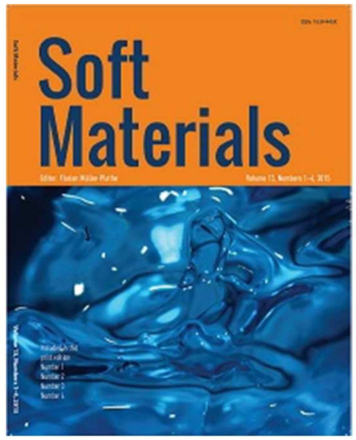

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On the Feasibility of Identifying First Order Ogden Constitutive Parameters of Gelatin Gels from Flat Punch Indentation Tests

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Answers to Reviewer(s)' Comments

Reviewer: 1
Comments to the Author

Soft Materials
Article: On the feasibility of identifying first order Ogden constitutive parameters of gelatin gels from flat punch indentation tests

Authors: L. Sanchez Fellay, L.A. Fasce, M. Czerner, E. Pardo and P.M. Frontini

Referee report

The paper deals with inverse identification of mechanical parameters of gelatin gels. The material model is chosen a priori (Ogden) and although general success is reported in terms of correct identification of the two parameters of this model, it is highlighted for which cases this method and material model fail to characterise the gels. I believe the paper would be of interest to the readers of Soft Materials. Some suggestions for improving the paper are listed below:

Authors thank the Reviewer's comments because they greatly help to improve the manuscript.

1) English can be improved in several places. Also I am not certain the term 're-identified' parameters is the right term to use throughout the script. How about 'inversely predicted' parameters?

English grammar was corrected. Changes were highlighted in red color in the revised version.

Regarding the use of the "re-identified" term, we give here a brief explanation and mention the changes introduced in the manuscript to clarify this issue:

In this work, we called "identified parameters" to those that are obtained by the inverse analysis using physical indentation curves as input data (ie. using experimental force and displacement measurements). In these cases, the values of the Ogden parameters that we want to identify by inverse analysis are unknown.
On the other hand, we called re-identified parameters to those that are recovered by inverse analysis using a simulated curve as input data. In these cases, we know the actual values of the Ogden parameters that have to be identified because we use them to generate the force-displacement curve. The initial guesses in the inverse analysis are assumed 0.5 and 1.5 times the known parameters values. This procedure, in which virtual experimental data calculated by numerical simulations with chosen parameters replace the real experimental measurements, is often called parameter re-identification and it was used for example by other authors in:
- Rauchs, G.: Optimization-based material parameter identification in indentation testing for finite strain elastoplasticity. Z. Angew. Math. Mech., 86, (2006), 539–562.
- Rauchs, G.; Bardon, J.: Identification of elasto-viscoplastic material parameters by indentation testing and combined finite element modelling and numerical optimization. Finite Elem. Anal. Des. 47, (2011), 653–667.
- Z. Chen, S. Diebels, Nanoindentation of hyperelastic polymer layers at finite deformation and parameter re-identification, Archive of Applied Mechanics, 82(2012) 1041-1056
- Z. Chen, S. Diebels, Nanoindentation of Soft Polymers: Modeling, Experiments and Parameter Identification, TECHNISCHE MECHANIK 34 (2014) 166 – 189.

We modified section 3.4.3 as follows:

"In this work, the averaged curve for spherical indentation of the sample denoted as 2-34 (10%w/w) in (14) was used as input data, the spherical indentation configuration was implemented in FEM and inverse analysis was applied. The identified parameters were compared with those reported by Gamompilas *et al.* (14) and then used to simulate the flat indentation response of the starch hydrogel. The simulated *P-d* curve for flat punch indentation was used as input data and Ogden parameters were re-identified. The initial guesses (α_0, μ_0) for the inverse analysis were assumed to be 0.5 and 1.5 times the known parameters (*ie.* the α and μ values inversely predicted from spherical indentation). The stability of the solution was analyzed by evaluating errors in the re-identified parameters yielded by possible errors in the input data. These errors were introduced by adding random noise within 1 and 2% of the maximum force to the simulated *P*-*d* curve."

And, section 4.1 as follows:

"… These initial guesses (μ_0, α_0) were assumed to be 0.5 and 1.5 times their known values (μ=3.67kPa, α=-6.67). For both set of initial guesses, Ogden parameters could be accurately re-identified. The stability of the solution was analyzed using polluted input data in the inverse analysis. …"

The use of "identified" and "re-identified" was checked in the whole paper to avoid misunderstandings.

2) page 3, line 1: change a to Greek font

Done

3) page 7, line 24: correct L/D=25 mm to L=D=25 mm. Also in the same section, how did the authors mitigate frictional effects at the loading platens? Would need to use a lubricant?

The correction of L=D=25mm was done in the text.

To avoid friction between gelatin gel sample and compression platens, before testing we used a Teflon spray, which formed a film, on the metal surfaces. As well, we lubricated the flat punch with the same spray.

This information was included in the revised manuscript as follows:

In section 3.2

"Teflon spray was applied at the interface between sample and compression platens in order to avoid friction."

In section 3.3

"Teflon spray was spread over the punch before testing to diminish frictional effects."

4) section 3.3: give sample dimensions

The text was accordingly modified as follows:

"Experiments consisted in penetrating a cylindrical gel sample (L=D=25mm) up to 4mm from the surface while the force and displacements were continuously recorded."

5) page 8, line 55: what does 'tip displacement-into surface range' mean?

In indentation experiments (both physical and simulations), the flat punch penetrated up to 4mm (or 3mm for GeBo10-G sample) into sample surface.

The text was accordingly modified as follows:

"…for every of *n* intervals in which the total displacement was divided."

6) The study investigated eight different gels. This could be turned into a real strength in comparing the obtained stress-strain results. Instead minimal comparison and use of the data was made in that

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respect. Why did the authors choose to study these materials then? What was the motivation? A more in depth discussion would be good.

We choose eight self-supporting gelatin gels of different formulations to cover a wide range of mechanical behavior. We have been working with gelatin gels for some years and know their behavior very well. These gels exhibit different gel strength (stiffness) and swelling behavior.
Gel samples differ in gelatin concentration, gelatin source, type of solvent and type of cross-links (only physical/physical and chemical). It is known from related literature that all these variables are able to alter the stiffness of gelatin gels.

Even it was not the aim of this paper, we agree with the Reviewer that the original version lacks of interpretation of the obtained parameters in terms of gels' formulation. Moreover, the gel selection was not explained and we apologize for the mistake.

We introduced several changes in the revised manuscript and included a list of related literature to justify the selection of the prepared formulations.

In the Introduction section:

"Eight self-supporting gelatin gels displaying distinct mechanical responses were prepared by modification of their basic formulations. Flat punch indentation and uniaxial compression tests were carried out to characterize the gels."

In section 3.1:

"Gelatin gels based on two gelatin sources were prepared at two powder concentrations, two solvent compositions and, with and without chemical crosslinking agent. It has been widely reported that the mechanical behavior of gelatin gels, especially their stiffness, is linked to the latter variables (25-30)."

In section 4.1:

"For both gelatin sources, the initial shear modulus increases with increasing gelatin powder content and glycerol in gel's formulation (Table II). These results are explained by the universal power law relationship that appears to exist between the modulus and the concentration of triple helices (C_{hel}), which act as the physical crosslinking points of the gelatin gel network (25-27). Increasing gelatin concentration and adding α-helicogenic solvents like glycerol enhance the amount of triple-helix segments and therefore increases the gel stiffness. The initial shear moduli of porcine gelatin gels are higher than their analogous of bovine gels (Table II) because porcine protein chains have higher amounts of glycine-proline-hydroxyproline sequences that are responsible of forming and stabilizing the triple-helix segments in the gel (28). The addition of GTA promotes the formation of chemical cross-links, which leads to the enhancement of the gel stiffness (29-30).
There is not a clear trend between the strain hardening capability of the gels and the modified formulation variables. All α values are within (3.7 to 4.7) or (-1.4 to -1.9) range (Table II). The incorporation of GTA markedly enlarges the strain at break of gelatin gels (Figure 5)."

New References:

-Joly-Duhamel, C. ; Hellio, D.; Djabourov, M. (2002) All gelatin networks: 1. Biodiversity and physical chemistry, *Langmuir* 18: 7208-7217.

-Joly-Duhamel, C.; Hellio, D.; Ajdari, A. ; Djabourov, M. (2002) All gelatin networks: 2. The master curve for elasticity, *Langmuir* 18: 7158-7166.

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-Gornall, J.L.; Terentjev, E. (2008) Helix-coil transition of gelatin: helical morphology and stability, *Soft Matter* 4: 544–549.

-Courty, S.; Gornall, J.L.; Terentjev, E. M. (2006) Mechanically Induced Helix-Coil Transition in Biopolymer Networks, *Biophys. J.* 90: 1019-1027.

- Hellio, D.; Djabourov, M. (2006) Physically and chemically crosslinked gelatin gels, *Macromol. Symp* 241 :23-27.

- Bigi, A.; Panzavolta, S.; Rubini, K. (2004) Relationship between triple-helix content and mechanical properties of gelatin films, *Biomaterials* 25: 5675-5680.

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7) Care must be taken with the material abbreviations as these are not consistent in the text. Also state in captions of Figure 5 and 6 that the abbreviations are explained in Table I.

Sample denomination was checked in the whole manuscript. Figure captions were modified according to Reviewer's suggestion.

8) page 11, line 43: Any explanation as to why BoGe10-G cracked earlier?

The selection of 4mm as maximum applied depth in flat punch indentation experiments was initially based on the Bloom Test. However, this condition is applied to gels having a concentration of 6.67%, tested at 10ºC and with no additives. It is expected that gels do not crack under these conditions. Obviously, this condition is too severe for BoGe10-G sample tested at room temperature. However, the determination of the gel strength by flat punch indentation can be done up to any specified penetration depth. If cracking occurs, Ogden model and simulations will not describe the real behavior. Therefore, we decided to decrease the penetration depth up to 3mm for BoGe10-G experiments.
We think that, comparing BoGe10 and BoGe10-G behaviors, the increase in gel stiffness due to the addition of glycerol caused a local increase in the stress field developed in the circumferential contact line, exceeding the gel toughness. This is because, for a given depth, the higher the gel modulus, the higher the stress developed.
We know from a the results of a work in progress, that the addition of glycerol caused an increase in shear modulus larger than the increase in fracture toughness (determined by the Wire Cutting method)
The measured Gc and μ values are as follows:

	μ (kPa)	Gc (N/m)
GeBo10	6.91	7.15
Gebo10-G	15.4	9.5

We believe that this feature is out of the scope of the present work. We will publish these results as soon as possible in another work leading with the link between textural and elastic properties and fracture toughness in gelatin gels.

9) As Ogden seems to fail for the gels that had alpha larger than 2, have the authors considered using another hyperelastic potential to avoid this difficulty?

We made some experiences using the Eight Chain Model developed in the group of Mary Boyce at the MIT. As it occurred with the Ogden model, the flat punch indentation response resulted insensitive to the parameter related with strain hardening capability (λ lock) in the range measured for gelatin gels. We decided to not include these results in the manuscript.
Zhang et al. (see please Ref. 20 in the revised manuscript)) used several hyperelastic models (Money-Rivlin, Arruda-Boyce and Fung) to identify hyperelastic parameters from spherical indentation data. They concluded that the possibility of determining other properties besides the initial shear modulus relies on the actual parameter values. They reported that parameters can be identified by inverse analysis without significant errors only if the values of θ (Mooney Rivlin) and b (Fung) are large or λ_m (Arruda-Boyce) is small. These finding are in agreement with our results. So,

we think that the lack of sensitivity is due to the gelatin gel behavior itself rather than the Ogden hyperelastic function. Note that, as mentioned in the original manuscript, the identification of parameters for other physical gels (like starch gels) using the Ogden model is possible.

A brief comment was added in section 5.2 as follows:

"Zhang et al. applied Mooney-Rivlin, Arruda-Boyce and Fung hyperelastic models to analyze spherical indentation data using inverse method (20). They found that constitutive model parameters associated with the strain hardening phenomena (θ in Mooney-Rivlin, *b* in Fung and λ_m in Arruda-Boyce models) can be univocally extracted only in a certain specific parameter range, as demonstrated here for α in the case of Ogden model."

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10) I do not get what the authors mean by strain softening. All the curves look like they show strain hardening. They refer to Figure 4b on page 14, line 22 but still it is not clear. Unless they authors can explain and justify using this term, I suggest removing figure 10.

The Reviewer is right; the writing of this part of the paper is rather confused. In fact, all materials show strain hardening. Those Ogden materials with $\alpha < 2$ display larger strain hardening than that predicted by a NeoHookean solid with the same μ. On the other hand, we refereed to "strain softening" the hardening displayed below the one predicted by the Neo-Hookean solid. In the revised version, we denoted these behaviors as H-NH and L-NH, respectively.

Section 5.1 was re-written. As well, Figure 4-b in the original version was actually Figure 10-b, we corrected the Figure number and sincerely apologize for the mistake. Figure 10-c) was also modified according with the new definitions.

"Uniaxial compression curves arisen from Eq. (2) for various fictitious Ogden materials are plotted in Figure 10 together with the Neo-Hookean solid responses ($\mu_{NH}=\mu$). The mathematical expression of the stress-stretch ratio relationship (Eq. 2) clearly states that the stress scales with μ for all strains. At large strains, any value of α predicts strain hardening over the Neo-Hookean solid behavior (Figure 10-*a*); hereafter called H-NH. However, at lower strains ($0.8 > \lambda > 1$ range) only $\alpha < 2$ yields to H-NH, while $\alpha > 2$ predicts strain hardening below the Neo-Hookean solid behavior (Figure 10-*b*); hereafter called L-NH. At very low strains ($0.97 < \lambda < 1$), uniaxial compression curves predicted by both Ogden and Neo-Hookean models overlap, meaning that α parameter has practically no effect on the response (Figure 10-*b*). The behavior of α parameter is overviewed in the map shown in Figure 10-*c*). This map was constructed comparing the stresses evaluated from Ogden (Eq. (2) for different values of α) and Neo-Hookean models for the whole range of stretch ratios ($0 < \lambda < 1$). For $\alpha > 2$, the strain level at which H-NH occurs decreases with increasing the α parameter value. $\alpha > 500$ show H-NH in the whole deformation range (results not included in the map). For $-0.9 < \alpha < 2$, a transition from H-NH to L-NH occurs in $0.5 > \lambda > 0$ range. $\alpha < -0.9$ displays H-NH in the whole deformation range."

11) Similarly the work described in the last paragraph of section 5.2. Can the authors clarify or omit in the final paper?
The analysis was completely re-written as follows:

"To verify that the inverse method failure is not linked to the trust region algorithm, parameters re-identification is performed using the minimization procedure based on the *P-d* library data described in section 3.4.4. An example of the values adopted by the objective function $\omega(x)$ is shown in Figure 12. In the shown example, a single simulated *P-d* curve for an Ogden material with α >2 is used as P^{i}_{exp} in Eq. (3). The flattened shape of the plotted ω(x) surface in the "α axis" (Figure 12) confirms the lack of sensitivity of the α parameter.

To complete the sensitivity analysis, re-identification of Ogden parameters by the minimization procedure based on the *P-d* library data is finally carried out using more realistic input data. Examples of Ogden materials that display α <2 and α >2 are considered. For each case, 30 simulated *P-d* curves polluted with two levels of randomly distributed noise, which resemble experimentally measured curves, are used. Then 30 objective function surfaces are determined and 30 sets of Ogden parameters are identified. The average values of the Ogden parameters are listed in Table IV together with the standard deviation. As judged from the standard deviation values, Ogden parameters can be re-identified with high accuracy for the case of α < 2 despite the *P-d* data pollution.

On the other hand, a large discrepancy between the known and re-identified α parameter is observed in the case of α > 2. The error in the solution increases with increasing the noise level in the input P-d data."

12) State what 'normalized parameter' refers to in Figures 4.

The captions of Figures 4 and 11 were modified as follows:

"**Figure 4.** Evolution of Ogden parameters with iteration number in the inverse method starting from two sets of initial guesses and using different input data: a) simulated flat punch *P-d* curve for a modified starch hydrogel (μ=3.67kPa; α= -6.67); b) P-d curve polluted with two levels of noise (1% and 2%). Ogden parameters are plotted normalized by their known values."

"**Figure 11.** Evolution of Ogden parameters with iteration number in the inverse method starting from for two sets of initial guesses and using different input data: a) simulated flat punch *P-d* curve for fictitious Ogden material (μ=20kPa; α= 3); b) *P-d* curve polluted with two levels of noise (1% and 2%). Ogden parameters are plotted normalized by their known values."

As well, the Y and X axis of both figures were modified to gain clarity.

13) Some curves in Figures 8 are not very clear. Can you change symbol styles so that they are clearly visible?

Figures 8-a) and b) were improved.

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Title:

On the Feasibility of Identifying First Order Ogden Constitutive Parameters of Gelatin Gels from Flat Punch Indentation Tests

Abbreviated Title:

Ogden Parameters Identification from Flat Indentation

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Authors:

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Key words:

INDENTATION, FINITE ELEMENT MODELING, HYPERELASTIC MODEL PARAMETERS, GELATIN GELS.

Short Abstract (100 words)

The feasibility of extracting First Order Ogden constitutive parameters of gelatin gels from experimental flat punch indentation curves is analyzed. Eight gelatin gel samples of different formulations are evaluated. Identification of constitutive parameters is carried out by an inverse method which combines finite element modeling simulations and numerical optimization. Parameters are compared with those obtained from uniaxial compression. A parametric study of the influence of model parameters on the shape of curves and a sensitivity analysis of parameter re-identification is performed. Accurate extraction of parameters is possible if the domain in which α parameter stands is lower than 2.

Extended Abstract

In this work, the feasibility of extracting First Order Ogden constitutive parameters of gelatin gels from experimental flat punch indentation curves is considered. The adopted indentation configuration is that used in the technological Bloom test for gelatins. Eight gelatin gel samples of different formulations are prepared and evaluated. Identification of constitutive parameters (μ and α) is performed by an inverse method which combines finite element modeling simulations and numerical optimization. Parameters are compared with those obtained from uniaxial compression using the analytical expression for the stress-stretch ratio relationship. Under uniaxial compression, gelatin gels display strain hardening over the Neo-Hookean model prediction. Multiple set of Ogden parameters are identified depending on the initial guesses. A single set of Ogden parameters is identified from flat punch indentation curves, which not always matches with those obtained in uniaxial compression. The origin of such discrepancy is investigated through a parametric study of the influence of μ and α on the shape of simulated flat punch indentation and uniaxial compression curves. Also, a sensitivity analysis of parameters re-identification is performed for two different

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ranges of α ($\alpha < 2$ and $\alpha > 2$). Flat punch indentation response is insensitive to α in the $\alpha > 2$ range. For such cases, α parameter can not be accurately re-identified. For the tested gelatin gels, α values extracted from flat punch indentation curves appear to be in the insensitive range. However, the uniaxial compression responses can be well described using the parameters identified from experimental flat punch indentation tests at least up to a stretch ratio of about 0.65. For gels that display appreciable strain hardening at low deformations, *ie.* $\alpha < 2$, first order Ogden constitutive parameters can be accurately identified from flat punch indentation tests. This test configuration is appealing since, opposite to spherical indentation configuration, the non-linearity of the load-depth curve is solely due to strain hardening of the material unlike that displayed by a Neo-Hookean solid.

1. Introduction

Nowadays, gelatin gels are a kind of soft materials that find an increasing number of structural applications in food, pharmaceutical, ballistic and biomedical industries, and especially in tissue engineering (1-6). Comprehending deformation behavior of soft matter is of great importance for design and selection of suitable materials for such specific applications.

A simple method widely used to mechanically characterize gels and other soft materials is the puncture test, in which a probe penetrates the sample to a required depth (7-9). Data are interpreted in terms of a gel strength value. For gelatins, gel strength is conventionally referred to as Bloom. The Bloom test is used by the gelatin producers and end-users as a technological key quality indicator (10). A cylindrical flat-ended punch of specific dimensions is introduced 4 mm into a gelatin sample prepared under specified concentration, time and temperature conditions, and the force exerted expressed in grams constitutes the Bloom number. The Bloom number is a function of structure and molecular mass of gelatin and it is assumed to be representative of its mechanical behavior (11, 12).

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Gelatin gels often undergo large deformation due to their low stiffness (1-100~kPa) and display highly non-linear hyperelastic response. Hence, the use of a single parameter to characterize the mechanical behavior for structural applications such as Bloom number or gel strength appears to be inadequate. This issue is clearly exemplified in Figure 1, in which two gel samples (A and B) that exhibit different behavior display the same Bloom number. The work expended in penetrating the punch is higher for material B than for A. Moreover, after 4 mm of penetration depth, the larger strain hardening capability of material A makes itself stiffer than material B, unlike the opposite stands for lower deformation levels.

The Ogden model has been shown to correctly capture the hyperelastic behavior of soft materials including hydrogels (13-15), and living tissues (16-18). The model parameters can be extracted from the mechanical response measured in experimental tests (19). Among the variety of tests available, the indentation configuration has many advantages over the others: it can be applied to non-self supporting gels, it does not require specimens with particular shapes, it is easy to implement, it is non destructive and it can be applied to measure local or global properties depending on the scale size of the punch. The main disadvantage is that the identification of parameters is not as simple as for uniform strain distribution like uniaxial compression. The deformation state developed beneath the punch is multiaxial and depending on the tip geometry can be also non symmetrical. These characteristics together with the non-linearity of the hyperelastic gel behavior make the extraction of constitutive parameters mathematically complex. Since it is not possible to develop an explicit analytical expression relating the indentation force-displacement curve with the material constitutive parameters, the use of inverse analysis assisted by finite element modeling (FEM) is compulsory.

Determining hyperelastic constitutive parameters from indentation data constitutes one of the main topics of current investigations involved with mechanics of soft matter (14, 20-22). Challenges such as proper model selection or strain and stress states required to identify physically meaningful material parameters are still subjects of debate.

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A successful precedent in using indentation data to extract first order Ogden parameters has been introduced by Gamompilas *et al* (14). They used spherical indentation load-depth curves, FEM simulations and an optimization algorithm to identify Ogden parameters of modified-starch based hydrogels of different stiffness (0.06 to 55kPa). They found an excellent agreement between parameters obtained from spherical indentation and shear or uniaxial compression curves.

Encouraged by the good results of Gamompilas *et. al* work (14), we explored the possibility of identifying First Order Ogden parameters of gelatin gels from indentation data obtained in the flat punch configuration of the Bloom test. Eight self-supporting gelatin gels displaying distinct mechanical responses were prepared by modification of their basic formulations. Flat punch indentation and uniaxial compression tests were carried out to characterize the gels. The identification of constitutive parameters from flat punch indentation curves was performed combining FEM simulations and a minimization procedure. The method was validated using the data of starch gels reported by Gamompilas *et al.* in ref. (14). On the other hand, extraction of parameters from uniaxial compression was performed by fitting the Ogden stress-stretch ratio analytical expression to experimental curves. The constitutive parameters thrown by both tests configurations were compared and the capability of the parameters identified from indentation data to predict the uniaxial compression response was analyzed. FEM simulations were used to investigate the effect of μ and α parameters on the shape of the indentation and uniaxial compression curves. A sensitivity analysis of re-identification of Ogden parameters was finally performed to establish the applicability range of the proposed methodology.

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2. Constitutive model description

The Ogden constitutive model is able to represent the response of an isotropic hyperelastic solid. The Ogden model is included in the group of constitutive models in which the stress-strain relationship derives from a strain energy density function. The Ogden strain energy density is expressed as (23):

$$U = \sum_{i=1}^{n} \frac{2\mu_i}{\alpha_i^2}\left(\lambda_1^{-\alpha_i} + \lambda_2^{-\alpha_i} + \lambda_3^{-\alpha_i} - 3\right) + \sum_{i=1}^{n} K_i \left(J^{el} - 1\right)^{2i} \quad (1)$$

Where α, μ and K are the constitutive parameters, λ are the principal stretches and J is the determinant of the stretch tensor. For incompressible materials, a value of $K \approx 10^5$MPa is assumed (24) and deformation satisfies J=1 so that the second term in Eq. 1 is neglected. For the particular case a one term Ogden energy density function (n=1), the model comprises only two parameters, the strain hardening exponent, α, and the shear modulus, μ.

Under a uniaxial compression state, the nominal stress (σ) as a function of the stretch ratio (λ) for a first order Ogden material has the following mathematical form:

$$\sigma = \frac{2\mu}{\alpha}\left(\lambda^{(\alpha-1)} - \lambda^{\left(-\frac{\alpha}{2}-1\right)}\right) \quad (2)$$

In equations (1) and (2), the α parameter can take either positive or negative values.

The first order Ogden model reduces to the Neo-Hookean solid for α = 2. The Neo-Hookean constitutive model is a single parameter model (μ=μ_{NH}) that can be used to describe the change from initially linear to non-linear stress-strain relationship of hyperelastic materials. But, it is known that the model fails in predicting deformation behavior at large strains.

3. Experimental and Methodology

3.1 Preparation of Gelatin Gel Samples

Gelatin gels based on two gelatin sources were prepared at two powder concentrations, two solvent compositions and, with and without chemical crosslinking agent. It has been widely reported that the mechanical behavior of gelatin gels, especially their stiffness, is linked to the latter variables (25-30).

Bovine hide gelatin (Type B, Bloom 200) and pork skin gelatin (Type A, Bloom 250) kindly supplied by Rousselot Argentina S.A. were used. pH 7, pH 10 buffer solutions and binary mixtures of

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glycerol/buffer solutions (40 % w/w) were used as solvents. The solvent pH was adjusted over the gelatin isoionic point to restrict the reordering of gelatin into triple-helix. Glutaraldehyde (GTA) was used as the chemical crosslinking agent.

The gels were prepared by dissolving gelatin powders in the proper solvent under continuous stirring during 15 min at 50ºC. To obtain chemically crosslinked gelatin gels a GTA solution (0.75 % w/w of gelatin powder) previously prepared was also added to the gelatin solution and homogenized. Then, gelatin solutions were poured into specially designed cylindrical Delrin® molds (height=25 mm, diameter=25 mm) and allowed to cool at room temperature to form the gels. Samples were wrapped in film to minimize drying out and stored at 4ºC during 48 hours. Before testing, samples were conditioned at 21ºC during 2 hours. Details of gelatin gels formulations and sample denomination are listed in Table I.

3.2 Uniaxial compression tests

Uniaxial compression test were carried out at 21ºC in an INSTRON 4467 universal testing machine using a load cell of 0.5kN. Cylindrical gel samples (L/D=25mm) were deformed up to fracture at a crosshead speed of 5mm/min. Teflon spray was applied at the interface between sample and compression platens in order to avoid friction.

Raw force –displacement (P-d) data were converted to nominal stress- stretch ratio (σ–λ). Identification of Ogden parameters was performed directly by fitting Eq. (2) to the experimental σ–λ data by Least Squares method.

3.3 Flat Indentation experiments

Indentation experiments were carried out at 21ºC under displacement control conditions using an INSTRON 3369 universal testing machine equipped with a load cell of 0.1kN. A flat ended cylindrical plunger having 10mm of diameter made of polymethylmethacrylate (PMMA), analogous to the one utilized in the Bloom Test, was used. Teflon spray was spread over the punch before testing to

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diminish frictional effects. The indenter penetration speed was 5mm/min. Experiments consisted in penetrating a cylindrical gel sample (L=D=25mm) up to 4 mm from the surface while the force and displacements were continuously recorded.

3.4. Identification of Constitutive Parameters from Indentation curves

3.4.1 Finite Element Modeling and Forward Analysis

Finite element indentation simulations were performed using the commercial software ABAQUS. The test configuration was a 5 mm radii cylindrical flat ended punch indenting on cylindrical samples with L=D=25mm. An axysimmetrical model was assumed and the mesh was constructed using linear quadrilateral elements, as shown in Figure 2-a). 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 computational time. The punch was assumed as a rigid body and the contact between punch and sample surface was considered frictionless. The ABAQUS material library was used since it includes the Ogden constitutive material model described by Eq. 1. During simulated indentation experiments, a downward displacement along the Y axis and into-the-surface was imposed to the indenter tip, as shown in Figure 2-b). The indentation curve was obtained by the normal reaction force (P) as a function of the tip displacement in the Y axis (d). To test the mesh sensitivity, the mesh was refined until the simulated force-depth (P-d) curves overlapped.

In addition, the case of the spherical indentation test configuration used in (14) was implemented in ABAQUS. This configuration was used to validate the used inverse method.

3.4.2 Inverse Analysis

The inverse analysis was formulated by the minimization of an objective function with respect to the unknown constitutive parameters. It was defined as the quadratic discrepancy between simulated curves obtained by FEM and experimentally measured ones (section 4.5), as defined in Eq. (3):

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$$\omega_{(x)} = \sum_{i=1}^{n} \left(P_{FA}^{i} - P_{\exp}^{i} \right)^2 \quad (3)$$

where $\omega_{(x)}$ is the objective function, x is a vector that collects the unknown constitutive parameters, P_{FA}^{i} are the force data points calculated by forward analysis and $P_{\exp}^{i}$ the experimentally measured force points for every of *n* intervals in which the total tip displacement was divided.

The minimization procedure was performed by the Trust Region algorithm (29-32). The solution procedure started with the selection of a suitable set of initial parameters (α_0, μ_0), which were iteratively improved by the optimization algorithm.

3.4.3 Method Validation

Spherical indentation data of a modified sago starch based hydrogel reported by Gampompilas *et. al* (14) were chosen to validate the inverse method. It was previously shown (14) that first order Ogden model correctly describes the response of this type of hydrogels under uniaxial compression and spherical indentation for a wide range of gels' stiffness. Moreover, parameters could be identified with high accuracy using FEM simulations and an optimization procedure based on the Levenberg-Maquardt method (14).

In this work, the averaged curve for spherical indentation of the sample denoted as 2-34 (10%w/w) in (14) was used as input data, the spherical indentation configuration was implemented in FEM and inverse analysis was applied. The identified parameters were compared with those reported by Gamompilas *et al.* (14) and then used to simulate the flat indentation response of the starch hydrogel. The simulated P-d curve for flat punch indentation was used as input data and Ogden parameters were re-identified. The initial guesses (α_0, μ_0) for the inverse analysis were assumed to be 0.5 and 1.5 times the known parameters (*ie.* the α and μ values inversely predicted from spherical indentation). The stability of the solution was analyzed by evaluating errors in the re-identified parameters yielded by possible errors in the input data. These errors were introduced by adding

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random noise within 1 and 2% of the maximum force to the simulated *P-d* curve for flat punch indentation.

3.4.4 Sensitivity Analysis

The sensitivity analysis of re-identified Ogden parameters was performed using the trust region method and also considering another minimization approach. In the later, the re-identification of constitutive model parameters was carried out by searching the minimum value of Eq. (3) using a library data as P_{FA}^{i} points. The library contains 11000 indentation *P-d* curves, which were FEM simulated by varying μ from 10 to 100 kPa and α from -10 to 10.

4. Results

4.1 Method validation

First order Ogden parameters were extracted by the inverse method using as input data the load-depth curve for a modified starch hydrogel. The identified parameters (μ=3.67kPa, α=-6.67) practically coincide with the ones reported by Gamompilas *et al*: (μ=3.3kPa, α=-4.9) for indentation and (μ=3.1kPa, α=-6.2) for uniaxial compression configurations. Figure 3 plots the starch hydrogel experimental data together with the simulated spherical indentation curves arisen from the identified parameters. The identified parameters adequately describe the spherical indentation response of this hydrogel sample.

Ogden parameters were re-identified using as input data the load-depth curve simulated for flat punch indentation with the previously identified parameters (μ=3.67kPa, α=-6.67). The evolution of parameters with the number of iteration is shown in Figure 4-a) for two different sets of initials guesses. These initial guesses (μ_0, α_0) were assumed to be 0.5 and 1.5 times their known values (μ=3.67kPa, α=-6.67). For both set of initial guesses, parameters could be accurately re-identified.

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The stability of the solution was analyzed using polluted input data in the inverse analysis. Figure 4-b) shows the evolution of parameters with iteration number from the same sets of initial guesses than in Figure 4-a). The errors in the re-identified parameters slightly increase with increasing the noise in the indentation P-d curve. The relative errors in the re-identified parameters were 0.29 and 1.55% for μ and α, respectively for a 2% noised indentation curve.

The previous results confirm that α and μ parameters can be identified from flat punch indentation data with high accuracy and moreover, that this solution is stable.

4.1 Uniaxial compression response of gelatin gels

Typical σ–λ curves obtained in uniaxial compression experiments are shown in Figure 5. All gels exhibited non-linear elastic response with strain hardening. The shape of the curves depends on gel formulation.

The Neo-Hookean model was initially used to fit the experimental data in the $0.95<\lambda<1$ range; the obtained μ_{NH} values are reported in Table II. Then, first order Ogden parameters were identified by fitting Eq. (2) to experimental data. The model was capable to capture the uniaxial compression response of all gels, but multiple sets of Ogden constitutive parameters were obtained, depending on the initial guess α_0 and the fitting λ range. The two set of values reported in Table II were obtained for $\lambda_{rupture} < \lambda < 1$ range and assuming the initial guesses μ_0 and α_0 as μ_{NH} and, 2 or -2, respectively. In all cases, one set displays $\alpha > 2$ while the other has $\alpha < 2$. Both sets practically yield the same regression coefficient. The multiplicity of "optimal" set of Ogden parameters is known and inherent to the Ogden strain energy function (19).

For both gelatin sources, the initial shear modulus increases with increasing gelatin powder content and glycerol in gel's formulation (Table II). These results are explained by the universal power law relationship that appears to exist between the modulus and the concentration of triple helices (C_{hel}), which act as the physical crosslinking points of the gelatin gel network (25-27). Increasing gelatin

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concentration and adding α-helicogenic solvents like glycerol enhance the amount of triple-helix segments and therefore increases the gel stiffness. The initial shear moduli of porcine gelatin gels are higher than their analogous of bovine gels (Table II) because porcine protein chains have higher amounts of glycine-proline-hydroxyproline sequences that are responsible of forming and stabilizing the triple-helix segments in the gel (28). The addition of GTA promotes the formation of chemical cross-links, which leads to the enhancement of the gel stiffness (29-30).

There is not a clear trend between the strain hardening capability of the gels and the modified formulation variables. All α values are within (3.7 to 4.7) or (-1.4 to -1.9) range (Table II). The incorporation of GTA markedly enlarges the strain at break of gelatin gels (Figure 5).

4.2 Flat punch indentation response of gelatin gels

The average P-d curves registered for all gel samples are shown in Figure 6. Up to the imposed displacement (4mm) samples showed no signs of cracking at the contact region between the punch and the hydrogel surface, with the exception of the BoGe10-G sample. For this case, the maximum displacement achieved in the experiment was reduced to 3mm to avoid cracking. All gelatin gels showed an almost linear response. The shape of the P-d curve depends on gelatin concentration, solvent type and addition of chemical crosslinking agent. It was observed that under the performed conditions, all samples were able to recover their original shape once the load was removed.

The inverse method was applied to identify the Ogden parameters from flat indentation curves of all gelatin gel samples. For each sample, the average indentation curve shown in Figure 6 was used as input data. The identified parameters are listed in Table III. Contrary to uniaxial compression, single sets of parameters were identified despite starting from different initial guesses.

Initial shear modulus values follow the same trend observed in Table II with gelatin source and concentration, solvent type and GTA presence. Contrary to what was observed for the modified

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starch hydrogel, all gelatin gels displayed α values higher than 2 with the sole exception of the PoGe20 sample.

4.3 Comparison of Ogden parameters obtained in uniaxial compression and flat indentation tests

A direct comparison of the Ogden parameters identified from both test configurations (Tables II and III) is given in Figure 7. Overall, different sets of parameter were identified, being in most cases the values obtained from flat punch indentation quite similar to the set with α>2 obtained from uniaxial compression. However, in some cases, parameters greatly differ.

The capability of the identified parameters from flat indentation curves to predict the uniaxial compression response is shown in Figure 8. Surprisingly, the predicted curve shows a very good agreement with the experimentally measured curves in the whole deformation range for BoGe10, BoGe10-X, PoGe10, PoGe10-G and PoGe20 samples. For the rest of the samples, the prediction is accurate only in lower deformation range (1>λ>0.65). The extrapolated response of the Neo-Hookean solid in the whole stretch ratio range (calculated with μ_{NH} parameters given in Table II) is also plotted in Figure 8. In all cases, the magnitudes of both experimental and Ogden model stresses are higher than the extrapolated Neo-Hookean responses. This means that all gelatin gel samples undergo strain hardening over the Neo-Hookean solid behavior. The observed strain hardening is well predicted using the Ogden parameters identified from flat indentation tests at least up to a certain deformation level.

The deformation field developed under the indenter is quite complex as shown in Figure 2-c. At an indentation depth of 4mm, the maximum strain achieved (logarithmic strain ~0.4) is comparable with a stretch ratio in the compressive state of ~0.67. But, most of the deformed material volume is sustaining a strain equivalent to λ~0.8. The difference in strain levels partially explains the discrepancy observed between parameters identified from both test configurations.

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5. Discussion

5.1 Influence of Ogden parameters on flat indentation and uniaxial compression curves

FEM simulated curves were used to analyze how Ogden parameters individually affect the shape of the flat indentation P-d curve. Figure 9 shows simulated P-d curves for selected values of μ and α. Each parameter influences the indentation curve in very different way.

The μ parameter governs the strength of the material; it gives the initial resistance and acts in the whole displacement range (Figure 9-*a*). According to dimensionless analysis, which states that physical laws do not depend on the arbitrariness in the choice of units of physical quantities, the indentation response of a first order Ogden material can be written, applying the pi-theorem, as:

$$P-d=\mu\, d_f^{\,2}\prod\left(\frac{d}{d_f},\alpha\right) \tag{4}$$

where d_f is the target tip displacement (*ie.* d_f =4mm). Therefore, it clearly emerges from Eq. (4) that the indentation response is directly proportional to the shear modulus of the material. If simulated curves are normalized by the μ parameter, they overlap in a single curve for each value of α parameter.

The effect of α parameter on the indentation response is not straightforward (Figure 9–b). A Neo-Hookean solid (α = 2) displays an almost linear response to flat punch indentation, whereas for Ogden materials the non-linearity of the curve increase with decreasing the value of α below 2. Within the examined displacement range (up to 4mm), the indentation curve is practically unaffected by α parameter for α > 2. From this analysis, it clearly emerges that flat punch indentation curve is sensitive to α parameter only for Ogden materials that display α values much lower than 2. This is the case of the modified sago and maize starch gel samples studied in (14), for which α varied from -3 to -6.

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Uniaxial compression curves arisen from Eq. (2) for various fictitious Ogden materials are plotted in Figure 10 together with the Neo-Hookean solid responses ($\mu_{NH}=\mu$). The mathematical expression of the stress-stretch ratio relationship (Eq. 2) clearly states that the stress scales with μ for all strains. At large strains, any value of α predicts strain hardening over the Neo-Hookean solid behavior (Figure 10-*a*); hereafter called H-NH. However, at lower strains (0.8 >λ >1 range) only $\alpha < 2$ yields to H-NH, while $\alpha > 2$ predicts strain hardening below the Neo-Hookean solid behavior (Figure 10-*b*); hereafter called L-NH. At very low strains (0.97<λ < 1), uniaxial compression curves predicted by both Ogden and Neo-Hookean models overlap, meaning that α parameter has practically no effect on the response (Figure 10-*b*). The behavior of α parameter is overviewed in the map shown in Figure 10-*c*). This map was constructed comparing the stresses evaluated from Ogden (Eq. (2) for different values of α) and Neo-Hookean models for the whole range of stretch ratios ($0 < \lambda < 1$). For $\alpha > 2$, the strain level at which H-NH occurs decreases with increasing the α parameter value. $\alpha > 500$ show H-NH in the whole deformation range (results not included in the map). For $-0.9 < \alpha < 2$, a transition from H-NH to L-NH occurs in $0.5 > \lambda > 0$ range. $\alpha < -0.9$ displays H-NH in the whole deformation range.

5.2 Re-identification of Ogden parameters from flat indentation curves: Sensitivity analysis

As previously shown in Figure 4, the inverse method is accurate to re-identify Ogden parameters for $\alpha = -6.67$. The high accuracy in parameters re-identification extends to Ogden materials that exhibit $\alpha < 2$. For sure, this is because the non-linearity of the flat punch indentation curve given by strain hardening is significant in these cases (Figure 9-b).

The results of the sensitivity analysis of Ogden parameter re-identification for materials that display $\alpha > 2$ are shown in Figures 11 and 12. Figure 11-a) exemplifies the evolution of Ogden parameters with iteration number when the inverse method starts from two different sets of initial guesses (0.5 and 1.5 times the known parameters used to simulate the input P-d data). μ is always re-identified with high accuracy but α can only be recovered if the initial guess α_0 is somewhat underestimated. The failure in re-identifying α is attributed to the lack of sensitivity of the flat punch indentation curve

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to α>2, as previously shown in Figure 9-b) and discussed in section 5.1. The lack of accuracy in parameters re-identification gets worst if errors in the input P-d data are introduced. This is demonstrated Figure 11-b) where the evolution of Ogden parameters using P-d data polluted with two levels of noise (1% and 2%) is shown. The inverse analysis solution is unstable and the method yields errors of about 45% in the α parameter.

To verify that the inverse method failure is not linked to the trust region algorithm, parameters re-identification is performed using the minimization procedure based on the P-d library data described in section 3.4.4. An example of the values adopted by the objective function ω(x) is shown in Figure 12. In the shown example, a single simulated P-d curve for an Ogden material with α>2 is used as P^{i}_{exp} in Eq. (3). The flattened shape of the plotted ω(x) surface in the "α axis" (Figure 12) confirms the lack of sensitivity of the α parameter.

To complete the sensitivity analysis, re-identification of Ogden parameters by the minimization procedure based on the P-d library data is finally carried out using more realistic input data. Examples of Ogden materials that display α<2 and α>2 are considered. For each case, 30 simulated P-d curves polluted with two levels of randomly distributed noise, which resemble experimentally measured curves, are used. Then 30 objective function surfaces are determined and 30 sets of Ogden parameters are identified. The average values of the Ogden parameters are listed in Table IV together with the standard deviation. As judged from the standard deviation values, Ogden parameters can be re-identified with high accuracy for the case of α< 2 despite the P-d data pollution.

On the other hand, a large discrepancy between the known and re-identified α parameter is observed in the case of α> 2. The error in the solution increases with increasing the noise level in the input P-d data.

Zhang et al. applied Mooney-Rivlin, Arruda-Boyce and Fung hyperelastic models to analyze spherical indentation data using inverse method (20). They found that constitutive model parameters

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associated with the strain hardening phenomena (θ in Mooney-Rivlin, b in Fung and λ_m in Arruda-Boyce models) can be univocally extracted only in a certain specific parameter range, as demonstrated here for α in the case of Ogden model.

6. CONCLUSIONS

In this work, the feasibility of extracting first order Ogden model parameters from flat punch indentation test is analyzed. To this aim, literature data for a starch hydrogel, physical measurements for eight gelatin gels of different mechanical behavior and FEM simulations of fictitious Ogden materials are used. The identified parameters are compared with those thrown by the uniaxial compression test. The following main conclusions arise:

Flat punch indentation configuration is very useful to characterize soft materials responding to First order Ogden model. For this configuration, non-linearity of the load-displacement curve arises from strain hardening over the Neo-Hookean almost linear response.

For the tested gelatin gels, two set of "optimal" Ogden parameters are identified from uniaxial compression curves (one with $\alpha > 2$ and one with $\alpha < 2$), while a unique set is identified from flat indentation curves (in most cases with $\alpha > 2$). Parameters values arisen from both test configurations generally differ. The source of discrepancy can be partially attributed to the different strain level experienced by the gels in each test configuration.

The parametric study of the effect of Ogden model parameters on the shape of flat punch indentation and uniaxial compression curves reveals that any value of α predicts strain hardening over the Neo-Hookean model at large strains but only $\alpha < -0.9$ does it within the whole deformation range. The flat punch indentation curve is almost insensitive to α parameter for $\alpha > 2$. Strain hardening over the Neo-Hookean model in flat punch indentation is described with α values lower than 2.

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No limitations in the identification of the μ parameter from flat punch indentation curve are found. This parameter can be accurately identified even if P-d data are polluted as expected in experimental measurements. On the other hand, due to the lack of sensitivity in the indentation response, α can only be accurately identified in cases where $\alpha < 2$.

Gelatin gel samples develop strain hardening at large deformation levels, therefore large errors in the α parameter identified from flat punch indentation is found. However, the uniaxial compression is well predicted up to λ=0.65 with the Ogden parameters identified from flat punch indentation curves.

In summary, accurate extraction of first order Ogden model parameters from flat punch indentation curves can be carried out if the strain hardening of the gels is appreciable at low strains and the domain in which α parameter stands is lower than 2.

7. Acknowledgments

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9. Tables

Table I. Details of gelatin gel formulations and sample denomination.

Denomination	Gelatin source, concentration	Solvent	Glutaraldehyde
BoGe10	Bovine gelatin, 10 % w/w	pH 7 buffer	No
BoGe10-G	Bovine gelatin, 10 % w/w	mixture pH 7 buffer/glycerol (40% w/w)	No
BoGe10-X	Bovine gelatin, 10 % w/w	pH 7 buffer	Yes
BoGe20	Bovine gelatin, 20 % w/w	pH 7 buffer	No
PoGe10	Porcine gelatin, 10 % w/w	pH 10 buffer	No
PoGe10-G	Porcine gelatin, 10 % w/w	mixture pH 10 buffer/glycerol (40% w/w)	No
PoGe10-X	Porcine gelatin, 10 % w/w	pH 10 buffer	Yes
PoGe20	Porcine gelatin, 20 % w/w	pH 10 buffer	No

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Table II. Constitutive parameters arisen from fitting uniaxial compression data to Eq. (2): Neo-Hookean (α=2) and first order Ogden model parameters of gelatin gels.

	Model (Fitting range)				
	NeoHookean model (0.95<λ<1)		Ogden model (~0.3<λ<1)		
Hydrogel / Parameter	μ_{NH} (kPa)	R^2	μ (kPa)	α	R^2
BoGe10	6.69 (±0.03)	0.98075	6.92 (±0.01)	4.67 (±0.01)	0.9997
			4.99 (±0.01)	-1.45 (±0.01)	0.9991
BoGe10-G	12.68 (±0.10)	0.98384	15.40 (±0.08)	4.15 (±0.02)	0.9962
			9.49 (±0.06)	-1.80 (±0.01)	0.9966
BoGe10-X	8.43 (±0.03)	0.99165	13.68 (±0.09)	3.54 (±0.02)	0.9912
			8.07 (±0.06)	-1.61 (±0.01)	0.9931
BoGe20	20.09 (±0.47)	0.88471	23.06 (±0.03)	4.03 (±0.01)	0.9999
			16.01 (±0.12)	-1.41 (±0.02)	0.9992
PoGe10	8.41 (±0.18)	0.80382	11.48 (±0.06)	4.22 (±0.04)	0.98407
			8.03 (±0.06)	-1.45 (±0.02)	0.98591
PoGe10-G	14.09 (±0.23)	0.95754	18.25 (±0.10)	3.99 (±0.02)	0.9973
			10.94 (±0.07)	-1.79 (±0.01)	0.9977
PoGe10-X	10.79 (±0.04)	0.98987	13.89 (±0.02)	4.42 (±0.01)	0.9989
			8.69 (±0.01)	-1.88 (±0.01)	0.9993
PoGe20	30.9 (±0.05)	0.9924	34.83 (±0.11)	3.73 (±0.01)	0.9939
			22.72(±0.01)	-1.49 (±0.01)	0.9940

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Table III. Identified first order Ogden model parameters from cylindrical flat punch indentation data for gelatin gels.

Hydrogel / Parameter	μ (kPa)	α
BoGe10	8.19	4.34
BoGe10-G	14.58	5.43
BoGe10-X	9.73	4.14
BoGe20	24.85	2.01
PoGe10	11.03	4.35
PoGe10-G	19.61	3.45
PoGe10-X	12.46	6.26
PoGe20	30.39	-0.82

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Table IV. Results of inverse analysis by minimization of the objective function (Eq. 3) using the *P-d* library data. The mean values of the 30 experiments with the standard deviation in brackets are reported.

Known values	Identified first order Ogden parameters			
	Noise 1%		Noise 2%	
	μ (kPa)	α	μ (kPa)	α
μ= 20kPa, α= -3	20.08 (±0.21)	-2.96 (±0.05)	20.09 (±0.85)	-2.88 (±0.21)
μ= 20kPa, α= 3	20.08 (±0.90)	3.15 (±1.02)	20.08 (±1.22)	3.21 (±1.35)

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10. Captions to Figures

Figure 1. Flat punch indentation response of two fictitious materials (A and B) that exhibit different hyperelastic behavior but the same gel strength. Curves were simulated considering the Bloom Test configuration and first order Ogden model with different parameters within a realistic range for gels (A: μ=10kPa, α=-5; B: μ=34kPa, α=-1).

Figure 2. Finite element model of cylindrical flat ended punch indentations: *a*) Sketch of final mesh; *b*) Von Mises stress distribution and c) Maximum logarithmic strain in plane principal for a first order Ogden material (μ=10kPa, α=-3).

Figure 3. Indentation response of a sago starch hydrogel: Experimental data taken from (14) and simulated load versus normalized displacement curves. The inside plot shows the Von Mises stress distribution for spherical indentation at maximum displacement.

Figure 4. Evolution of Ogden parameters with iteration number in the inverse method starting from for two sets of initial guesses and using different input data: a) simulated flat punch *P-d* curve for a modified starch hydrogel (μ=3.67kPa; α= -6.67); b) *P-d* curve polluted with two levels of noise (1% and 2%). Ogden parameters are plotted normalized by their known values.

Figure 5. Typical stress-stretch ratio curves obtained from uniaxial compression tests of gelatin gels of: a) bovine source and b) porcine source.

Figure 6. Average P-d curves obtained in flat indentation experiments of gelatin gels of: a) bovine source and b) porcine source.

Figure 7. Comparison of Ogden parameters obtained in uniaxial compression and flat indentation tests: *a*) shear modulus; *b*) strain hardening capability.

Figure 8. Comparison of experimental uniaxial compression curves and predicted curves using Ogden parameters identified from flat indentation tests of gelatin gels of: a) bovine source and b) porcine source.

Figure 9. FEM simulation experiments for different Ogden parameters: a) P-d curves obtained varying μ for a given α (-1.3); b) P-d obtained varying α for a given μ (10kPa).

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Figure 10. Uniaxial compression response of Ogden materials: *a)* Stress- stretch ratio obtained for various α and a given μ (10kPa) for large deformation states ; *b)* Stress- stretch ratio obtained various α and a given μ (10kPa) for small deformation states; *c)* Map of deformation characteristic as a function of α parameter and stretch ratio. The map is valid for any value of μ parameter. See text for H-NH and L-NH definitions.

Figure 11. Evolution of Ogden parameters with iteration number in the inverse method starting from for two sets of initial guesses and using different input data: a) simulated flat punch *P-d* curve for fictitious Ogden material (μ=20kPa; α= 3); b) *P-d* curve polluted with two levels of noise (1% and 2%). Ogden parameters are plotted normalized by their known values.

Figure 12. Example of minimization of the objective function (Eq. 3) using a P-d library data to identify first order Ogden parameters. Results for the case of μ=20kPa and α=3.

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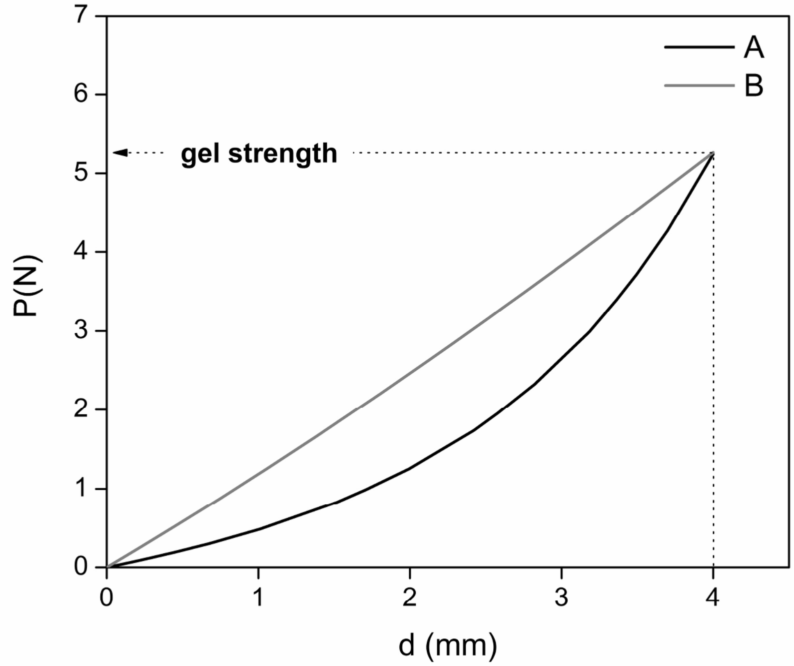

Figure 1. Flat punch indentation response of two fictitious materials (A and B) that exhibit different hyperelastic behavior but the same gel strength. Curves were simulated considering the Bloom Test configuration and first order Ogden model with different parameters within a realistic range for gels (A: μ=10kPa, α=-5; B: μ=34kPa, α=-1).
208x160mm (300 x 300 DPI)

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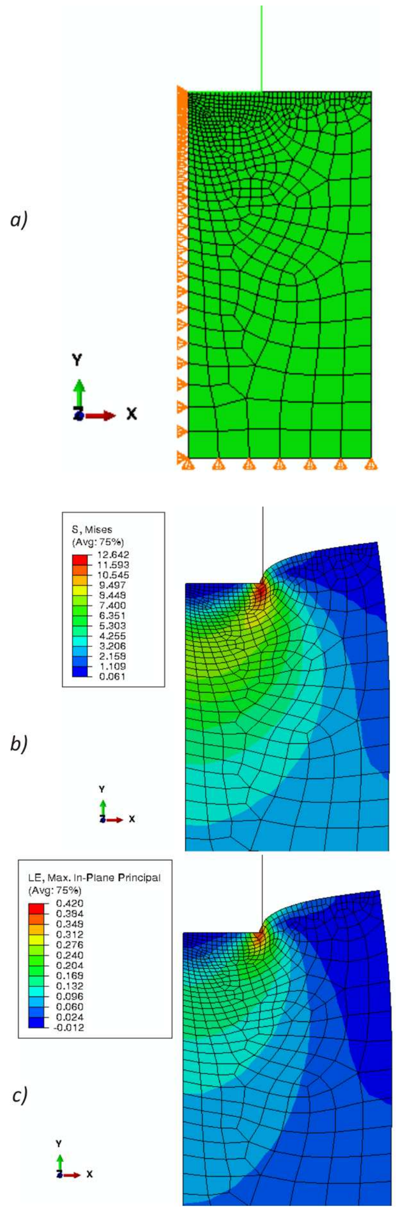

Figure 2. Finite element model of cylindrical flat ended punch indentations: a) Sketch of final mesh; b) Von Mises stress distribution and c) Maximum logarithmic strain in plane principal for a first order Ogden material (μ=10kPa, α=-3).
74x229mm (300 x 300 DPI)

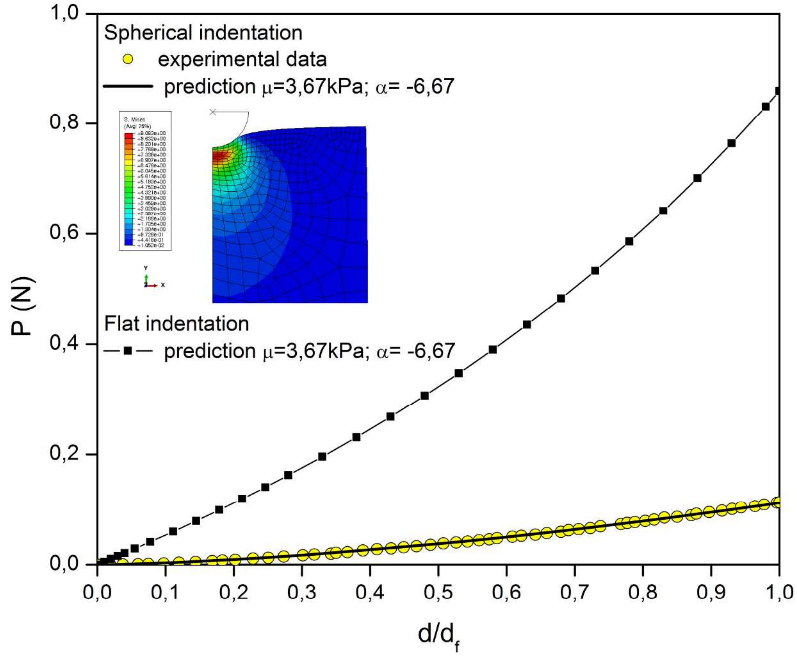

Figure 3. Indentation response of a sago starch hydrogel: Experimental data taken from (14) and simulated load versus normalized displacement curves. The inside plot shows the Von Mises stress distribution for spherical indentation at maximum displacement.
203x156mm (300 x 300 DPI)

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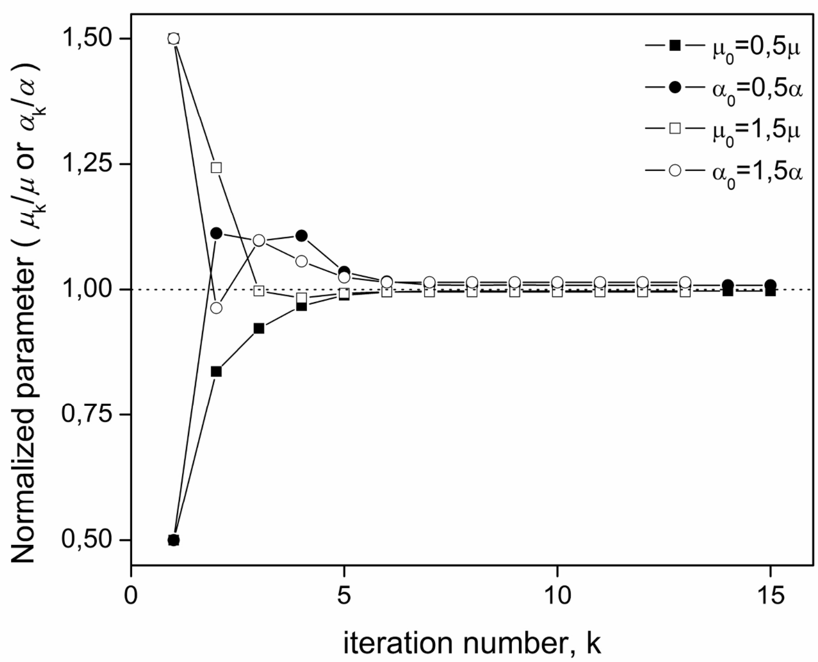

Figure 4: Evolution of Ogden parameters with iteration number in the inverse method starting from for two sets of initial guesses and using different input data: a) simulated flat punch P-d curve for a modified starch hydrogel (μ=3.67kPa; α= -6.67); b) P-d curve polluted with two levels of noise (1% and 2%). Ogden parameters are plotted normalized by their known values.
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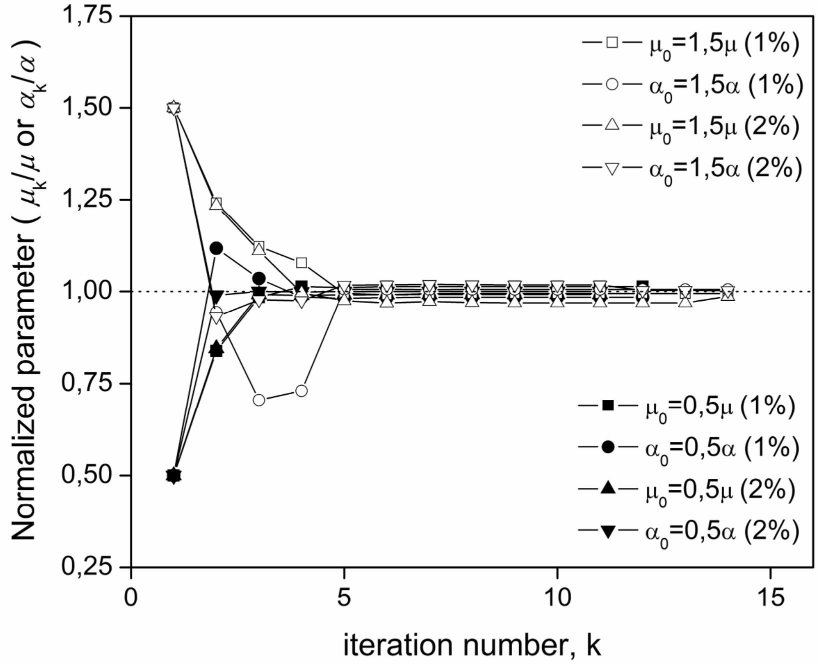

Figure 4: Evolution of Ogden parameters with iteration number in the inverse method starting from for two sets of initial guesses and using different input data: a) simulated flat punch P-d curve for a modified starch hydrogel (μ=3.67kPa; α= -6.67); b) P-d curve polluted with two levels of noise (1% and 2%). Ogden parameters are plotted normalized by their known values.
208x160mm (300 x 300 DPI)

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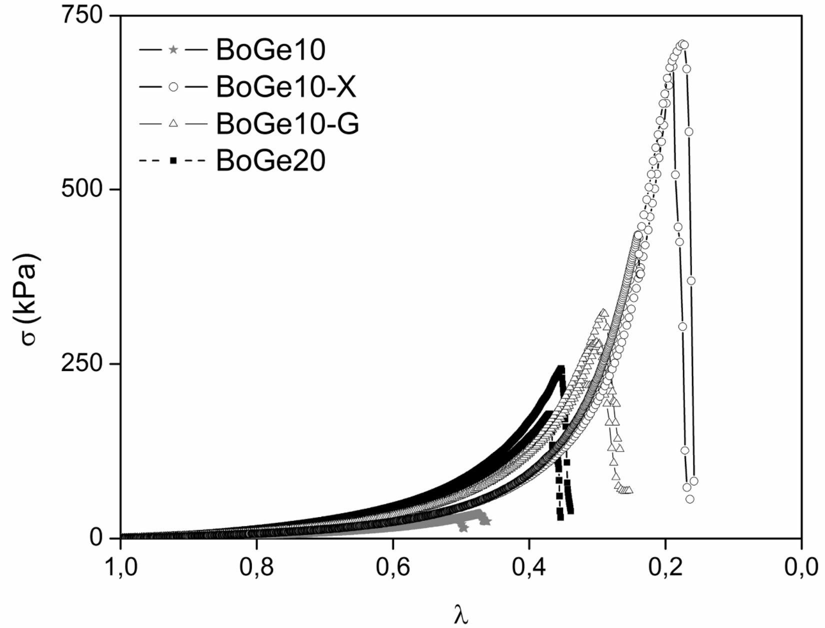

Figure 5. Typical stress-stretch ratio curves obtained from uniaxial compression tests of gelatin gels of: a) bovine source and b) porcine source.
204x148mm (300 x 300 DPI)

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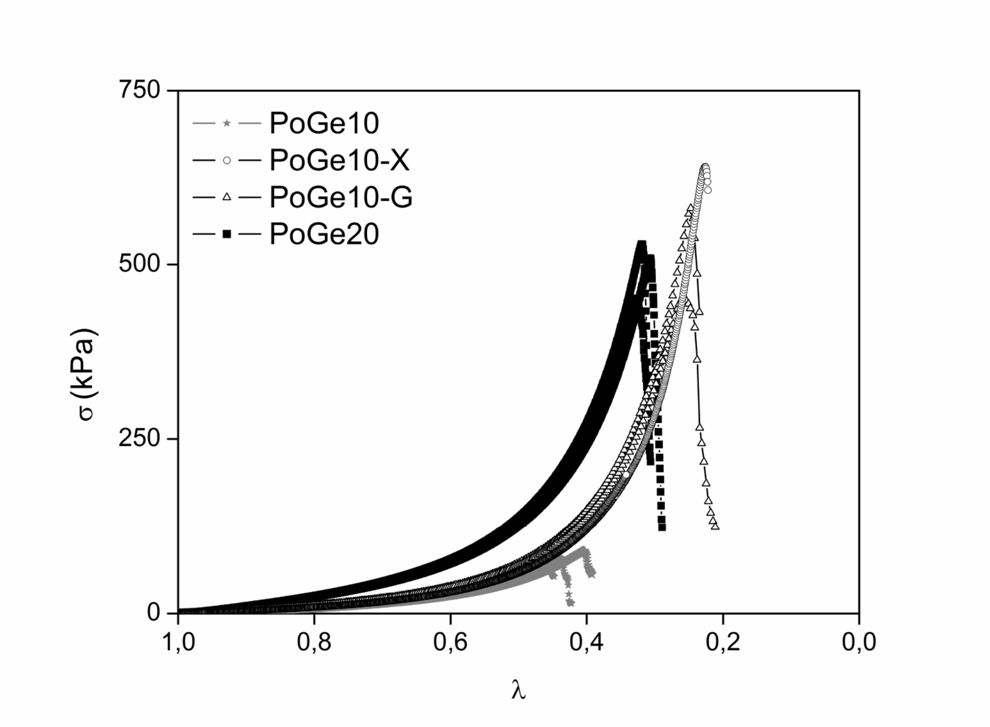

Figure 5 (continue). Typical stress-stretch ratio curves obtained from uniaxial compression tests of gelatin gels of: a) bovine source and b) porcine source.
204x148mm (300 x 300 DPI)

For Peer Review Only

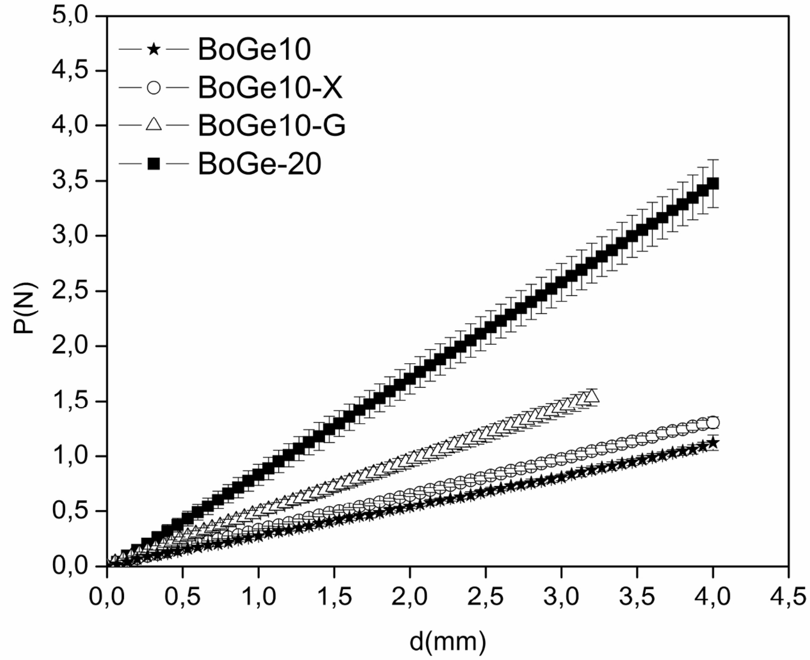

Figure 6. Average P-d curves obtained in flat indentation experiments of gelatin gels of: a) bovine source and b) porcine source.
208x160mm (300 x 300 DPI)

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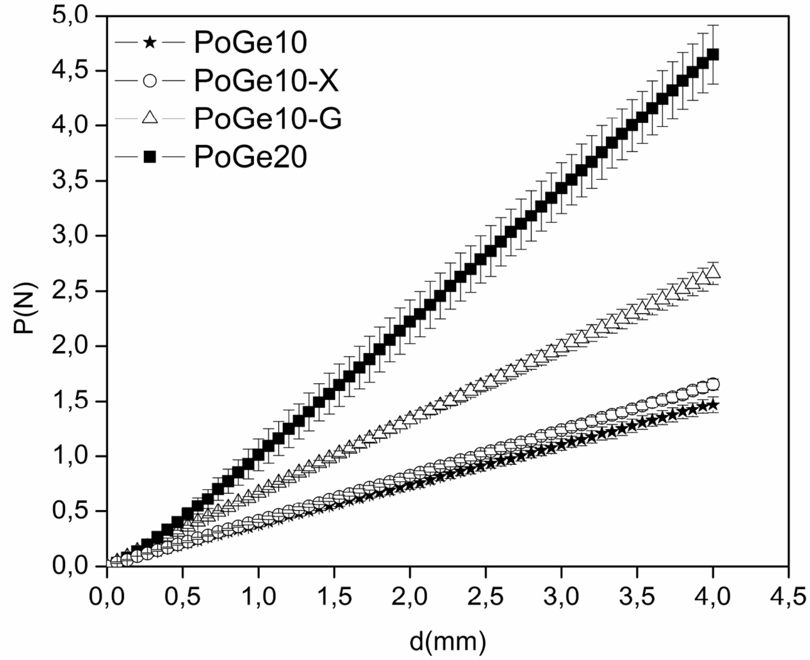

Figure 6 (continue). Average P-d curves obtained in flat indentation experiments of gelatin gels of: a) bovine source and b) porcine source.
208x160mm (300 x 300 DPI)

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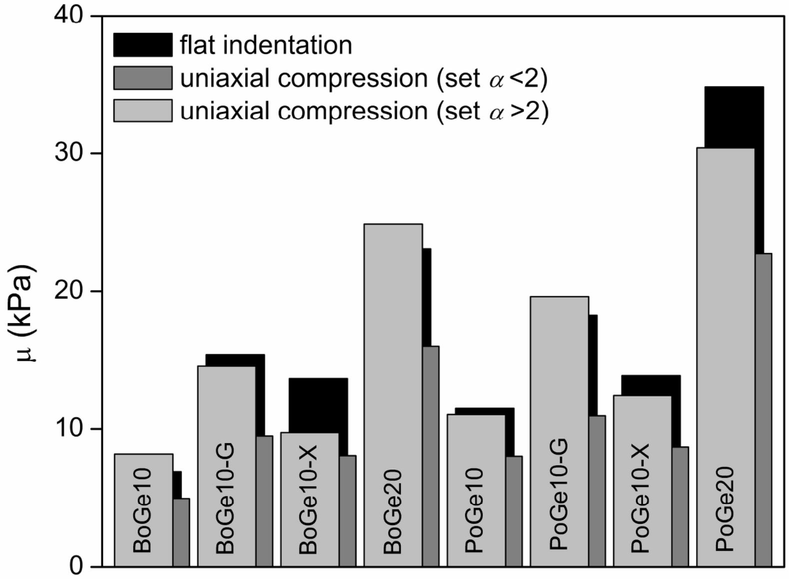

Figure 7. Comparison of Ogden parameters obtained in uniaxial compression and flat indentation tests: a) shear modulus; b) strain hardening capability.
208x160mm (300 x 300 DPI)

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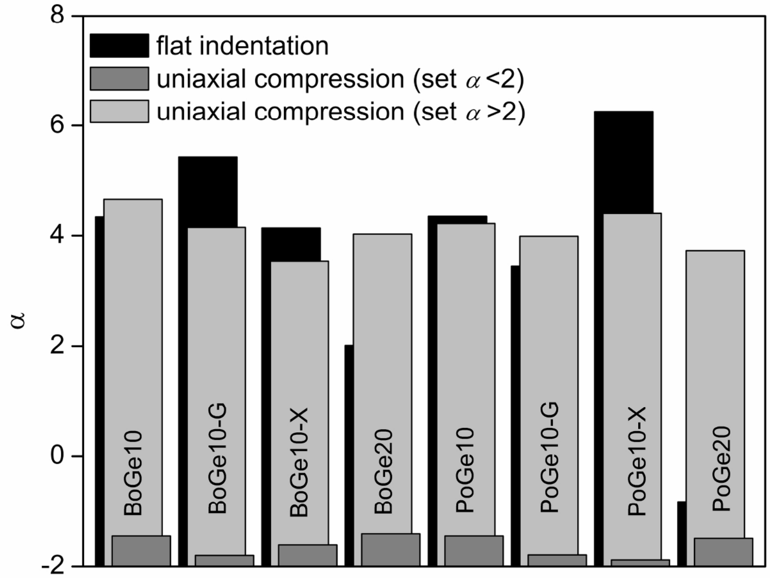

Figure 7 (continue). Comparison of Ogden parameters obtained in uniaxial compression and flat indentation tests: a) shear modulus; b) strain hardening capability.
208x160mm (300 x 300 DPI)

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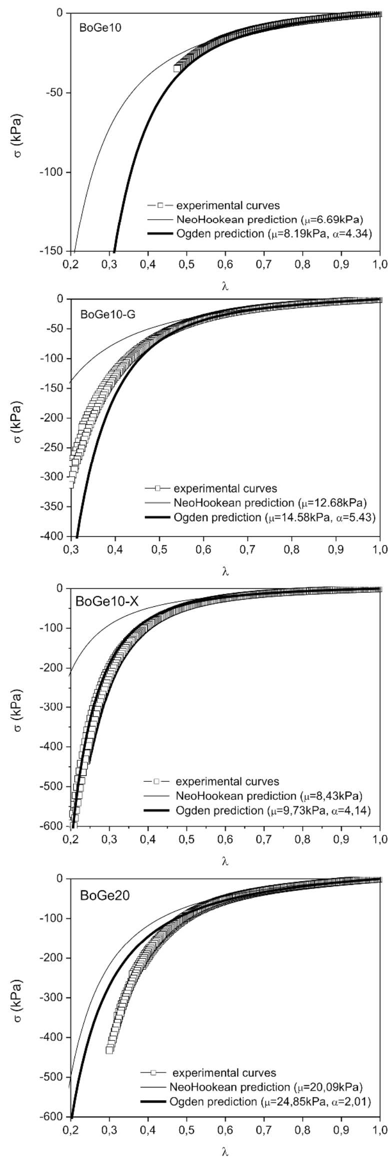

Figure 8: Comparison of experimental uniaxial compression curves and predicted curves using Ogden parameters identified from flat indentation tests of gelatin gels of: a) bovine source and b) porcine source.
223x625mm (600 x 600 DPI)

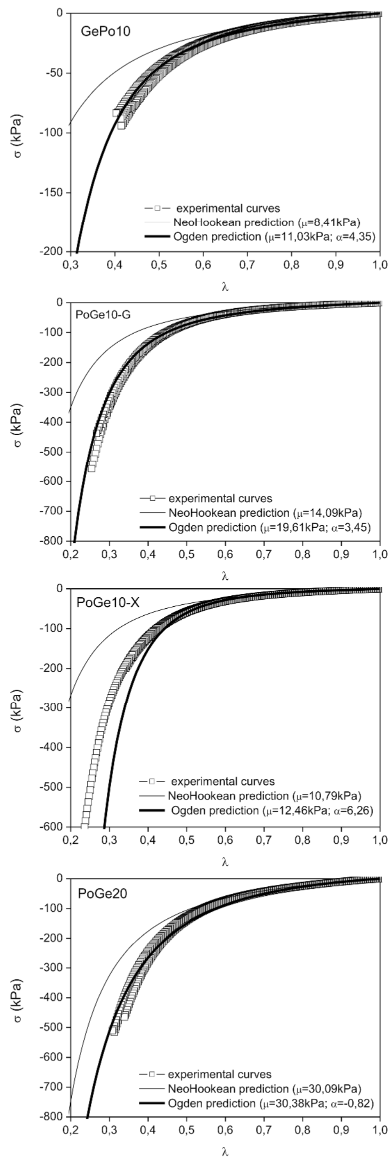

Figure 8: Comparison of experimental uniaxial compression curves and predicted curves using Ogden parameters identified from flat indentation tests of gelatin gels of: a) bovine source and b) porcine source.
223x623mm (600 x 600 DPI)

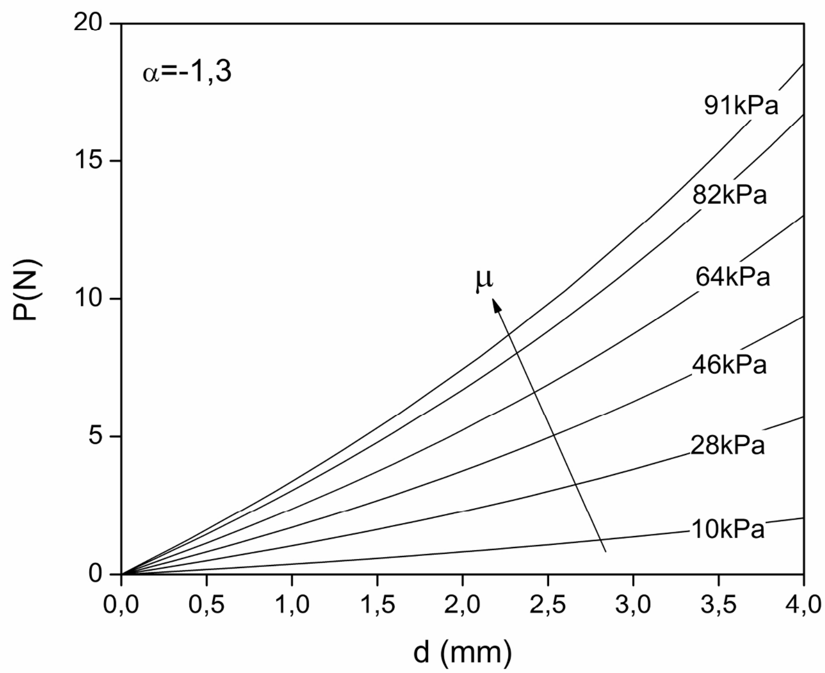

Figure 9. FEM simulation experiments for different Ogden parameters: a) P-d curves obtained varying μ for a given α (.-1.3), b) P-d obtained varying α for a given μ (10kPa) (.
208x160mm (300 x 300 DPI)

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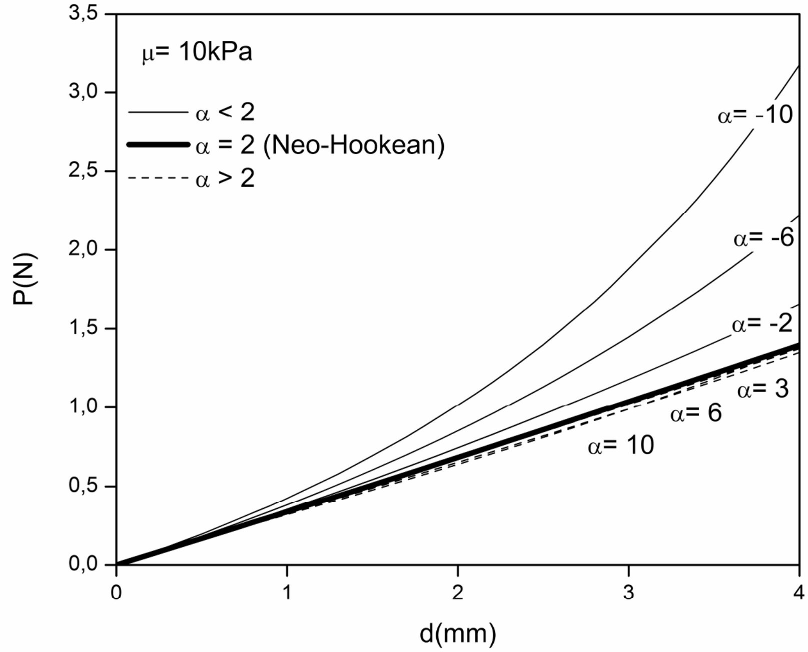

Figure 9 (continue). FEM simulation experiments for different Ogden parameters: a) P-d curves obtained varying μ for a given α (.-1.3), b) P-d obtained varying α for a given μ (10kPa) (.
208x160mm (300 x 300 DPI)

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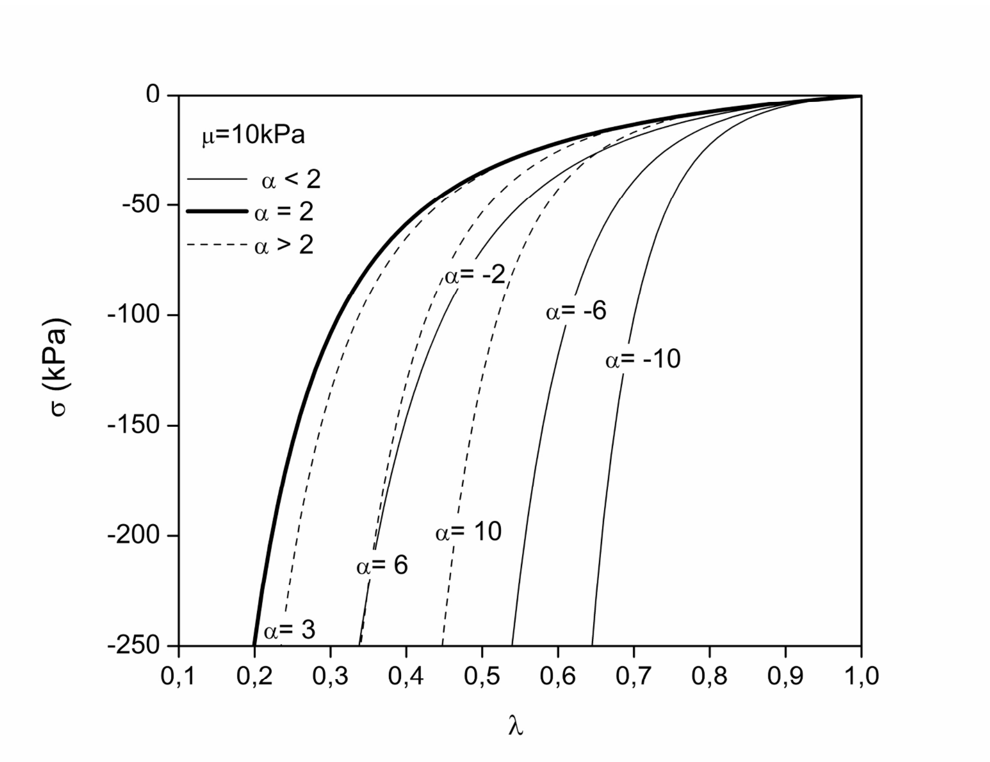

Figure 10. Uniaxial compression response of Ogden materials: a) Stress- stretch ratio obtained for various α and a given μ (10kPa) for large deformation states ; b) Stress- stretch ratio obtained various α and a given μ (10kPa) for small deformation states;c) Map of deformation characteristic as a function of α parameter and stretch ratio. The map is valid for any value of μ parameter. See text for H-NH and L-NH definitions.
208x160mm (300 x 300 DPI)

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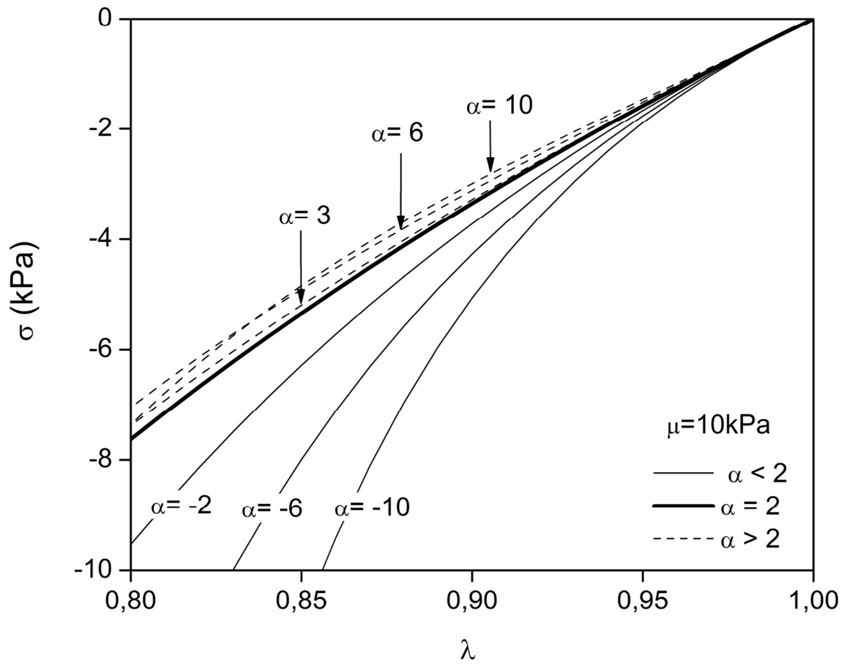

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208x160mm (300 x 300 DPI)

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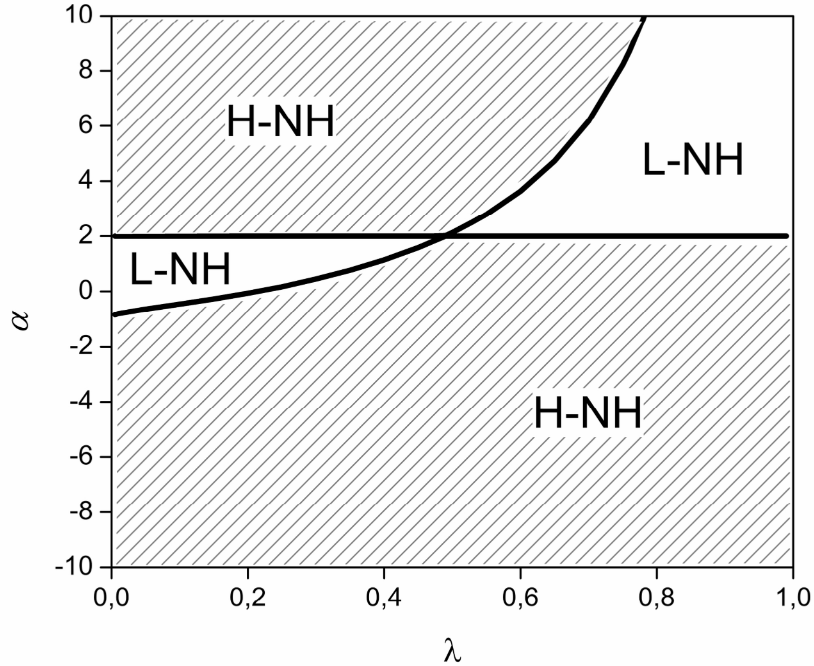

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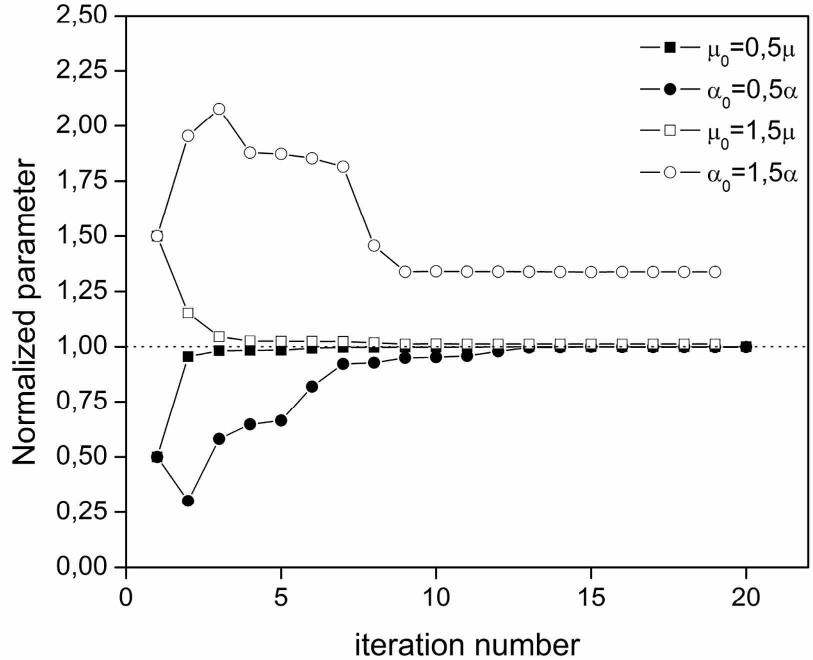

Figure 11. Evolution of Ogden parameters with iteration number from two sets of initial guesses for the case of fictitious Ogden materials: a) the simulated P-d curve (μ=20kPa, α= 3) was used as input data; b) Two levels of noise (1% and 2%) were added to P-d curve.
208x160mm (300 x 300 DPI)

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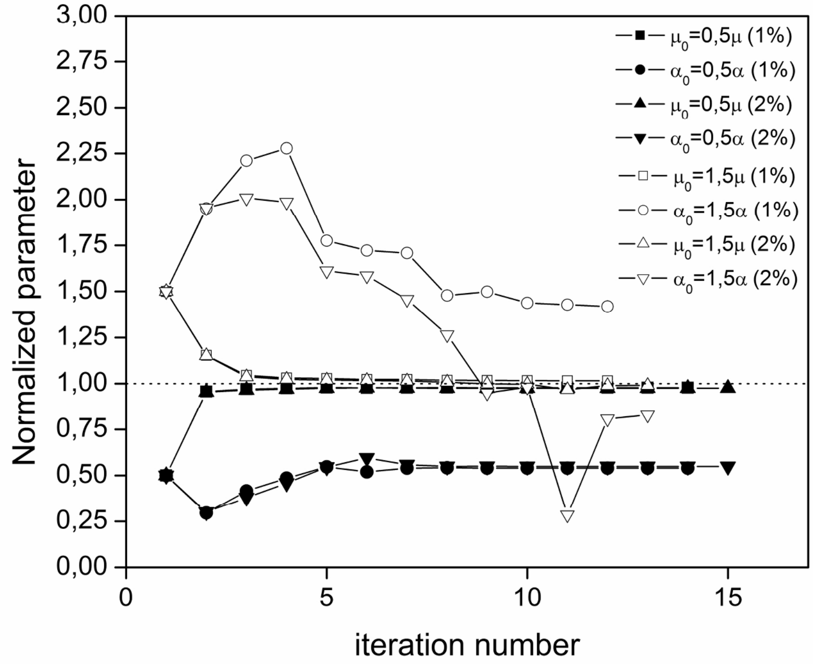

Figure 11 (continue). Evolution of Ogden parameters with iteration number from two sets of initial guesses for the case of fictitious Ogden materials: a) the simulated P-d curve (μ=20kPa, α= 3) was used as input data; b) Two levels of noise (1% and 2%) were added to P-d curve.
208x160mm (300 x 300 DPI)

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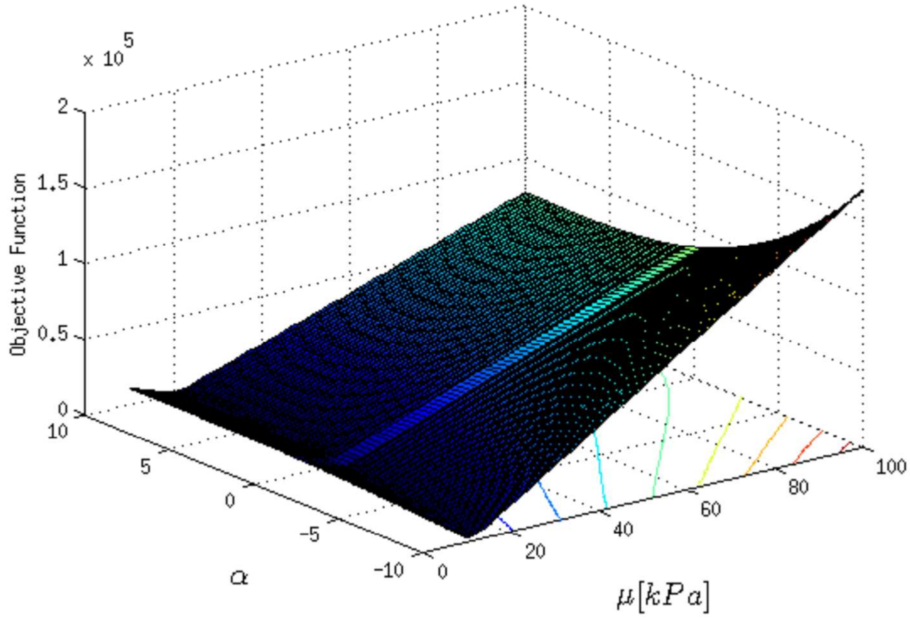

Figure 12. Example of minimization of the objective function (Eq. 3) using a P-d library data to identify first order Ogden parameters. Results for the case of μ=20kPa and α=3.
118x79mm (300 x 300 DPI)

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