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## AN EXPERIMENTAL METHOD OF CHARACTERIZATION OF DEFORMABLE POROUS MEDIA

Mario BOULOS<sup>\*</sup>, Wahbi JOMAA<sup>\*</sup>, Alain SOMMIER<sup>\*</sup>, Denis BRUNEAU<sup>\*</sup>

**Abstract:** A porous medium saturated with liquid and placed within a medium that undergoes a change in pressure reacts by shrinking. If the space contains the same liquid as the pores of the sample, then after a certain lapse of time the sample dilates. By measuring this dilation kinetic the specimen's permeability can be approximated. This experimental method is called Dynamic Pressurisation. We set up an experimental apparatus to measure the permeability and the different agarose gel compressibility moduli. The liquid contained inside the gel pores is water. We have realized experiments in water and others in oil. In Scherer's method the flow is considered only in the radial direction. To find the real permeability value we have built a numerical model considering that both the liquid and the solid are compressible. The simulations were compared to the experimental results and have allowed finding the real value of the permeability by considering the flow in both radial and axial directions.

### 1. INTRODUCTION

The principle of Dynamic Pressurisation (DP) has been applied to aerogels by Gross and Scherer [1]. The theory was developed by Scherer [2], and then improved by Grasley et al. [3] so that it could be applied to cimentitious materials. Christopher et al. [4] [5] modified the geometry of the sample so that the permeability of materials that are not very permeable could be measured.

The advantage of DP is that it can be applied to both strong and weak permeabilities. For rigid materials like cement it requires much less time than other experimental methods, which prevents the properties of the sample from changing during the course of the experiment [6] [7]. The dynamic pressurisation method remains a reliable method which can be applied to all sample types.

In this paper we describe the experimental apparatus that has been developed and the measurement method used. We review the dynamic pressurization principle and the theoretical bases for exploiting the data obtained and give the results for agar gels.

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In his method, Scherer [2] considers that the flow only in the radial direction of the cylindrical specimen. In other words, the cylinder has an infinite height comparing to his diameter. Which is why the value of the permeability obtained is approximated. In order to find the correct this approximated value and to find the real permeability we have built a numerical module that takes into account the compressibility of both the liquid and solid phases. The simulations were compared to the experimental results in a first time and have allowed finding the real value of the permeability by considering the flow in the radial and the axial directions.

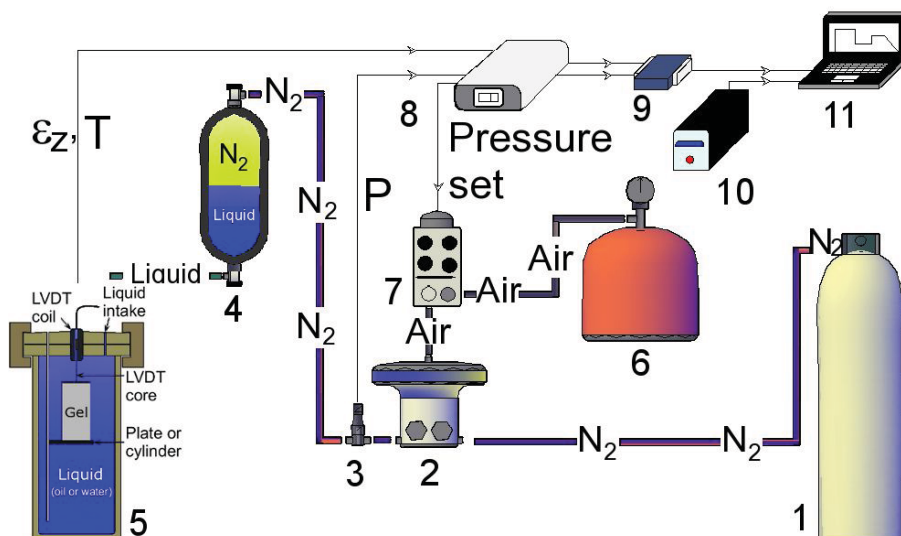
## 2. MATERIALS AND METHOD

### 2.1. EXPERIMENTAL APPARATUS

Figure 1 shows a diagram of the experimental apparatus. The experiment consisted of equilibrating a sample in a tank under pressure (or an autoclave), then altering the pressure in the system while measuring the axial strain of the gel, written as  $\epsilon_z$ . The cylindrical sample was placed in the autoclave filled with liquid (water or oil) on a porous plate. Its variation in height was measured using an LVDT sensor with the core resting on the upper surface of the gel through a metal plate (Model SX12C, diameter 12 mm, pressure range 250 bar and  $T = 85^\circ\text{C}$ ).

Pressure in the system was controlled by the nitrogen, which was in contact with the liquid inside the interface bottle (4) (see Figure 1). Adjustments were made using a gas regulator with a membrane (component 2 in Figure 1). The regulator receives the nitrogen from the bottle (1) at a pressure of 200 bar and reduces it by means of a membrane which opens and closes to allow the correct amount of gas to pass through. Pressure is regulated either manually or automatically by the regulator (8) which sends instructions to the converter (7).

The values for height, pressure and temperature were obtained via a USB 6009 National Instrument data acquisition card. Control/command software was specially developed under labVIEW to record data at adjustable time intervals and to manage the pressure cycle applied to the sample.



**Figure 1: Diagram of the experimental apparatus for applying DP to agar gel**

## 2.2. METHOD AND ANALYSIS OF RESULTS

The analysis of the results in water are discussed in detail in [2]. The most convenient way of doing the experiment is to impose an instantaneous increase in pressure. In other words, the pressure on the tank ( $P_C$ ) changes from atmospheric pressure ( $P_0$ ) to the imposed pressure ( $P_A$ ).

As for the tests in oil, The sample was placed in the autoclave on a sintered base. The apparatus was filled with oil and we programmed in the pressure cycle to be applied to the sample.

The instruction  $\Delta P$  ( $\Delta P = P_{\text{applied}} - P_{\text{atmospheric}}$ ) included the following phases:

- a long equilibration phase at atmospheric pressure (20 hours),
- a gradual increase in  $\Delta P$  from 0 to 59 bar (90 minutes),  
so that the Young's modulus could be calculated at every moment and any variation in relation to the applied pressure could be studied,
- a period of constant pressure (one hour),
- a sudden drop in pressure from 60 bar to atmospheric pressure,
- a phase at atmospheric pressure (one hour).

In this case the contraction of the samples in the oil was due to isostatic compression (the applied pressure was the same in all directions). The Young's modulus was calculated from the elasticity or compressibility modulus  $C$ , which associates variations in pressure and volume for a homogeneous and isotropic material, in the following equation:

$$\Delta P = -C \frac{\Delta V}{V_0}, \quad (1)$$

$$E = -3V_0(1-2\nu) \frac{\Delta P}{\Delta V}. \quad (2)$$

For a homogeneous and isotropic material, strains in the three directions can be considered equal [8]. The volumetric strain is then equal to three times the only information we have in terms of variation in displacement,  $\epsilon_z$ . According to equation (2), the Young's modulus is defined as:

$$E = -h_0(1-2\nu) \frac{\Delta P}{\Delta h} \quad (3)$$

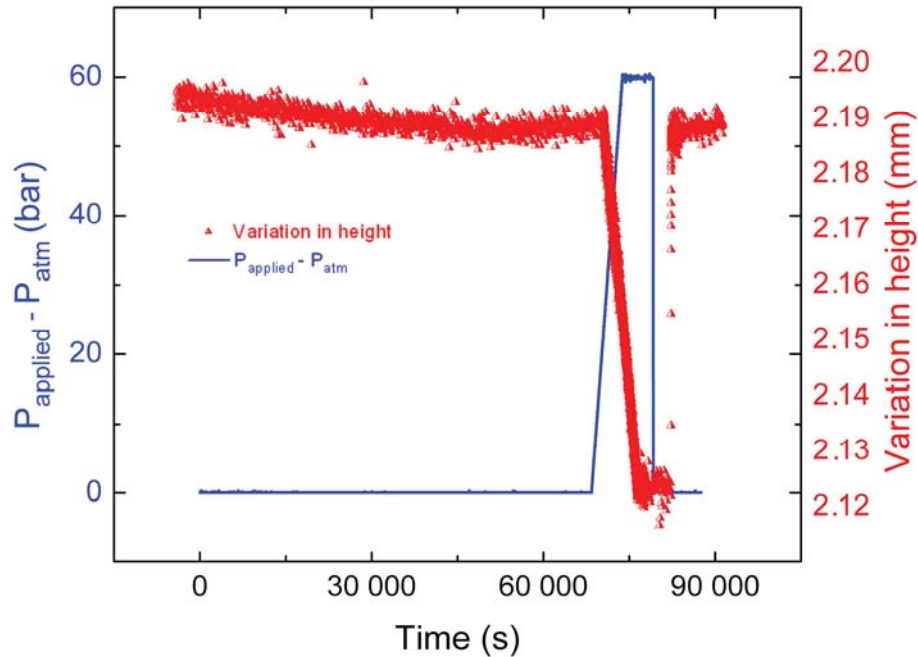
These tests gave the Young's modulus for the non-drained material as the water cannot come out of the sample to enter the autoclave which is filled with oil (different liquid from that in the pores of the porous medium). The tests in oil were carried out using cylindrical samples of agar gel (height 3.5 cm, diameter 1.8cm). These samples were prepared according to a standard protocol described in [9] for agar.

## 3. RESULTS AND DISCUSSION

### 3.1. IN OIL

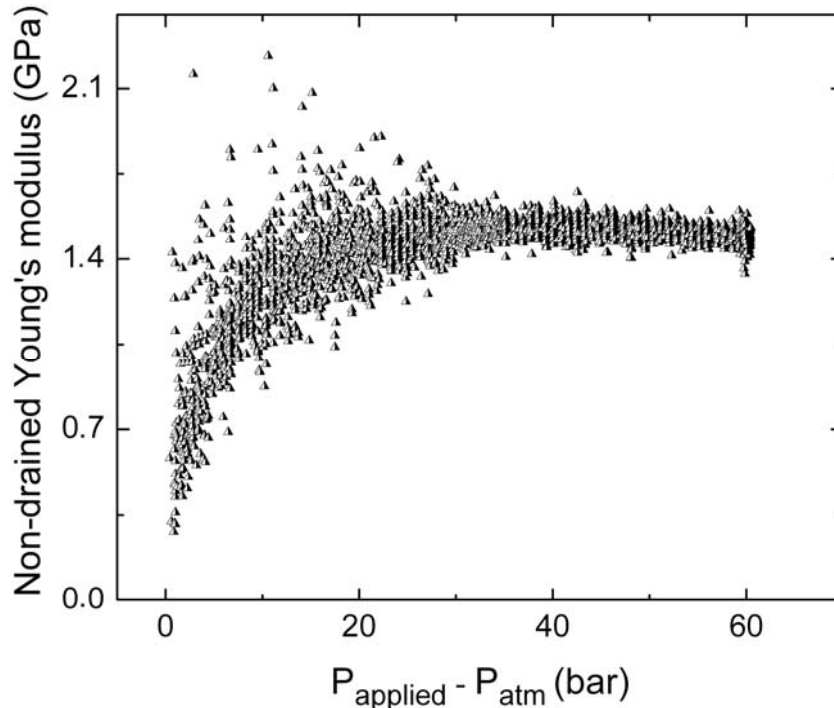
From the tests in oil we were able to measure the non-drained Young's modulus of the porous medium. Permeability could not be calculated as the water inside the gels was not able to leave or enter. Thus the experiment in oil revealed the physical behaviour of the gel. In Figure 2, we show an experimental graph of the variation in height of an agar gel sample as a function of the difference in applied pressure. We note that the majority of

time is devoted to equilibration of the gel which in this case displays a linear elastic behaviour.

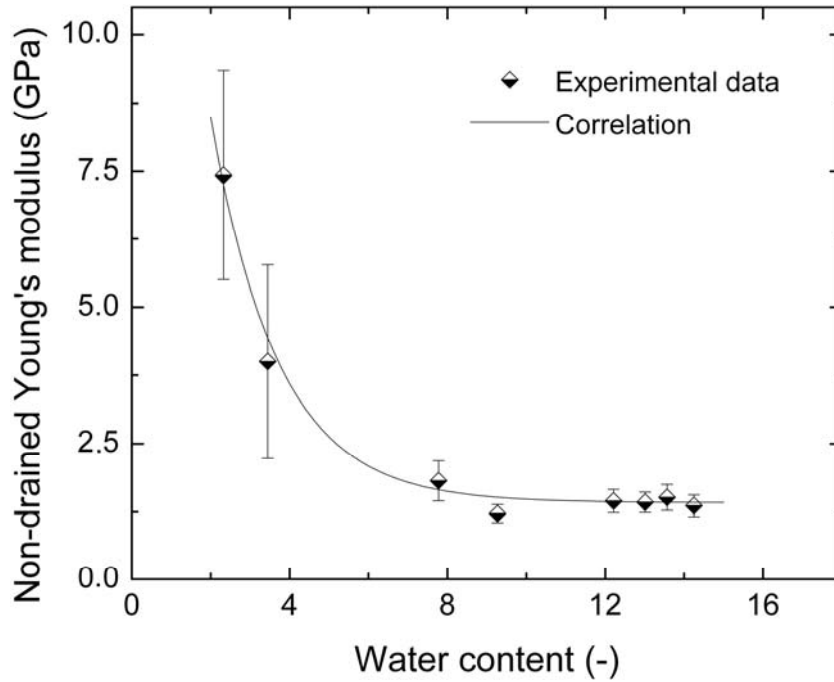


**Figure 2: Variation in height of agar gel with the applied pressure in oil**

The variation in non-drained Young's modulus as a function of  $\Delta P$  is shown in Figure 3. When looking at this curve, we note that the value of  $E$  varies for  $0 < \Delta P < 30$  bar before stabilising. The non-drained Young's modulus is calculated by averaging the values of  $E$  in the stable phase. Figure 4 shows the variation in non-drained Young's modulus of the agar gel as a function of water content. Errors in relation to the Young's modulus determined from the DP experiment were relatively large when water content was low. This is related to the low values for displacement given the sensitivity of the LVDT sensor.



**Figure 3: Non-drained Young's modulus in function of the applied pressure**

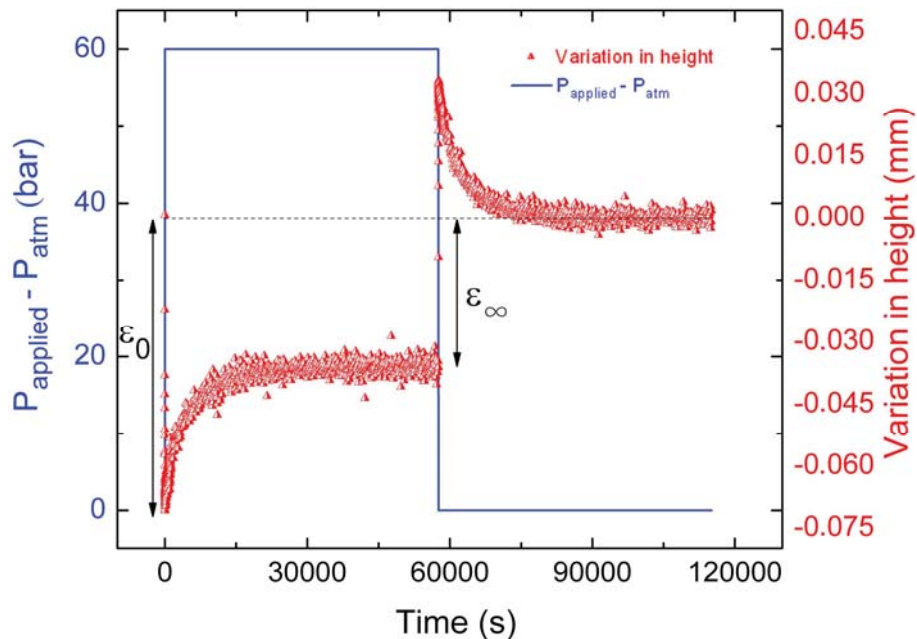


**Figure 4: Non-drained Young's modulus in function of water content**

### 3.2. IN WATER

According to Sherer's theory in [2], the system contains five equations for six unknowns: the compressibility moduli of the solid and liquid phases and of the porous medium ( $C_s$ ,  $C_l$  and  $C_m$ ), Biot's number ( $b$ ), Biot's modulus ( $M$ ) and  $\lambda$ . In order to close the problem we focused on the values of the drained Young's modulus obtained in the load relaxation experiment. The main equations are those relating the initial and final strains to the compressibility moduli of the porous medium and the solid phase respectively.

Figure 5 shows the variation in height of an agar gel sample equilibrated during the two phases of instantaneous compression and relaxation.



**Figure 5: Measuring initial and final strains of an agar gel in water**

Permeability ( $K$ ) was calculated from relaxation time  $\tau_v$  represented by equation (34) in [2] and determined by adjusting the experimental curve for relaxation of the gel once all the coefficients had been calculated.

The variation in water content was always low because of the absorption of water by the sample. Table 1 shows  $C_l$ ,  $C_s$ ,  $C_m$  and  $b$  for each value of  $W$ . We note that the value of  $b$  is always close to 1, which fits the theoretical value for gels. The values of  $M, \lambda$ ,  $\tau_v$  and  $K$  as a function of  $W$  are given in table 2. The value of  $\lambda$ , like  $b$ , is also very close to 1, which fits with the theory [2].

Water content (-)	$C_l$ (GPa)	$C_s$ (GPa)	$C_m$ (GPa)	$b$ (-)
14.800	0.778	1.38	0.00172	0.998
14.673	0.945	1.69	0.00168	0.999
14.542	0.974	1.82	0.00164	0.999
14.607	1.001	1.86	0.00166	0.999
13.520	0.987	1.98	0.00134	0.999

**Table 1:  $C_l$ ,  $C_s$ ,  $C_m$  and  $b$  as a function of water content**

Water content (-)	$M$ (GPa)	$\lambda$ (-)	$\tau_v$ (s)	$K$ (m <sup>2</sup> )
14.800	0.796	0.999	20719.234	3.02E-18
14.673	0.966	0.999	21211.887	3.06E-18
14.542	0.986	0.999	17109.89	3.89E-18
14.607	1.003	0.999	20011.135	3.17E-18
13.520	1.001	0.999	36211.916	2.16E-18

**Table 2:  $M$ ,  $\lambda$ ,  $\tau_v$  and  $K$  as a function of water content**

Lastly, we compared permeability values measured using the DP methods in the water and those using the load relaxation method in table 3. We noted slight differences between the permeability values obtained in the two different experiments but for experimental values the deviation remained minimal.

Water content (-)	Permeability (m <sup>2</sup> ) by the DP method	Permeability (m <sup>2</sup> ) by the relaxation
14.800	3.02E-18	9.51E-18
14.673	3.06E-18	1.68E-19
14.542	3.89E-18	2.13E-19
14.607	3.17E-18	1.89E-19
13.520	2.16E-18	1.16E-18

**Table 3: Comparison of  $K$  values measured in the DP and relaxation procedures**

#### 4. MODELLING AND CORRECTION OF THE PERMEABILITY VALUE

The experiments applying Dynamic Pressurisation used the theory of Scherer which is based on Biot's module [10]. This module was discussed in detail by Coussy [10], it considers the liquid and solid phases of the porous medium as compressible and is compatible with big deformation. However, this model uses a Lagrangian scale (study between two instants without following the evolution). We constructed a model that considers the liquid and solid phases as compressible and uses a eulerian scale.

The energy balance and the mechanical equilibrium are the same as in the Biot's module. As for the mass balance, the gradient of the deformation velocity of the solid phase appears in our module in difference of Biot's mass balance.

Our mass balance equation is based on the mass balance of the porous medium using the work of Coussy [11] specially the relation that relates the variation of the porosity to the variation of volumetric strain and of the intrinsic liquid pressure.

$$\frac{\partial \phi_l}{\partial t} = b \frac{\partial \epsilon}{\partial t} + \frac{1}{N} \frac{\partial \langle P_l \rangle^l}{\partial t} \quad (4)$$

Our mass balance module equations are presented in table 4.

Equation type	Equation Expression
Liquid mass conservation	$\frac{1}{M} \frac{\partial \langle P_l \rangle^l}{\partial t} + b \frac{\partial \epsilon}{\partial t} + \vec{\nabla} \cdot \left( \phi_l \langle \vec{V}_s \rangle^s - \frac{\bar{K}}{\mu_l} \vec{\nabla} \langle P_l \rangle^l \right) = 0$
Medium momentum conservation	$\vec{\nabla} \cdot (E \bar{\epsilon}) - \vec{\nabla} \cdot (\langle P_l \rangle^l \bar{I}) = 0$
Medium energy conservation	$\frac{\partial \langle H \rangle}{\partial t} + \vec{\nabla} \cdot \left( \langle \rho C_p \rangle \langle \vec{V}_s \rangle^s - (\rho C_p)_l \frac{\bar{K}}{\mu_l} \vec{\nabla} \langle P_l \rangle^l \right) \langle T \rangle - \bar{\lambda} \cdot \vec{\nabla} \langle T \rangle = 0$

**Table 4: the model equations**

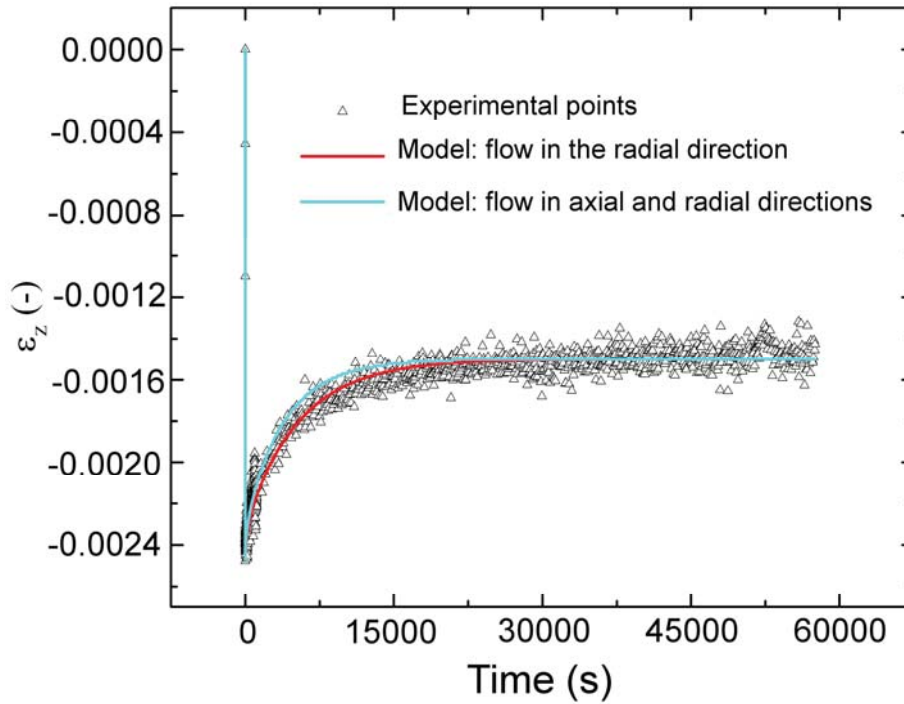
Where  $M$  is the Biot's module,  $b$  is the Biot's coefficient,  $\epsilon$  is the volumetric strain,  $\bar{K}$  is the permeability tensor,  $\mu_l$  is the liquid viscosity,  $\phi_l$  is the porosity of the porous medium,  $\langle \vec{V}_s \rangle^s$  is the intrinsic deformation velocity of the solid phase,  $E$  is the Young's modulus,  $\bar{\epsilon}$  is the stress tensor,  $\langle P_l \rangle^l$  is the intrinsic liquid pressure,  $H$  is the enthalpy,  $\bar{\lambda}$  is the thermal conductivity tensor and  $T$  is the temperature.

The boundary conditions are those of the DP experiment, by considering the flow only in the radial direction in a first time and in both axial and radial directions in a second time allowing us to correct the permeability value measured experimentally.

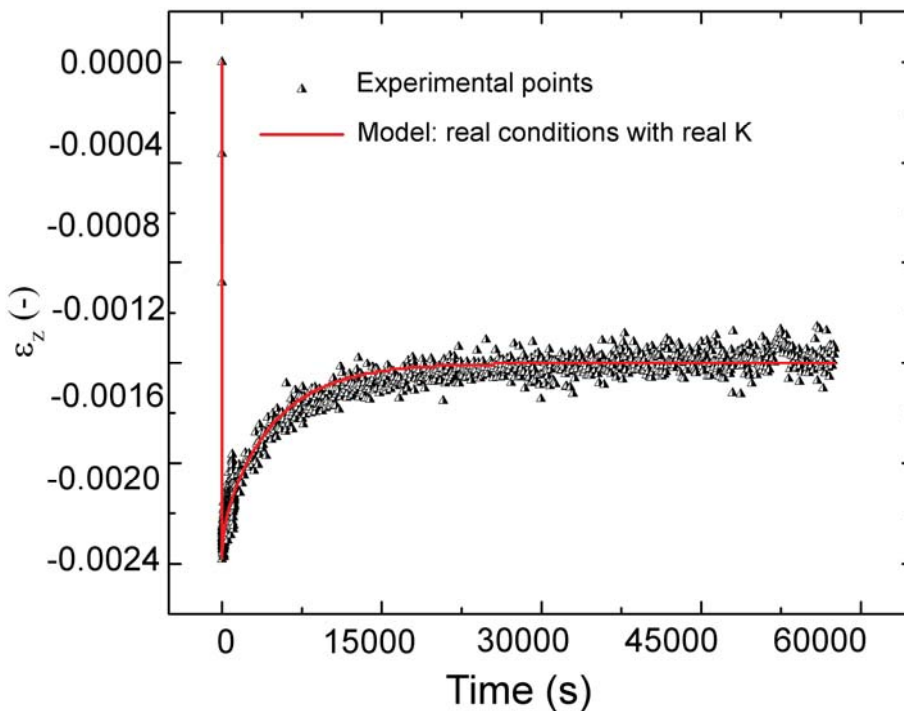
Figure 6 shows the experimental points of a DP experiment on an agar gel, the model curve considering the flow only on the radial direction according to Scherer's model and the model curve considering the real case in which the flow is in both radial and axial directions.

Figure 6 clearly shows that the model fits exactly with the experience by considering Schere's boundary conditions, but in the real case, where the flow is in both radial and axial directions the curves do not fit quite well. The model will allow one to correct the permeability value by changing it until the model curve with the real boundary conditions fits exactly with the experimental points like shown in figure 7.





**Figure 6: Different boundary conditions model curves and experimental points**



**Figure 7: Correcting the permeability value**

## 5. CONCLUSION

Dynamic pressurisation (DP), the method described here, is an experimental method which can be used to measure simultaneously the compressibility modulus of liquid and solid phases and the permeability of the porous medium. The distinctive feature of DP is

that the non-drained compressibility modulus of the medium can be measured when a different liquid is used from that in the pores (e.g. oil), and the drained and non-drained moduli can be measured when the same liquid is used as in the pores.

The principle based on Biot's theory [10] and developed by Coussy [11] was described by Scherer [2]. When a saturated and equilibrated porous medium in a vessel filled with liquid undergoes a sudden change in pressure, it contracts in accordance with its compressibility modulus.

If the liquid in the vessel is the same as in the pores, it enters the pores from the vessel due to a difference in pressure and the sample dilates without returning to its initial position. The final strain depends on the rigidity of the solid grains (thus the compressibility modulus of the solid phase can be measured) and the kinetics of dilatation give permeability. If the liquids are different, only the non-drained compressibility modulus of the medium is measured.

DP was applied to gels by Gross et al. [1] who measured only permeability and not the other parameters, and to cement by Grasley et al. [3] by measuring all the parameters. However, the LVDT sensor was introduced into the cement sample, in a position which was impossible for the gels because of their structure. Our experimental procedure is a mixture of the two systems; it can be applied to a gel and it ensures that all parameters can be measured.

Elasticity and reversibility were tested and demonstrated for the agar gel. Next, we presented the results. In the case of oil, isostatic compression equations were used, whereas for the agar gel in water, we applied the Scherer formulae. Resolving the equation system was based on the values for the drained Young's modulus obtained from the load relaxation experiment. The permeability values were then compared with those obtained using the load relaxation method.

The information provided using Scherer's approach to the compressibility modulus has very great precision. However, the permeability value is only an estimate.

For this reason, a new model based on the theory of Biot [10] by changing to eulerian scale coordinates was established using the work of Coussy [11]. The simulations were carried out with the same boundary condition of Scherer's model in a first time using the experimental parameters, a good agreement between the experience and the model is noticed. The value of the permeability is corrected by considering the flow in both radial and axial directions which is the real case.

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