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MICRO-DRILLING RESISTANCE MEASUREMENT: A NEW TECHNIQUE TO ESTIMATE THE POROSITY OF A BUILDING STONE.

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ABSTRACT

This study aimed to find the relationship between the drilling resistance (DR) of a stone and its porosity. 40 sandstone and limestone samples (20 each lithotype) were taken into consideration in this study. The samples were mineralogically and petrographically investigated using X-ray diffraction (XRD) and polarized light microscopy. The sandstone samples were identified as quartz arenite that is composed of Quartz, Kaolinite and Calcite, while the limestone samples were identified as micritic limestone that is composed of Calcite. The average DR of the samples was measured using a Drilling Resistance Measurement System (DRMS), and the porosities were determined using a RILEM standard test method. A mathematical relationship between the DR and porosity was derived for both stone types. It was found to be a linear inverse relationship in both cases. The derived relationships were used to calculate the porosities of additional sandstone and limestone samples based on their measured average DR. The values of calculated porosities showed acceptable accuracy when compared with measured values. Therefore, this technique is recommended to estimate the porosity of stones with similar mineralogical compositions.

KEYWORDS: porosity, drilling resistance measurement system, sandstone, Petra, limestone, archaeological sites, mineralogy, petrography, calibration curve.

INTRODUCTION

The corrosion of porous building materials does not only occur on the weathering surface but also under the surface in the pore spaces. These represent the preferred affected area for physico-chemical and biological weathering processes. The porosity influences the movement of water, water vapor, and salt solutions in the stone. Furthermore, the inner zone of stone being attacked chemically is a function of porosity. It was proven that the chemical deterioration of more porous building stones is higher than that of denser types (Efes, 1979; Snethlage, 1982; Ordaz & Espertm, 1985; Fitzner, 1988; Fitzner, 1990; Sousa Luis et al., 2005). Therefore, stone porosity was classified as a main physical property affecting weathering and deterioration of building stones by water and gases (Robertson, 1982). Consequently, the porosity of stone could be categorized as a principal indicative property that should be measured to evaluate its durability.

On the other hand, determining the mechanical properties of stones is vital to study the decay process of monumental stones, to evaluate their conservation state and to assess the conservation action that must be applied (Exadaktylos et al., 2000). Several methodologies can be used to measure the mechanical properties of building stones. These measurements are usually performed with expensive and complex apparatus and need a large number of specimens with regular shape. These conditions are not achievable in most studies on historical monuments (Fritsch & Schamberg, 1986; Guidetti et al., 1995; Tiano et al., 2000a). Therefore, the use of Drilling Resistance Measurement was proposed to overcome these problems (Garrod & Massey, 2000; Singer et al., 2000).

Drilling Resistance Measurement was invented in 1908 by the German scientist Julius Hiscshwald (1845-1929) and developed considerably during the last 15 years (Von Plehwe-Leisen et al., 1994, Pamplona et al., 2007). It is a sensitive, reliable and micro destructive technique in which a thin drill (3-5 mm) is used. The evaluation of the hardness is related to the drilling penetration force: the force necessary to drill a hole with specific operative conditions such as the Penetration Rate (PR) and the Rotational Speed (RS), which remain constant during the test. The profiles of drilling resistance provide information about the consolidating effect and the penetration depth of a product (Tiano et al., 2000; Bourgès, 2006), The main fields of the application of DR measurements in conservation have been to assess:

1. The effect of the weathering on materials by drawing the weathering profile of stone, which is a graphical presentation for the relationship between depth and drilling resistance,
2. The mechanical durability of building stone,
3. The inner microstructure of stone and other materials and
4.The absorption and effectiveness of consolidantion treatment.

Very few relations between drilling resistance and other mechanical and physical properties have been reported in the literature. Leonhardt & Kiessl (1990) defined the relation between flexural strength and Young's modulus. Alfes et al. (1992) showed the correlation between penetration hardness and compressive strength on sandstones. Tiano et al. (2000a) confirmed the relation of drilling resistance with uniaxial compressive strength (Bourgès, 2006).

On the other side, several methodologies, including theoretical and empirical approaches, have been developed to estimate the porosity of building stone. For example, RILEM 1980-Test No. I.1, DIN EN 772-4, ISRM (1972), and UNE-EN 1936: 2007. In addition to these conventional techniques, various innovative measurement techniques have been suggested; Merrill (1994), Bowers et al. (1995) and Marica et al. (2006) showed that magnetic resonance imaging can be used to make nondestructive experimental measurements of fluid flow velocity and rock porosity of sandstone. Al-Harthi et al. (1999) showed that image analysis technique is a reliable method to estimate the porosity of stone, Griffiths (1976) suggested the application of electrical resistivity measurements to determine the porosity and permeability in sandstones, Guo and Cawley (1994), Papida et al. (2000) and Goueygou et al. (2009) correlated between the ultrasonic velocity and porosity. They found that the porosity could nondestructively be measured by the application of ultrasonic attenuation measurements. Van Geet et al. (2003) and Cnudde et al. (2009) suggested the use of microfocus X-ray computed tomography (μCT) to measure the porosity of sedimentary rocks. Ruggieri et al. (2009) mentioned that air pycnometry is considered the most suitable and accurate technique to obtain reliable measures of porosity. Maukoa et al. (2009) recommended the use of confocal laser scanning microscopy (CLSM) for the characterization of porosity in marble.

This study aims to derive a mathematical relationship between the DR of sandstone and limestone and their porosities. The benefit of such relationship, will be in calculating the porosity of natural stone without a need to apply a time-consuming procedure or take large samples, which will make it recommended over conventional techniques in the studies on historical monuments.

2. SAMPLES AND METHODS

Taking into account the fact that sandstone and limestone are widely used as building materials in many areas around the World and some of the most important culturally significant monuments and buildings throughout the World were built using these types of rocks, they have been chosen for this study. 20 sandstone cubic samples from the Disi Sandstone (Ordovician) in Petra were taken for the purpose of this study. This formation is a parts of the Nubian Sandstone, which is exposed in Saudi Arabia, Egypt, Jordan and Palestine (Bender, 1974). In the first three countries, this unit was used to build (or carve) very important archaeological structures such as Mada'in Salih in northern Saudi Arabia, most of the monuments in Upper Egypt and Petra monuments (Al-Naddaf, 2009). Moreover, 20 limestone cubic samples were taken from the Massive Limestone Formation (Upper Cretaceous) exposing in northern Jordan. Limestone from this formation was used for the construction of many important archaeological sites, e. g. the Decapolis. Furthermore, it is the main source for building stone for modern constructions in Jordan.

The cubic samples (5x5x5 cm^3) were taken from sound quarry blocks with no bedding.

Since the drilling resistance of a stone is affected by the hardness of the minerals composing it (Pamplona et al., 2007), all of the studied samples should be chosen to have similar mineralogical composition, therefore, the studied samples were investigated with Shimadzu Lab X, XRD 6000 X- Ray Difractometer. Powder diffraction patterns were obtained by applying the following conditions: CuK_{α} radiation (1.5418 Å) with 30 kV, 30 mA energy and Graphite Monochromator. Samples showed different mineralogical content were excluded. For petrographic examination, 4 x 4 cm thin sections were prepared and studied by Leica Polarizing Microscope.

The instrument used in this study to determine the drilling resistance is a Drilling Resistance Measurement System DRMS Cordless 2005 designed and built by SINT Technology within the EC Hardrock Project (SMT4 - CT96 - 2065). The machine is available at the Conservation Laboratory of the Faculty of Archaeology and Anthropology at Yarmouk University in Jordan. The operative conditions adopted in this study are those suggested by Tiano et al. (2000b) for some varieties of sandstone and limestone as follows: measuring depth: 0 – 10 mm, rotation speed: 600 rpm, penetration rate: 20 mm/min for sandstone and 10mm/min for limestone . A specially made 5 mm diamond, two-lip end mil drill bits (Diaber, Italy) were used in this study. In each sample, at least 12 holes were made (at least 2 each face) and the average drilling resistance in the depth between 2 and 9 mm was calculated. To avoid the wear effect on the drilling bit, which causes more drilling resistance, the DR of calibration materials (Artificial Reference Sample, ARS) was measured before and after conducting DR measurement on the samples, the drill bits were replaced when there was a difference between the DR of the ARS measured before and after.

DR measurements were conducted horizontally (parallel to bedding if exists), under such conditions the drilling will be only in one lithotype, i. e. no mixed lithologies, and consequently, a higher degree of correlation will be obtained.

The porosity of the studied samples was measured according to the procedures of RILEM, 1980, Tests No. I.1 as follows: After drying to a constant mass, the samples were placed in an evacuation vessel; the pressure was gradually lowered. This low pressure was maintained constant for 24 hours in order to remove the air contained in pores of the samples. Then distilled water was slowly introduced into the vessel until the samples were completely immersed. The samples were left for another 24 hours under water at atmospheric pressure. Then they were weighed separately in water (hydrostatic weight). The samples were quickly wiped with a dampened cloth and the mass of each sample saturated with water was measured.

The following formula was used to calculate the porosity:

$\varrho = (M2-M1)/(M2-M3)*100$ in Vol %

ϱ: Porosity

M1: Dry Weight

M2: Wet Weight

M3: Weight taken under water (hydrostatic weight).

3. RESULTS AND DISCUSSION

The studied samples, both sandstone and limestone, showed no significant differences concerning their mineralogical composition. The XRD pattern of the studied samples showed that the sandstone samples are composed of Quartz as a major mineral, Kaolinite as a minor mineral and traces of Calcite , while the limestone samples are composed of Calcite only (fig. 1). The petrographic examination showed that the sandstone samples are classified as quartz arenite with cementing material composed mainly of silica and the limestone samples are classified as micritic limestone.

The porosity of the studied samples varies considerably, it ranges between 2 and 22 % for sandstone and between 1 and 18 % for limestone, the same can be said about the average drilling resistance which varies between 2 and 72 N for sandstone and

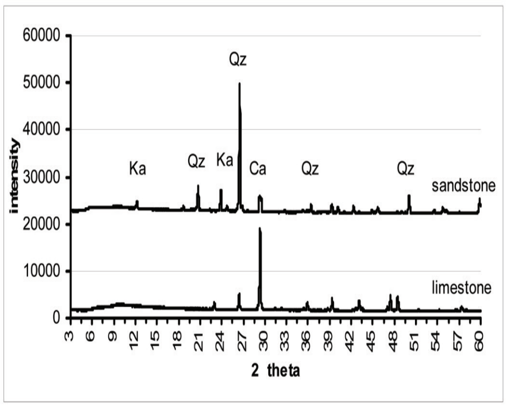

Figure 1: representative powder XRD pattern for sandstone and limestone. Qz is quartz, Ca is calcite and Ka is Kaolinite.

Table 1: Porosity (%) and average drilling resistance (N) calculated between a depth of 2 – 9 mm for sandstone (S.st) and limestone (L.st) samples.

Sample No.	Average Drilling Resistance (N)	Porosity (%)	Sample No.	Average Drilling Resistance (N)	Porosity (%)
S.st1	67.87	3.04	**L.st1**	28.0	9.0
S.st2	62.21	4.88	**L.st2**	60.0	1.6
S.st3	2.28	22.00	**L.st3**	22.0	11.2
S.st4	40.96	11.98	**L.st4**	50.0	3.2
S.st5	50.99	8.03	**L.st5**	3.5	16.5
S.st6	50.00	8.00	**L.st6**	14.5	12.5
S.st7	72.06	2.25	**L.st7**	40.0	6.2
S.st8	13.58	17.20	**L.st8**	64.0	1.1
S.st9	33.01	10.40	**L.st9**	8.3	15.4
S.st10	35.34	13.50	**L.st10**	18.0	11.1
S.st11	52.00	7.70	**L.st11**	30.0	8.1
S.st12	4.20	20.20	**L.st12**	49.0	2.8
S.st13	7.30	18.50	**L.st13**	25.0	10.0
S.st14	11.50	17.30	**L.st14**	39.0	5.9
S.st15	5.10	21.00	**L.st15**	20.0	10.8
S.st16	20.00	16.20	**L.st16**	45.0	4.5
S.st17	58.30	6.40	**L.st17**	58.0	1.8
S.st18	65.00	3.50	**L.st18**	12.0	13.6
S.st19	39.80	12.20	**L.st19**	35.0	7.0
S.st20	30.50	14.10	**L.st20**	1.7	17.8

between 2 and 64 N for limestone (Table 1). The correlation coefficient between the porosity and the average drilling resistance was calculated, it had a very high negative value equals to - 0.99 for both sandstone and limestone (fig. 2). Therefore, it can be concluded that as porosity increases drilling resistance of sandstone decreases. The relationship between the two parameters can be given as in equation 1 for sandstone and in equation 2 for limestone:

$P = 21.5 - 0.27\ DR$ with $R^2 = 0.98$ (1)

$P = 16.86 - 0.27\ DR$ with $R^2 = 0.98$ (2)

Where P is the porosity (%), DR is the drilling resistance (N) and R^2 is the Coefficient of Determination.

In the case of sandstone, for DR in the range between 2 and 80 N, the relationship between DR and the porosity is linear. Equation 1 is valid only for sandstone samples having a DR less than 79 N, applying this equation for sandstone with

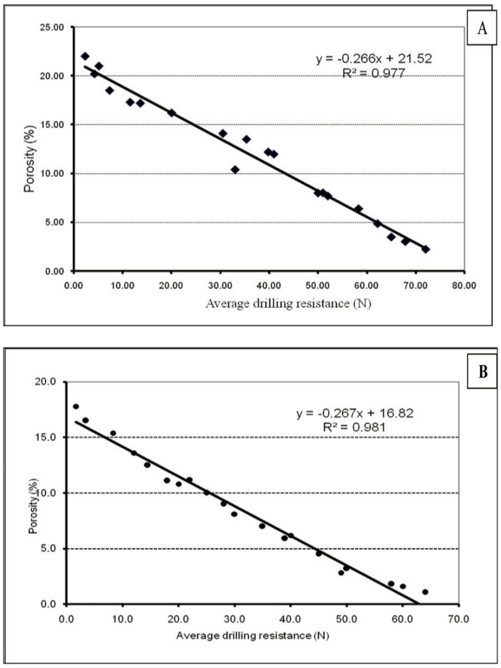

Figure 2: Scatter diagram showing the reverse linear relationship between porosity (%) and average DR (N) for sandstone (a) and limestone (b).

higher DR will give a negative value for porosity, which is not possible. The maximum porosity of sandstone that can be estimated by this equation is about 21 %. While in the case of limestone, for DR in the range between about 2 and 60 N, the relationship between DR and the porosity is linear. Equation 2 is valid only if the limestone has DR less than 63 N, applying this equation for limestone with higher DR will result a negative value for the porosity. The maximum porosity of limestone that can be estimated by this equation is about 17 %.

To test the validity of these equations, the drilling resistance of other sandstone and limestone samples, with compositions similar to those of the samples used to draw the calibration curve, was measured. Their porosities were calculated by applying equations 1 and 2. After that the porosities have been measured and the results have been compared. The results are shown in Table 2, from which it can be seen that the difference between the calculated and measured porosities is less than 10 %.

Sample No.	Average DR (N)	Calculated Porosity (%)	Measured Porosity (%)	Difference % between calculated and measured porosity
Ts.st 1	42.3	10.3	11.1	8.1
Ts.st 2	56.8	6.4	6.1	4.9
Ts.st 3	12.3	18.2	17.8	2.5
Ts.st 4	27.1	14.3	15.1	5.5
Ts.st 5	18.5	16.6	17.1	3.0
T.l.st.1	15.1	12.8	11.7	8.5
T.l.st.2	3.2	16.0	16.3	2.1
T.l.st.3	10.3	14.1	15.2	8.0
T.l.st.4	16.1	12.5	11.3	9.8
T.l.st.5	8.3	14.6	13.5	7.6

Table 2: DR, porosity calculated by applying equation 1 for sandstone and equation 2 for limestone and measured porosity.

3. CONCLUSION

The following general conclusions may be drawn on the use of DR to estimate the porosity of sandstone and limestone:

1. The porosity of sandstone and limestone can be estimated by measuring their DR especially where immersion testing is not possible. The difference between the porosity calculated from the DR and the porosity measured by applying the conventional methods is less than 10%.
2. By applying the conventional methods to measure the porosity, only the bulk porosity, i. e. the porosity of the whole sample, can be measured, if there is differences in the porosity within the sample itself, these differences cannot be seen, while by application of DR

measurement, any differences in the porosity within a sample can be detected.

3. To apply DR measurement to estimate the porosity of a stone, a calibration curve for DR vs. porosity is needed. This calibration curve is valid only for stones with similar mineralogical composition-petrography. i. e. each lithotype needs its own calibration curve., we believe that a calibration curve made for limestone cannot be used for another type of stone such as travertine, although both have the same mineralogical composition. The operative conditions, i. e. penetration rate, revolution speed and the type of drill bit, applied to measure the DR of samples should be similar to those applied to draw the calibration curve; otherwise, correction is needed.
4. DR measurement can estimate the total porosity, but gives no idea about the pore size distribution of the studied samples.
5. The porosity measured by RILEM, 1980, Tests No. I.1, is the water accessible porosity, i. e. the isolated pores are not considered. Therefore, the presence of a high portion of such pores will decrease the accuracy of the porosity calculated from the DR, because this type of pores contributes to the decrease of the DR, but is not measurable by the conventional method.
6. Due to the fact that the application of DR measurement to estimate the porosity of a stone needs a calibration curve, it can be said that this method is feasible in a case where the porosity of a large number of samples from a lithotype is needed to be measured, but only limited samples are available for such a measurement.

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