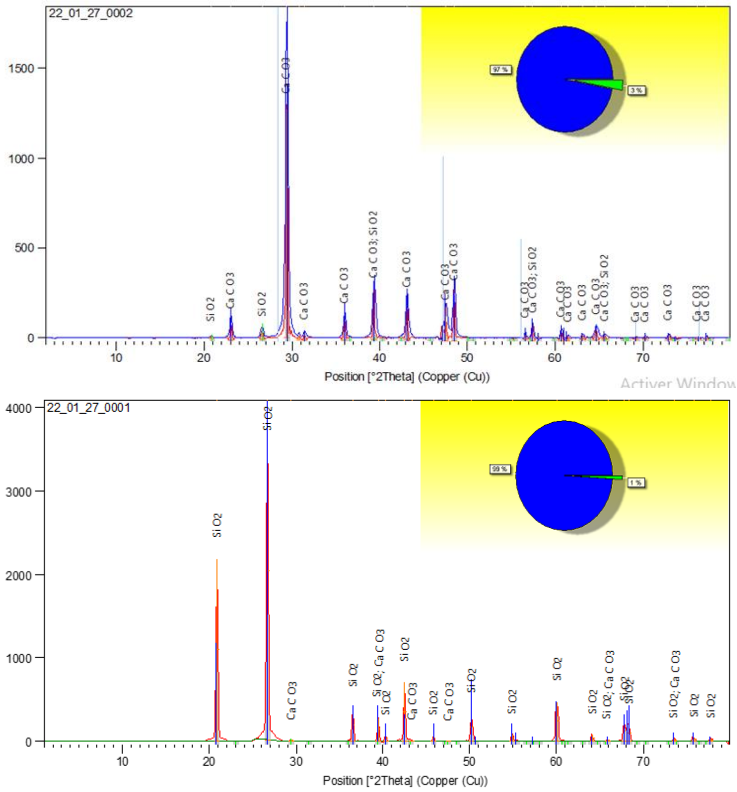


Fig5: FTIR spectra of our samples

X-ray diffraction (XRD) technique :

The XRD analysis of sample R01 indicated that the sample is mainly composed of SiO_2 quartz, with a hexagonal crystal structure under the $P3_221$ space group[11, 17]. Quartz was identified in this sample based on the diffraction angles of 2θ 20.897°, 26.668°, 36.587°, 39.503°, 40.331°, 42.502°, 45.847°, 50.195°, 54.909°, 60.034°, 64.146°, 65.887°, 67.789°, 68.334°, 73.518°, 75.757°, and 77.753°, corresponding to the following crystalline planes: (100), (101), (110), (102), (202), (211), (113), (300), (212), (203), (104), (302), and (220)[18, 19]. In addition, calcite was detected at diffraction angles of 2θ 29.423°, 43.387°, and 47.576°, corresponding to the crystal planes (104), (202), and (018), respectively. On the other hand, XRD analysis of sample R02 revealed that its primary constituent is $CaCO_3$ calcite with a Rhombohedral crystalline structure under the R-3c space group. Calcite was identified in this sample based on the diffraction angles of 23.021°, 29.354°, 35.962°, 39.402°, 43.143°, 47.555°, 48.514°, 56.610°, 57.430°, 60.709°, 61.426°, 63.134°, 64.683°, 65.666°, and 72.948°, corresponding to the crystal planes (012), (104), (110), (113), (202), (018), (116), (211), (122), (214), (214), (119), (125), (300), (0012), and (128)[20]. The detection of quartz in sample R02 was also confirmed by the 2θ angles of 20.797° and 26.587°, which correspond to the crystal planes (100) and (101), respectively. These findings were consistent with the results obtained from other analytical techniques and further validated by the XRD analysis.[21]

Fig06: The XRD spectrum of R01 and R02



Thin Blades

Petrography And Diagenesis

Field sample: R01

The microfacies shows a sandstone composed of monocrystalline quartz crystals, distributed according to a unimodal distribution. These quartz grains are angular to sub-angular. We also note the presence of chalcedony, a variety of microcrystalline quartz that forms in sedimentary and volcanic rocks. Under the light microscope, it appears as small rounded masses, with circular patterns and a crystalline structure consisting of deformed quartz crystals and hues ranging from white to gray.

Field sample: R02

This microfacies is essentially composed of crystalline dolomite (dolomicrosparite to dolosparite crystals).

The microfacies comprises a xenomorphic to idiomorphic mosaic dolomite (crystals have curved to planar outlines); the mosaic is equi-crystalline. This proves that the replacement of calcite by dolomite is complete.

Rare iron inclusions and calcite relics coat the edges of some dolomite crystals. This diagenetic phenomenon is typical of burial dolomitization during mesogenesis. We notice an abundance of dolomite in xenomorphic mosaic compared to that in idiomorphic mosaic, this is fundamentally linked to the CRT factor (Critical Roughening Temperature) or critical hardening temperature, which affects the texture of dolomite crystals (Gregg & Sibley, 1984). According to crystal growth theory, at low CRT, the appearance of thin, planar surfaces is favored. This implies an idiomorphic mosaic, whereas at temperatures beyond the CRT hard, curved surfaces are favored. Such a mosaic is called an xenomorph mosaic. According to Gregg & Sibley, 1984, the CRT for dolomite is between 50° and 100°C.

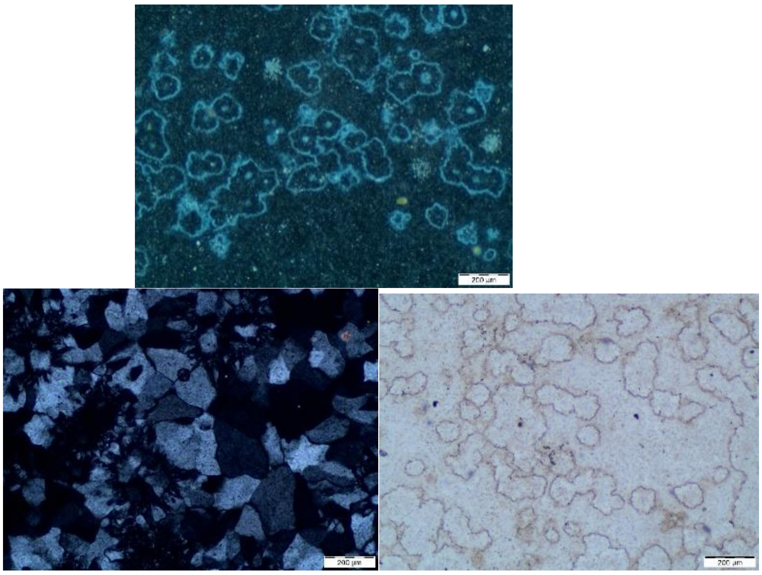


Fig07:a microscope photos of sample R01 with different dimentions

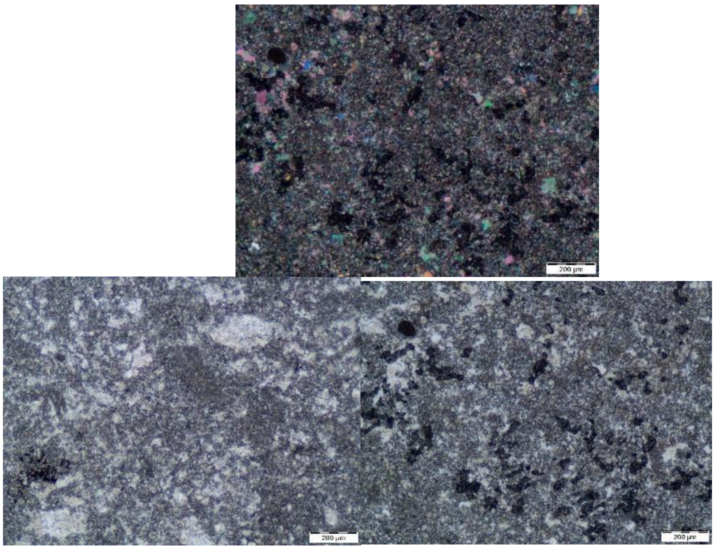


Fig08: a microscope photos of sample R02 with different dimentions

Conclusion

This study represents the pioneering effort in conducting a comparative analysis of rock samples sourced from the Khangat Sidi Nadji region in Biskra. Utilizing X-ray fluorescence technology, distinct differences between the two samples were revealed. Sample R01 was identified to predominantly consist of 98% quartz, while sample R02 showcased a significant presence of calcite.

Verification of these findings was conducted through FTIR spectroscopy, which confirmed the prevalence of quartz in sample R01, evidenced by the areas of maximum absorption. Conversely, sample R02 exhibited prominent calcite absorption zones. These outcomes were congruent with X-ray diffraction analysis, wherein the X'pert Highscore program was utilized for processing. The first sample exhibited a high concentration of quartz, characterized by a hexagonal crystal structure within space group P$3_2$21, while the second sample showed negligible quartz presence but a notable abundance of calcite, with a rhombohedral crystal structure under space group R3c, accompanied by trace elements.

This technique, XRD, stands as a reliable method for both qualitative and quantitative analysis of multi-component mixtures like rocks, representing its widespread utilization.

The outcomes derived from the preceding methodologies were reaffirmed by employing the thin blades technique, providing additional evidence that delineated the compositional disparities between the two samples.

The diverse hues of these rocks directly stem from their chemical composition and the trace minerals they harbor. Khangat Sidi Nadji's rock formations offer valuable reservoirs of quartz and calcite, crucial raw materials in various industrial sectors such as glass production, semiconductors, and optical contrasts. Moreover, these rocks hold immense significance across scientific disciplines encompassing earth sciences, geophysics, and geology."

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