

LABORATORY METHODS OF DETERMINATION OF PORE
STRUCTURE PARAMETERS: FORMATION FACTOR AND
TORTUOSITY OF PERMEABLE MEDIA BY ELECTRIC METHODS

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The paper presents the methods of determination of structural parameters of porous materials: formation factor and tortuosity, based on conductometric techniques. The basic experimental procedures are reviewed and factors, which influence the relative conductivity of electrolyte saturated samples such as type of experimental technique, electrolyte and electrodes, length of sample, are presented. The role of surface conductance, polarization effects and frequency are analyzed.

1. INTRODUCTION

Due to the growing interest of engineers in natural porous materials (soils, rocks, bones, etc.) and wide applicability of artificial porous materials in different fields of technology (e.g. industrial filters, materials used in catalysis, composite porous materials, etc.) and medicine (e.g. porous implants, filtering media), there is an increasing need to develop models and methods of experimental studies of various properties of porous materials. In particular it concerns structural properties of the media and the parameters describing internal pore structure of the materials from the point of view of transport of fluids and chemicals.

This paper deals with methods of laboratory measurement of electrical properties of porous materials saturated with electrolytes, that are performed in order to determine two pore structure parameters: formation factor and tortuosity. The significance of the discussed measuring methods results from the fact that they are simple and noninvasive, and the determined structure parameters characterize not only the macroscopic electric properties but also play an essential role

in description of the transport phenomena (flow of fluid and migration of chemicals in pore fluid) as well as in modeling of dynamic processes taking place in saturated porous materials [9].

Different methods developed for determination of the formation factor or tortuosity are based on measurements of AC electrical conductivity of electrolyte materials saturated with, using signals of particular frequency or applying measurements within a range of frequencies. The studies performed for variable frequencies show that in the range from about 1 kHz to hundreds of kHz one can disregard the effects related to electrodes polarization, and that the results are free from the influence of impedance of stray and of dispersion phenomena connected with different kinds of polarization at the skeleton-electrolyte interface or in the electrolyte.

Tightly related to the conductometric methods is the technique, which is used to determine complex permittivity, impedance or admittance of porous materials in wide range of frequencies. The technique known as electric spectroscopy is particularly focused on identification of equivalent electric models of the studied materials. Such models must incorporate the role of different electrochemical effects related to composite nature of multicomponent materials. The method of electric spectroscopy is applied to evaluate the internal structure and composition of the materials. The results may be also used in determination of conductivity and structure parameters of porous materials as well as in specifying the optimum conditions: frequency, voltage, electrodes, etc. in measurements of conductivity.

Besides studies of the formation factor and tortuosity, the electrical methods are used in case of saturated porous materials in order to estimate two other structural parameters: specific surface and fractal diameter [15]. While the specific surface is evaluated through measurement of electrical conductivity of porous materials as a function of electrolyte concentration at very low concentrations, the fractal diameter is determined through the analysis of electrical response of the material in wide range of frequencies.

The aim of the present paper is to review the electrical laboratory methods of measurement of formation factor and tortuosity of porous materials as well as to analyze basic problems encountered in such studies. The discussed methods assume that the skeleton is made of dielectric material and samples are completely filled with electrolyte. In the second section basic definitions characterizing the electrical and structural properties of porous materials are given. Next, the techniques of electric measurements with application of bridge and direct methods for two-, three- and four- electrode systems are reviewed. The fourth section is concentrated on the analysis of results of conductometric studies for chosen porous materials. The factors influencing the measured conductivity, its frequency dependence, and models derived within the low frequency methods of electric spectroscopy are discussed. A comparison of different definitions

of the formation factor and tortuosity of porous materials is presented in the Appendix.

2. BASIC DEFINITIONS

The measurements of AC electric properties of electrolytes and porous materials saturated with the electrolytes are made to determine complex admittance, impedance or permittivity of the media.

- The admittance (complex conductivity), Y , is measured in the parallel mode and its components are conductance, G , and a susceptance, B , i.e. $Y = G + jB$.
- The impedance (complex resistivity), Z , is measured in the series mode while components of Z are resistance, R , and reactance, X , where $Z = R + jX$. The admittance is reciprocal of the impedance, i.e. $Y = 1/Z$.
- An alternative measure of electric (dielectric) properties of materials is permittivity, κ , (or complex dielectric constant) which is related to the complex conductivity, σ , as

$$\kappa = \frac{\sigma}{i\omega\epsilon_0},$$

where ω is angular frequency, ϵ_0 is permittivity of vacuum, $\sigma = Yt/A$, and A and t denote the cross-section and thickness of the sample, respectively.

Pore structure parameters describe the internal geometry of porous media and determine the properties which are significant for transport of fluids, migration of chemicals in the materials, as well as determine certain dynamic and thermal properties of the media. Below, the most important structural parameters of porous materials are defined assuming isotropy and homogeneity of the materials.

- The volume porosity, ϕ is defined as the ratio of the volume of pores in a representative sample to the total volume of the sample.
- The specific surface, S_w , is defined as the ratio of the surface area of the interface in the porous sample and volume of the sample.
- The permeability, k , denotes the proportionality constant in Darcy's law relating discharge (filtration) velocity, v , to the gradient of pore pressure, p , i.e.

$$(2.1) \quad v = \frac{k}{\mu} \nabla p,$$

where μ denotes dynamic viscosity of pore fluid.

- The formation factor of porous medium, FF , with a non-conducting material of skeleton is the ratio of electric conductivity of solution of electrolyte, σ , to the conductivity of porous sample saturated with the solution, σ_p ,

$$(2.2) \quad FF = \frac{\sigma}{\sigma_p}.$$

- The tortuosity, T , has different meanings in the literature concerning transport in porous media (see Appendix A) but in relation to electric methods of characterization of porous materials it is defined as the product of the formation factor and the porosity,

$$(2.3) \quad T = \phi \cdot FF.$$

3. MEASUREMENT METHODS

According to the above definitions, if volume porosity of the material is known, the determination of the formation factor and tortuosity require measurements of conductivity of electrolyte and of the porous material saturated with the electrolyte. The measurements of conductivity of saturated porous samples are performed in systems which use the RLC bridge or a direct technique. The chamber containing sample usually has a cylindrical shape and is made, if possible, from a non-conducting material (in some cases, for studies proceeding at very high pressure and temperature, the cell is made from steel). The sample is placed between electrodes made of e.g. copper, stainless steel, platinum or nickel. The systems of electrodes are composed of two, three or four elements (see Fig. 1). The two and four electrode systems apply in cases when the diameter of sample is equal to the diameter of electrodes and the measuring chamber is made from non-conducting material. The three electrode systems are used in cases when samples have diameters larger than the diameter of electrodes or when conductivity of the measuring chamber cannot be neglected.

3.1. Two-electrode system.

The two-electrode system, as the simplest one, is most frequently used in particular for studies of granular materials. The drawback of the system is that the polarization of electrodes influences the results of measurements. In the case of measurements realized in a single direction (useful for porous materials with isotropy of internal structure), the cylindrical chamber with a pair of electrodes is applied (see Fig. 2), e.g. [19].

In studies of materials with anisotropic pore structure, the two-electrode measurements can be applied in a setup with a chamber containing a few pairs of electrodes enabling measurements in different directions (see Fig. 3), [12].

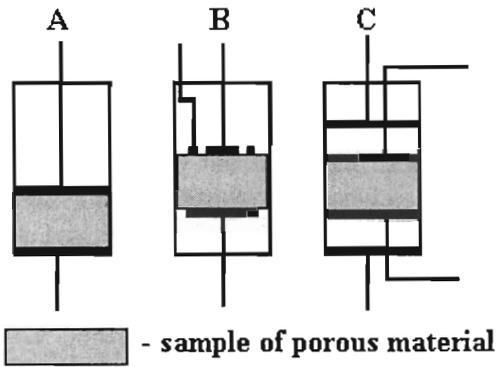


FIG. 1. Configurations of electrodes in studies of electric properties of porous materials. A: two-electrode system, B: three-electrode system, C: four-electrode system.

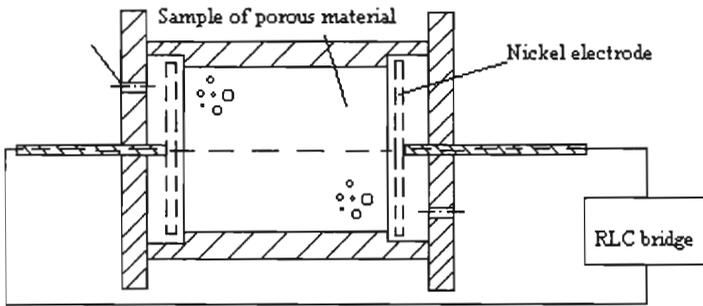


FIG. 2. Measuring chamber with two-electrode system, [19].

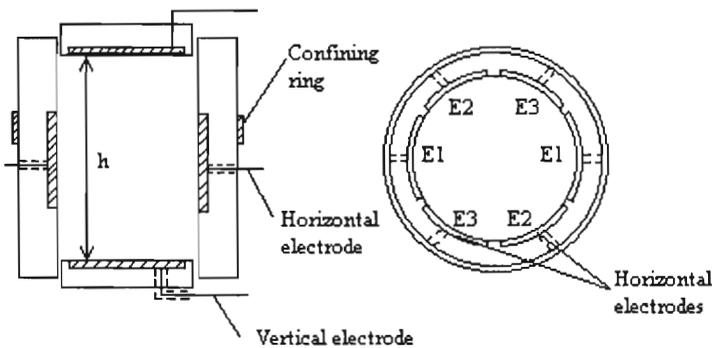


FIG. 3. Measuring system designed for studies of anisotropic porous materials.

3.2. Three-electrode system.

The three-electrode system is mostly applied when the size of electrodes is smaller than the diameter of sample or when the material of the chamber is conducting electric current. The external electrode is the screen one which guarantees that the lines of an electric field between the measuring electrodes are parallel (see Fig. 4). While the polarization of electrodes does influence the result of measurement, the advantage of the three-electrode system is the independence of the size of electrodes on the dimensions of samples (the latter may not be smaller than the external diameter of electrodes).

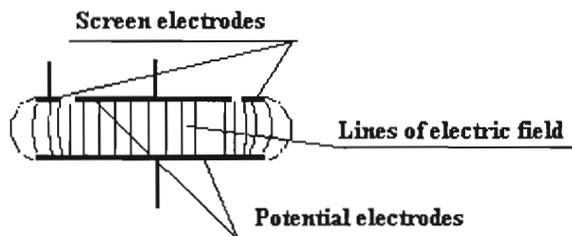


FIG. 4. Schematic of lines of electric field in the three-electrode system.

3.3. Four-electrode system.

The four-electrode system is used to separate the regions close to electrodes where electrochemical effects of polarization of electrodes takes place from the body of saturated porous material [6]. The current (external) and potential (internal) electrodes are separated by a layer of porous material (ceramic) in which polarization regions are contained (see Fig. 5). Diameters of both pairs of the electrodes are equal to the diameter of sample. While the current electrodes could be made from non-permeable material, the potential electrodes should be permeable for ions.

A variance of the four-electrode system consists of the two current electrodes and a pair of rods (see Fig. 6). Such system has been applied for measurement of electric parameters of unconsolidated clays, [1].

3.4. Measurement of conductivity of electrolyte.

In order to determine the conductivity of electrolyte used in studies of porous materials, one can apply a measuring chamber used for testing of porous materials. However, the standard measurements of conductivity of electrolytes used in electrochemistry are performed with a special cell shown in Fig. 7. It is worth

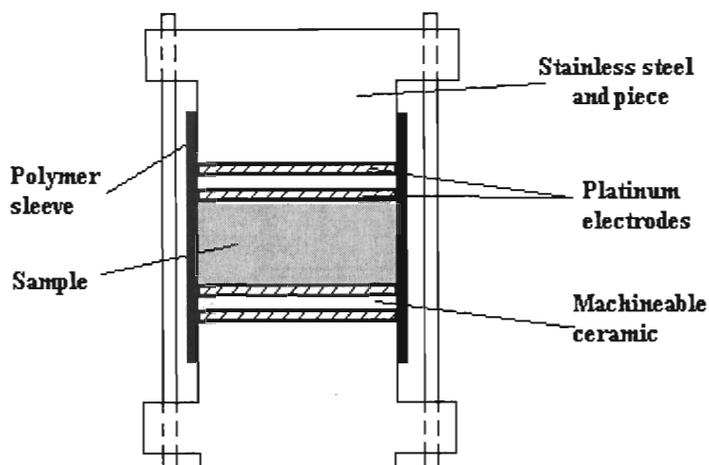


FIG. 5. Measuring chamber with four-electrode system.

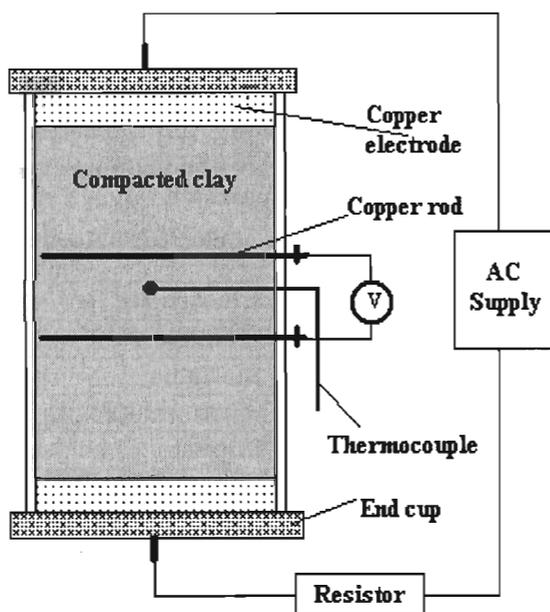


FIG. 6. Measuring chamber designed for studies of compacted clays.

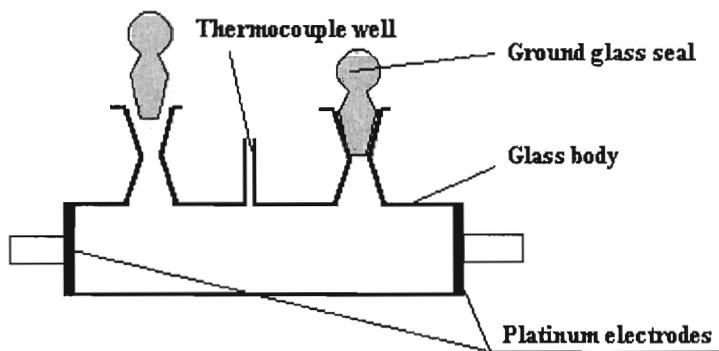


FIG. 7. Standard measuring cell for determination of conductivity of electrolyte.

noticing that in the first case the immediate correction of measurement with respect to the geometry of the cell is realized.

3.5. Electrolytes and electrodes.

The selection of electrolyte and electrodes in studies of conductivity of electrolyte saturated porous material must take into consideration two types of polarization: polarization at the interface between fluid and solid and the electrode polarization. The analysis of interaction of electrolytes with mineral materials and with typical materials of electrodes [e.g. 17] indicates the electrolyte and material of electrode selection for relatively good couples, see Table 1.

Table 1. Recommended materials of electrode-electrolyte systems

Electrolyte	Electrodes
Potassium chloride (KCl)	Stainless steel
Sodium chloride (NaCl)	Platinum
Silver nitrate (AgNO ₃)	Silver plated with silver chloride
Cupric sulphate (CuSO ₄)	Copper

3.6. Experimental setup.

The application of the two-electrode system allows one to utilize in measurements of components of the complex impedance or admittance the RLC bridge (see Fig. 8A). The other electrode systems require application of direct methods, which in some cases are also used with the two-electrode system (see Fig. 8B). Along with computerization of measuring devices, the conductometric techniques

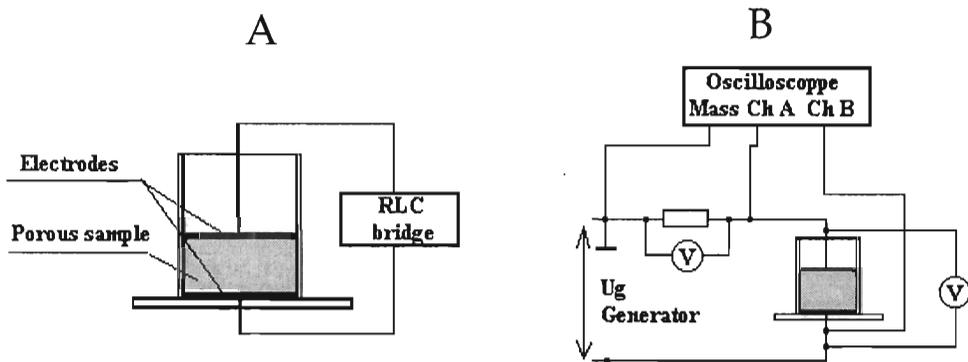


FIG. 8. Measurement setup.

are frequently equipped with AD cards, which makes the direct methods more flexible than application of the RLC bridge.

4. ANALYSIS OF RESULTS

4.1. Factors influencing measurements of conductivity of electrolyte-saturated material.

Determination of the formation factor and tortuosity based on measurements of electric conductivity is possible in cases when the total conductivity of electrolyte-saturated sample is controlled by ionic conductivity of the electrolyte filling the pore volume e.g. [6]. It takes place for materials with a non-conducting skeleton and relatively high concentration of electrolytes. At higher concentrations of electrolytes, the conductivity of saturated porous samples is proportional to the conductivity of electrolyte and, as the result, to the concentration. For the case of low concentration, the conductivity of a sample filled with the electrolyte is usually a non-linear function of the conductivity of electrolyte and for very low concentration it approaches the characteristic for the given material value. Such results are explained by effects of sorption of ions on the interface and the resultant contribution of surface conductivity in the total conductivity of porous materials. The example of the visible influence of surface effects (conductivity) on the total conductance of electrolyte-saturated glass beads with different average size of grains is shown in Fig. 9 [11].

The other two factors that must be taken into account in the conductometry and influence accuracy of the measurement of structure parameters of porous materials are: the electrode polarization (significant particularly for frequencies below 1000 Hz), and the charge buildup on the relatively non-conducting surfaces of the porous skeleton – “Maxwell-Wagner” effect (it occurs at frequencies

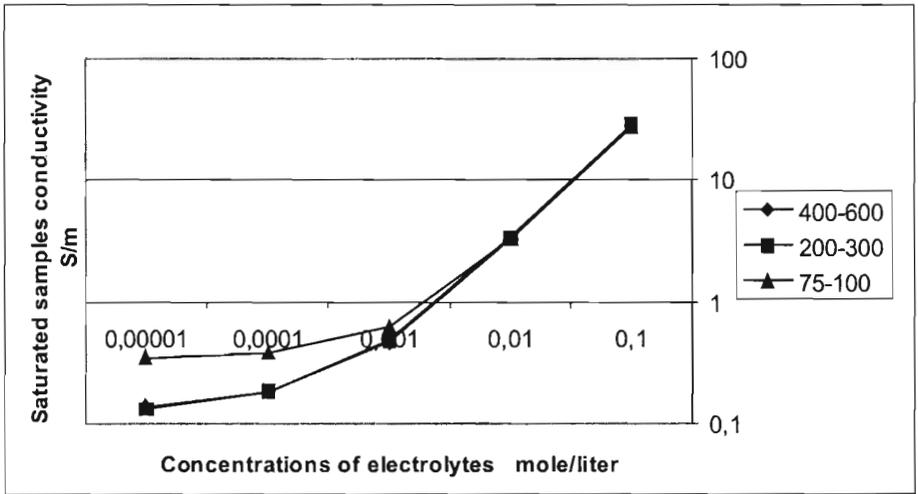


FIG. 9. Electrical conductivity of porous material made from three different glass beads saturated with solution of KCl versus concentrations.

between 100 Hz and 1 MHz) [7]. The electrode polarization is a set of complex electrochemical effects caused by electron-ion charge transfer at the electrode-electrolyte interface. While in the case of the four-electrode system the electrode polarization can be disregarded since the polarization does not appear at the potential electrodes, the measurements with the two- and three-electrode systems must take it into account and if necessary, should be corrected for the polarization e.g. [12].

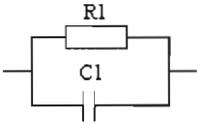
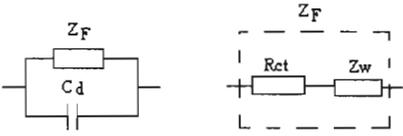
Yet another factor influencing conductivity of the saturated porous materials is the surface topography of skeleton (grains) related to the roughness of the internal surface of the skeleton and influencing the motion of ions in the pore space.

4.2. Models of electrolyte - saturated porous materials.

In general, two types of models are considered to describe the behavior of materials studied by electric methods: the equivalent electrical models and empirical models. The first ones are electric circuits composed of resistors and capacitors for which the electric response corresponds to the observed behavior of saturated porous samples. The elements of the systems are identified with certain properties of measured samples being important in ionic conduction. The empirical models are mathematical formulae, which can predict complex behavior of the studied materials (electric properties) without referring to a particular physical model.

The system composed of the porous sample saturated with electrolyte along with the attached electrodes is usually modeled as a series connection of the impedance of the sample (material) and the impedance of the near-electrode layers. The two most frequently considered models of the near-electrode layer are shown in Table 2.

Table 2. Equivalent electric models of the near-electrode layer

Equivalent electric model of the near-electrode layer	Reference
 <p style="margin-left: 40px;"> $R1$ - effective resistance for the overall electrode reaction; $C1$ - Double layer capacity of the electrode </p>	<p>J. E. Bauerle, (1969),</p>
 <p style="margin-left: 40px;"> Zf - Faradaic impedance Cd - capacitance of the electrode/electrolyte solution; Rct - the resistance of charge transfer; Zw - Warburg impedance </p>	<p>M. Shi <i>et al.</i>, (1999),</p>

The first model represents a parallel connection of the effective resistance of the set of electro-chemical near-electrode reactions and the capacitor corresponding to the double electric (near-electrode) layer. The second model includes, in addition in series with the resistor, the diffusive impedance (Warburg impedance)[18].

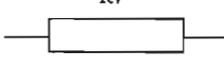
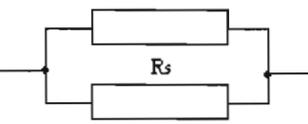
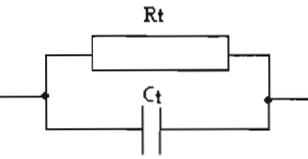
The impedance of the material due to differences in physical properties of the solid skeleton and electrolyte and various range of considered frequencies is modeled in a number of ways through equivalent electric models or empirical models, see Table 3. The models may take into account both the volumetric and surface conductivity as well as different polarization mechanisms.

4.3. Experimental data - examples.

In what follows, a set of representative results of experimental studies performed in order to determine the pore structure parameters: formation factor or tortuosity for different porous materials are discussed. The analysis is concentrated on main physical and technical factors, that influence the measured properties of porous materials including possible sources of inaccuracy of the measurements of structure parameters.

In Fig. 10 the conductivity of saturated Danish beach sand (SiO_2) with the average grains diameter $250 \mu m$ as a function of frequency is presented, NET-

Table 3. Equivalent electric models of porous material filled with electrolyte

Equivalent electrical model of saturated porous sample	Mathematical formula for impedance	Reference
	$Z_m = R_v$ where: R_v – volume resistance of electrolyte filling a pore space of porous material	Archie (1942)
	$Z_m = (R_v * R_s) / (R_v + R_s)$ where: R_v – volume resistance of porous material filled with electrolyte; R_s – surface resistance of ions existing on material/electrolyte interface	P. Glover <i>et al.</i> (1994)
 Debye model	$Z_m = R_t + X_c$ where: R_t – surface and volume resistance of porous material filled with electrolyte; C_t – capacitance of Maxwell-Wagner effect	C. Ruffet <i>et al.</i> (1991)
Cole-Cole model	$\rho(\omega) = \frac{\rho_o}{1 + (i\omega\rho_o\varepsilon_c)^{1-q}}$	C. Ruffet <i>et al.</i> (1991)

TELBLAD *et al.* 1995. The electrolyte is water solution of NaCl with conductivity 63 [mS/m]. The conductivity of the material is almost constant in the range of frequencies from 10^2 to 10^6 [Hz]. The lower value of conductivity for lower frequencies is assigned to the diffusion effect and polarization, while the higher value of conductivity for higher frequencies ($>10^6$ Hz) is explained by inaccuracy of measurements [13].

In Fig. 11 the conductivity of Danish beach sand of two samples with different thickness is shown as a function of conductivity of electrolyte (water solution of NaCl) that fills the porous material [13]. Except the results corresponding to very low concentration, the obtained data exhibit a linear dependence of conductivity of samples on conductivity of the electrolyte. Additionally, it is visible that the thickness of the specimens has no major influence on measured conductivities of the specimens.

In Fig. 12 the dependence of the formation factor on the porosity of specimens of Danish beach sand with different grain size distribution is presented. The obtained dependence is in good agreement in the whole range of porosity with empirically established Archie's law, $FF = \phi^{-m}$. This law relates the formation

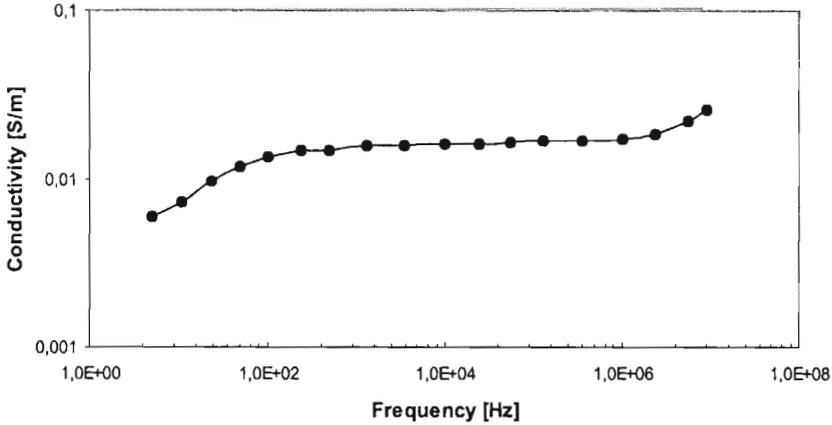


FIG. 10. Conductivity of saturated Danish beach sand as a function of frequency.

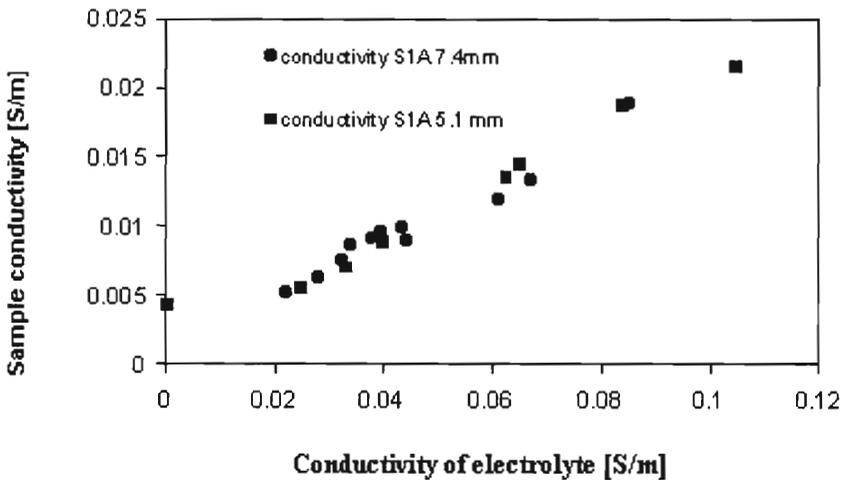


FIG. 11. Conductivity of saturated Danish beach sands as a function of water conductivity.

factor and porosity through cementation exponent, m , which for the results shown in Fig. 12 is close to 1.

Experimental studies show that the tortuosity significantly depends on the grain size distribution of porous materials, represented by the standard deviation of the size of grain. Sinuosity of the pore space increases with wider distribution of size of solid particles, and the example of such dependence for Danish beach sand is shown in Fig. 13

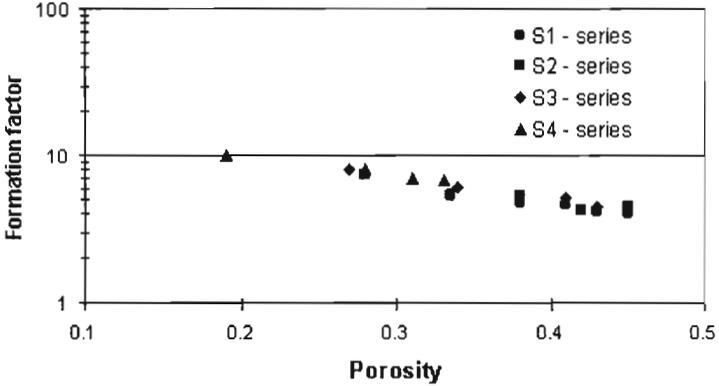


FIG. 12. The formation factor of Danish beach sand as a function of porosity.

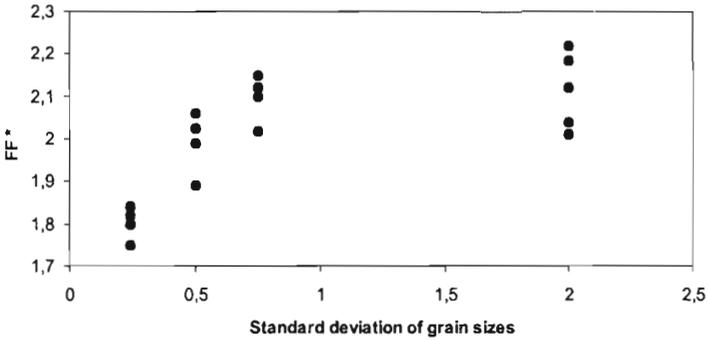


FIG. 13. The tortuosity of Danish beach sand as a function of the standard deviation of grain size distribution.

The conductivity of saturated porous materials is in general influenced by surface effects – sorption and surface conductivity. The example of such dependence for measurements done with constant frequency 1 kHz is shown in Fig. 14 [7].

It results from Fig. 14 that for the lower range of concentrations, the conductivity of material saturated with electrolyte is no longer proportional to the conductivity of pore fluid but approaches a characteristic value for the given material, which represents the surface conduction. The effect of surface conduction is present in the whole range of concentrations of electrolyte, but its influence on the values of total conductivity of the sample is significant only for low concentrations of electrolyte. The surface conductivity is caused by high mobility of ions concentrated in the Gouy-Chapman double layer.

In cases when the volume conduction of saturated porous material does not dominate over the surface conduction, the relative conductance (the ratio of

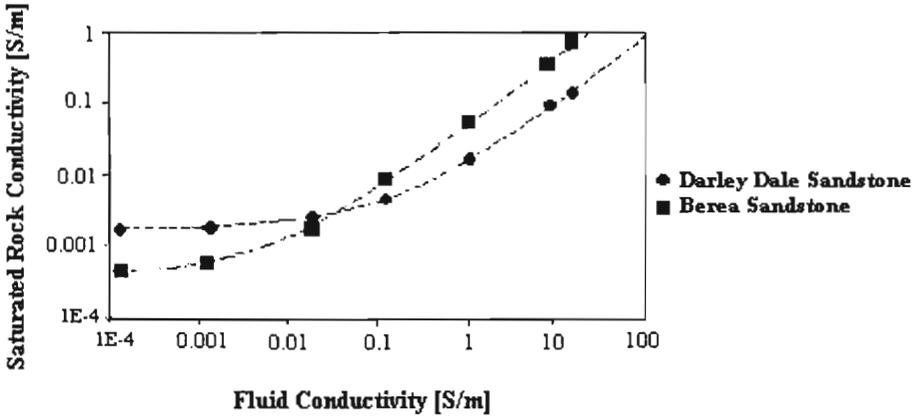


FIG. 14. Electrical conductivity of Berea and Dale sandstones saturated with NaCl solutions at various concentrations as a function of the electrical conductivity of the electrolyte solutions.

conductivity of electrolyte and of electrolyte-saturated sample) should not be identified with the structure parameter – the formation factor.

In Fig. 15 the dependence of the relative conductance, F , on diameters of glass beads saturated with water solution of KCl in the range of concentration from 0.00001 to 0.1 [mole/liter], is presented for measurements made in a two-electrode system [11]. The value of the relative conductance for concentration 0.1 and 0.01 [mole/liter] is almost constant in the whole range of diameters of the measured granular material.

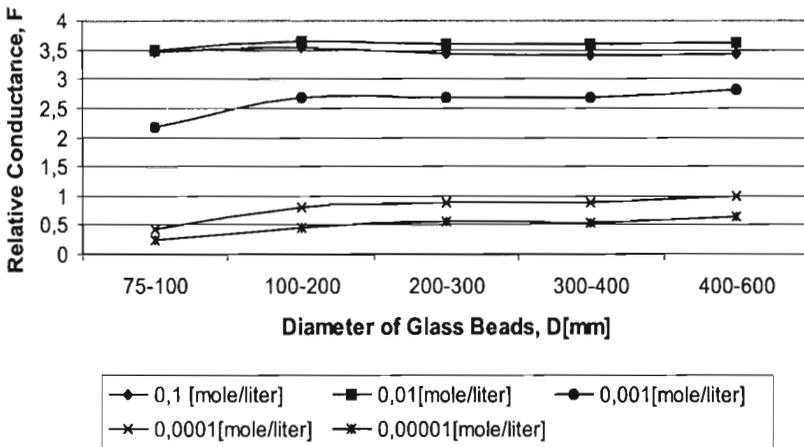


FIG. 15. Diagram of relative conductance, F as function of diameters of glass beads, D for various concentrations of KCl.

The absence of changes of the relative conductance of saturated glass beads in the range of diameters from 75 to 600 μm for KCL, for concentration 0.1 and 0.01 [mole/liter], and the fact that the porosity is approximately constant (the value of porosity does not depend on the volume of grains but on their configuration) result in the validity of empirical Archie's Law [2]), which determines the relation between the formation factor and the porosity.

A set of experimental data for pore structure parameters: porosity, formation factor, tortuosity and permeability of various natural porous materials is given in Table 4. The smallest value of tortuosity for given materials have the glass beads with a diameter 8 [mm] and granular glass material in the range of diameters from 300 to 600 μm . The largest value of tortuosity, $T = 3.4$, exhibits the Greenhorn limestone.

Table 4. A set of experimental results for various porous materials [16, 8]

Material	Porosity [-]	Formation factor [-]	Tortuosity [-]	Permeability [10^{-15}m^2]
Granular glass 300-600 μm	0.39	3.71	1.45	
White sand 125-210 μm	0.403	3.97	1.60	22,8
Glass beads 8 mm	0.435	3.29	1.43	14,000,00
Garden loam	0.491	4.17	2.05	
Cecil clay (19,6% H_2O)	0.475	5.01	2.38	
Greenhorn limestone	0.340	10	3.4	3-9
White sandstone	0.260	7.69	2.0	6.050
Limy sandstone	0.191	21.1	4.02	190

APPENDIX A – FORMATION FACTOR AND TORTUOSITY – DEFINITIONS AND MODELS

Formation factor

The formation factor, FF , can be defined with the help of conductivity of porous sample and of the electrolyte (see eq. [2.2]) or equivalently as the ratio of the electrical resistivity, ρ_o of a porous material saturated with electrolyte to the electrical resistivity of the electrolyte ρ_w [2]

$$(A.1) \quad FF = \rho_o / \rho_w.$$

Although the above definition of the formation factor is general and refers to all porous materials independently of their microstructure, in the literature one can find a number of works that propose simple theoretical models relating the factor with more elementary geometrical parameters of porous medium: porosity n , length of sample L , average length of pathways through pores L_e , area of

cross-section available to the current flow A_e , etc. Such models are derived e.g. by considering the resistance of the sample R_o , as connected to the parameters L_e and A_e :

$$(A.2) \quad R_o = \rho_w \frac{L_e}{A_e}.$$

Taking into account the fact that the resistance can be also expressed as

$$(A.3) \quad R_o = \rho_o \frac{L}{A}$$

where A is the total area of the cross-section of the sample, the expression for the formation factor FF in terms of the introduced geometrical parameters is obtained by combining equations (A.1), (A.2) and (A.3)

$$(A.4) \quad FF = \frac{L_e}{L} \frac{A}{A_e}.$$

In order to eliminate from the relation (A.4) the ratio of the surface areas $\frac{A}{A_e}$, some idealized models of pore geometry along with the resultant equations for A_e and FF gathered by Bear, 1972, are given in Table 5.

Tortuosity

The tortuosity of porous materials is intuitively understood as a measure of the actual path length through a sample of the material for fluid particles or ions referred to the length of the sample. More specifically, the parameter may be defined in relation to different transport phenomena such as 1) ionic current, 2) flow of ideal or viscous fluid, and 3) diffusion of chemicals in pore fluid.

1. Tortuosity as related to ionic current.

The tortuosity related to ionic conduction in porous medium is defined as the product of formation factor, FF and porosity, ϕ (see Equation [2.3]).

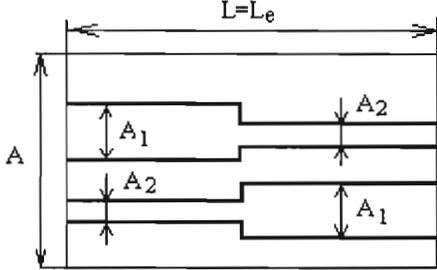
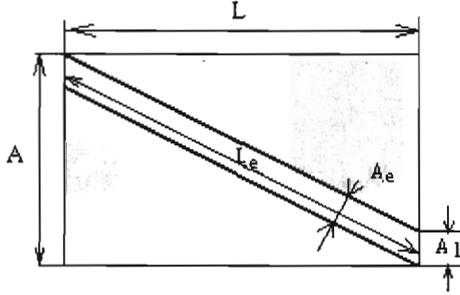
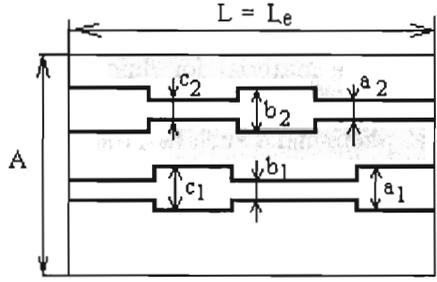
$$(A.5) \quad T = FF * \phi.$$

2. Tortuosity and flow of viscous fluid.

The analysis of equation for viscous fluid flow through capillary model of porous medium has led to Kozeny-Carman equation (see [4]) which involved the factor called the tortuosity, T , defined as

$$(A.6) \quad T = \left(\frac{L_e}{L} \right)^2.$$

Table 5. Porous medium models used for relating FF , A_e and A

Porous medium model	Authors and relationships for A_e and FF
	<p>Wyllie and Spangler, 1952</p> $A_e = A_1 + A_2 = \phi A$ $FF = \frac{1}{\phi} \frac{L_e}{L}$
	<p>Cornell and Katz, 1953</p> $A_e = A_1 \frac{L}{L_e} = \phi A \frac{L}{L_e}$ $FF = \frac{1}{\phi} \left(\frac{L_e}{L} \right)^2$
	<p>Wyllie and Gardner, 1958</p> $A_e = \phi^2 A$ $FF = \frac{1}{\phi^2} \frac{L_e}{L}$

3. Tortuosity and diffusion

When a process of mass diffusion of a chemical in pore fluid takes place, the relationship between the effective or macroscopic coefficient of diffusion D_e and the coefficient of molecular diffusion of the chemical in pore fluid, D_m , is expressed through porosity and tortuosity in the form, [8],

$$(A.7) \quad D_e = \frac{\phi}{T} D_m.$$

Such definition of tortuosity has the meaning identical with in the definition given by the electric formation factor (2.3).

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Received November 28, 2000.
