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Determination of Elastic Modulus of Gelatin Gels by Indentation Experiments

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Abstract

Mechanical characterization of hydrogels is a challenging task because they are much softer than metals, ceramics or polymers. The elastic modulus of hydrogels is within 10^0-10^2kPa range. Because they easily break and slump under their own weight, tensile and bending tests are not suitable configurations to assess elastic modulus. This work reports on the determination of elastic modulus of a gelatin gel by indentation experiments. Indentation is very simple configuration, it is of technological importance and it can be applied at different length scales with high accuracy. The gelatin hydrogel behavior is first calibrated by uniaxial compression and low strain rheological measurements. It behaves as a hyperelastic solid with strain hardening capability at large strains and shows no dependence with frequency in the linear viscoelastic range. It can be properly characterized by the First order Ogden material model. Indentation experiments are carried out at macro and nanoscales using spherical and flat-ended cylindrical punches. Elastic contact solutions and inverse analysis accounting for hyperelasticity are used to extract the elastic modulus from experimental force-depth curves. Adhesion between punch and hydrogel influences the indentation response and affects the accuracy of elastic modulus determination in a larger extent than the assumption of linear elasticity. Adhesion leads to overestimation of elastic modulus values. The influence of adhesive forces increases with decreasing the length scale. A markedly decay of elastic modulus with increasing maximum load is observed at nanoscale. A hybrid model based on Hertz elastic contact solution and Johnson-Kendal-Roberts model for adhesion is used to determine elastic modulus. This model yields an elastic modulus in good agreement with that obtained from uniaxial compression test.

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Keywords: Depth Sensing Indentation, Soft Materials, Hydrogels, Elastic Modulus, Adhesion

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1. Introduction

The mechanical characterization of soft materials is of great importance for a broad range of applications in food industry, ballistic, biomedicine and tissue engineering. Determination of mechanical properties of this class of solids is a challenging task because they are much softer than metals, ceramics and glassy polymers. Evaluation of low deformation properties such as elastic modulus is fundamental in the utmost application fields of soft materials. Because they easily break and slump under their own weight, conventional tensile and bending tests are not suitable configurations. Uniaxial compression, shear strain oscillatory and indentation tests appear more appropriate.

The indentation configuration is very simple and it can be applied at different length scales, allowing the determination of global and local material properties with high accuracy. In nanoindentation tests, the size scale of the indenter and the resolution of displacements and forces allow to investigate material heterogeneity on the order of microns.

Indentation at macroscale is of technological importance for the food industry. For example, the puncture test is the most frequently used method for textural evaluation of fruits, vegetables and gels. The Bloom test is used to determine a technological quantity (the bloom number) that is related to the stiffness of gelatin gels (BSI 757, 1975). Depth sensing indentation across size scales (nano to macro) is of scientific significance for biomedical applications and tissue engineering. The large-scale spherical indentation test has been used to calibrate material models to simulate the response of biological soft tissues in the development of medical devices (Kerkod et al., 2002). Nanoindentation experiments constitute a powerful tool for mapping spatially mechanical properties in soft tissues and other biomaterials at the micro and nanoscales (Ebenstein and Pruitt, 2006).

The determination of material parameters from indentation data is not as straightforward as for other test configurations in which the stress-strain field is uniform such as uniaxial tensile or compression. A complex stress state is developed under the indenter, which complicates extracting material parameters from the measured data. A standard procedure for soft materials does not yet exist. As well, the current methodologies for modeling punch-sample interactions are still limited.

Elastic modulus can be ideally evaluated from elastic contact solutions introduced by Hertz (1863). However, the actual indentation response of soft materials is affected by hyperelasticity, viscoleasticity and other issues such as adhesion and friction between the indenter tip and sample surface. Soft materials often undergo large deformation exhibiting strong non-linear responses. Adhesion can affect the governing displacement-based Hertzian contact area approximation used to determine the contact area which, in turn, affects the mechanical property calculations.

The combination of inverse analysis and finite element simulations of the indentation problem may constitute a helpful way of determining material parameters. This methodology allows the assumption of a material constitutive model such as neo-Hookean, Ogden, Money-Rivlin, Yeoh, Fung, Arruda Boyce, etc. (see for example the work by Zhang et al., 2013). The accuracy of the combined method in determining the model parameters relies on the idealization of the indentation problem and the potentiality of each constitutive model.

This paper aims to contribute to the development of robust methods to determine material properties of soft materials using indentation experiments. In particular, the present work deals with the determination of elastic modulus at different length scales: macro and nano. A bovine gelatin hydrogel is used as an example of soft material that exhibit hyperelastic behavior. First, the material behavior is calibrated by traditional uniaxial compression and small shear strain oscillatory measurements. Then, the initial elastic modulus is obtained from the experimental indentation force-displacement curves employing different approaches. The results from Hertzian elastic contact solutions, inverse analysis and finite element modeling accounting for hyperelasticity and friction, and from a hybrid model accounting for adhesion effects at the nanoscale are discussed and compared.

2. Experimental and Methods

2.1. Material and Sample Preparation

Bovine hide gelatin type B, Bloom 200, isoionic point (Ip) 4.7-5.4 kindly supplied by Rousselot (Argentina) was used to prepare hydrogel samples. Gelatin powder (20%wt) was dissolved in a pH 7 buffer solution with continuous stirring at 50ºC. The solution was then poured into cylindrical Delrin® moulds and kept at room temperature during

15min to form the gel. Samples were wrapped in film in order to minimize drying out and stored at 4ºC during 48 h. Hydrogel samples were conditioned at 21ºC during 2h previously to mechanical testing.

2.2. Uniaxial compression tests

Uniaxial compression tests were performed in an INSTRON 4469 universal testing machine at a loading rate of 5mm/min. A 0.5 kN load cell was used and teflon spray was applied onto compression platens to avoid friction effects. Samples of height (H=25mm) and diameter (D=25mm) were used. They were loaded up to fracture and the obtained load (P) vs. displacement (v) data were converted to true stress (σ) vs. true strain (ε) curves assuming that the material was incompressible (poisson coefficient ν=0.5). Data were also expressed in terms of true stress (σ) – stretch ratio ($\lambda = e^{\varepsilon}$).

2.3. Rheology tests

Small strain rheology experiments were carried out in an Anton Paarrheometer type Physica MCR-301. Oscillation measurements were performed using two parallel plates (D=25mmm) and a gap of 2mm (sample height). The spectrum obtained using a frequency of 1Hz was used to select a % strain that verify being in the viscoelastic range (0.1%). At this strain, the storage (G′) and loss (G′′) shear moduli were measured for a wide range of frequencies up to 50Hz at 21ºC.

2.4. Macroindentation tests

Macro-indentation experiments were carried out in an INSTRON 4469 universal testing machine at a crosshead speed of 5mm/min. Two different punch geometries were used, a cylindrical flat-faced plunger with a diameter of 10 mm and a spherical indenter with a diameter of 5.9 mm. A Load cell of 0.1 kN was used. Experiments were performed up to 4 mm and 2.8 mm displacement for the cylindrical and spherical geometries, respectively.

2.5. Nanoindentation tests

Nanoindentation experiments were performed in a TriboindenterHysitron and a diamond spherical tip of 500µm nominal radius was used. The indentation test involved a complete loading-holding-unloading cycle under displacement controlled conditions with the continuous measurement of load and displacement. The pre-load was set as 0.1µN, the holding time at 15s and the maximum indentation depth achieved was varied between 200 and 4000nm. Experiments were carried out from the Imaging Module in order to verify the initial contact between sample surface and tip.

2.6. Constitutive Model

The First order Ogden constitutive model was chosen to describe the response of the gelatin hydrogel. This model is included in the group of constitutive models in which the stress-strain relationship derives from a strain energy density function. It models the response of an isotropic, hyper-elastic solid with strain hardening. The model comprises only two parameters, the strain hardening exponent, α, and the shear modulus, μ. More negative α values indicate large strain hardening capability, while less negative ones stands for more linear elastic behavior. The energy density function is:

$$U = \frac{2\mu}{\alpha^2}(\lambda_1^{-\alpha} + \lambda_2^{-\alpha} + \lambda_3^{-\alpha} - 3) \tag{1}$$

while, for uniaxial compression configuration, the nominal stress-stretch relationship ($\sigma_N - \lambda$) , results:

$$\sigma_N = \frac{2\mu}{\alpha}(\lambda^{\alpha-1} - \lambda^{-\frac{1}{2}\alpha-1}) \tag{2}$$

2.7. Finite Element Modeling and Inverse Method

Due to the complex stress-state developed in the indentation test configuration, a straight relationship between stress and strain like that given by Eq. (2) is not available. Therefore, in order to extract constitutive parameters (α, μ) and hence calculate the initial elastic modulus (E=3μ) from force-depth data an inverse method was applied. An optimization algorithm which minimizes the quadratic discrepancy between experimentally measured data and pseudo-experimental load-depth curves obtained via Finite Element Modeling (FEM) simulations was implemented.

FEM indentation simulations were performed using the commercial software ABAQUS. The same two test configurations employed in the macro-indentation tests were used: a 5 mm radius cylindrical punch and a 5.9 mm radii spherical punch indenting on cylindrical samples with H=D=25mm. In both indentation configurations, an axysimmetrical model was assumed and the mesh was constructed using linear quadrilateral elements as shown in Figure 1. A fine mesh concentrated towards the gel surface was used close to the contact zone while a coarse mesh was used outside this region to economize computation time. The indenters were assumed as rigid bodies and the contact between tip and sample surface was considered frictionless. The gel behavior was assumed to obey First Order Ogden model (Eq. 1). In order to test the mesh quality, the mesh was refined until the extracted load-displacement curve coincided.

a) *b)*

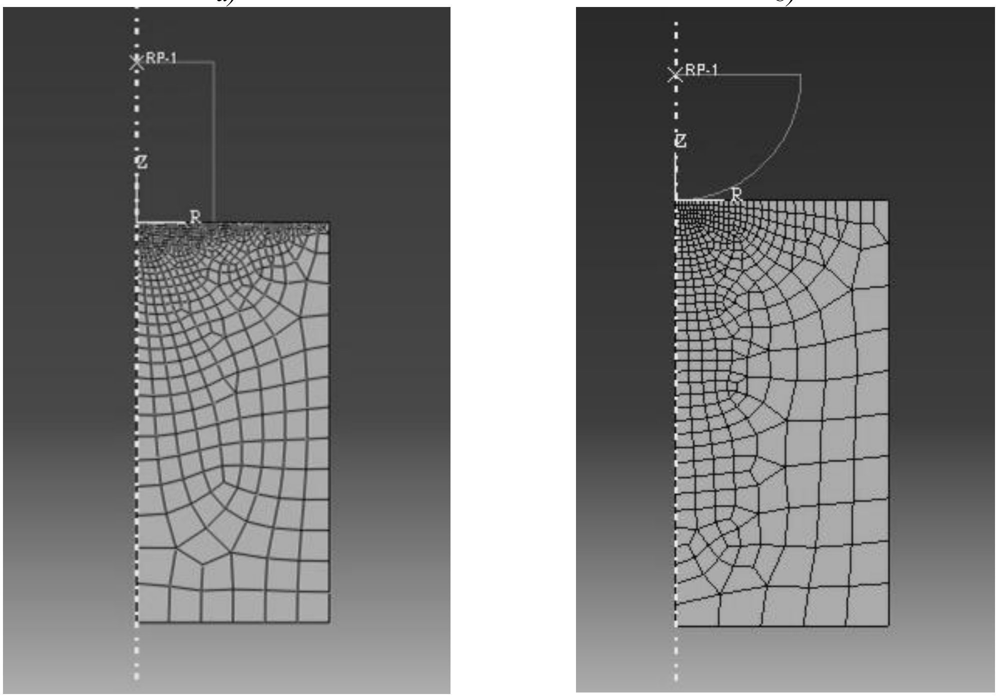

Fig. 1. Mesh used in FEM indentation simulations with (a) flat-ended cylindrical punch and (b) spherical punch.

In the inverse analysis the minimization of the objective function with respect to the unknown constitutive parameters (α, μ) was carried out following the Trust Region method [Coleman and Li (1996)].

3. Results and Discussion

3.1. Calibration of hydrogel behavior

The stress-strain behavior of hydrogel sample was assessed by traditional uniaxial compression experiments. Adhesion and friction effects between sample surface and compression platens were avoided by the use of lubrication. Fig. 2-*a*) shows a typical nominal stress – strain response determined at a strain rate of 0.2min^{-1}. Experiments performed at other rates in the quasi-static range showed that gel behavior was independent of strain rate. As well, exploratory loading-unloading cycles demonstrated that, before facture, deformation was completely reversible(data not shown here). Therefore, the gel behavior was non-linear elastic and showed strain hardening.

The uniaxial compression response could be accurately modeled by Ogden constitutive equation (Eq. 2), resulting α = -1.4 and ***E*=43.2kPa**. The use of the constitutive equation allowed us to determine the elastic modulus instead of determining a secant modulus from an arbitrary stress value [Tung, 2001].

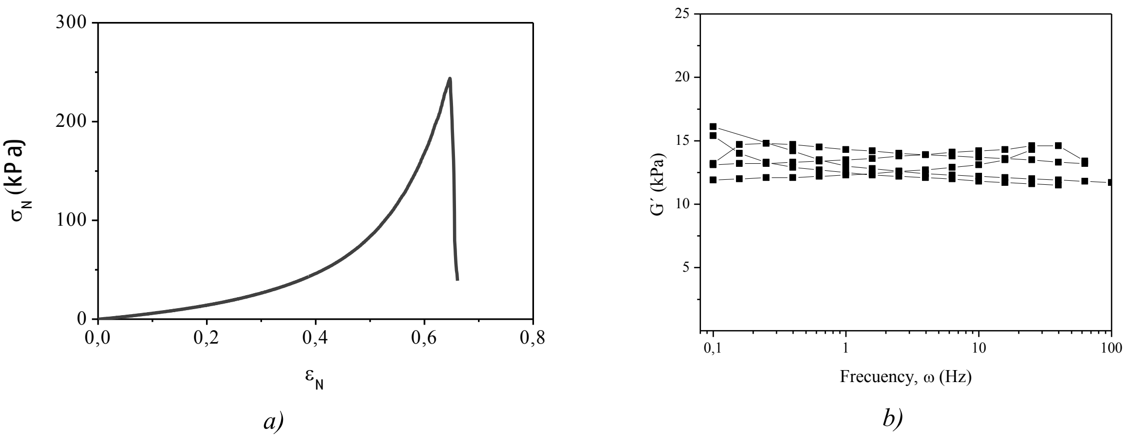

Fig. 2. Results from material behavior calibration experiments: a) Typical stress-strain curve measured in uniaxial compression; b) Storage modulus values obtained from rheology experiments at a maximum strain of 1% . Data are plotted in the semi-log scale to show reproducibility.

The negative α value indicates that the modulus at low strains increases with increasing deformation, as [Gamompilas et. al, 2010]:

$$E = 3\mu(1+\frac{\alpha\,\varepsilon}{2}) \tag{3}$$

The reported *E* value (43.2kPa) is that for very low deformation level (E≈3μ).

Fig.2-*b*) shows the frequency- dependence of the shear storage modulus (G′) recorded in the rheology tests. G′ remains constant in the 0.01 to 100 Hz range with an average value of 13.1kPa. Hence, the elastic modulus arisen from rheology experiments is ***E*=39.3kPa**, which practically coincides with the obtained in uniaxial compression.

3.2. Macroindentation

Fig. 3 shows load (*P*) – depth (*h*) curves obtained in macro-indentation experiments for both indenter configurations. It can be observed that the shape of the curves depends on the indenter geometry. The curves for flat-ended cylindrical punch appear almost linear while the ones for the spherical punch are highly non-linear. This can be attributed to the difference in the contact area with penetration depth of both types of indenters. The contact area of a cylindrical flat-ended punch is constant while it increases with penetration depth for a spherical punch in elastic contact with a flat surface. The contact radius, a, is given by Eq. (4) for a rigid sphere and by Eq. (5) for a rigid cylinder (Fisher-Cripps, 2000):

$$a = \sqrt{2Rh_p - h_p^2} \tag{4}$$

$$a = R \tag{5}$$

where R is the indenter radius and $h_p$is the depth of the circle of contact, which differs from the total depth due to sink-in of the material surface.

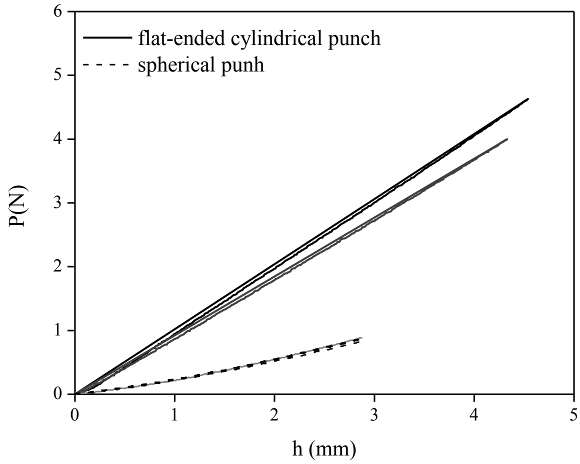

Fig. 3. Load-depth curves measured in macroindentation experiments.

Curves shown in Fig. 3 were used as input data in the inverse analysis procedure to obtain the Ogden parameters reported in Table 1. The E values obtained from both macro-indentation configurations practically coincide but they are larger than the ones determined in uniaxial compression experiments. The α parameter determined from different tests shows large discrepancies due to the lack of sensitivity of this parameter at low deformation levels.

Table 1. Ogden constitutive parameters and Elastic modulus obtained from inverse analysis of macro-indentation data.

Indenter Geometry	μ (kPa)	α	E (kPa)
Flat-ended cylindrical punch	21.9±2.1	-0.13± 0.18	65.7
Spherical punch	20.5±2.8	-3.73±2.53	61.5

For comparison, macro-indentation P-h curves were also analyzed considering the elastic contact solutions for a rigid indenter and half-space. For the case of the cylindrical indenter, the load-depth relationship is [Timoshenko and Goodier, 1970]:

$$P = \frac{E\,2\,R\,h}{\left(1-\nu^2\right)} \tag{6}$$

An average E value of **73.4kPa** was determined by fitting the experimental data obtained with the flat-ended cylindrical indenter to Eq. (5) and considering the Poisson coefficient, ν, equal to 0.5 (incompressible solid).

For the case of a spherical indenter, the load-depth relationship is usually interpreted using the Hertzian contact theory for low indenter displacements is (Johnson, 1999):

$$P = \frac{16}{9} E\, R^{1/2} h^{3/2} \tag{7}$$

An average E value of **67kPa** was obtained after fitting the experimental curves measured with the spherical indenter up to h/R=0.2. The fitting range was selected according to the results of [Zhang et al., 2013] work. The E value tends to increase with broadening the h/R range.

The E values determined from the elastic contact solutions (Eq. 6 and 7) are slightly larger than the ones obtained assuming the First Order Ogden constitutive model in the inverse method. This result shows that the elastic modulus is somewhat overestimated if the hydrogel behavior is assumed elastic instead of hyperelastic.

A simulated indentation curve obtained using the Ogden parameters arisen from uniaxial compression experiments are compared with the experimental values in Fig. 4-*a*). It can be observed that for any indentation depth the measured load values are larger than the predicted ones, explaining the difference in elastic modulus found between uniaxial compression and macroindentation data. This discrepancy may be due to different issues of the indentation process not considered in the idealized FEM simulations.

The main issue may be the presence of adhesive forces contemplated neither in FEM simulations nor in elastic contact solutions. Adhesion forces increase the contact radius, a, for a particular applied load P over that predicted by the Hertz equations (Fisher-Cripps, 2000). Adhesive force is significant for very compliant samples even when the contacts are large. On the contrary, the adhesion force becomes significant only at very small contacts for samples with large elastic modulus. It has already documented that adhesion forces could yield to overestimation of elastic modulus in depth sensing indentation experiments [Gupta et al., 2007; Liao et al., 2010; Fisher-Cripps, 2000].

Friction between indenter and sample materials could also lead to overestimation of elastic modulus. In order to analyze this effect on the indentation response of hydrogel samples reasonable friction coefficients between 0.1 and 0.3 were incorporated in FEM indentation simulations. The effect of friction is exemplified in Fig. 4-*b*). It is observed that it has a small influence on the indentation curve and hence on the determination of elastic modulus (see the calibrated parameters included in Fig. 4-*b*). This result is in good agreement with the works of Zhang et al. (2013) and Lee et al. (2003), meaning that the existence friction has little influence on the indentation response of hyperelastic solids.

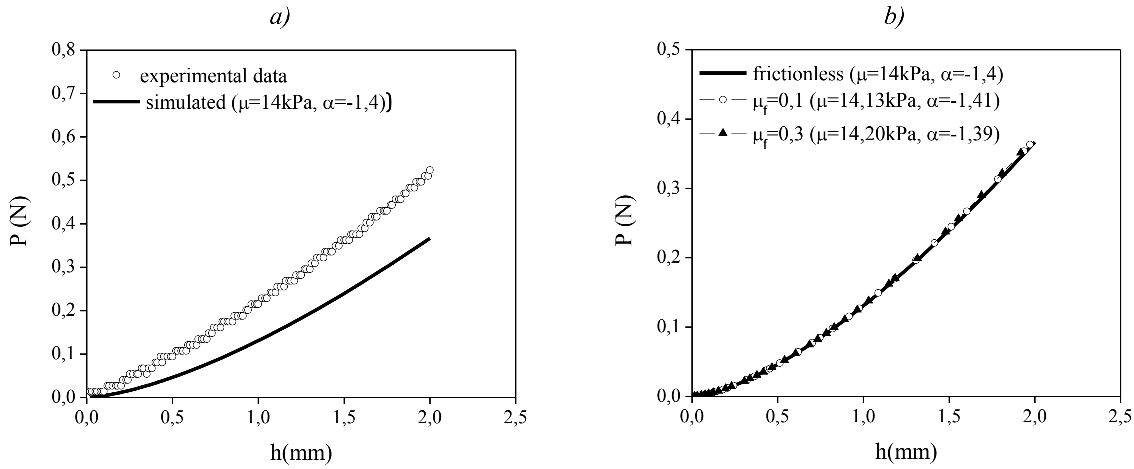

Fig. 4. Finite Element Modeling simulations of hydrogel indentation response for spherical punch: *a*) Comparison with measured curves considering constitutive parameters arisen from uniaxial compression; *b*) Effect of friction on *P-h* curves and calibrated parameters (α, μ).

Other effect is the difference in mechanical behavior that could exhibit the hydrogel sample under different stress-states. The Ogden constitutive model parameters were determined from uniaxial compression data and assumed valid under the complex stress-state under the indenter. One known disadvantage of Ogden models is that generate different material parameters from different stress configurations [ABAQUS, 2001]. Fig. 5 shows that there are regions near the indenter in which the stress-state is positive despite the major volume of material beneath the indenter is subjected to compressive stresses, which are actually larger than tensile ones. Lin et al. (2009) proposed indentation force-depth relations for several hyperelastic models assuming compressive stress states and validating them via finite element simulations. This means that tensile contributions to the overall indentation response can be neglected without large errors. Therefore adhesion appears as the major issue affecting the accurate determination of elastic modulus.

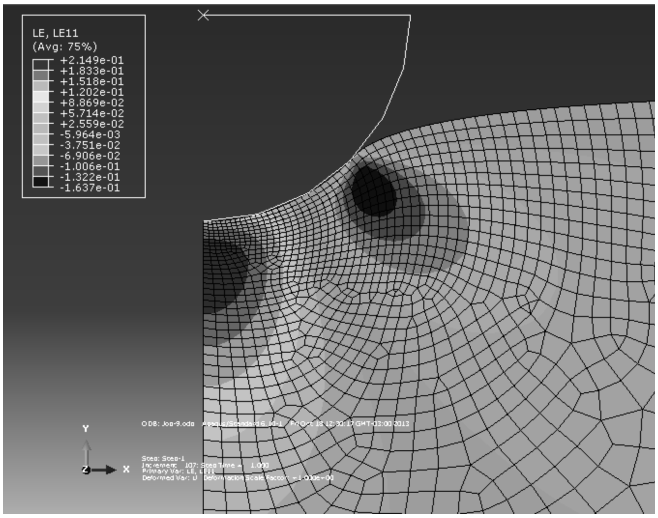

Fig. 5. Logarithmic strain distribution in the *x* direction obtained by FEM simulation of the spherical indentation response of an hydrogel. The presence of elongation deformations is denoted.

3.3. Nanoindentation

Fig. 6-*a*) and *b*) shows typical load-depth curves measured in nanoindentation experiments. Load-depth curves appear almost linear and the loading and un-loading curves do not match each other due to a relaxation process occurring at the imposed maximum indentation depth. The negative forces registered during unloading at depth approaching initial contact indicates the presence of adhesive forces between the hydrogel surface and the diamond tip [Gupta et al., 2007].

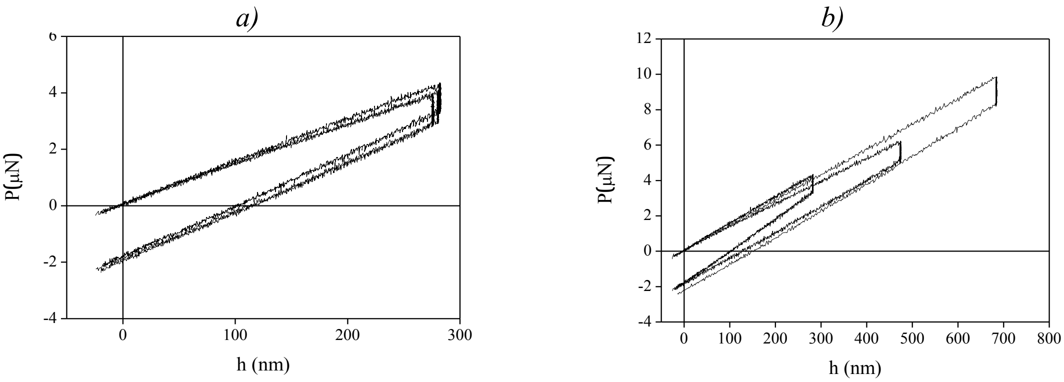

Fig. 6. Load-depth curves obtained in Nano-indentation experiments: Data replication at h=300nm (*a*) and Data at different maximum indentation depths (*b*).

As very low deformation levels were achieved in nanoindentation experiments (h/R<0.008), the material behavior was assumed to be elastic instead of hyperelastic. Accordingly, two approaches were used to obtain elastic modulus values: the Hertz´s elastic solution and the Oliver-Pharr method [Oliver and Pharr, 1992]. The Hertz's solution yields:

$$E_r = \sqrt{\frac{S^3}{6RP_{max}}} \tag{8}$$

where E_r is the reduced elastic modulus, P_{max} is the force achieved at maximum indentation depth and S is the contact stiffness obtained from the initial slope of the unloading curve. In the Oliver-Pharr approach:

$$E_r = \frac{\sqrt{\pi}}{2}\frac{S}{\sqrt{A_c\left(h_c\right)}} \tag{9}$$

where $A_c(h_c)$ is the actual contact area of the spherical diamond tip. Given that the indenter can be considered perfectly rigid and the hydrogel sample incompressible (ν=0.5), the relationship between E_r and E is:

$$E = E_r\left(1-\nu^2\right) = 0.75E_r \tag{10}$$

Fig. 7 shows the calculated elastic modulus values as a function of maximum indentation depth. It is observed that both approaches yield almost the same elastic modulus. The scattering in the E values decreases as the indentation depth increases due to the less influence of the surface roughness in the contact area. Values display a decreasing trend with increasing maximum indentation depth. This trend was also observed in nanoindentation experiments performed on polydimethylsiloxane elastomer (PDMS) (Gupta et al., 2007; Liao et al., 2010). The decreasing trend is a consequence of the adhesive forces acting between the punch and the sample surface.

By coupling adhesive interactions within the Johnson-Kendall-Roberts adhesion contact model and the Hertz's elastic contact solutions, Liao et al. (2010) proposed a hybrid model that accurately fit the decreasing trend in elastic modulus values of PDMS. The model allows the determination of E and the work of adhesion, $\Delta\gamma$, from a given set of experiments.

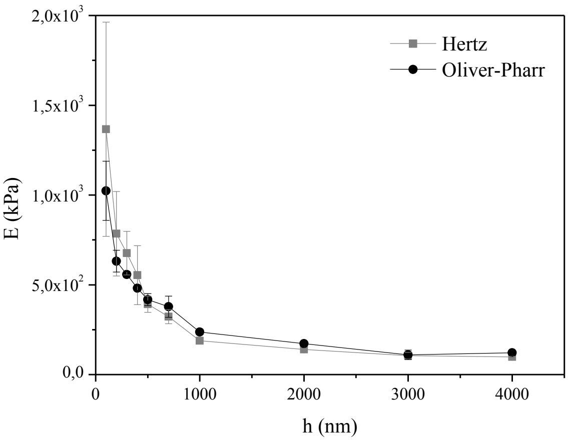

Fig. 7. Elastic modulus values estimated from Hertz´s and Oliver-Pharr models as a function of maximum indentation depth in nanoindnetation experiments.

The hybrid model is represented by a complex equation that relates E^{Hertz} with the maximum applied load (P_{max}) through two fitting parameters: the adhesion force (P_{ad}) and the elastic modulus E (Liao et al., 2010). A value of **42kPa** is obtained by fitting the equation to experimental data using minimum least squares. The work of adhesion $\Delta\gamma$ is 93.4mJ/m^2, showing that the adhesive forces are of the same order of magnitude than indentation forces. The elastic modulus value is in excellent agreement with that obtained in uniaxial compression test

4. Conclusions

The following conclusions are drawn from the present study:

- The elastic modulus values obtained from the inverse method and macroindentation data are larger than those obtained from uniaxial compression or small strain oscillatory experiments due to adhesion between indenter and sample materials.
- The assumption of elasticity instead of hyperelasticity slightly overestimates the elastic modulus value at the macroscale. Pure elastic behavior can be assumed without errors to analyze nanoindentation data due to the low *h/R* range achieved.
- Friction has little effect on the indentation response of hydrogels and hence hardly influences the calculus of elastic modulus.
- The impact of adhesion effect increases with reducing the indentation length scale. The indentation response at the nanoscale is strongly affected by adhesion forces.
- The hybrid model proposed by Liao et al. (2010) for a synthetic elastomer is suitable to describe the decreasing trend of elastic modulus with penetration depth for the hydrogel. The thrown elastic modulus value coincides with the elastic modulus determined from lubricated uniaxial compression data.

Further work will be carried out in order to incorporate adhesion effects in the FEM indentation simulations since it appears as the major issue complicating the extraction of elastic modulus values from experimental data via inverse analysis.

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