POROZITATEA, PERMEABILITATEA ŞI DENSITATEA ÎN VRAC A ROCILOR ŞI RELAȚIILE ACESTORA BAZATE PE MĂSURĂTORI DE LABORATOR
POROSITY, PERMEABILITY AND BULK DENSITY OF ROCKS AND THEIR RELATIONSHIPS BASED ON LABORATORY MEASUREMENTS

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Physical properties of rocks are measured and analyzed, and their relationships are discussed in this paper. Permeability and mercury porosimetry methods, porosity, and pore size distribution are determined. Furthermore, bulk and particle densities of rocks are determined. The morphology of the porous medium has been approached by mercury porosimetry which gives an appearance to the pore distribution of the material. The permeability of a variety of natural materials is characterized using a relatively new laboratory apparatus. Permeability and porosity are in close relation, and it could be assumed that its relationship is linear, i.e., with increasing porosity, permeability increases as well. This relationship is influenced by other rock properties, such as the amount of open and closed pores within the rock sample, size, and distribution of pores. From this point of view, it is necessary to study these physical properties of natural materials as well, because this enables an overall analysis of rocks and their possible use for construction.

Keywords: *rocks, permeability, porosity, mercury porosimetry, bulk density, TinyPerm II*

1. Introduction

The fluid transport into porous materials is an area of study relevant to many scientific and engineering fields such as hydrogeology, physics, and geo-environmental, petroleum, and chemical engineering. Knowledge of permeability and porosity is critical to accurately predicting fluid transport. As a result, there is a great interest among scientists and engineers in quantifying permeability characteristics, porosity of natural and manmade materials for many practical applications related to oil extraction, groundwater flow and contaminant transport [1,2].

Permeability and porosity are two of the primary properties that control the movement and storage of fluids in rocks. They represent an important characteristic of materials. On the basis of the known permeability and porosity, possible influences of water on an engineering construction are considered.

Furthermore, knowledge of permeability and porosity is necessary at water leakages, at the structural foundation in order to evaluate affluent to a foundation pit, and in terms of the design of waterproofing of buildings. Permeability and porosity are also very important indicators for the utilization of various kinds of rocks [3].

Mainly calcarenite rocks are used for various purposes in the building industry, for the renovation of historical monuments, stonework, and sculptures, etc. The use of marbles and granites is related to the architecture: floor coverings and facades, decorated as columns, and balusters; but also with planning the manufacture of street furniture: benches. Permeability and porosity have an impact on rock weathering, which affects the field of engineering utilization.

Permeability is one of the rock properties that are necessary for considering the solving of hydrological and hydrogeological problems by methods of numerical and physical modelling [4, 5].

Devices such as surface gas permeameters provide reliable measurements and may be used to determine other material properties. These permeameters, however, are only capable of material testing at single points in time, and establishing a large, high density map can be extremely time consuming. As a result, it would be both critical and helpful to know the extent (minimum distance) between sampling points needed to “accurately” predict material properties for natural and manmade materials with varying levels of heterogeneity.

A surface permeability probing method, developed by Valek, et al. [6], demonstrated the applicability of using a surface gas permeameter for historic conservation including: the weathering and decay process associated with masonry materials, characterization of existing materials to

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seek compatible replacement material, and investigation of the carbonation process in lime mortars in historic and modern masonry. While this method was highlighted as being non-destructive and able to measure a wide range of permeability values, it was found that measuring low permeability material was seldom non-destructive.

In this paper, we characterize the permeability of a variety of natural materials using a relatively new laboratory apparatus. The apparatus is unique in that it is non-destructive and capable of measuring a wide range of surface gas permeability on building materials. Discussion of the laboratory methods used for measuring permeability and porosity and their relationship is carried out in this paper.

2. Materials and methods

The study includes 13 varieties of building stone extracted from different regions of Morocco, deliberately varied. The geographical distribution of selected samples is shown in Figure 1. Measurements of permeability and porosity are performed on 13 samples. These measures will help to better understand and identify the characteristics of the porous network of materials used in the construction.

Porosity of porous medium describes the fraction of the void space in the rock, where the voids may contain air or water. The porosity is defined as the ratio of the volume of voids expressed as a percentage of the total (bulk) volume of a rock, including the solid and void components. Porosity is calculated from the derived formula:

$$N = \left(1 - \frac{\rho_d}{\rho}\right) \times 100 \tag{1}$$

where ρ_d is bulk density of the dry specimen and ρ is particle density.

Bulk density can be determined from a regular specimen by a stereo-metric method. Our tests are carried out on 13 samples. Particle density, an average mass per unit volume of the solid particles in a rock sample, is usually determined by applying the mercury intrusion [7]

Permeability and porosity depend on pores in the rock. There are two discerned typologies of pores in rocks: closed and open pores. Closed pores are completely isolated from the external surface, not allowing the access of external fluids in either the liquid or gaseous phase. Closed pores influence parameters such as density and mechanical and thermal properties. Open pores are connected to the external surface and are therefore accessible to fluids, depending on the pore characteristics/size and the nature of fluid. Open pores can be further divided into dead-end or interconnected pores. The percentage of interconnected pores within the rock is known as effective porosity. Effective porosity excludes isolated pores and the pore volume occupied by water that is adsorbed on clay minerals or other grains. Total porosity, determined from formula (1), is the total void space in the rock, whether or not it contributes to fluid flow.

Effective porosity is typically less than total porosity. The character of porosity alters with the genesis of rocks and strongly determines its physical properties, e.g., permeability, adsorption properties, mechanical strength, or durability. On

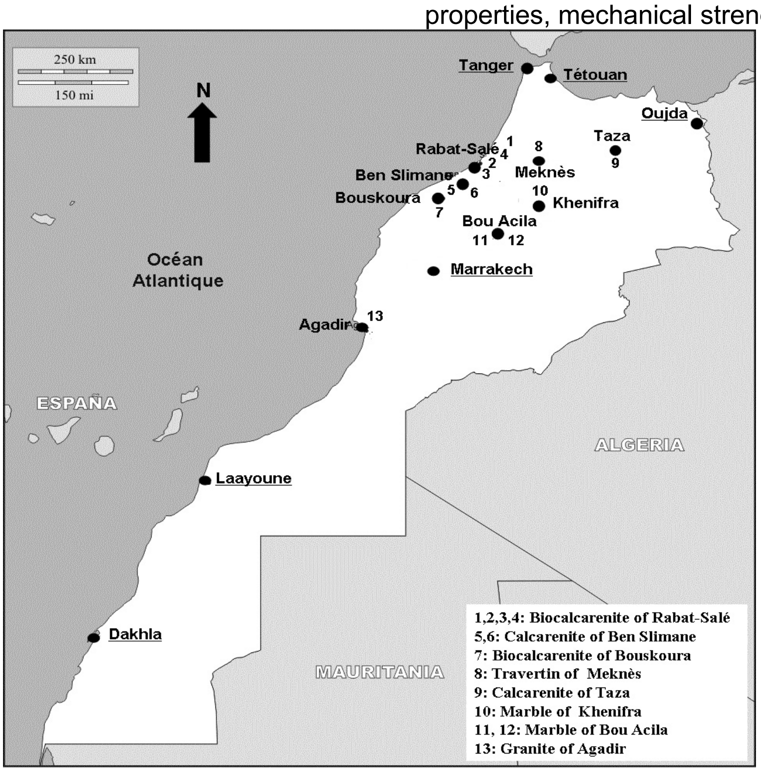

Fig. 1 - Geographical distribution of studied rocks.

the basis of the known character of porosity, predicting rock behaviour under different environmental conditions and its usage are considered.

One of the most important parameters is the pore size and pore size distribution. Pores are classified according to four groups depending on the access size: micropores, with a size less than 2 nm diameter; mesopores, ranging between 2 and 50 nm diameter; macropores, which are in range from 50 nm to 7500 nm diameter and rough pores in size over 7500 nm [8].

2.1. Mercury porosimetry

This technique consists in injecting the non-wetting fluid (mercury), under various pressures in previously desiccated and degassed samples [9]. It allows porosity and pore size to be estimated by measuring the volume of injected mercury and the injection pressure. The applied pressure is connected to the threshold access of the pore by the Young- Laplace equation:

$$P = \frac{2\sigma\cos\varphi}{R} \qquad (2)$$

where P [Pa] is an actual pressure, R [nm] half-length distance of two opposite walls of a pore expressed by an effective radius, σ surface tension of mercury [$480 \cdot 10^{-3}$ $N \cdot m^{-1}$] and φ contact angle [141.3°].

These porosimetry measurements are performed using an apparatus Micromeritics Pore Sizer 9320 which makes it possible to inject mercury with pressures ranging between 0.001 and 300 MPa. So the access threshold ranges between 400 and 0.003 µm. This technique determines the connected porous volume and its distribution according to the injection pressure and the thresholds access.

Cylindrical samples, of 25 mm length and 20 mm in diameter, are dried at 60°C, weighed and placed in an injection cell. After a degasification step under a 50 µm mercury depression, the injection cell is filled with mercury, and then the vacuum is broken gradually until atmospheric pressure. The intrusion measurement, i.e., the volume of mercury injected into the sample, is made for low pressures (between 0.001 and 0.15 MPa) and for high pressures (between 0.15 and 300 MPa). The pressure rises are carried out in stages; after each stage, the injected mercury volume is measured. From these data, it is possible to determine the saturation curve according to the injection pressure.

2.2. Permeability

We have used a relatively new surface gas permeameter for making gas permeability measurements on the surface of the substrate specimens described above. The permeameter, TinyPerm-II, made by New England Research, Inc.. It is a unique hand-held device that can characterize the permeability of rocks and soils or the effective hydraulic aperture of fractures in situ on outcrops as well as on laboratory specimens [10]. The apparatus is capable of making permeability measurements ranging from 0.01 to 10 darcies for matrices and 10 µm to 2 mm fracture apertures [10]. Although the measurements could have been made in the field, all measurements reported here are made on the specimens in a laboratory.

This device uses Darcy's law to compute the permeability. Brown and Smith [11] show the permeability can be determined using the following relationship:

$$\frac{Q}{P_0} = -\frac{kA}{\mu L} \qquad (3)$$

where Q is the net air flow into the piston syringe, P_0 is the applied pressure which remains constant, A is the inlet area for air flow, µ is the viscosity of the gas (air), and L is the length. Since the values of A, L and µ are known; and Q and P_0 are measured, equation (3) may be solved for permeability, k.

The testing procedure is straightforward; the operator presses a rubber nozzle against the specimen and withdraws air from it with a single stroke of a syringe. As air is pulled from the sample, a microcontroller unit simultaneously monitors the syringe volume and the transient vacuum pulse is created at the sample surface. Using signal processing algorithms, the micro-controller computes the response function of the sample/instrument system. Key characteristics of this response are displayed on the liquid crystal display (LCD) screen. Theory shows a relationship between the response function and permeability; and either matrix permeability or effective fracture flow aperture may be determined from the calibration charts and tables provided [10, 11].

The response function is related to permeability K:

$$T = -0.8206\log_{10}(K) + 12.8737 \qquad (4)$$

where T is the value of the response function and the recorded output from the mini-permeameter. For each sample point 3–6 measurements are made in order to ensure the quality of the data. For each sample point, an average of the measurements is calculated and used as the representative value for that point.

In total, 13 various rock samples are tested, and values of the permeability, particle and bulk densities, and porosity are listed in Table 1. The values of the presented rock properties are predominantly determined as an arithmetic average of rock specimen tests. For each specimen, the permeability, particle, and bulk

Table 1

Physical properties of rocks

Rock class	Types of rock	Location	Rock code	Permeability K (m^2)	Bulk density ρ_d (g/cm^3)	Particle density ρ (g/cm^3)	Porosity N (%)
Sedimentary	Biocalcarenite	Rabat-Salé	1	2.28E-11	1.64	2.38	31.07
	Biocalcarenite	Rabat-Salé	2	3.34E-11	1.59	2.38	33.15
	Biocalcarenite	Rabat-Salé	3	1.74E-11	1.68	2.39	29.82
	Biocalcarenite	Rabat-Salé	4	5.04E-11	1.60	2.49	35.83
	Calcarenite	Ben Slimane	5	2.2E-15	2.45	2.72	9.84
	Calcarenite	Ben Slimane	6	1.8E-15	2.55	2.76	7.61
	Biocalcarenite	Bouskoura	7	1.46E-11	2.25	2.81	19.89
	Travertin	Meknès	8	3.13E-16	2.34	2.47	5.26
	Calcarenite	Taza	9	8.01E-14	2.54	2.96	14.08
Metamorphic	Marble (Black)	Khenifra	10	1.24E-19	2.71	2.72	0.3
	Marble	Bou Acila	11	3.72E-18	2.72	2.73	0.39
	Marble	Bou Acila	12	2.14E-18	2.74	2.75	0.26
Magmatic	Granite	Agadir	13	5.08E-17	2.69	2.71	0.59

densities are measured, and porosity calculated. The morphology of the porous medium has been approached by mercury porosimetry which gives an appearance to the pore distribution of the material. The samples have a sufficient volume to be representative of the material. The results are shown in Figures 2, 3, 4 and Table 2.

3. Results and discussion

The results of the permeability tests are analysed using the method of least-squares regression. The correlation coefficient (R^2) is determined for this regression.

The permeability is correlated with porosity. The plot indicating the correlation is shown in Figure 2. It is seen that there is a linear relation between permeability and porosity. The correlation coefficient is acceptably high, suggesting a sufficient correlation between the two variables for engineering use.

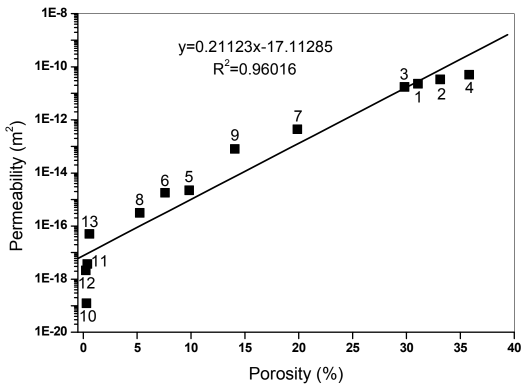

Fig. 2 - Relationship between permeability and porosity of rocks.

Permeability and porosity are in a close relationship that depends on the amount of the void space in the tested material. It is widely accepted that permeability is determined by microstructure, which is, in this context, defined in terms of pore and crack structures. So it could be supposed that with increasing porosity, the permeability should increase as well. But there are some other facts to note when speaking about this relationship. Therefore, permeability of porous material is influenced not only by porosity, but also by shape and the arrangement of pores, or by the amount of clayey component [8, 12].

Firstly, it is necessary to distinguish between total and effective porosity. We are not able to make assumptions on permeability of tested material from values of total porosity, due to the fact that it is the total void space in the rock. A rock may be highly porous, but if the voids are not interconnected, fluids within the closed (isolated) pores cannot leak.

Secondly, pore size distribution is important. To clarify the relationship between permeability and porosity, pore size and pore size distribution are determined for selected rock samples. Pore dimensions cover a very wide range. Within our research, two samples of calcarenite, which have approximately the same order values of permeability, but different porosities, are tested by mercury porosimetry for pore size distribution. Results are shown in Figures 3 and 4.

As we can see from Figures 3 and 4, sample 2 has a more uniform distribution in the range of pore size. The rough pores of this sample should not exceed 70% in total. It has a very broad spectrum of porosity with significant mesoporosity (pore diameter access between 1 and 100 μm). The prevailing part of pores belongs to the rough pores, which can create main transporting ways for liquid. In the case of sample 7, the distribution of pore size is different. The rough pores of this sample about 30 % in total. The dominant part of pore belongs to the mesoporosity, and microporosity, which is also very important.

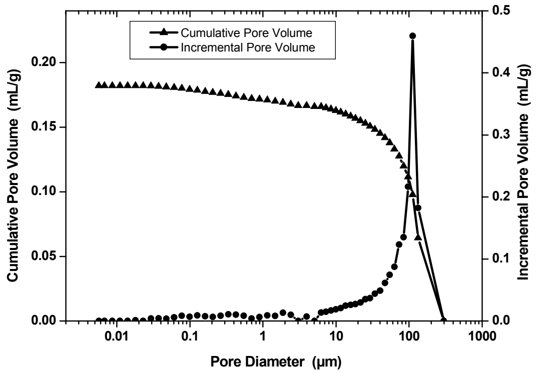

Fig. 3 - Pore size distribution of calcarenite from Rabat-Salé (sample 2).

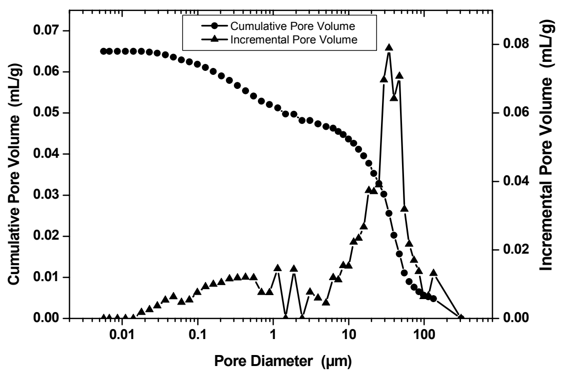

Fig. 4 - Pore size distribution of calcarenite from Bouskoura (sample 7).

The distribution of pore volumes of sample 2 is unimodal (a single dominant family of pores resulting in a single point of inflection in the curve of injection). It is possible to determine a radius access or threshold pore: it corresponds to the radius access of pore that to a low pressure increment provides access to a large pore volume. It is graphically determined on the first injection curve as the radius corresponding to the inflection point of the curve or by the method of tangents [7, 13]. The threshold pore can also be viewed on the curves representing the increment of mercury injected for each access radius. The injection curves with multiple turning points illustrate

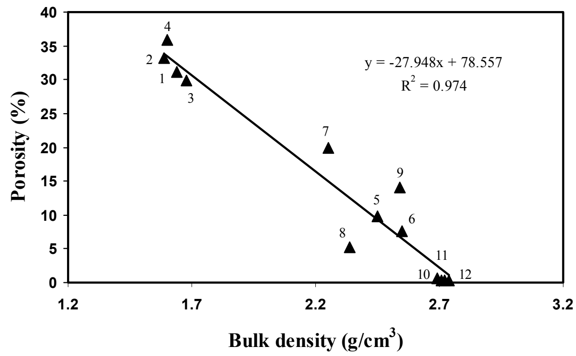

Fig. 5 - Relationship between bulk density and porosity of rocks.

multimodal porous networks where several families ray access to the pores coexists.

Average pore diameter is usually used as a representative parameter of the pore size distribution. In case of sample 2, average pore size diameter is 1.96 µm; for sample 7, average pore size diameter is 0.57 µm (Table 2).

It is well known that mineral admixtures affect permeability. The basis for this effect can be understood in terms of the formation of a large amount of porosity in the mesopore range. This assertion requires further experimental verification.

Finally, there are some other characteristics of samples we have observed, such as the relationship between bulk density and porosity. We have obtained the generally known fact from rock samples. The relationship between bulk density and porosity of all the 13 tested samples can be seen in Figure 5. We can clearly identify that with decreasing bulk density, the porosity of the sample increases. It is due to the small differences in particle densities, which are not dependent on porosity, but only on modal composition. The modal compositions of calcarenite samples are assumed to be approximately the same.

The relationship between bulk density and porosity of all tested samples is shown in Figure 5.

Calcarenite has high porosity. The permeability of the rocks above depends on the structured nature of minerals forming the solid matrix. The permeability of these rocks is in the range of 10^{-11}.

Tested samples of granite and marble are fine-grained rocks, so they have low porosity, and this fact causes a low permeability as well.

Table 2

Physical properties of tow samples calcarenite.

Types of rock	location	Rock code	Permeability k (m^2)	Bulk density ρ_d (g/cm^3)	Particle density ρ (g/cm^3)	Porosity N (%)	Average pore size diameter (µm)
Calcarenite	Rabat-Salé	2	3.34E-11	1.59	2.38	33.15	1.96
Calcarenite	Bouskoura	7	1.46E-11	2.25	2.81	19.89	0.57

4. Conclusion

Physical properties of 13 rock samples are measured and analyzed in an integrated manner. Laboratory measurements have been carried out on the following physical properties: permeability, porosity, pore size distributions and bulk density. The graphs of permeability against porosity have been presented. From the graphs it can be seen that, permeability of the rocks is directly proportional to porosity.

The interest of this study is focused on samples of rocks extracted from different regions of Morocco to pick up the physical properties of rocks that experienced a rarity of technical studies. It provides results on the permeability and porosity for these well-characterized rocks. The results obtained show that the permeability of porous material is influenced not only by porosity, but also by the shape and arrangement of pores, or by the amount of clayey component. Only effective porosity can influence permeability, because only open pores are interconnected and allow leaking water through. Another important factor is pore size distribution. By evaluating the relationship between porosity and permeability, it is also necessary to take into account rock bulk and particle density.

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