Mediterranean Arhaeology and Archaeometry, Vol. 12, No 2, pp.213-224
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PETROGRAPHY, XRD AND WET CHEMISTRY OF SASSANID ROCK RELIEFS AT KHAN TAKHTI (230 AD), IRAN: A CASE STUDY

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Received: 11/01/2012
Accepted: 11/03/2012
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ABSTRACT

At Khan Takhti at North West of Iran a sculpted monument commemorating a war waged against the Romans by Shapour I, the second Sassanid king, has been archaeometrically studied. This relief monument carved into the rock in the mountains has faced a considerable destruction process. Petrography, wet chemistry experiments and X-ray diffraction (XRD) analysis were performed to identify rock samples for conservation and preservation purposes. The rock used in this monument is limestone and dolomite. According to XRD analysis, a significant amount of quartz is present in the stone structure. The results of analysis and stone type identification show that a significant quantity of surface sediments and dissolved sulphate, chloride and nitrate salts have been deposited on the surface of the monument due to its proximity to the salty Urmia Lake, which is considered an important factor in the destruction process.

KEYWORDS: XRD analysis, petrography, wet chemistry, rock reliefs, technical studies, Khan Tkhti, Sassanid reliefs, Iran

1. INTRODUCTION

The Sassanid dynasty overcoming "Ardavan V" last king of the Parthian dynasty in 247 AD, became a powerful new alliance based on a political unity government instead of Parthian feudal. (Yarshater, 1983)

Artaxerxes policy was to take back the lands of West Asia to the Mediterranean and Black Sea from Rome , the policy of the Roman Emperors was to be Alexander's successors for taking control of Asia Minor and West Asia to India. However, The main reason can be determined as taking control the lands of Armenia, Mesopotamia, Syria and the Euphrates. (Zaryab-e-Khoei, 1975, 31)

The economic reasons had more to political campaigns, Because of the importance of north ways (Silk Route) between east and west, Roman Empire had tried to take control over it to avoid paying transit tax. On the other hand, Sassanid Empire had tried to reach Mediterranean region and Black sea to recover Achemanid power.

"Alexander Severus," the King of Rome in support of "Khosrow" who was powerful ruler of Armenia sent an army to Iran against Artaxerxes. The campaign ended with the victory of Sassanid army and Ardeshir attacked Armenia and Alexander occupied the place. This event is depicted in form of stone reliefs on the Salmas Mountain which is Khan Takhti reliefs as a case study in this research. (Mashkour, 1986, 86-91)

Khan Takhti is a village located in the Anzal Rural Community, a suburban area of Urmia town. An important carved monument associated with a war fought against the Romans by Shapour I, the second Sassanid king, is located near this village. This structure has been carved in embossed form near a mountain known as Sourat Bourni (Dehghan, 1993: 216; Fig. 1). It shows two figures on horseback receiving a floral crown from a person on foot standing in front of them. The images of some captives standing below the two riders can also be seen. Some have attributed the pictures to Ardeshir I and Shahpur I, while others associate them with his son, Bahram II. The engravings are not stylistically or aesthetically comparable to other works of art created in the Sassanid era and the beautiful carvings of Tagh-e-Bostan, a Sassanid monument in Ghasr-e-Shirin.

There are two well-known stories about the Khan Takhti reliefs. One says that the Khan Tkahti reliefs shows the first Sassanid king Artaxerxes (Ardeshir) Babakan and his son (Shapur) in front of the defeated Armenian rulers. If so, this is as a result of the same event that happened in 230 AD, and this relief also belongs to that period. Another story says that the Khan Takhti relief shows Sassanid kings in front of captive Romans. However, literature review

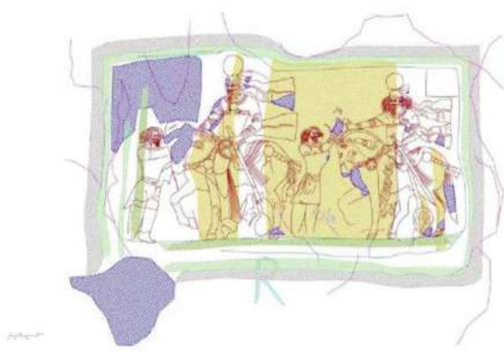

Figure 1: Khan Takhti Relief and Documentation of its damages (Source of documentation: Technical Office, West Azarbayjan's Cultural Heritage Organization)

proposes the probability of the first story more than the second one. (Bakhshaei, 2011)

Urmia Lake, located in Salmas in the West Azarbaijan Plateau and measuring about 150 km from north to south, is unusual in being saturated with various salts, bearing very close affinities regarding depth, morphology, water chemistry, sediment types, environmental system and water cycle to the Great Salt Lake in Utah, USA (Iran's Geological Organization, 1999). The proximity of the lake to the monument means that the latter is influenced by environmental factors, mostly evident in the form of residual effects (Fig 2).

Figure 2: Position of the Khan Takhti relief with regard to Urmia Lake (source of image: google earth)

Rock monuments are found with great diversity in Iran and are of special historical value. The element of time in technical studies of stone/rock monuments is interrelated with the geological background of such engraved surfaces. The way geological structures have emerged, the types of rock found in the structure of rocky walls and the environmental conditions all play vital roles in the survival or deterioration of these structures over time.

According to the literature, the quality of the Khan Takhti engravings and the clarity of the images in these lithographs suggest that less skill was used in creating them than similar works found in other places especially in Fars province. One of the reasons for this uniqueness is related to the kind of rock used in these lithographs, but as most lithographs have been carved on limestone substrates, no explicit opinion can be based on this argument. Another reason is that the Shahpur region (contemporarily known as Salmas) was remote from the centre of Sassanid supremacy and as a result these carvings were sculpted by semi-skilled lithographers. According to another belief, this lithograph was made prior to the establishment of Sassanid rule in Iran, so it was not possible to recruit skilled and experienced artisans; therefore the delicacy of the work was inferior to that of similar work elsewhere. (Bakhshaei, 2011)

According to the authors' observations during field research, important events have normally been selected for commemoration in such structures and lithographs, to capture the public attention. It is likely that this lithograph was created to narrate and commemorate an incident which had happened in the region. Visual evidence suggests that compared to similar carvings, less time was spent in creating this structure and that it was in fact crafted hastily.

The main difference between this engraving and those works contemporaneous with it is related to the way that the patterns have been embossed. Producing surfaces at different levels in most historical embossed patterns is done in such a way as to convey a realistic sense of volume or a perspectival image to the observer, so each pattern is carved according to its real appearance. For instance, human and animal bodies are crafted with a curved slope starting from the most prominent parts and ending in the lowest parts, representing a more stylized and realistic view of the pictures. Besides, the degree to which a pattern is embossed is proportionate to the pattern, resulting in a multi-plan lithography. In the Khan Takhti relief, by contrast, embossed surfaces are directly connected to lower surfaces with a steep and level slope, making the embossed patterns or reliefs look flat and artificial. The number of differences in surface levels can be determined only in the two plans. Furthermore, to add decorative detail, no more than a few lines have been drawn on the embossed surfaces.

Sassanid kings ruled in Iran for over 400 years and the first 85 years of their rule (i.e. 224 to 309 AD) witnessed many changes: Seven kings came to throne and were overthrown, the Sassanids won many victories and suffered many defeats, depicted by thirty embossed engravings in Iran's mountains and rocks. An investigation of these embossed works also reveals the characteristics associated with the artistic environment of the time. Generally accepted principles, rules and styles specific to a school often undergo very little change. As a result, a pattern or image created by two different artists may indicate the school of art, the artist's personal history, his training and his repertoire of imagery.

The literature contains a number of research papers reporting the use of X-ray diffraction (XRD) analysis and polarizing light microscopy to identify the composition of stone monuments (Jimenes-Lopez et al., 2007; Lazzarini et al., 2007; Vacchiano et al., 2008; Liu & Zhang 2007). Kumar and Rakesh (1999) note that X-ray diffraction analysis and observations by electronic microscopes have been employed to study the quality of materials used in the construction of rocky structures. In addition, student theses by Ayasi (2003) and Mehdiabadi (1996) discuss the successful use of the wet chemistry method to indentify the types of rock used in embossed structures.

A number of questions arise here as to the structure of the stone used in embossed carvings which suffer great structural damage. Is the stone structure an effective factor in the degradation process? To what extent will the type of stone affect its deterioration?

The main topic of this technical study is the rocky bed of the Khan Takhti reliefs, which was investigated using a number of different methods: petrography, XRD analysis and wet chemistry tests.

Although stone is often a symbol of eternity and durability, most varieties of natural stone are very sensitive to the environment and show severe signs of damage and decay over time (Winkler, 1997). This has been long recognized and recorded (Schaffer 1932). Understanding the mechanisms of decay requires consideration of many factors, from ambient conditions to stone properties (Smith et al., 2008). Effective diagnosis of the present condition of the built stone heritage is the first step in its preservation (Warke et al., 2003). It involves various methods of in situ description and testing, as well as laboratory analysis of material samples.

2. MATERIALS AND TECHNIQUES

The identification of some of the materials used in structures is possible through the use of wet chemistry tests. This method is often associated with taking samples and as a result damaging or destroying the structures. In the present project, wet chemistry tests were employed as part of a suite of methods in order to complete and confirm the results obtained. These experiments were conducted to identify the rock types employed and the surface sediments.

In order to perform wet chemistry analysis, three areas adjacent to the structure were sampled. The main reason was to prevent any damage to the surface of the main structure caused by the sampling. In order to obtain reliable results for a wide statistical population, samples were taken from three different sections.

The properties of stone may be tested either in situ or in the laboratory. The former methodology is concerned with the in situ identification of stone types and the nondestructive or low destructive testing of natural stone at the monumental scale. Laboratory analyses include a wide range of tests, in most cases using destructive methods, the most common being petrography, analysis of the mineralogical or chemical composition of stone samples and the testing of physical properties. The main aims of all such analyses are to identify the stone types, to establish their present condition and to provide data on the long-term behaviour of natural stones. The other aspect of stone testing is the selection of replacement stones with appropriate characteristics, compatible with those found in the monument (Přikryl, 2006).

Based on the possibility of the existence of limestone, a number of tests were performed to identify the major ions existing in the limestone rocks (i.e. Ca^{2+} and CO_3^{2-}). Part of each of the three samples was converted into powder and used in these tests. The volume of powder of each sample used in these tests was about 2 cm^3 and the remaining part of each sample was kept for further analysis.

A calcite and carbonate identification test was performed according to a method proposed by Svehla (1992), then, based on the possibility of the existence of chloride, sulphate, phosphate and nitrate ions, indicated by the dissolution rate, the displacement ability and chemical and physical destructive ability of these four ions were studied. Sediment sampling was carried out according to place of accumulation and colour variation of the ions. To do this, three points were selected on the surface of the structure with the highest accumulations of surface sediments. An area of about 10 cm^2 was sampled for each section, providing powder samples weighing 3 gr. A third of each sample was used in wet chemistry testing and the rest was preserved for subsequent analysis. The identification tests for chloride (Cl^-), sulphate $(SO4)^{2-}$ and phosphate $(PO4)^{3-}$ ions were performed according to the method proposed by Svehla (1992).

At the next stage, the sample was XRD analyzed to detect differences in the crystalline shapes of various minerals and provide qualitative and quantitative results. Petrological characterization of the stone by XRD and polarizing light microscopy is the best way to conduct such research (Liritzis et al. 2007 and Domingo et al., 2008). If a like-for-like replacement stone is required, it is important to characterize the composition and petrographic features of the original stone (Hyslop, 2006).

X-ray analysis of the samples was next performed using XRD, which can be used to identify minerals (Park 2009). To perform XRD, the rock samples were converted into powder and analyzed by X-ray beam.

To complement the results of the XRD analysis and wet chemistry tests, petrographic observations of samples were made to record microscopic features, to identify their mineral components, to discover the arrangement of these components and to observe features related to appearance such as colour, shape, size and porosity. This stage included tests performed under polarized light, a technique which is employed to analyze rocks, soil and sand components. Microscopic cross-sections of rocks are prepared and the samples are imaged by electronic (polarization) microscopes. Petrographic analysis by thin-section analysis is an accurate method of analyzing rocks and its value lies not only in detecting aesthetic aspects of stone but also in determining hidden geotechnical inconsistencies causing the rapid destruction of rocks (Hyslop, 2004). The employment of this technique to select a stone for the subsequent restoration is very effective in the long term (e.g. Warnes, 1926; Ashurst & Kelly, 1980).

Careful sampling is necessary to ensure that samples are representative, and in many cases a number of samples are required to characterize variations in the stone. Taking thin sections from a series of small diameter cores is an effective means of obtaining a representative set of samples.

3. RESULTS AND DISCUSSION

Khan Takhti relief has valuable feature with regard to golden proportions. The following images show golden divisions in the Khan Takhti reliefs in the form of golden rectangle. Although Khan Takhti is suffering from weakness in performing details of reliefs, golden rectangle show that composition and layout of the scene is composed very well.

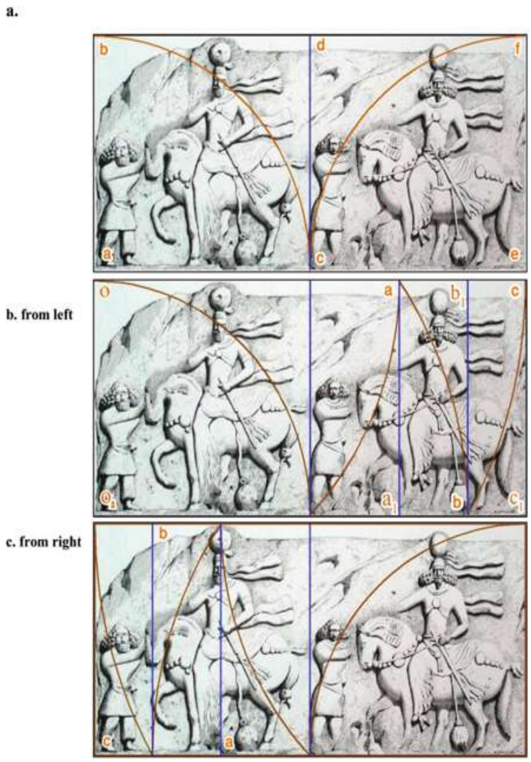

Figure 4: Golden proportions in Khan Takhti reliefs

3.1 Sampling

To achieve results in a wider statistical community, sampoling carried out from different points. So, three samples of stone taken from bellow, left and right sides of the reliefs. Figure 5 shows locations of sampling. In addition, samples of sediment accumulation based on the location and the color difference is they do. Three points from the level of accumulation of surface deposits which have been selected for this purpose. Surface sediments samples taken about 10 cm for each sample which was in about 3 gr weight. One-third the weight of each of these three samples were used for wet chemical tests and the rest used for other analyzes. Figure 6 shows locations of surface sampling.

Figure 5: Location of stone sampling

Figure 6: location of surface sediment sampling

3.2 Identification of types of stone

Given the structure of the stone beds of these rock engravings, it was believed that limestone was likely to be present, so tests were performed to identify the major ions existing in calcareous rocks, i.e. calcite (Ca^{2+}) and carbonate (Ca_3^{2-}). The results of the experiment indicated that the ion identification test was positive in all three samples.

$Ca^{2+} + C_2O_3^{2-} \longrightarrow CaC_2O_4$

$CO_3^{2-} + HCl^{-} \longrightarrow CO_2+H_2O+Cl^{-}$

Given the existence of Ca^{2+} and Ca_3^{2-} ions in the samples, it can be concluded that they were of limestone rocks (See Table 1).

Table 1: Result of wet chemistry tests

Ion	Ca^{2+}	$(CO_3)^{2-}$
Result	+	+

3.3 ***Investigating the existence of damaging ions in embossed surface sediments***

In surface sediment samples taken from three different embossed sites, chloride and sulphate ions were detected, but there appeared to be no phosphate ions present.

Nitrate ions were identified in the sample taken from points A and C, due to penetration of these ions along with the humidity rising from the surrounding area into parts of the relief. These ions are found in abundance in the soil of farms and cemeteries and also in large quantities in organic waste. The remnants of bird droppings were spread on parts of the embossed surface. The rock edges of upper parts of the engraving were a good place for birds to perch and it is likely that the existence of nitrate ions in the upper parts was due to the penetration of bird waste into the surface sediment beds (See Table 2).

$$Cl^- + AgNO_3 \rightarrow AgCl + NO_3^-$$

$$^{-2}(SO_4) + BaCl_2 \longrightarrow BaSO_4\downarrow$$

Table 2: Result of wet chemistry tests on sediments

Ion	Cl^-	$(PO_4)^{3-}$	$(NO_3)^-$	$(SO_4)^{2-}$
Result of sample A	+	-	+	+
Result of test on sample B	+	-	-	+
Result of test on sample C	+	-	+	+

As the results of field studies indicate, this rock was grey in appearance, containing microcrystal minerals and lodes of calcite called grey calcite stone.

The following results were obtained through examination of microscopic cross-sections of rock. The bed consisted of microcrystal micrite minerals (calcite) of a dark cream colour, making up a large part of the cross-section. This mineral was extremely microcrystalline and its components were less than 4 microns across. The veins in the cross-section were filled with secondary macro-crystalline calcite minerals (asperite). The mineral is placed in porosity and is deposited along with the fluid and then takes a sedimentary form. Crystals of dolomite mineral with a rhomboid (diamond) shape can be seen on the surface and were found in abundance in the cross-section. This mineral is characteristically similar to calcite, differing only in appearance and prominence. It is classified by Falk (1995) (see, Fahimifar and Hamed, 2001) as a new dolostone or dolomite limestone with calcite veins (Fig. 7).

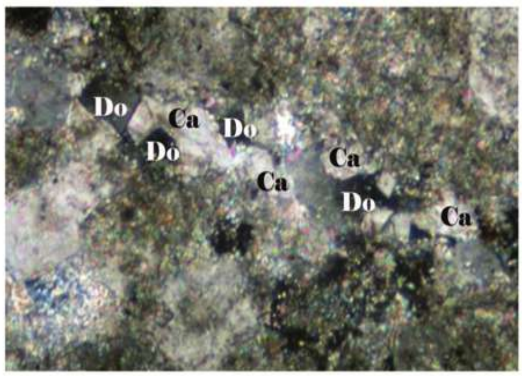

Figure 7: Petrography of stone sample, calcite and dolomite crystals under polarized light, 500x

The composition of carbonate rocks (calcium carbonate) is based on calcite and they include dolomite (calcium magnesium carbonate), also called limestone. One of the samples, containing dolomite mixtures is dolomite named specifically as dolomite. Dolomite and limestone rocks are of essentially similar composition. The composition of the samples can be classified as follows:

- Veins (granulation) or density (mass) of calcium carbonate containing tiny and large compounds in addition to fossils, pellets or intra-calcites.

- Carbonate mud (micrite or microcrystalline carbonates).
- Clastic quartz, clay, or other noncarbonated minerals (including anhydrite, plaster, limestone, glauconite, feldspar, etc).

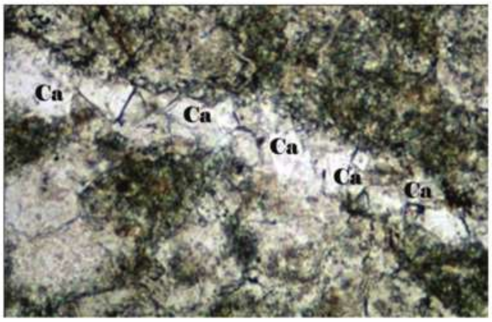

Fig. 8: Calcite veins (white area) under normal light, 500x

- Large carbonate crystals deposited often as in the slot, often called asperite (Reedy, 2008).

The formation of dolomite in calcareous sedimentary rocks leads to a reduction of rock volume. This in turn causes secondary porosity and thus will increase the risk of damage to the rock. The fossil residuals can be seen in the form of brown semicircular marks in the cross-section (Fig. 8).

Calcareous rocks are formed by chemical and biological processes in sedimentary areas. About 95% of these rocks are formed as the result of the activity of living organisms and are diversified by the evolution of organisms over time (Faiznia, 1998: 68).

Based on the results of the XRD analysis, calcite and dolomite phases were identified in the rock structures, indicating that the samples had a calcareous structure. The stones used in the Khan Takhti reliefs are thus composed of dolomite limestone and are free of silica impurities (Fig. 9 and Table 3).

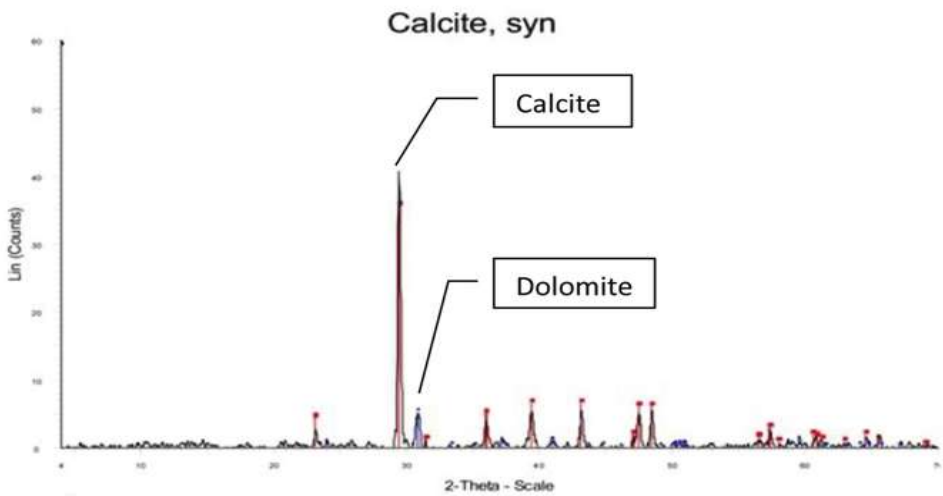

Figure 9: XRD analysis of stone sample, red line indicates calcite and blue line indicates dolomite

Table 3: XRD results of rock samples

No	Compound	Amount (%)
1	Calcite	77.8
2	Dolomite	22.2

The primary mineral found in limestone is calcite (rhomboid calcium carbonate stabilized at an initial constant temperature). Other carbonate minerals may be added, in addition to aragonite (orthorhombic calcium carbonate formed in sea water), dolomite (rhomboidal calcium magnesium carbonate), magnesite (magnesium carbonate) and siderite (iron carbonate) (Reedy, 2008).

Calcareous rocks are divided into four categories: pure limestones, impure calcareous rocks, dolomite calcareous stones and impure dolomite calcareous stones. Pure calcareous rocks are made of pure calcium carbonate and are amorphous. The existing carbonate is crystallized due to adjacent metamorphism and makes up a type of stone called crystalline limestone or marble. Most marbles are white, because carbon materials are destroyed in the transformation process (Sohrabi et al., 1995: 97).

If impure calcareous rocks contain silica or clay impurities, they form mineral marbles, due to adjacent metamorphism. The environmental temperature leads to a different morphology in dolomite limestones. The carved cross-sections of these stones make very delicate and interesting patterns. Impure dolomite calcareous rocks are mostly made of magnesium silicates. In other words, impurities such as SiO2 and magnesium oxides are clearly visible in these rocks (Zarian, 1964: 112-115).

Following the experiments done for stone identification, the surface sediments were also identified to determine the extent of

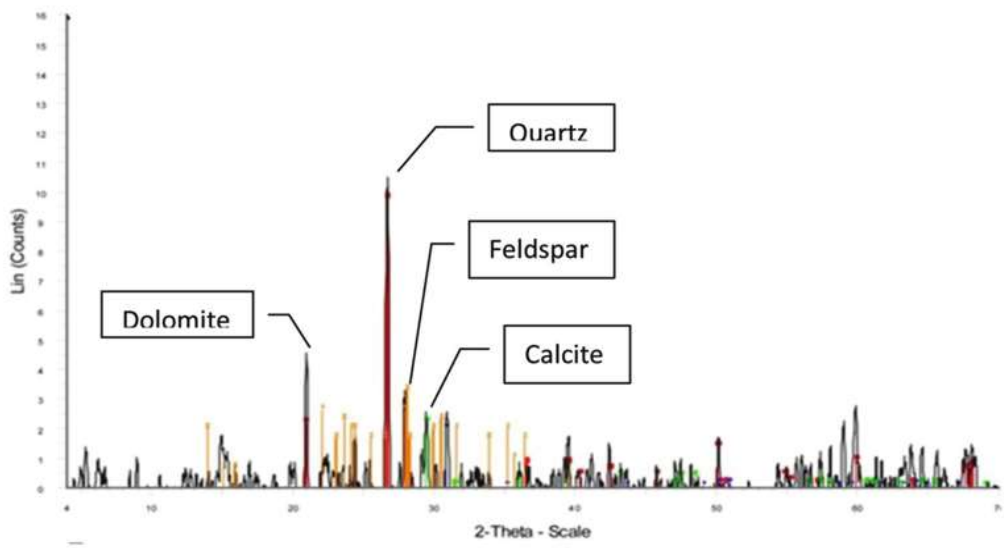

Figure 10: XRD analysis of sediments, indication of lines are as Red: Quartz, Blue: Dolomite, Green: Calcite and Yellow: Feldspar

Table 4: XRD results for sediments

No	Compound	Amount (%)
1	Calcite	12.1
2	Dolomite	21.8
3	Quartz	30.1
4	Feldspar	36

their effect on the degeneration process (Fig. 10 and Table 4).

The existence of calcite and dolomite in surface sediments can be clearly attributed to the erosion of these rocks. Dolomite had a higher numerical value than calcite and its potential instability caused a greater volume of the material to be disposed in the form of eroded products.

Quartz and feldspar are more likely to have a solid origin. The movement of particles contained in these compounds by the wind and their landing on the stone surface may account for the existence of such particles in surface sediments on these embossed engravings.

CONCLUSION

According to the results of the studies reported here, the samples consisted of dolomite and calcareous rocks, while the sediments on their surface included chloride, nitrate and sulphate ions. The existence of calcite, dolomite, feldspar and quartz phases has led to a great diversity in the composition of the rock, which has lost its resistance under the influence of environmental factors, especially water, resulting in the initiation of a degeneration process in the stone. Various ions such as $(NO_3)^-$ and $^{2-}(SO_4)$ were detected on the surface and are assumed to be present as a result of salt deposits. Possible sources of chloride salts are the surrounding soils and the transfer of salts contained in the nearby salt lake by atmospheric currents. The destructive effects of salts dissolved in water has been thoroughly explicated in all resources associated with the protection and restoration of stone structures, but the existence of such compounds in deposits formed on the surface of embossed engravings does not necessarily suggest destruction of the monument and this is the subject of much controversy.

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