Predicting the Mechanical Behaviour of Starch Gels through Inverse Analysis of Indentation Data

C. Gamonpilas[1,2*], M.N. Charalambides[1*], J.G. Williams[1,3], P.J. Dooling[4], S.R. Gibbon[4]

[1] Imperial College London, Mechanical Engineering Department, London SW7 2AZ, U.K.
[2] National Metal and Materials Technology Center (MTEC), 114 Paholyothin Road, Pathumthani 12120, Thailand
[3] School of Aerospace, Mechanical and Mechatronic Engineering J07, The University of Sydney, Sydney, NSW 2006, Australia
[4] Akzo Nobel Research Development and Innovation, Wilton Applied Research Group, Wilton, Redcar, TS10 4RF, U.K.

* Email: chaiwutg@mtec.or.th, m.charalambides@imperial.ac.uk
Fax: x66.2564.6446 or x44.2078238845

Received: 22.5.2009, Final version: 9.12.2009

Abstract:
Two types of starch gels made with various starch/water concentrations were studied in terms of their mechanical behaviour. Indentation tests were performed which revealed a rate independent load-deflection response. An inverse analysis based on the Marquardt-Levenberg optimisation algorithm and Finite Element Analysis was used to derive the stress-strain behaviour from the indentation data. The inverse predictions for the stress-strain curves are in good agreement with the direct measurements from uniaxial compression and shear tests up to high values of strain. The validity of the method was proven for both self-supporting and non self-supporting gels, with initial moduli ranging from a very small 60 Pa to 55 kPa. Thus the indentation characterisation method is proven as a powerful, fast and efficient way of evaluating and/or monitoring the behaviour of gels.

Zusammenfassung:
Zwei aus unterschiedlichen Stärke/Wasser-Konzentrationen bestehende Stärke-Gel-Arten wurden in Bezug auf ihr mechanisches Verhalten hin untersucht. Eindrucktests wurden durchgeführt, die eine ratenunabhängige Kraftablenkung aufzeigten. Mittels einer auf dem Marquardt-Levenberg-Optimierungsalgorithmus und der Finite-Elemente-Analyse basierenden inversen Analyse wurde das Spannungs-Dehnungs-Verhalten von den Eindruckdaten abgeleitet. Die inversen Voraussagen für die Spannungs-Dehnungs-Kurven stehen bis zu hohen Dehnungswerten in gutem Einklang mit den aus direkten Messungen ermittelten uniaxialen Kompressions- und Scherversuchsdaten. Die Gültigkeit der Methode wurde sowohl für die selbsttragenden als auch für die nicht selbsttragenden Gele bewiesen, wobei initiale Moduli Druckwerten ausgesetzt wurden, die von sehr niedrigen 60 Pa bis zu 55 kPa reichten. Somit ist erwiesen, dass die Eindruckcharakterisierung eine leistungsfähige, schnelle und effiziente Methode der Beurteilung und/oder Überwachung des Verhaltens von Gelen ist.

Résumé:
Deux types de gels d'amidon, obtenus avec des concentrations variées en amidon et eau, ont été étudiés du point de vue de leur comportement mécanique. Des tests d'indentation ont été réalisés révélant une réponse déflection-charge indépendante de la vitesse. Une analyse inverse basée sur un algorithme d'optimisation de Marquardt-Levenberg et une analyse d'élément fini, a été utilisée afin de dériver le comportement contrainte-déformation à partir des données d'indentation. Les prédictions inverses pour les courbes de contrainte-déformation sont en bon accord avec les mesures directes obtenues avec des tests de cisaillement et de compression uni-axiale, jusqu'à des valeurs élevées de déformation. La validité de la méthode est démontrée pour des gels qui se supportent ou non, avec des modules initiaux allant d'un très petit 60 Pa jusqu'à 55 kPa. Ainsi donc, la méthode de caractérisation par indentation se présente comme un moyen puissant, rapide et efficace d'évaluer ou contrôler le comportement des gels.

Key words: starch gels, constitutive behaviour, indentation, inverse analysis, finite element analysis, food rheology

 DOI: 10.3933/ApplRheol-20-33283

1 INTRODUCTION

Starch is a very common ingredient in food products. It is widely used as a thickening, stabilizing or gelling agent in food products due to its inexpensive and abundant supply [1-2]. In its native state, starch is present in the form of granules, composed of an amylopectin skeleton in an interpenetrating amylose matrix. When dry starch granules are dissolved in water and then heated, they absorb water and swell as much as 200% in granule size [3]. Subsequently, amylose separates from amylopectin and leaches out of granules. Then, on cooling and during a subsequent storage, the initially ordered structures in starch dispersions are partially recovered resulting in gelation and crystallinity of both amylose and amylopectin. Starch gels can be seen as composite materials consisting of an amylose network reinforced by the swollen starch granules [4]. Either solid (self-supporting) or weak (non-self supporting) gels can be formed depending on the amount of starch in relation to the dissolved water. When added to food products, starch can greatly affect their structure and mechanical behaviour. Knowledge on the role of starch in food products and the ability to predict accurately its deformation properties using standard techniques are therefore crucial for achieving the desired mechanical properties.

Many types of tests are available for characterising the mechanical behaviour of soft materials such as gels or foods. These tests can be categorised into those with a uniform strain distribution such as the uniaxial compression and tension tests, and those with non-uniform strain distribution, such as the indentation test. The former tests are well-established for conventional structural materials because the measured loads and displacements can be converted easily into the stress–strain response using simple expressions. However, such tests can sometimes be difficult to apply to soft foods due to the low stiffness and limitations in size and shape. On the other hand, the indentation test offers many advantages because it is quick and simple, it does not require samples with specific shape requirements and is not greatly affected by friction between sample and loading platens, as in the case of uniaxial compression [5]. Indentation tests can be performed on a large scale using high throughput screening techniques where large numbers of different product formulations/recipes can be automatically tested using high speed robotic indenters on small samples moving on conveyor belts. However, the mechanical behaviour of food is usually very non-linear, hence there are no theories that can be easily applied to relate the indentation load–displacement response to the stress–strain properties [6, 7]. Thus, there is a need for converting indentation load–depth data for non linear materials into the fundamental material properties using an inverse parameter identification technique.

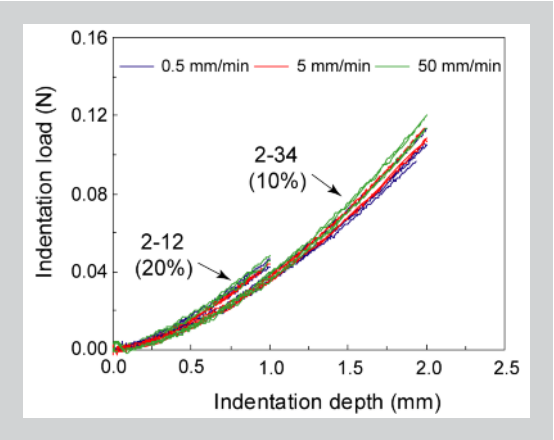
Inverse analysis techniques have gained widespread use in many engineering applications, including signal processing, inertial navigation, radar tracking, manufacturing and many other aspects [8–10]. The use of inverse analysis techniques to determine the material properties from indentation is still relatively new. Little work has been published regarding the use of this technique in conjunction with the indentation test. Until recently, the inverse approach and instrumented indentation technique have been used to successfully evaluate the material parameters of functionally graded materials [11], elastoplastic [12] and viscoelastic materials [5]. The aim of this work is to use inverse analysis with experimental data derived from indentation tests to derive the stress-strain characteristics of two types of starch gels made with varying starch granule concentrations. The results obtained from the inverse calculations will be validated by independent compression and shear experiments.

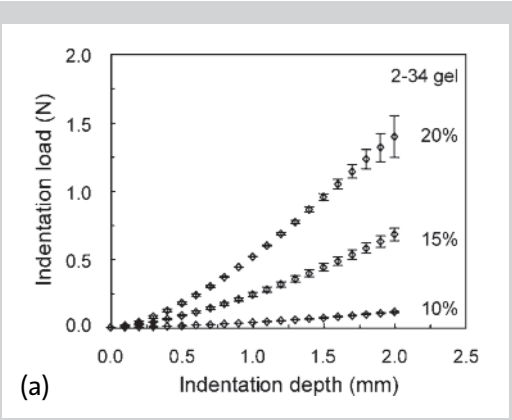
2 MATERIALS AND PREPARATION

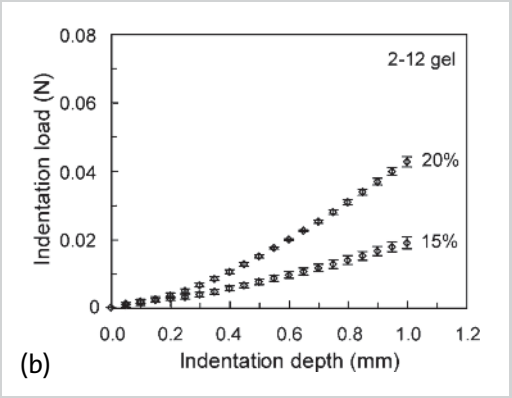
Two starch gels here called 2-34 (modified sago starch) and 2-12 (modified maize starch), were investigated. Prior to making the gel samples, the powders were dried overnight in an oven at 110°C to remove any moisture present. From this point, the powders were kept in a desiccator and were only removed just before testing. Starch gels were made by mixing the powder with distilled water in a beaker. The beaker was then placed in a tub of boili ng water. The mixing solution was stirred well for two minutes during which the gel reached its gelatinization state. The heating time was kept constant for every batch to ensure consistency and to avoid over cooking. Before transferring the solution into moulds, it was kept in

Figure 1 (left): Experimental measurements of indentation response for 2-34 (10 %) and 2-12 (20 %).

Figure 2: Indentation response of (a) 2-34 and (b) 2-12 at various starch concentrations (%w/w).







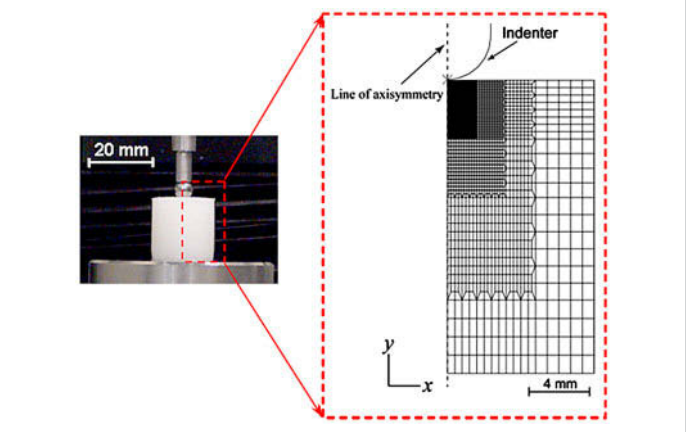
the beaker at approximately 90°C for a further 1 minute for the 2-34 gel and 18 minutes for the 2-12 gel. During this period, the beaker was sealed with a cling film to maintain the water content of the solution. The solution was then allowed to solidify over night, i.e. for approximately 15 hours, at room temperature, in cylindrical PTFE moulds of 20 mm diameter and 20 mm height. The gel settling time and temperature were kept approximately constant across batches to avoid any effects of storage and ageing. Various powder concentrations were studied e.g. 5, 10, 15 and 20 %w/w (ratio of weight of powder to weight of water) for the 2-34 and 10, 15 and 20 % w/w for the 2-12. It is worth noting that 2-34 (5 %) and 2-12 (10 %) gels are non-self supporting.

3 EXPERIMENTS

All the experiments detailed below were performed at 21°C and 50 % relative humidity. Indentation tests were performed on an Instron 5543 testing machine using a spherical indenter of diameter 5.90 mm at the three loading speeds of 0.5, 5 and 50 mm/min. Due to the small loads involved, a 10N load cell was used in all indentation measurements. Maximum loading depths of 1 and 2 mm were applied to 2-12 and 2-34 gels, respectively. A smaller depth was used for 2-12 as it was found that this gel was more brittle and therefore broke earlier than the 2-34. In order to validate the suggested inverse analysis methodology (see later section), uniaxial compression tests were also performed on all self-supporting gels by moving the Instron crosshead at constant strain rates of 0.1, 1 and 10 /min. Silicon oil was used throughout the tests in order to minimise friction between the compression platens and the sample and to eliminate sample height effects [13, 14]. A 100N load cell was used. The storage (G') and loss (G'') moduli as well as the shear stress-strain behaviour of non-self supporting gels were measured using a parallel plate geometry with a diameter of 40 mm and a gap of 1 mm (AR2000ex , TA instruments). These gels were too weak to be tested in compression and this is the reason why shear experiments were performed instead. A frequency sweep (0.1 - 63 rad/s) test within the linear viscoelastic range, i.e. with a 2% strain amplitude, was conducted. In addition, a constant shear rate test was also performed at a rate of 0.05 /sec until rupture of the gel was obtained.

4 INDENTATION TEST RESULTS

The indentation load-depth response of 2-34 (10 %) and 2-12 (20 %) gels is shown in Figure 1. It is observed that the indentation response of these gels does not depend on speed. This was true for all other gels investigated in this study. In addition, the 2-34 (modified sago) starch gel appears to be stiffer than the 2-12 (modified maize) gel; a smaller concentration of starch leads to almost identical response as shown in Figure 1. The initial granule size of the two starches was found to be different from light microscopy, i.e. in the range of 10 - 50 μm for 2-34 and less than 10 μm for 2-12. The 2-34 granules also take longer to cook and disintegrate which would suggest that they are, perhaps, stiffer, which could explain why the indentation response of such gels also seems stiffer. The average curves for the two gels at all concentrations are shown in Figure 2. As expected from composite theory [15], the gels with higher starch concentration are stiffer. The aim now is to derive accurate stress-strain characteristics from the data shown in Figure 2 using inverse analyses.



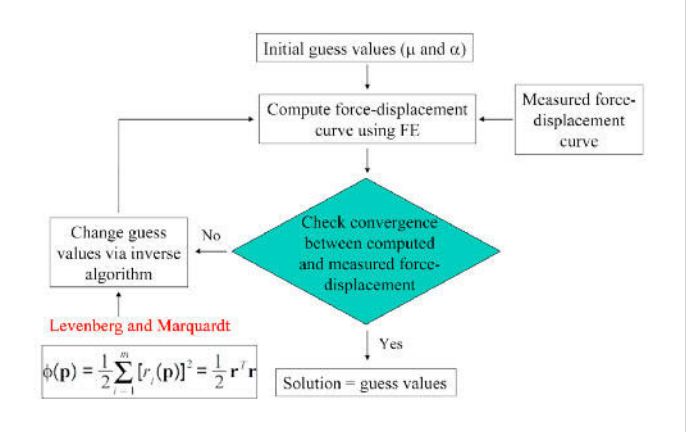


Figure 3 (left): Illustration of finite element analysis of the indentation problem.

Figure 4: Inverse analysis procedure.

5 INVERSE ANALYSIS METHOD

In order to perform the inverse analysis, a solution for the indentation response of the material in question is needed. Therefore, one needs to determine the form of a constitutive model which fits the observed material behaviour accurately. For this reason, independent loading-unloading tests were performed under uniaxial compression loading (data not shown here) which showed reversible response of the starch gels; samples always visually reverted to their original shape upon removal of load. Based on the latter tests and the data shown in Figure 2, the assumption was made that the gels deform in a non-linear elastic manner and are rate independent. This finding agrees with previous work by Luyten and Van Vliet [16] and Ikeda and co-workers [17].

Having established that the gels are non linear elastic, the problem arises that there exists no analytical solution for the indentation problem of non linear elastic materials. Therefore, the solution was obtained via a finite element (FE) simulation of the indentation test. The commercial FE software code ABAQUS [18] was used. The 2D model makes use of the axi-symmetric geometry of the sample as shown in Figure 3. A very fine mesh is used in the vicinity of contact region due to the large deformation of the region. A gradually coarser mesh is applied away from the contact region in order to reduce the total number of degrees of freedom and computational time. A mesh sensitivity study was performed and the final mesh used is shown in Figure 3. The spherical indenter is modelled as a rigid body. The contact between the indenter and the specimen was assumed to be frictionless. During the analysis, the indenter was loaded by means of a downward displacement along the *y* axis. The Ogden hyperelastic material model was chosen for the gel [18]. Under a uniaxial deformation state, the true stress, σ, is defined as:

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

where λ is the stretch ratio and $\lambda = exp(\epsilon)$ where $\epsilon$is the true strain, μ is the initial shear modulus such that Young's modulus, *E*, is given by $E = 3\mu$ and α is the Ogden constant. It is worth noting here that even though the Ogden model was chosen in this work, this does not limit the applicability of the described methodology to this particular model. One could in fact choose any of the several forms of hyperelastic models described in terms of a strain energy potential (e.g. Mooney-Rivlin, Van der Waals etc [18]).

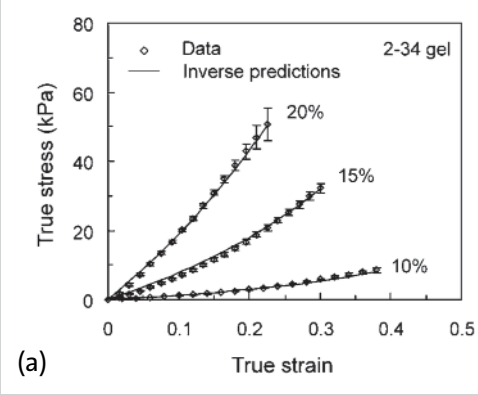
Initially, 'guesses' are entered in the FE for the unknown material parameters, μ and α. The corresponding indentation response is obtained by the normal reaction force on the rigid indenter as a function of the vertical displacement of the indenter. This FE derived response is compared with the experimental data of Figure 2 and using an optimisation algorithm (described below), the material parameters used in the numerical simulations are iteratively updated until the FE response and the experimentally measured one are within some pre-set convergence tolerance criteria.

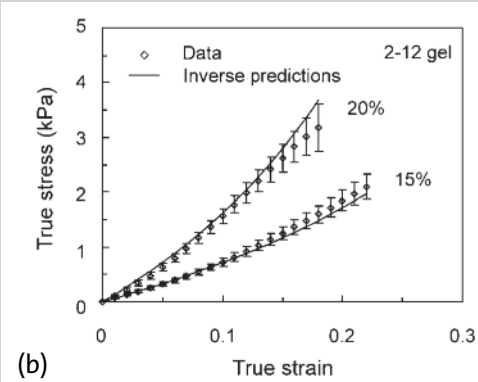
The optimisation algorithm that was used for the inverse analysis is the Levenberg-Marquardt (LM) method. The LM method is proven to be an effective and popular way of solving non-linear least optimisation problems and is described extensively in the work of Schnur and Zabaras [19]. Its use within the context of this work is illustrated by the flow chart shown in Figure 4. Essentially, it processes the experimental data and attempts to obtain the best estimates for unknown state variables based on non linear optimisation theory. The latter is based on minimising an error function, ϕ, with respect to the parameter, $\boldsymbol{p}$, as:

$$\Phi(\boldsymbol{p}) = \frac{1}{2}\sum_{i=1}^{m}[r_i(\boldsymbol{p})]^2 = \frac{1}{2}\boldsymbol{r}^T\boldsymbol{r} \quad (2)$$

Figure 5 (left): Inverse analysis predictions of stress-strain response from indentation data for (a) 2-34 and (b) 2-12 at various powder concentrations. Data points are from uniaxial compression experiments performed to validate the inverse analysis predictions.

Figure 6: Stress-strain curves as obtained from uniaxial compression tests for 2-34 (10 %) and 2-12 (20 %).

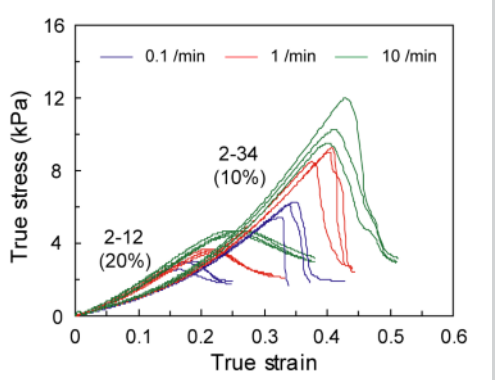




where $\boldsymbol{p}$ is a vector which contains the unknown parameters and m is the number of measurements. The vector $\boldsymbol{r}$ is defined as:

$$\boldsymbol{r} = \boldsymbol{p}_i - \hat{\boldsymbol{p}} \tag{3}$$

where $\boldsymbol{p}_i$ and $\hat{\boldsymbol{p}}$ are the numerically generated and measured indentation load, respectively, at the specified indenter displacement values. In order to reduce the computational effort in performing the finite element calculations within each iteration during the inverse analysis, a reference database was created. The database contains numerically computed indentation load-displacement data for different combinations of μ and α. These combinations consist of the parametric values of μ (in kPa) = 1, 6, 11, 16, 21, 26, 31, 36, 41, 46, 51, 56, 61, 66 and of α = -10, - 8, - 6, - 4, - 2, 2, 4, 6, 8, 10. During the inverse analysis procedure, the load-displacement values for any combinations of μ and α were interpolated between the points in the database. The inverse algorithm outlined here was coded in the commercial MATLAB [20] program.



6 DERIVED STRESS-STRAIN CHARACTERISTICS FROM INDENTATION DATA

The stress-strain behaviour of both starch gels at all concentrations was derived using the inverse methodology outlined in the previous section. The results for the self supporting gels are described first. These are shown in Figure 5. The derived material parameters for 2-34 and 2-12 gels at various concentrations are summarised in Table 1.

In order to validate the inverse predictions, independent uniaxial compression experiments were performed as described in the Experiments section. Figure 6 shows the typical compression behaviour of 2-34 (10 %) and 2-12 (20 %) gels at three constant strain rates of 0.1, 1 and 10 /min. In agreement with the observations made from the indentation data shown in Figure 1, the stress-strain curves are independent of rate and the 2-34 starch seems to form stiffer gels. Another interesting observation from this figure is that fracture stress and fracture strain increase with an increase in the deformation rate i.e. both quantities are rate dependent. This phenomenon is discussed further elsewhere [21]. The data points corresponding to the average response of each gel are shown in Figure 5 together with the inverse analysis predictions. An excellent agreement is observed for all cases, validating the inverse analysis method. In addition, similar to

Table 1 (above): Inverse predictions (using indentation data) of material parameters of 2-34 and 2-12 gels at various powder concentrations.

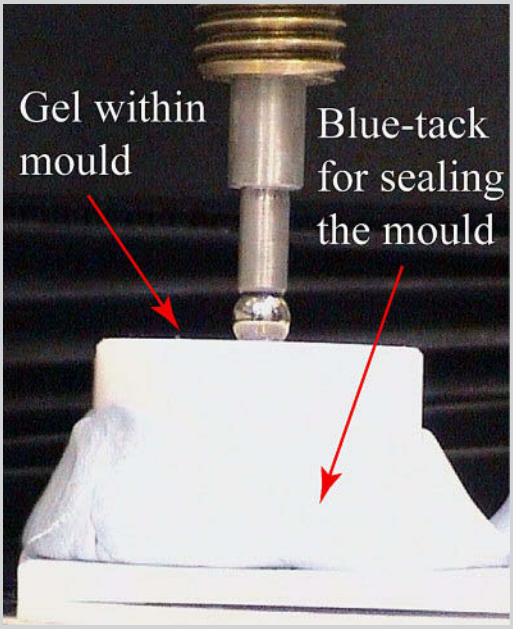
Ogden	2-34 (10%)	2-34 (15%)	2-34 (20%)	2-12 (15%)	2-12 (20%)
μ (kPa)	3.3	22.9	56.5	2.1	5.0
α	- 4.9	- 4.1	- 3.7	- 4.9	- 2.9

Table 2 (middle): Material parameters of 2-34 and 2-12 gels at various powder concentrations, determined by fitting the experimental compression data of Figure 5 to the Ogden model.

Ogden	2-34 (10%)	2-34 (15%)	2-34 (20%)	2-12 (15%)	2-12 (20%)
μ (kPa)	3.1	21.9	55.3	2.2	4.7
α	- 6.2	- 5.1	- 4.4	- 6.6	- 3.7

Table 3 (below): Percentage difference between material parameters derived from inverse analysis of indentation data (Table 1) and compression data (Table 2).

Ogden	2-34 (10%)	2-34 (15%)	2-34 (20%)	2-12 (15%)	2-12 (20%)
μ (kPa)	8.7%	4.4%	2.2%	-4.2%	7.5%
α	- 20.5%	- 19.7%	- 15.5%	- 26.1%	- 21.5%



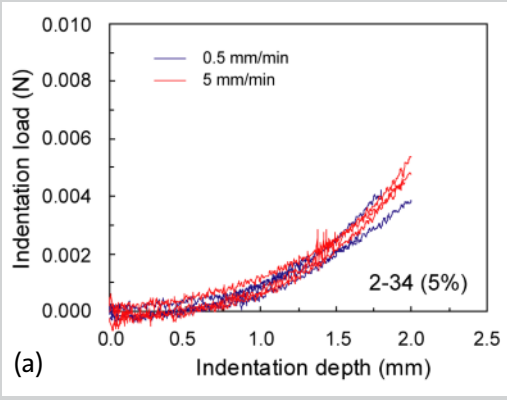
indentation, the expected increase in the stress-strain curve with starch concentration is observed.

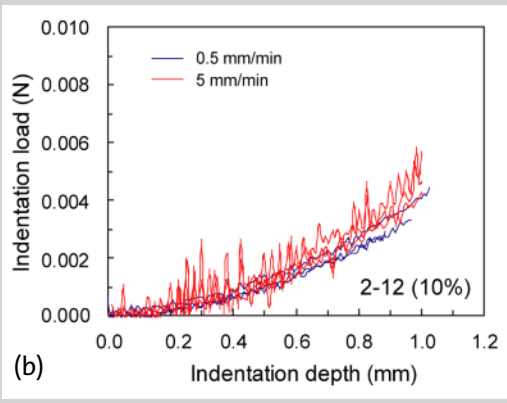
In order to compare with the data of Table 1, the compression data of Figure 5 were fitted with the Ogden model given by Equation 1, using a Solver function in Excel. Values for the material constants, μ and α, for each material are tabulated in Table 2. The percentage difference between the parameter values in Tables 1 and 2 are given in Table 3. For all gels studied, the inverse analysis predicted the values of μ and α within maximum differences of 8.7 and 26.1 %, respectively, as compared to the compression values. The reason for the larger difference seen in the values of α is due to the low sensitivity of the stress-strain curve on this parameter when the latter is in the range of - 3 to - 5.

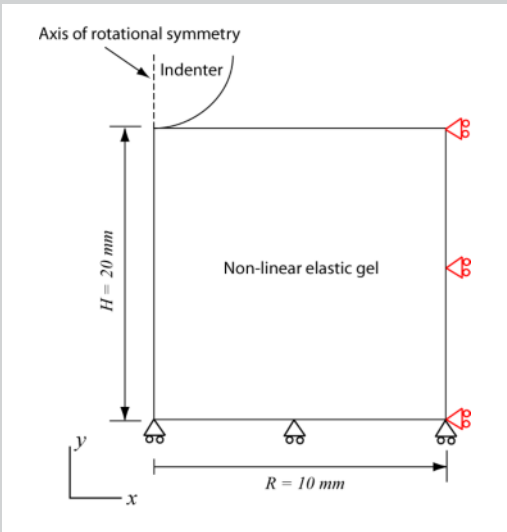
7 NON-SELF SUPPORTING GELS: 2-34 (5 %) AND 2-12 (10 %) W/W

A challenging task in this work was how to characterise the two non-self supporting gels using indentation data. In order to solve this problem, indentation tests were conducted within the mould containing the gel (see Figure 7); it was not possible to extract these gels from their moulds without damaging them. Even if one could extract them from the moulds, the samples would not be able to support themselves when left standing on a surface.

The results are presented in Figure 8 for 2-34 (5 %) and 2-12 (10 %), respectively. Although it is apparent that the behaviour is rate-independent and similar to that of the self-supporting gels, large fluctuations in the measurements are obtained. These are caused by the very small recorded loads and the dynamic effects from the







machine as it is driven by a single screw mechanism. Therefore the scale of the fluctuation increases with the rate of loading.

In order to perform the inverse analysis on the indentation data obtained from tests where the gels are left within the mould, the *FE* model described in Section 5 needs to be modified by prescribing an additional constraint along the boundary between the sample and the mould, as shown in Figure 9. A new database was subsequently created which also included smaller values for the initial shear modulus μ. The indentation data shown in Figure 8 were 'smoothed' by fitting with a simple quadratic equation. Using

Figure 7 (left): Indentation tests within a mould for non-self supporting gels.

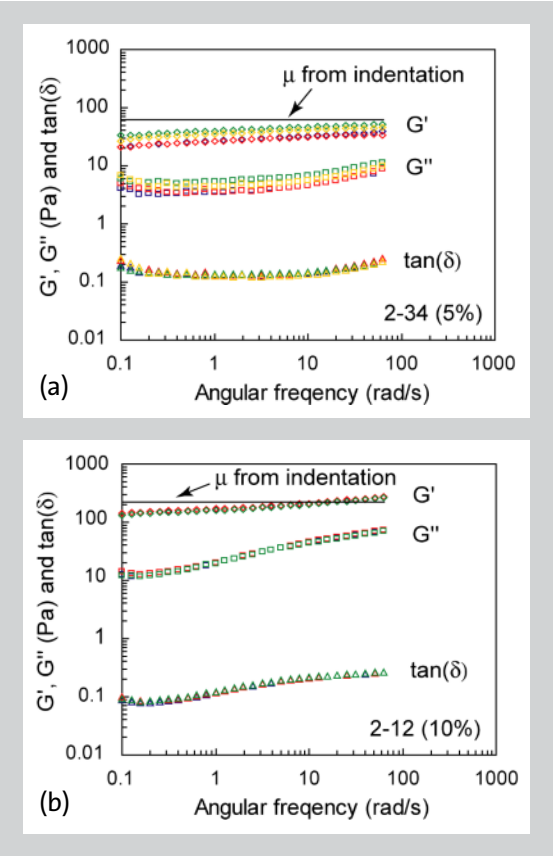
Figure 8 (right above): Indentation response of two non-self supporting gels (a) 2-34 (5 %) and (b) 2-12 (10 %).

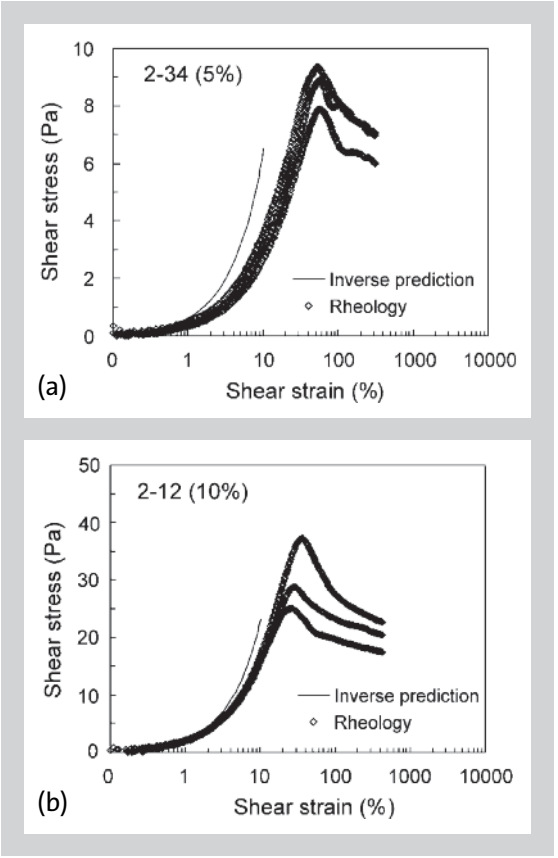
Figure 9 (right below): A schematic finite element model of an indentation on a non-self supporting gel.

Figure 10 (left): Moduli G', G'' and tan δ spectra as a function of frequency for (a) 2-34 (5 %) and (b) 2-12 (10 %).

Figure 11: Rheological behaviour of 2-34 (5 %) and 2-12 (10 %) in comparison with those predicted from inverse predictions (using indentation data).

Table 4: Inverse predictions (from indentation data) of material parameters of the non-self supporting gels.





this smoothed response, the inverse predictions were then performed on both gels to obtain their μ and α parameters. The results are summarised in Table 4.

Ogden	2-34 (5%)	2-12 (10%)
μ (Pa)	62.7	223.8
α	- 9.9	- 9.1

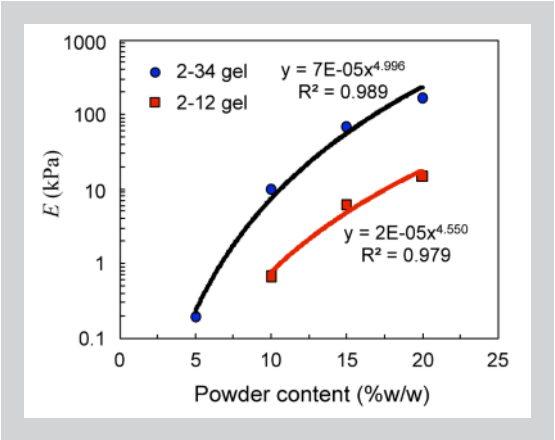
As uniaxial compression tests were not possible for the non-self supporting gels, rheometric shear tests were performed instead, in an effort to validate the inverse predictions. The curves of *G'*, *G''* and *tan*(δ) as a function of frequency are shown in Figures 10(a) and 10(b) for 2-34 (5%) and 2-12 (10%), respectively. It can be seen that *G'* is almost constant and a great deal larger than *G''* in the entire frequency domain and *tan*(δ) is small and almost constant. These spectra are typical of those usually observed for gels [22, 23]. When increasing the shear strain by applying a very low steady shear rate to the gel, the shear stress started to increase proportionally up to the shear strain of about 1 % for both gels (see Figure 11). Beyond this point, the shear stress increased more rapidly with the shear strain and dropped sharply at a shear strain of approximately 0.4 for 2-34 (5 %) and 0.2 for 2-12 (10 %) gels. These results are similar to the work by Michon and co-workers [22]. In order to compare the predictions from the inverse analysis with the data in Figure 11, the parameters shown in Table 4 were used to calculate the corresponding shear response. Under a simple shear condition, the shear stress, τ , is related to λ as shown below [24]:

$$\tau = \frac{2\mu}{\alpha}\left(\frac{\lambda^{\alpha+1}}{\lambda^2+1} - \frac{\lambda^{-\alpha+1}}{\lambda^2+1}\right) \quad (4)$$

where λ is related to the shear strain, γ, through

$$\lambda = \frac{\gamma}{2} + \frac{\sqrt{\gamma^2+4}}{2}$$

The comparison is shown in Figure 11 and the agreement between the inverse predictions and the experimental rheological data is reasonable for both materials. The discrepancies in the predictions may be due to experimental difficulties in performing both tests. On the one hand, it could be the result of large experimental scatter in indentation data as the level of load measured was comparable with the inherent vibration of the machine. On the other hand, there could be some slippage occurring during the rheological measurements, hence a weaker response. In addition, the μ values shown in Table 4 are plotted together with the storage moduli in Figure 10. It is apparent that the predicted μ values for



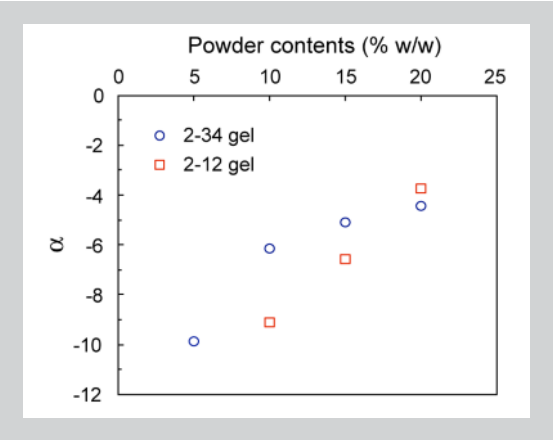


Figure 12 (left): Dependence of E on the powder content (%w/w) for both self supporting and non-self supporting gels. Lines are fitted power laws.

Figure 13: Dependence of α on the powder content (%w/w) for both self supporting and non-self supporting gels.

both gels are in good agreement with the storage moduli obtained from the rheological tests.

Figure 12 shows the dependence of E (calculated from 3μ) on the % powder content for both self-supporting and non-self supporting gels. A semi-logarithmic scale was chosen as the rise in E with increasing % powder content is so dramatic. Power laws were fitted to the data as shown in Figure 12 and for both types of gels, the power exponent was close to 5. This is very close to the values quoted by Genovese and Rao [25]. In addition, Figure 13 shows the dependence of α on % powder content for all seven gels that were investigated. There is a clear trend with α becoming 'less negative' as the powder concentration increases. The parameter α is an indication for the strain hardening part of the stress-strain curve, with less negative values indicating more linear stress-strain curves without significant strain hardening. At small strains, the modulus expression derived from Equation 1 can be expressed as:

$$E = 3\mu\left(1+\frac{\alpha\epsilon}{2}\right) \tag{5}$$

Clearly, this is linear for $\alpha = 0$. In addition, for $\alpha < 0$, the compression modulus increases with strain whilst the tensile one decreases. For $\alpha > 0$, the reverse is true and the modulus increases for all cases at large strains.

8 CONCLUSIONS

The mechanical behaviour of two starch gels made from various starch/water ratios has been obtained using an inverse analysis on data obtained from indentation tests. The inverse predictions were validated with independent compression and shear data for self-supporting and non-self supporting gels, respectively. Excellent agreement was obtained for all of the gels studied up to high values of strain. The method was tested on a large number of gels with initial shear moduli varying in the large range of 60 Pa to 55 kPa. This is a clear demonstration that the method is a powerful tool for obtaining the stress-strain characteristics of such materials.

ACKNOWLEDGEMENTS

Support for this work has been provided by ICI plc under the SRF scheme. ABAQUS was provided under academic license by HKS Inc., Providence, Rhode Island, USA.

REFERENCES

[1] Galliard T: Starch: Properties and potential, John Wiley & Sons, UK (1987).
[2] Imeson A: Thickening and gelling agents for food, Aspen publishers Inc, Maryland (1997).
[3] Patel BK, Seetharaman K: Effect of heating rate on starch granule morphology and size, Carbohyd. Polym. 65 (2006) 381-385.
[4] Carnali JO, Zhou Y: An examination of the composite model for starch gels, J. Rheol. 40 (1996) 221-234.
[5] Goh SM, Charalambides MN, Williams JG: Characterisation of non-linear viscoelastic materials via the indentation techniques, Rheolo. Acta 44 (2004) 47-54.
[6] Sneddon IN: The relation between load and penetration in the axisymmetric Boussinesq problem for a punch of arbitrary profile, Int. J. Eng. Sci. 3 (1965) 47–57.
[7] Sakai M: Time-dependent viscoelastic relation between load and penetration for an axisymmetric indenter, Phil Mag A 82 (2002) 1841-1849.
[8] Grewal MS, Andrews AP: Kalman Filtering: Theory and practice, Prentice-Hall Inc., New Jersey (1993).
[9] Gabriel C: 2nd Workshop on inverse problems in rheology and related experimental techniques, Appl. Rheol. 11 (2001) 284-285.
[10] Anderssen B: Inverse problems and emerging techniques in materials characterization, Appl. Rheol. 10 (2000) 194-195.
[11] Nakamura T, Wang T, Sampath S: Determination of properties of graded materials by inverse analysis and instrumented indentation, Acta Mater. 48 (2002) 4293-4309.
[12] Gamonpilas C, Busso EP: Characterization of elastoplastic properties based on inverse analy-

sis and finite element modelling of two separate indenters, J. Eng. Mater. Tech. 129 (2007) 603-608.
[13] Christianson DD, Casiraghi EM, Bagley EB: Uniaxial compression of bonded and lubricated gels, J. Rheol. 29 (1985) 671-684.
[14] Charalambides MN, Goh SM, Wanigasooriya L, Williams JG, Xiao W: Effect of friction on uniaxial compression of bread dough, J. Mater. Sci. 40 (2005) 3375-3381.
[15] Stapountzi OA, Charalambides MN, Williams JG: Micromechanical models for stiffness prediction of alumina trihydrate (ATH) reinforced poly (methyl methacrylate) (PMMA): Effect of filler volume fraction and temperature, Comp. Sci. Tech. 69 (2009) 2015-2023.
[16] Luyten H, Van Vliet T: Fracture properties of starch gels and their rate dependency, J. Texture Stud. 26 (1995) 281-298.
[17] Ikeda S, Yabuzoe T, Takaya T and Nishinari K: Effects of sugars on gelatinisation and retrogradation of corn starch, in: Starch: Advances in structure and function, edited by Barsby TL, Donald AM and Frazier PJ, Royal Society of Chemistry, Cambridge, UK (2001) 67-77.
[18] ABAQUS Version 6.4, Hibbitt Karlsson and Sorensen Inc, Providence, RI (2004).
[19] Schnur DS, Zabaras N: An inverse analysis for determining elastic material properties and a material interface, Int. J. Numer. Methods in Eng. 33 (1992) 2039-2057.
[20] MATLAB Version 6.5, The Mathworks, INC, USA (2002).
[21] Gamonpilas C, Charalambides MN, Williams JG: Determination of large deformation and fracture behaviour of starch gels from conventional and wire cutting experiments, J. Mater. Sci. 44 (2009) 4976–4986.
[22] Michon C, Chapuis C, Langendorff V, Boulenguer P, Cuvelier G: Strain-hardening properties of physical weak gels of biopolymers, Food Hydrocolloid. 18 (2004) 999-1005.
[23] OuldEleya MM, Gunasekaran S: Rheology of fluid foods for dysphagic patients, Appl. Rheol. 17 (2007) 33137-33146.
[24] Treloar LRG: The physics of rubber elasticity, Oxford University Press, UK (1975).
[25] Genovese DB, Rao MA: Role of starch granule characteristics on rheology of starch dispersions with and without amylose, Cereal Chem. 80 (2003) 350-355.