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Characterization of Basalt for Conservation Use from Cultural Heritage Site of Umm El-Jimāl in Jordan

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Abstract

The present study is a multi-analytical approach to the characterization of several basalt stone samples, from Umm el-Jimāl Cultural Heritage site, northern Jordan, which represents ancient trade routes between Arabian Peninsula and Syria. In particular, Micro-X-ray Fluorescence Mapping as new in the mineralogy, X-ray Diffraction, Energy Dispersive X-ray spectroscopy are used for the determination of the petrological, geochemical, and mineralogical characteristics of the basalt used in this archeological site for conservation purposes. The results reveal that it belongs to the alkaline trachy-basalt to basanite-tephrite type. With predominant quartz, olivine, pyroxene, and plagioclase (albite) as major elements, the vesicular texture is occupied with secondary minerals such as biotite, gypsum, and calcite.

Keywords: Umm el-Jimāl, Cultural Heritage, Basalt, X-Ray Diffraction, Micro-X-ray Fluorescence

1. Introduction

Umm el-Jimāl is a cultural heritage site, situated northeast of Jordan, 70 km to the north of the Amman city and just at the southern border of the Syrian Hauran plain (Fig. 1). Nowadays, the Umm el-Jimāl is divided into two areas: the archaeological site, the village of Umm el-Jimāl. The oldest archaeological evidence goes back to the first century. Throughout ancient times, the place was never emptied and it was inhabited from the first to the eighth century AD (De Vries, 1990b). Umm el-Jimāl was considered the gateway to the desert and the major link to the neighboring cities, the possibility that the first inhabitants of it are the Nabateans, who commuted with their caravans between the south of Jordan, from Petra to Syria via Wadi Sirhan toward Damascus in the first century BC (Bowersock, 1973; De Vries, 1981; De Vries et al., 1998). When the Romans invaded the region in the second century AD, the town eventually became a part of the Roman Limes Arabius, this limes represented the desert borders of the Empire, and it was located in the Arab province of Petra or a system of fortification and roads, or line of protection and fortification that the Roman system of frontier district protected the Roman province of Arabia, now Jordan and southeastern Syria. Nevertheless, in Umm el-Jimāl, it can be supposed that the inhabitants, to some extent, were existed in relative independence, in the 6th and 5th centuries, reached the height of its prosperity as a rich Byzantine city of perhaps 5,000 inhabitants. In

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the followed centuries, the population of Umm el-Jimāl has rebuilt and used the basalt structures until their probable decline and gradual abandonment in the late 8th century (De Vries, 1990a, 1998; Young, 2003; De Vries et al., 1998). The area is characterized by volcanic activity that took place through six periods between the Oligocene and Miocene ages until the Holocene (Ilani et al., 2001). The most known of these volcanoes is Jebal Aritain (Al-Muheisen, 2009; Horsfield, 1937). The basalt plain covers an area of about 45.000km², including 11.000 km² in north-eastern Jordan (Al-Muheisen, 2009). The city of Umm al-Jimāl is part of the volcanic zone extending between Saudi Arabia, Jordan, and Syria, which is located at an altitude of more than 650 meters above sea level and is part of the volcanic zone in Jordanian Hurat Ash Shaam (Al-Malabeh, 1994a; PINT, 2006; Shaw et al., 2007; Tarawneh, 2000; Shaw et al., 2003) (Fig. 2). The region has a semi-arid climate, based on 150-200 mm annual rainfall (Al-Muheisen, 2009; Corbett and Reynolds, 1957).

Currently, archaeological researchers continue to work out the crux of the matter of Umm el-Jimāl's illuminating the everyday history of antiquity. Even if it is located on the semi-arid volcanic plateau in Hauran, the inhabitants have developed a well-devised water storage system (Al-Muheisen, 2009; De Vries, 1998). The Canals and reservoirs systems collect surface runoff during the wet season from long distances; can provide sufficient water to help thousands of people, animals, and their agricultural production. Umm el-Jimāl has also a unique architectural attraction. The old inhabitants of the town used abundant black basalt from the volcanic plains of the region to design and construct strong buildings. with basaltic stones (Brown, 2009; De Vries, 1981, 1985, 1990a, 1993, 1994).

Fig. 1. The panoramic view of the Umm el-Jimal cultural heritage site

Volcanic rocks were a popular working material for pharaonic building and monuments in ancient Egypt. Often people used the basalt for stone tools such as milling stones, statues, and construction. Among the oldest stone vessels made in Egypt in the Neolithic period were vases. The oldest stone containers made in Egypt in the Neolithic period were for example vases (Mallory-Greenough et al., 1999). Basalt is widely used throughout the world and seems to have older origins, which can be traced back to a pharaonic stone name that was already used in 2000 BC (Harrell, 1995). The basalt stones were already used in the Old Empire for road material in the necropolis that stretches from Giza to Saqqara in Egypt. The basalt probably came from the Fayum in Egypt, where the old quarry can still be found today. Throughout the pharaonic era, basalt was needed for figures and sarcophagi. Also, the Greek and Roman kings produced the sarcophagi from basalt. (Rapp, 2009) and found as stone tools in many archaeological sites in Iraq (Al-Zubaidi et al., 2021).

The main components of basalt, in general, are the plagioclase enriched with calcium, pyroxene, magnetite, and olivine. Gabbro is the chemical equivalent to Basalt (Rapp, 2009). The basalt is an

extrusive igneous rock since it cools rapidly when hot magma spreads on the surface of the crust, leaving a slight time to let the crystals start growing. Consequently, the texture of the extrusive rock is fine-grained. These volcanic rocks are a product of the explosions that occur during a volcanic eruption. Basalt is an excellent example of extrusive igneous rock, while Granite is an example of intrusive igneous rock, both depend on the cooling and crystallization of molten magma (Rapp, 2009; Trefil et al., 2005).

Fig. 2. The location of Harrat Ash Shaam basalt Plateau in Jordan, Syria, and Saudi Arabia

The main chemical components of igneous rocks are SiO_2 , Al_2O_3 , CaO , MgO , Na_2O , TiO_2 , and Fe_2O_3 , with small amounts of K_2O , MnO , and P_2O_5 . Silica is the main content to classify the igneous rock. The rich-silica igneous rocks are recognized as acidic (or felsic) rock, but the low-silica rocks are known as mafic. Generally, the rocks that are caused by explosive volcanoes are a higher concentration in silica than those that resulted from lava flux (Rapp, 2009; Smith, 2014). Basalt was dispersed widely over many different cultures worldwide for building, monuments, and tools in Egypt, Germany, and the Netherland (Stuart, 1993), Hawaii (McCoy and Gould, 1977), and Sicily (Leighton, 1989). The Romans revealed that a pulverized combination of lime and basaltic pumice would yield a new type of binding material that was distinctly superior to lime mortar. It was durable, stable, resisted an attack by seawater, and would set as hydraulic lime underwater as well as in the air. The Romans extracted their ash and pumice stone deposits in a volcanic area close to the town of Pozzuoli belongs to the commune of the city of Naples in Italy, and this hydraulic basalt mixture cement type is called pozzolana (Rapp, 2009).

Basalt is a common material for millstone made from the Neolithic, Roman Periods, and nowadays in Palestine, Israel, Syria, Jordan, Egypt, Greece, Sardinia, and Turkey (Williams-Thorpe, 1988; Peacock, 1989; Williams-Thorpe and Thorpe, 1993; Peacock, 1989; Lorenz and Wolfram, 2007). Basalt is the foremost rock type used by the peoples of northeastern Hashemite Kingdom of Jordan, for building or monumental stones concurrently with other tool implements including pestles, grindstones, millstone, mortars, axes, adzes, and mauls (Walker and Firestone, 2009). Basaltic rocks exhibit a subtle variety of textures and mineral-chemistry that may control their mechanical and physical properties as well as their use as masonry materials and components (Engidasew and Barbieri, 2014). Mössbauer spectroscopy is a good method to analyze iron ions in the bulk samples of basalt, to evaluate the four paramagnetic components assigned to Fe^{2+} in olivine ($(\text{Mg}, \text{Fe})_2\text{SiO}_4$), ilmenite (FeTiO_3), pyroxenes ($(\text{Mg}, \text{Fe}, \text{Ca})\text{SiO}_3$), and a Fe(III) constituent allocated in ferric iron that is generally not conceivable to allocate to any specific mineral classes (Gajbhiye and Date, 2009; Gunnlaugsson et al., 2006). Al-Malabeh had investigated the magnetic properties of Tertiary – Quaternary in Umm Al-Qutein Basaltic flows from Hara El-Jabban, in the Northeast of Jordan using the Superconducting Quantum Interference Device Magnetometry (SQUID), they confirm the presence of a mixture of both magnetic phases, ferrimagnet, and ferromagnet phases (Al-Malabeh et al., 2002). Al-Malabeh, studied the volcanogenic of Welded Pyroclastic Rocks in Jabal Aritain volcano, Northeastern Jordan. El-Hasan, and Al-Malabeh, investigated the geochemistry and petrology and geological settings of Al-Lajjoun Basaltic flows, in the southern part of Jordan (Al-Malabeh et al., 2003).

The basalt rocks in Jordanian Harrat Ash Shaam are mainly tertiary – quaternary lavas. They occupy 18 % of the area of Jordan and arranged according to Bender and Al-Malabeh in three major regions, depending on the type of their presence: 1) in the Jordan rift valley (Zara basalt), 2) in Jordan center. (El-Lajjun basalt) and 3) NE-Jordanian Harrat Ash Shaam (with an area 11,400 km²) which is a part of the major Harrat Ash Shaam, as illustrated in Fig. 3. Previous petrochemical analysis of the basaltic rocks indicated that the NE basalt plateau is constituted of alkali basalts and basanites with low amounts of nephelinites. (Al-Malabeh et al., 2003; Al-Malabeh, 1994b; Barberi et al., 1979; Bender, 1974; Al-Malabeh et al. 2009, Ibrahim and Al-Malabeh, 2006; Moffat, 1988). The geochronological age of Umm el-Jimāl basalt is correlated with the adjacent Umm Al-Qutein flow, which corresponds to the dating of K-Ar from the Late Pleistocene to the Holocene of about 9.86 Ma (Moffat, 1988). The aim of this study is to characterize the basalt in terms of minor and major element oxides in the cultural heritage site of Umm el-Jimāl for conservation purposes. Therefore, 18 basalt samples are collected and subjected to XRD analysis with the Energy Dispersive X-ray spectroscopy (EDS) and X-ray Fluorescence Spectrometer (XRF).

2. Materials and Methods

Eighteen basalt samples from different places were collected from Umm el-Jimāl archaeological site. The samples have been investigated first by μ -XRF as new analytical possibilities in basalt study. X-ray fluorescence of elemental analysis done with small spots needs only a simple preparation and permits protected sample handling. The elemental maps of the basalt were got swiftly and independent of the shape and the size of the specimen. Basalt samples have inhomogeneous minerals distribution. The analysis of their different compounds is important for understanding the mineralogical processes, exploration of their applications, and mineral resources. X-ray radiation is focused on a small sample area. A supplementary benefit of this method relatively has high sensitivity and precision for the detection of trace elements. (Haschke et al.; Haschke et al., 2002; Janssens et al., 2000; Lemberge et al., 2000). Micro X-ray fluorescence is applied excellently in the field of archaeology and cultural resources and related science with multiple material types, including the investigation of minerals to characterize the element distribution in detail to observe the spatial alteration in geological and archaeological

samples, to study various ancient buildings and monuments material objects. μ -XRF increases the analytical performance, identifying association among elements, including better sensitivity and resolution, a higher speed of the measurement, and enhanced usability. μ -XRF mapping was performed by the data of the whole analyzed area of $18.6 \times 22.5 \text{ mm}^2$ was acquired. The measurement was investigated via a Tornado M4 micro-XRF Spectrometer (Bruker Nano GmbH, Berlin, Germany) equipped with a rhodium (Rh) side window X-Ray tube with polycapillary lens giving a spot size down to $25 \text{ }\mu\text{m}$ focused a high intensity with a step of $15 \text{ }\mu\text{m}$ which marks in a pixel number of 720×900 . The measurement time was 3 ms per pixel, and then the total acquisition time of plotting for every element was two hours. A small area of the sample (up to 300 cm^2) is moved on a motor-powered stage in the M4 Tornado concluded a beam of an X-ray source equipped with a polycapillary lens. The beam size is matched by positioning the sample at a fixed distance from the polycapillary lens. In addition to high sensitivity for elements wide range, the system offers an intuitive graphical user interface.

The X-ray Diffraction (XRD) was employed for the mineral identification with an XRD; Xpert PRO interfacing (PANalytical, Almelo, The Netherlands), through Ni-filtered and $\text{CuK}\alpha$ radiator has a wavelength ($\lambda=1.5405 \text{ \AA}$), was used in all measurements. Patterns were obtained by step scanning from 20° to $70^\circ 2\theta$, with a dual goniometer speed of $0.03^\circ/\text{s}$, operating at 30 kV and ten mA at room temperature. The samples for XRD measurements were grounded with an agate mortar and pestle until we have a fine attained and the size required powder before analysis was obtained. The basalt samples quantitative analysis of basalt samples was performed using energy-dispersive X-ray spectroscopy (EDS) using a QUANTAX 200 Bruker detector at 12 kV for EDS analysis at IPHT Jena in Germany.

3. Results and Discussion

3.1. Chemical Analysis

Generally, the chemical compositions (SiO_2 , Al_2O_3 , Fe_2O_3 , MnO , TiO_2 , CaO , MgO , K_2O , Na_2O , and P_2O_5) of basalt samples were determined, the main components SiO_2 and Al_2O_3 are the dominant beside higher Fe_2O_3 and alkaline elements such as Na_2O and K_2O , besides that CaO is high too due to secondary filling of vesicular texture, the results are presented in Table 1.

Table 1. Geochemical composition of the basalt stones of 18 regions, from the *Umm el-Jimāl* (all in Wt%)

Sample ID	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	K_2O	Na_2O	P_2O_5	TiO_2	MnO	LOI	SUM
UJB-1	45.76	15.53	12.23	9.33	8.77	1.12	4.33	0.4	2.42	0.11	1.87	101.87
UJB-2	45.91	14.21	12.45	8.91	9.45	1.27	4.71	0.39	2.39	0.31	1.74	101.74
UJB-3	45.98	14.08	13.23	9.19	8.35	1.79	4.6	0.37	2.23	0.18	1.35	101.35
UJB-4	43.47	14.98	12.76	8.69	10.95	1.84	4.36	0.42	2.53	0.07	1.23	101.3
UJB-5	44.95	15.34	13.29	8.73	10.46	1.31	3.23	0.32	2.16	0.21	1.57	101.57
UJB-6	45.66	13.43	12.51	9.98	8.92	1.81	4.77	0.47	2.31	0.14	1.89	101.89
UJB-7	45.13	12.67	13.13	10.16	9.78	1.22	4.79	0.39	2.57	0.16	2.01	102.01
UJB-8	46.65	12.92	13.19	9.55	9.8	1.46	3.64	0.37	2.23	0.19	1.84	101.84
UJB-9	45.82	15.34	12.09	9.28	8.78	1.67	3.88	0.41	2.6	0.13	1.97	101.97
UJB-10	44.87	15.21	12.18	9.87	9.51	1.09	4.12	0.36	2.48	0.31	1.35	101.35
UJB-11	45.19	14.61	12.66	10.11	8.37	1.75	4.61	0.36	2.19	0.15	1.87	101.87
UJB-12	45.11	14.98	12.56	9.81	8.34	1.58	4.63	0.46	2.42	0.11	1.56	101.56
UJB-13	45.62	14.34	12.33	9.97	8.37	1.89	4.47	0.39	2.54	0.08	1.89	101.89
UJB-14	45.89	14.21	12.77	8.45	9.11	1.39	4.7	0.36	2.41	0.71	1.84	101.84
UJB-15	45.03	14.21	13.81	10.03	8.39	1.08	4.42	0.45	2.39	0.19	1.97	101.97
UJB-16	45.77	14.98	12.49	9.17	8.82	1.84	4.53	0.28	1.89	0.23	1.92	101.92
UJB-17	45.05	14.45	12.27	9.56	9.32	1.81	4.37	0.48	2.52	0.17	1.12	101.12
UJB-18	43.67	15.52	13.61	9.66	8.95	1.25	4.13	0.37	2.66	0.18	1.28	101.28

Table 2. Trace elements concentrations from Umm el-Jimāl basalt

Sample ID	Ni	Co	V	Pb	Sn	Mo	Sr	Rb	Y	Zr	Nb	Ba
UJB-1	211	55	222	6	11	4	577	5	21	103	16	269
UJB-2	199	45	208	7	13	4	534	7	20	112	14	249
UJB-3	214	61	203	6	15	6	529	8	16	106	13	255
UJB-4	222	55	201	5	15	5	554	4	21	104	14	246
UJB-5	215	57	199	5	14	5	540	8	20	102	13	249
UJB-6	221	68	208	7	13	6	627	9	16	104	15	255
UJB-7	217	49	214	8	15	6	740	8	17	102	14	239
UJB-8	211	55	222	4	14	6	627	11	18	113	13	269
UJB-9	199	45	208	5	14	5	505	9	21	102	15	260
UJB-10	214	61	203	6	14	4	517	9	18	106	15	232
UJB-11	222	55	201	7	15	5	543	10	21	104	13	249
UJB-12	215	57	199	7	15	4	523	9	20	105	15	255
UJB-13	221	68	208	6	16	6	524	8	16	111	15	255
UJB-14	217	49	214	8	15	5	522	11	21	117	14	249
UJB-15	230	61	192	6	14	6	614	9	20	104	13	255
UJB-16	244	56	187	6	14	4	537	8	16	118	15	239
UJB-17	230	61	192	7	15	6	547	9	20	102	13	249
UJB-18	244	56	187	5	16	6	521	7	19	105	15	255

All samples are very alike in their composition; obvious from the very narrow varieties of major and trace element concentrations. This is mainly illustrated by the narrow range in silica content between 43.67 and 46.65 wt% with an average of 45.31 wt%. This content falls in the averages described the basanite and basalt, Tephrite given by Bas et al. (1986); Ibrahim and Al-Malabeh, (2006) (Fig. 3).

Fig. 3. Total Alkali-silica diagram from Umm el-Jimāl Basaltic rock (TAS) (Le Bas et al. 1989)

The MgO content ranges from 8.32 to 10.95 wt% with an average of 9.14 wt%. The Mg-number (Mg#), which is defined by Jenner et al. (1987); Downes et al. (1995) as the molecular proportion of $Mg^{2+}/(Mg^{2+}+Fe^{2+})$, is usually used as a petrogenetic indicator for magma fractionation and its primitive nature. The Umm el-Jimāl basalt has a high Mg-number ranging between 0.65 and 0.73 with an average of 0.70. For Mg# calculations, the Fe_2O_3 equals to $TiO_2 + 1.5$ as recommended by Irvine and Baragar, (1971). Houghton and Wilson (1989) reported that a value of $Mg^{2+} > 0.7$ as a threshold that characterizes

the primitive magmas (Houghton and Wilson, 1989). Moreover, the total Fe-content of Umm el-Jimāl basalt, calculated as Fe_2O_3 , ranges between 12.18 and 13.81 wt% with an average of 12.75 wt% which is similar to the nearby Umm Al-Qutien basalt (Al-Malabeh et al., 2002).

Umm el-Jimāl basalt has K_2O and Na_2O averages of 4.354 wt% and 1.51 wt%, respectively. The total $\text{Na}_2\text{O} + \text{K}_2\text{O}$ values are similar through the specimens equal to 5.86 wt% on average. The average value of the $\text{Na}_2\text{O} / \text{K}_2\text{O}$ ratio is 2.88, reflecting the sodic affinity of the rock (Fig. 4). The plot of $\text{Na}_2\text{O} + \text{K}_2\text{O}$ versus SiO_2 shows the alkaline nature of the rock (Fig. 5). Similar basalt was recorded in El-Lajjoun south Jordan (El-Hasan and Al-Malabeh, 2008).

Fig. 4. Total Alkali-silica diagram from Umm el-Jimāl Basaltic rock

Fig. 5. K_2O vs Na_2O diagram showing the sodic affinity of the Umm el-Jimāl Basaltic rock (Middlemost, 1975)

Umm el-Jimāl basalt has relatively high Ni contents. Ni varies between 199 ppm and 244 ppm and averages 219 ppm. Rb content in Umm el-Jimāl basalt is relatively low, ranging from 4 to 11 ppm and averages 8 ppm. This value is lower than the value of 22 ppm reported for alkali basalt (Coleman and McGuire, 1988) and provides an argument against plagioclase fractionation. This was further supported by the high K/Rb ratio. Sr satisfied in the ranges from 505 to 740 with an average of 560 ppm. The Umm el-Jimāl basalt has Zr content ranging from 102 to 118 ppm. Nb content ranges between 13 and 16 ppm. The average Zr/Nb ratio is 7.6. Y content is low and it displays a very limited variation in the range of 16 to 21 ppm, with an average ratio Y/Nb equals 1.36. This ratio is inconsistent with the ratio of >1 reported by Pearce and Cann for the intra-continental alkali basalt (Pearce and Cann, 1973).

3.1.1. μ XRF Analysis

Classifying the chemical status of the basalt needs the distributions of the elements in the investigated area. All scans were done in a vacuum chamber of the Tornado M4 - μ -XRF. The color intensity in the images increases with the x-ray signal intensity. The results of μ -XRF measurement of a basalt sample (Basalt 1 of 2) (Fig. 6).

Fig. 6. The selected area for elemental mapping using micro X-ray fluorescence (m-XRF) of Ti, Al, Ca, Cr, Fe, Mg, Ni, P, S, and Si. color scales represent the normalized total $K\alpha$ fluorescence counts, proportional to the total metal present, from Dark (lowest) to light color (highest)

3.2. Petrography and Mineralogy

3.2.1. Mineralogical (XRD) analysis

The mineralogical investigation based on the XRD study for all investigated samples reveals the presence of plagioclase, nepheline, olivine (forsterite), augite, and hyroxysodalite (Fig. 7). The basalt rocks of Umm el-Jimāl is fine- to medium-grained, melanocratic and holocrystalline, with a dark gray color in the hand sample. The rocks show essentially typical mineralogical assemblages, such as those typical of alkali basalts or basanite, containing phenocrysts dominated by subordinate clinopyroxene, plagioclase, and olivine, and some opaque oxides distributed in a basalt mass of olivine, clinopyroxene, plagioclase, and oxides. The rocks show porphyritic, glomeroporphyritic, and intergranular textures. Some samples occasionally display a vesicular texture that is usually filled with secondary calcite (Fig. 5).

Fig. 7. XRD chart for the selected sample from Umm el-Jimāl archeological site basaltic rock

3.2.2. EDS Analysis

The EDS technique is the right tool for determining the elemental distribution within the rock matrix. It can identify the elements that are not uniformly distributed but found as hotspots within the measured area, such as the accessory mineral phases in basalt. During the investigation of Umm el-Jimal basaltic sample, trace elements such as Ti, Cu, and Zn, which cannot be detected in the sum spectrum, however, they can show relatively high intensity in the Maximum Pixel Spectrum (MPS), thus their distribution was not examined. Also, Cr has a significantly higher intensity in the MPS than in the sum spectrum, because it is concentrated in limited areas (hot spots). However, because these areas are large enough, the Cr peak can be detected in the sum spectrum, and the distribution of Cr can be determined. The content of the major elemental components of Umm- el-Jimal basalt is shown in Fig. 8, those are the elements that were identified using the MPS, such as Ti, Cu, and Zn. The distribution of Zn shows only a signal hotspot, which is similar to the elements Ti and Cu. They are also located only in isolated and scarce regions of the sample (Haschke et al. 2012).

Fig. 8. EDAX analysis for selected areas of Umm el-Jimāl basalt areas (1,2 and 3)

4. Conclusions

The combination of μ -XRF analysis and EDS supports the data collection of samples for the spatial distribution mapping of specific elements major and trace that revealed by recording a map represents the distribution of the elements of basalt surface while the beam is scanned the surface of the basalt object. μ -XRF offers good resolving power between the highest spatial resolution of EDS and the macro sample, apart from providing high accuracy for trace elemental analysis. The μ -XRF shows exactly the distribution of the minerals grains localization on the surface of the basalt object and to get the size of the grain precisely in both a thin or thick section within 2h to help the study of minerals by EDS.

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