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Some Objective Instrumental Methods for Evaluating the Texture of Solid Potato Tissue (*Solanum tuberosum* L.)

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ABSTRACT

The paper presents different objective instrumental methods for studying the rheological behaviour of either fresh or thermally treated potato tissue at large and small deformations. It describes the methodology followed and the equipment used to perform fundamental objective assays of compression, tension, shear, stress-relaxation and creep compliance, and imitative direct texture profile analysis (TPA). It also shows the manner in which the force-deformation, deformation-time and force-time curves are used to obtain different rheological and textural properties. Some examples are given of the type of information that can be derived from them. Some refined measurements and procedures that complement recognized methods are proposed to improve the texture measurement of potato tissue. Since the results of objective methods depend on the experimental setup, some of the factors to be considered in each one have been included. Tests are performed on fresh and thermally-treated potato specimens, and indications are given of the best objective methods and rheological properties to represent the firmness of either fresh potato tissue or potato tissue that has been subjected to the different thermal treatments involved in the frozen potato production process.

Keywords: objective methods, potato tissue, rheological properties, texture, viscoelasticity
Abbreviations: PME, pectin methylesterase; **RSM**, response surface methodology; **TPA**, texture profile analysis

OBJECTIVE

The main objective of the present study was to describe the methodology followed to carry out different instrumental objective methods of testing (either at large or small deformations) to measure the texture of potato tissue, as well as the type of information that can be derived from them. The objective methods presented here are not novel. Nevertheless, refined measurements and procedures complementary to well-known methods are proposed as improvements to optimize texture measurement of potato tissue. For that reason, suggested enhancements can be considered as the innovation of the methods employed in this paper. The choice of the method will be determined essentially by the purpose of the measurement. Results and references are included pointing out the best objective methods and/or rheological properties to represent firmness of potato tissue subjected to the different thermal treatments involved in the complete production process of frozen potato.

MATERIALS AND METHODS

Test material

Potatoes (*Solanum tuberosum* L. cv. 'Monalisa'), from Segovia (Spain), were used in this study. For reliable and reproducible results from mechanical tests some basic rules must be taken into consideration with test material, especially when using biological materials that are notoriously variable in their morphology and structure. In order to reduce scatter and enable a reliable comparison between samples, main sources of variation should be at least partially controlled. Product morphology is one of these sources, and test materials with similar structures must always be chosen. By choosing products with similar weights and specific weights, variability from this factor can be reduced (Canet 1980). In this study, only potatoes having weights (in grams) within the confidence interval ($153.83 \leq \mu \leq 186.56$) and specific weights (g cm^{-3}) within the interval ($1.0635 \leq \mu \leq 1.0796$); $P \leq 0.01$ were used. Also, the moisture content of the materials and the temperature both play an important part in affecting the mechanical parameters of foods, and storage can give rise to variability in the results (Alvarez and Canet 2000c). To minimize this, raw material should be stored for 2 months at the latest at about 4 ± 1°C, and 85% relative humidity in the absence of light to avoid sprouting and accumulations of sugars before the experiments (Nourian *et al*. 2003).

The effect of storage time on moisture content was examined over a period of two months. The moisture content of the tubers was 81.81% at 0 days in storage and was slightly lower after 60 days in storage, although the differences between values determined at 0 and 60 days were non-significant. It has been shown that moisture content of fresh potato tissue is practically unaffected by storage over 80 days (Canet *et al*. 2005), and therefore it was unexpected that the mechanical properties of potato samples under objective methods should be affected. Although the effect of starch/sugar levels in tubers on mechanical properties was not studied, the average value of starch in fresh tuber at 0 days in storage was 762 (g kg^{-1} fresh weight). Lower temperatures favoured conversion of starch to reducing sugars, which has been linked to induce undesirable colour changes in fried potatoes (Nourian *et al*. 2003). However, our experiments were conducted at 2-4 weeks over a two-months storage period, and therefore under the storage conditions used it was unexpected that mechanical properties should be affected by changes in starch/sugar levels.

Each day prior to testing, potatoes were reconditioned at room temperature for about overnight. Tubers were manually washed and then the different specimens were obtained as indicated below.

Specimen preparation procedures

Size and shape of the samples affect deformation evaluation (Gil 1991; Luyten *et al*. 1992). Sample dimensions influence the force-deformation data obtained when large forces are applied (Culioli and Sherman 1976; Chu and Peleg 1985; Canet and Sherman 1988; Gil 1991; Charalambides *et al*. 2001). Therefore, sample

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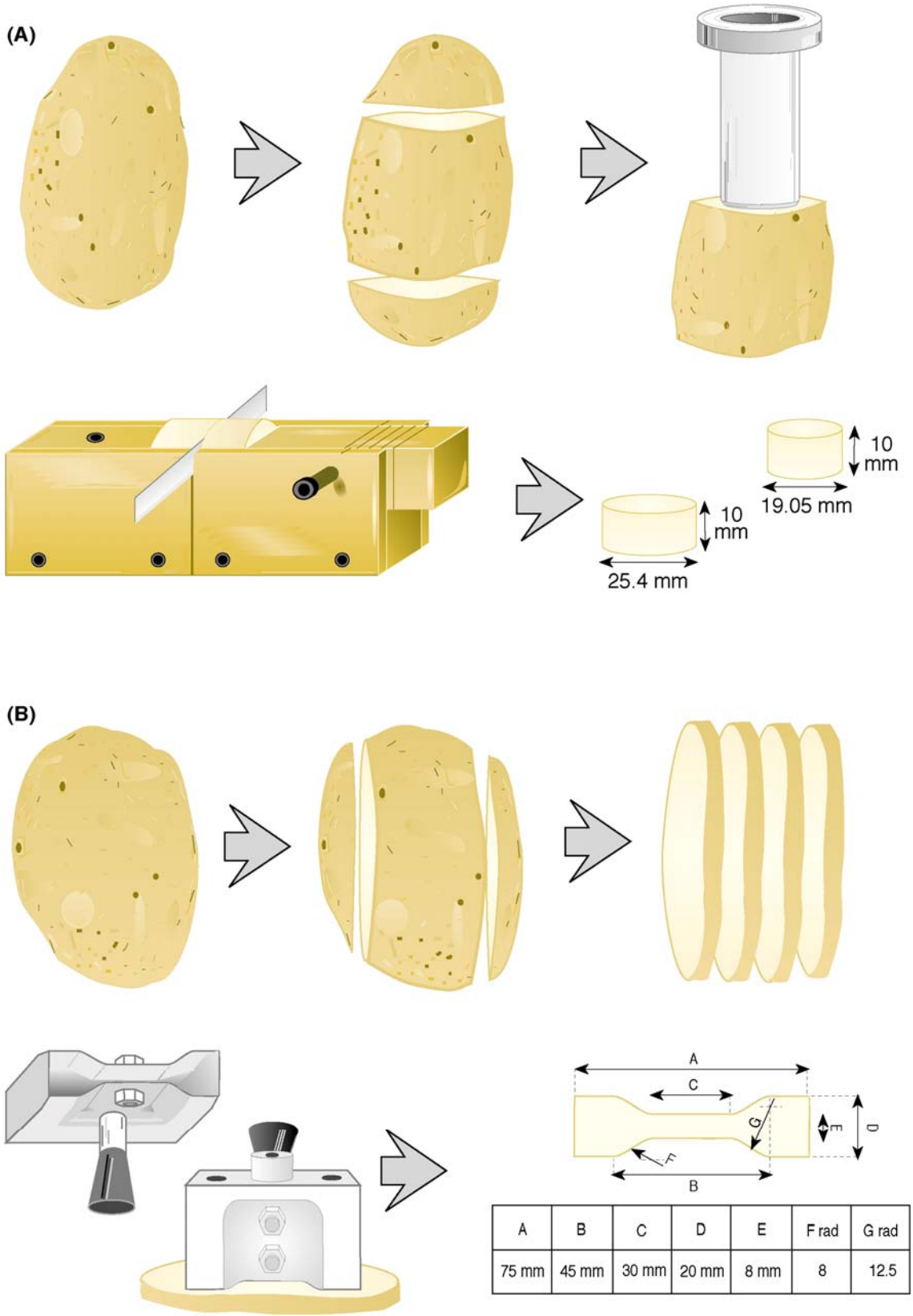

A	B	C	D	E	F rad	G rad
75 mm	45 mm	30 mm	20 mm	8 mm	8	12.5

Fig. 1 Preparation procedures for (**A**) cylindrical specimens (**B**) dumb-bell shaped specimens.

preparation procedure should assure high uniformity in the size and shape of the specimens. A novel methodology was developed to obtain repetitive cylindrical or dumb-bell shaped specimens, through two procedures involving three stages (**Fig. 1**). The distribution and arrangement of morphological structures contribute to the texture of the flesh (Khan and Vincent 1990). The cortex tissue of raw potatoes had 3-6% higher dry matter than pith tissue and showed 25-65% higher puncture force than the pith tissue (Anzaldúa-Morales *et al*. 1992). The proposed repetitive methodology described next allows avoiding the large textural differences reported to exist between the cortex and pith tissues.

To obtain cylindrical specimens, the first stage is to cut off the top and bottom ends of the potato by hand, leaving a central portion 40-50 mm in height as defined by two planes perpendicular to the longitudinal axis of the potato and parallel to one another. The second step is to obtain cylinders of tissue using standard cork borers operating perpendicular to the shear planes. Cork borers 19.05 mm and 25.4 mm in diameter were used (**Fig. 1A**). Finally,

the cylinders are cut to the required height using two stainless steel cells specially designed for these diameters (Gil 1991). For potato tissue, the height of the specimens is always fixed at 10 mm. These diameter/height ratios produce more homogeneous deformation in the samples tested (Canet *et al*. 2007a). Besides, for this height, repetitive rates of cooking and heating during thermal treatments are reached (Alvarez *et al*. 1997).

Another proposed improvement of this study is to obtain dumb-bell shaped specimens to carry out tension tests on potato tissue. For that, the first step is to cut off side sections of the potato with a slicer, following two planes parallel to the longitudinal axis of the potato and to one another. The second step is to obtain 5-mm thick slides by slicing manually parallel to the shear planes. Finally, slides are cut with a cutting die in fine steel working perpendicular to the shear plane. Dimensions and size of dumb-bell shaped specimens (**Fig. 1B**) correspond to the type A described in the Spanish Standard UNE 53023. Once the specimens for each mechanical test are obtained, heights and thicknesses are always checked with an automatic specimen measurement device (Mitutoyo Corporation Digimatic indicator type IDF-150 E 2/50 mm, 0.0001/0.001 mm). Only cylindrical specimens having heights (in mm) within the confidence interval ($9.9407 \leq \mu \leq 10.1777$) and dumb-bell shaped ones having thicknesses (in mm) within the interval ($4.8997 \leq \mu \leq 5.1421$); $P \leq 0.01$ were used in mechanical tests. Since the potato responds to damage by mobilizing sugars and increasing the osmotic pressure of the cells, the mechanical preparation needs to be timed accurately. Certainly, different studies have focused on the turgor pressure as the factor that to a very large extent controls the stiffness of liquid-filled cells (Alvarez *et al*. 2000). For this reason, the mechanical procedures of preparation of specimens were carried out under standard conditions such that the time from taking the whole potato to having a specimen in the test machine were minimum and constant (less than 3 min prior to testing).

Measurement equipments

Two texturometers were used to carry out all the different objective methods with the exception of creep compliance tests. Essentially, these machines consist of a fixed plate and a moving crosshead containing the load cell (Bourne *et al*. 1966). In these instruments, different attachments can be fitted to the underside of the crosshead. Which one is selected depends on how deformation takes place, how the forces in the interior are distributed and what is the predominant type of resulting tension. The three fundamental types of stress are compression, tension and shear (**Fig. 2**). These instruments deform the sample in a controlled manner, at a previously fixed and constant deformation rate. Many authors have pointed out the effect of deformation rate on rheological parameters because for many food materials the deformation properties are time-dependent (Canet and Sherman 1988; Gil 1991; Luyten *et al*. 1991, 1992). For this reason, the deformation rate should be kept constant in each test. Recently, uniaxial compression tests were performed on potato flesh (cv. 'Desiree'). Failure strain, engineering stress and energy at failure increased with deformation rate, whereas true stress and apparent elastic modulus decreased (Canet *et al*. 2007a). In addition, before the test commences, the testing cell should perform one blank run to allow the crosshead to reach the deformation rate selected. Data acquisition systems in these apparatuses record force variation as a function of time or deformation, and the different rheological parameters are derived from the curves.

Mechanical tests of uniaxial compression, shear and uniaxial tension were performed using a model 4501 Instron Universal Testing Machine (Instron, Canton, Mass., USA) with a load cell of 5-kN. A minimum of ten replicates (n = 10) of compression, shear and tension tests were done. Stress-relaxation and texture profile analysis (TPA) tests were carried out by using the TA-HD Texture Analyzer (Stable Micro Systems LTD, Godalming, UK) with a load cell of 2.5-kN. A minimum of five replicates (n = 5) of stress-relaxation and TPA tests were done. At this time, the automatic specimen measurement (ASM) option in the Instron model 4501, takes specimen dimensions, converts the data to an RS-232 format and outputs it to a computer. The option consists of the Digimatic indicator cited above and a Mitutoyo Digimatic multiplexer mod Mux-10. Using the ASM option (Instron series IX software), specimen dimensions can readily be used in calculations of rheological parameters. The Texture Expert software package (Texture Expert for Windows^{TM}, version 2.61) from the TA-HD Texture Analyzer provides the direct capture of specimen data. Compression, shear, tension, stress-relaxation and TPA tests were performed on cylindrical specimens (diameter, 25.04 mm, height, 10 mm), whereas tension tests were performed on dumb-bell shaped specimens. All mechanical tests were carried out at room temperature (20-21°C). During compression, shear and tension tests, this temperature was maintained by using an Instron programmable chamber (Mod. 3119-05, -70°C/+250°C), whereas during stress-relaxation and TPA tests the temperature was maintained using a Temperature Controlled Peltier Cabinet (XT/PC) coupled to a separate heat exchanger and PID control unit.

The equipment used to measure creep compliance is a viscoelastometer with parallel-plate geometry based on the one described by Sherman (1966). Alvarez *et al*. (1998) have described details of its principal components. In each experiment, two cylindrical specimens (diameter, 19.05 mm, height, 10 mm) are sandwiched between a mobile central plate and the two stationary plates. The weight to be used in the test will depend on the food being tested. In the present case, the imposed weight was selected so that it would be up to about 70% of the breaking strength of the tissues. It was exactly 30.031 g. From this weight, the constant applied shear stress can be accurately calculated; this was 516.27 Pa. Creep behavior of the specimens is measured over 2 min at room temperature (20-21°C), using a solvent trap to prevent water evaporation during testing. For creep compliance tests, a minimum of five replicates (n = 5) were performed. The improvement of the method was the development of two software systems to simplify the acquisition and analysis of creep behaviour. The software used to collect displacement-time data was developed by Rico (1995) with the LabWindows for DOS package in C language. Analysis of creep data is performed by a program developed in Quick-Basic 4.5 on the basis of the software described by Sherman (1970) using the graphic method of Inokuchi (1955). Full details of both software systems are reported elsewhere (Alvarez *et al*. 1998).

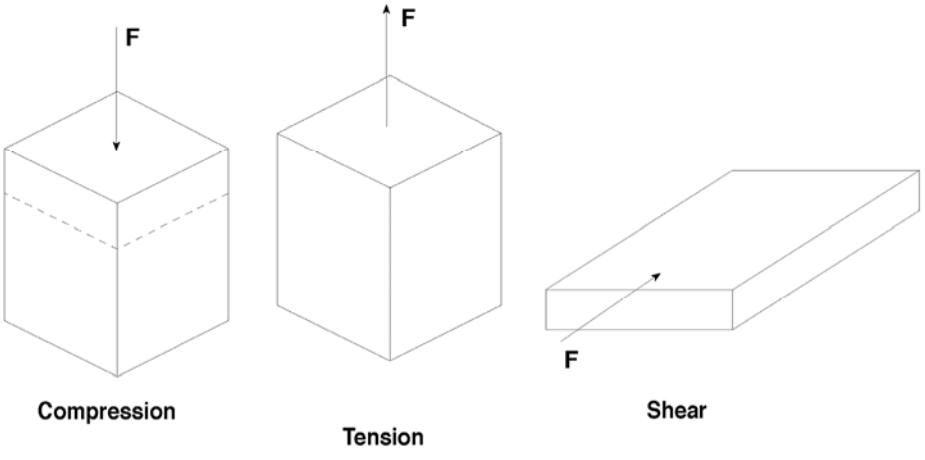

Fig. 2 Modes of action of forces on a solid.

RESULTS AND DISCUSSION

Uniaxial compression test

The uniaxial compression test is the most popular means of deriving the stress–strain properties of soft foods and of biological materials in general; other methods like texture profile analysis (TPA) and stress relaxation are based on the compression test (Canet *et al*. 2007a). Mechanical properties of foods are most commonly determined by compression tests, mainly because of the ease of specimen preparation and the simplicity of the test's performance (Bourne 1982; Luyten *et al*. 1992). As a result, there have been a considerable number of studies on the compressive strength of potatoes (Chu and Peleg 1985; Scanlon and Long 1995; Alvarez *et al*. 1997; Alvarez and Canet 1997). A standardized testing protocol for the determination of rheological properties by uniaxial compression tests at constant displacement rate of potatoes does not exist and needs to be established. The drawback of the compression test, however, is that the friction between the sample and the loading plates leads to an inhomogeneous stress–strain state in the sample (Charalambides *et al*. 2001).

A simple type of uniaxial compression test performed is shown in **Fig. 3**. Essentially, it consists of applying a deformation to the sample and the force resisting this deformation is developed within the specimen and registered by the load cell, so that the deformation of the load cell is read off as the force which causes a given deformation percentage. In most cases this percentage is high enough and the structure breaks. The test was carried out between the standard Instron stainless steel polished platens (upper plate: Catalog number 2830-009, with 57.3 mm in diameter and designed for fracture testing) using a 57 mm diameter flat compression plunger. The compression platen must be of equal or greater diameter than the cylindrical specimen. If the sample has a larger surface area than the diameter of the probe, then the probe can puncture or penetrate the sample. What's more, the strain state of the sample will be non-uniform and therefore essentially indeterminate, so that the test is meaningless (Bourne 1982; Hiller and Jeronimidis 1986). The conditions under which compression tests are performed vary widely in terms of specimen dimensions, magnitude of applied forces, deformation percentage and deformation rate (Canet and Sherman 1988; Gil 1991).

In order to minimize the influence of friction conditions, displacement rate and sample dimensions on the fracture parameters and behaviour in compression tests of cylindrical potato samples, experimental setting has been suggested as follows: surface of sample is not to be lubricated, displacement rate should be intermediate-high (200 mm min^{-1}), and samples have to be made with large diameter (Ø = 25.4 mm) and intermediate height (h = 10–15 mm) (Canet *et al*. 2007a). These conditions can pronounce other important parameters as variety effect that is masked by the variability in conditions. In this study, cylindrical samples of height (10 mm) and diameter (25.4 mm) were compressed under non-lubricated friction condition at 200 mm min^{-1}. A typical force-deformation curve from fresh potato specimen compression is included in **Fig. 3**.

From this, several different rheological parameters can be derived. Usually the maximum compression force required accomplishing breakage [F_c (N)] is measured and used as an index of textural quality. Apparent modulus of elasticity [E_c (MPa)] is also considered as a measurement of the firmness of the product (Finney *et al*. 1964; Canet 1980; Szczesniak 1987). This modulus should be defined as a modulus tangent to the curve in the linearly elastic region, since the force-deformation ratio is not perfectly linear in biological materials. The force F_{Ec} and the deformation ΔL are therefore determined at a point in the linear zone of the curve located two-thirds of the way to the point of maximum breaking force. When the area of the specimen's cross-section A_0 (πr^2) is known, apparent modulus of elasticity is

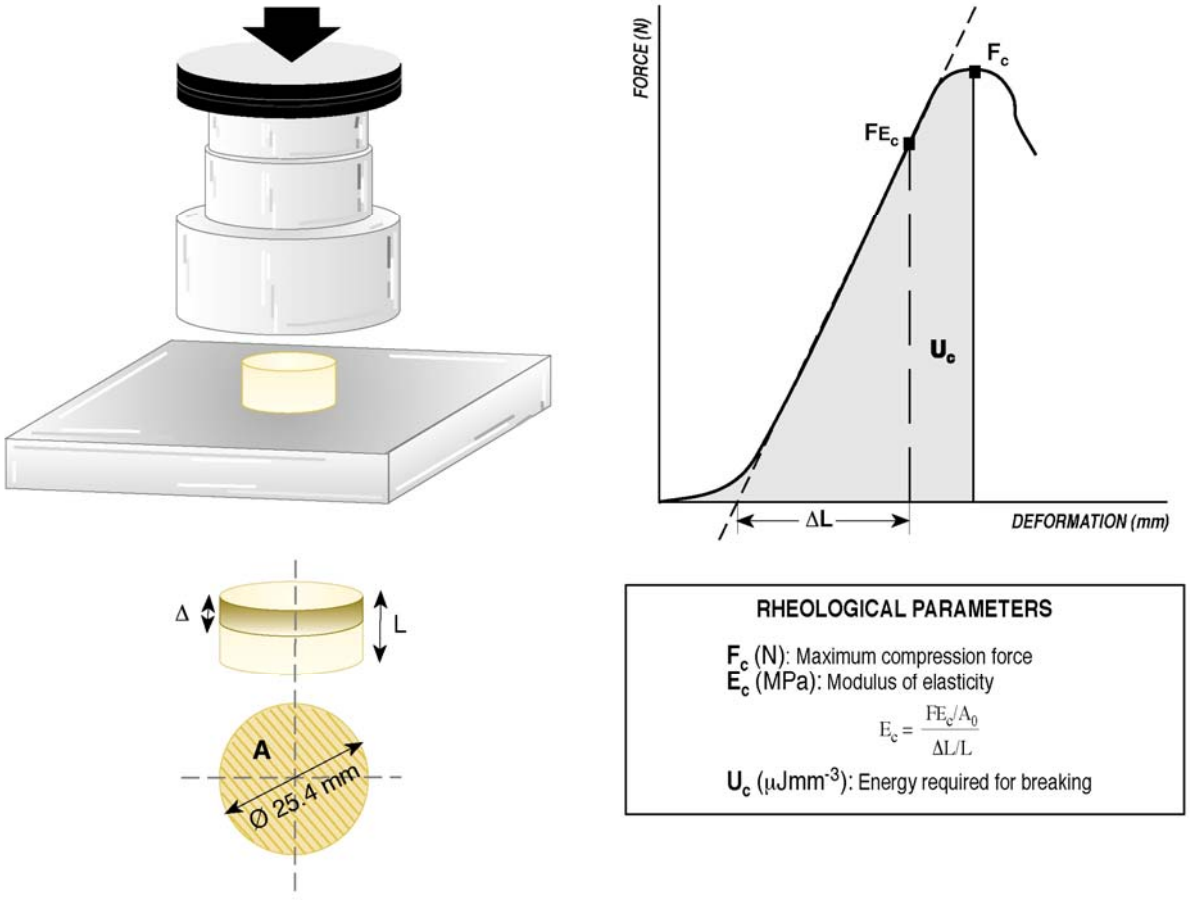

Fig. 3 Uniaxial compression test carried out on cylindrical potato specimens (diameter 25.4 mm, height 10 mm) using a 5 kN load cell and a 57 mm diameter flat compression plunger.

Table 1 Compression, tension, shear and stress relaxation rheological parameters for fresh and thermally-treated potato tissues.

Reference	Thermal treatment	Uniaxial compression test			Uniaxial tension test				Shear test		
		F_c (N)	E_c (MPa)	U_c (μJ mm^{-3})	F_t (N)	E_t (MPa)	U_t (μJ mm^{-3})	D_t (mm)	F_s (N)	G_s (kPa)	U_s (μJ mm^{-3})
Alvarez and Canet 1998b	Fresh	697.89	4.76	411.17	25.86	3.34	160.37	10.46	92.60	16.96	247.27
Alvarez and Canet 1998b	Cooking[a]	50.84	0.46	37.56	2.26	0.59	3.25	3.49	10.53	1.79	8.98
Alvarez *et al*. 1997	Freezing and thawing[b]	352.90	2.05	207.36	12.81	0.96	62.79	13.41	52.55	9.05	151.98
Alvarez *et al*. 1997	Four freeze/thaw cycles[c]	180.27	1.18	80.00	7.57	0.52	42.75	14.16	24.68	3.50	77.84
Alvarez *et al*. 1997	Pre-cooling and freezing and thawing[d]	421.94	2.78	176.33	18.25	0.95	106.70	-	62.13	10.00	184.04
Alvarez and Canet 1997	Pre-cooling and freezing to -6°C[e]	1538.30	6.84	1074.96	23.10	1.45	102.34	5.90	246.40	42.27	398.94
Alvarez and Canet 1998a	Fluctuations during frozen storage[f]	348.32	1.70	166.29	14.62	1.26	71.10	13.17	46.53	6.93	130.78
Alvarez and Canet 1999	Stepwise blanching, freezing and thawing[g]	256.50	1.00	126.93	6.11	0.54	27.08	12.08	27.57	4.57	89.12

Reference	Thermal treatment	First cycle of relaxation			Second cycle of relaxation			Third cycle of relaxation		
		F_{r1} (%)	S_{r1} (N s^{-1})	U_{r1} (N s)	F_{r2} (%)	S_{r2} (N s^{-1})	U_{r2} (N s)	F_{r3} (%)	S_{r3} (N s^{-1})	U_{r3} (N s)
Alvarez and Canet 1998b	Fresh	58.02	-2.56	8009.77	55.80	-3.11	10658.51	54.96	-3.24	11190.16
Alvarez and Canet 1998b	Cooking[a]	82.84	-0.59	534.40	79.56	-0.34	382.91	75.28	-0.22	309.94
Alvarez and Canet 1999	Stepwise blanching, freezing and thawing[g]	81.39	-0.32	221.70	80.40	-0.42	313.00	76.74	-0.42	377.40

[a]Cooking by immersion in water at 97°C for 15 min; [b]freezing rate (2°C min^{-1}) and thawing rate (0.5°C min^{-1}); [c]freeze/thaw cycles performed with freezing at 2°C min^{-1} and thawing at 0.5°C min^{-1}; [d]pre-cooling at 3°C for 30 min prior to freezing at 2°C min^{-1}and thawing at 0.5°C min^{-1}; [e]pre-cooling at 3°C for 30 min prior to freezing to -6°C at 2°C min^{-1}; [f]freezing at 2°C min^{-1}, sixteen temperature fluctuations in the range -24 to -18°C and thawing at 0.5°C min^{-1}; [g] First step at 60°C for 30 min, second step at boiling point for 2 min, freezing at 2°C min^{-1}and thawing at 0.5°C min^{-1}

determined as the ratio between engineering stress and unit deformation (Cauchy strain). A third rheological parameter used is the apparent energy required for breaking per unit of volume [U_c (μJ mm^{-3})], that is measured as the area under the force-deformation curve up to the rupture point. Recently, a method has been established for determining dimensionless area expansion ratio at failure, A_f/A_0 (actual maximum area of the deformed sample in contact with the loading platens/original area of the non-deformed sample) (Canet *et al*. 2007b). The determination of area expansion ratio at failure allows a direct determination of true stress (F/A_f) from engineering stress (F/A_0), justifying the use of this additional parameter in relation to failure under compression tests.

Table 1 shows average values of F_c, E_c, and U_c obtained for fresh and thermally- treated specimens. The differences are clearly due to the structural differences between cells of tissues. Compressive rheological parameters obtained are difficult to compare with bibliographic data due to the wide variation of the experimental conditions used. In potato tissue, compression tests have been used to study elastic behaviour and establish the relation between cell turgor pressure and apparent modulus of elasticity (Canet 1980; Szczesniak 1987). In fresh potato, the values obtained of the parameters 'maximum compression force' and 'apparent modulus of elasticity' reflect the mechanical response of the tissue to the internal cell pressure (Alvarez and Canet 1998b). In cooked tissue these parameters reflect the response to the intercellular cohesion and the elastic response of the cell walls disrupted by starch gelatinization (**Table 1**). The freezing process itself causes damage to cell structures, but more appropriate methods can be used in order to optimize quality. Alvarez *et al*. (1997) showed that when potato tissue was cooled at a low temperature (3°C) for a long time (30 min) prior to freezing, the maximum compression force required accomplishing breakage of the tissue increased at the different freezing rates tested. For example, note as F_c value for potato tissue was 352.90 N after freezing at 2°C min^{-1} and thawing at 0.5°C min^{-1}, whereas this value increased up to 421.94 N when a pre-cooling at 3°C for 30 min was performed prior to freezing. By dividing maximum compression force between sectional area (in this case, 506.71 mm^2), the stress at breakage or strength can be estimated; of the two values, the latter reaching up to 0.83 Pa when a pre-cooling is carried out, as against 0.70 Pa without pre-cooling. On the other hand, also the structural rigidity of samples subjected to four freeze/thaw cycles was less than half that of samples subjected to only one cycle (Alvarez *et al*. 1997). Compression test was the best objective method for the analysis of pre-cooling and freezing rate effects on mechanical strength of potato at temperatures ranging from –3 to –18°C (Alvarez and Canet 1997). In this study, rheological parameters from compression tests were more significantly affected by factors studied, with high correlations between all of them. In addition, maximum compression force (F_c), as a measure of mechanical damage, showed the highest level of significance for the effect of temperature fluctuations during frozen storage on the textural quality of potato (Alvarez and Canet 1998a). Therefore, this test may give good results in studies related to the effects of freezing and frozen storage in the texture and structure of potato.

Uniaxial tension test

The device used to carry out the uniaxial tension tests is shown in **Fig. 4**. Tension tests give a comparable (but opposite) deformation to compression tests. In contrast with compression, tension tests are far more difficult to perform, primarily because of the grip problem but also because the preparation of a suitable specimen for testing may require special considerations (Luyten *et al*. 1992; Chen *et al*. 1994). As mentioned above, the last problem was solved in this study by cutting out a dumb-bell shaped specimen and holding it at the wide ends, so that the sample is then more likely to break in the narrow center portion (Bourne 1982). Certainly, no data have been found in the literature on the application of tension tests to dumb-bell shaped potato specimens. In turn, the test cell consists of two compressed-air clamps (pressure of clamping used at the ends of the speci-

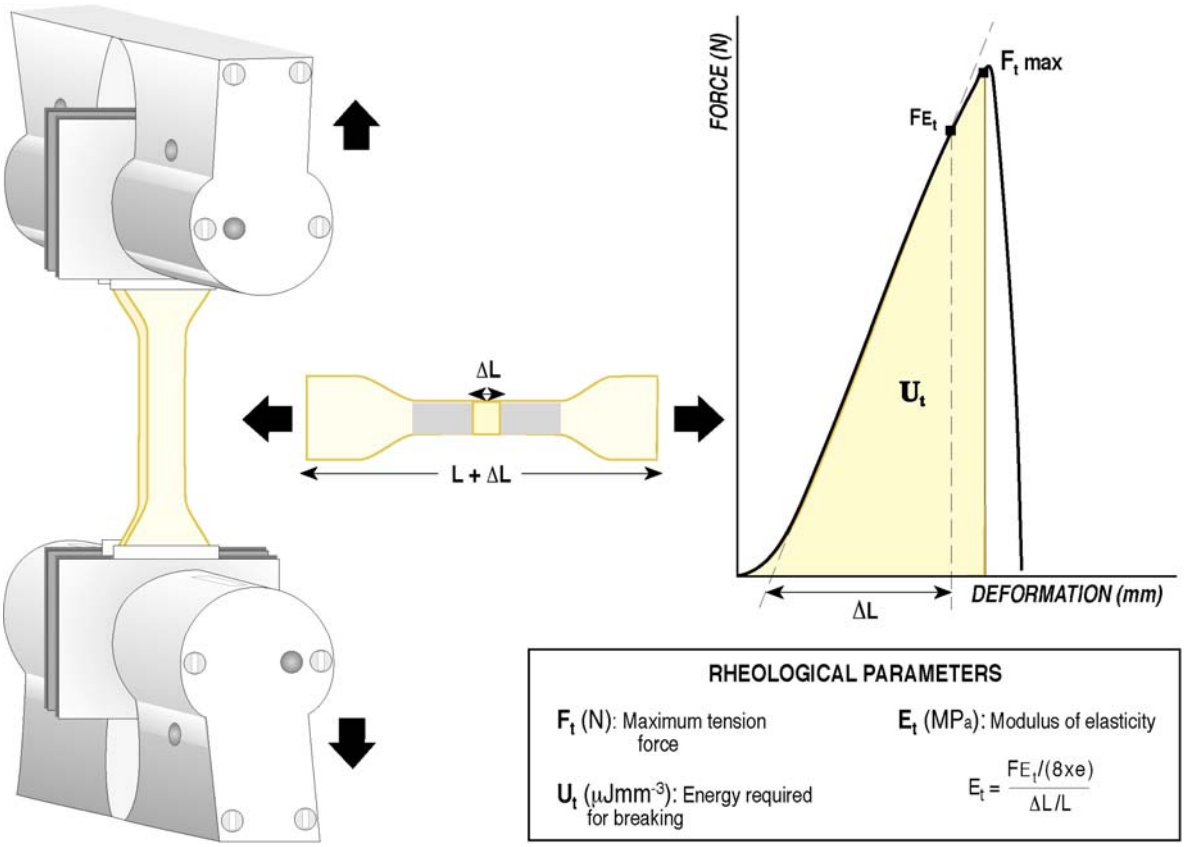

Fig. 4 Uniaxial tension test carried out on dumb-bell shaped potato specimens (type A, Spanish Standard UNE 53023) using a 5 kN load cell.

men: 0.15 MPa) fitted to the wide specimen ends by filter paper to minimize slippage and failure. For uniaxial tension, a deformation rate of 100 mm min^{-1} was used. A typical curve force-deformation from potato specimen tension is also included in **Fig. 4**. Different rheological parameters can be derived from the curves. The most usual parameter is maximum tension force required to accomplish breakage [F_t (N)]. As in compression, apparent modulus of elasticity [E_t (MPa)] can also be calculated as a modulus tangent to the curve in the linearly elastic zone. When the area of the specimen's cross-section A_0 ((8 x e) mm^2, where e is 5-mm thick)) is known, the apparent modulus of elasticity is determined as the ratio between engineering stress and unit deformation (strain). The area enclosed by the force-deformation curve which this mechanical test generates represents energy (force × distance). Great care is necessary to ensure that the curve encloses only the fracture energy and does not include stored elastic strain energy (Vincent 1994). The easiest way to ensure this is by notching the specimen and to unload the test specimen before it has broken completely. In this manner any elastic strain energy is discounted from the final reckoning, and the energy which the force-deformation curve encloses is that require propagating the crack. It is therefore one of the ways in which measurements can reliably be made on complex plant structures and materials (Atkins and Vincent 1984). We did not use notched specimens, so that the area under force-deformation will give only a measurement of the apparent energy required for breaking expressed per unit of volume [U_t (μJ mm^{-3})]. This calculated parameter cannot be taken as real fracture energy. In turn, for calculating tension energy, a 30-mm length of specimen in the neck region between the retained ends is taken as the main part of the specimen for which a uniform stress may be considered (Gere and Timoshenko 1986). Maximum deformation [D_t (mm)], representing the relative displacement of the points of a body until rupture, may be another useful parameter for reflecting the plastic deformation of the cell wall (Canet 1980).

For fresh and thermally- treated dumb-bell shaped specimens, the average values obtained of F_t, E_t, U_t, and D_t are also presented in **Table 1**. From tension, the maximum breaking force is interpreted as the mechanical response to internal cell pressure; apparent modulus of elasticity reflects the elastic response capacity of the cell wall under tension up to collapse and bursting of the cell (Alvarez and Canet 1998b). In cooked tissues, tension rheological parameters have been considered as a measure of adhesiveness (Bourne 1982). For comparative purposes it may be noted that in fresh tissue, E_t is lower than E_c estimated from compression tests, i.e., the opposite of what was found for cooked tissue, indicating that this large material deformation property in potato is not independent of the size and shape of the test-piece or of the method applied. As reported by Luyten *et al*. (1992), in order to understand the material properties at large deformations it is important to ensure that real properties determined, are not affected by the method chosen. Because the deformation in fracture increases with a decreasing rate of deformation and the viscous nature of viscoelastic materials are more pronounced for longer timescales (Luyten *et al*. 1991), the lower deformation rate used for tension tests could at least partially account for the lower value of the modulus of elasticity found in the tension test. Also, the different proportions of tissues present in the two types of specimen can account for this discrepancy (Alvarez and Canet 1998b). As reported by Vincent (1994), if two different tests give different results, this may be due to that the test specimen is responding in two different ways in two tests, indicating some influence from the structures within specimen.

Tension tests are used to measure the adhesion of a food to a surface and are widely used in bakery products and bread (Nussinovitch *et al*. 1990; Chen *et al*. 1994). Luyten *et al*. (1991) pointed out that the advantage of tension testing over compression is that the start of fracture can be readily observed because it is nearly always at the outside of the sample, while with uniaxial compression the start of

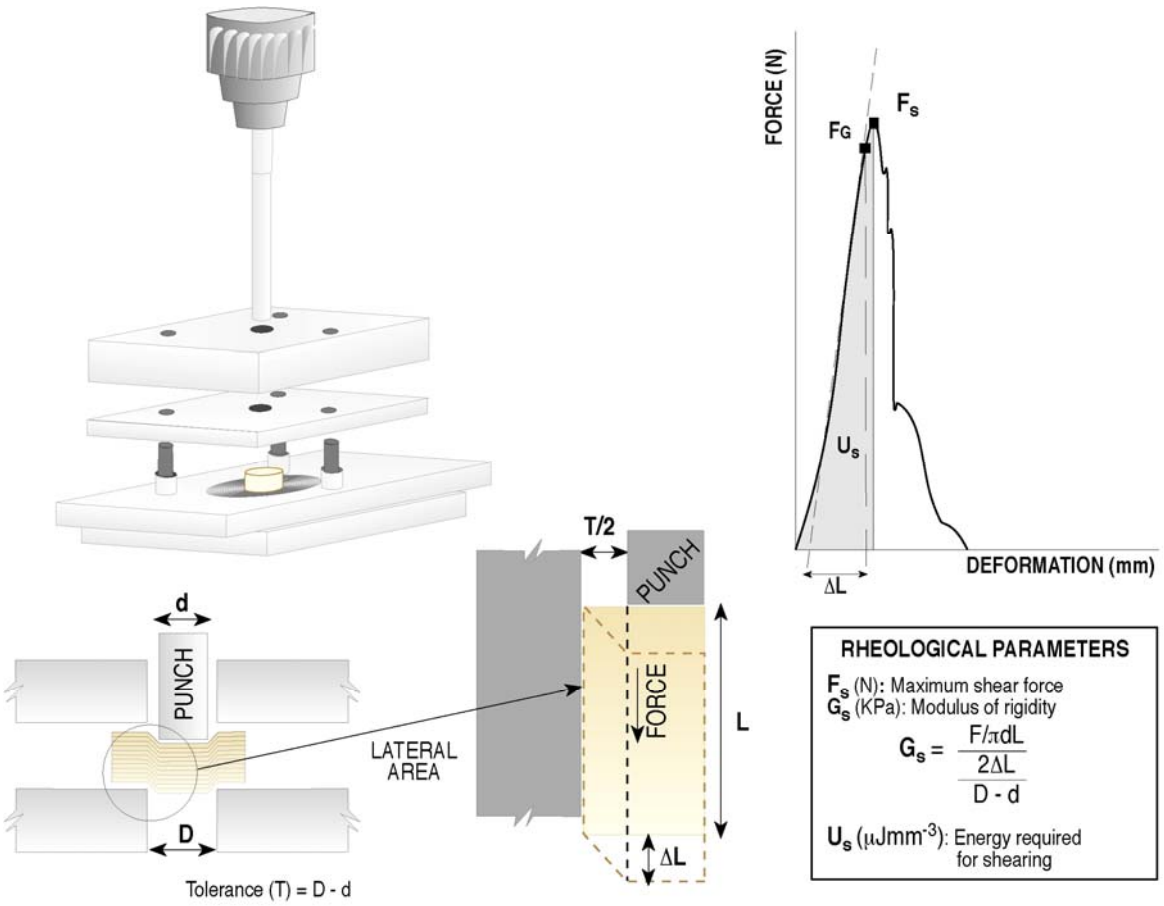

Fig. 5 Shear test carried out on cylindrical potato specimens (diameter 25.4 mm, height 10 mm) using a 5 kN load cell and one stainless steel cell specially designed for the test.

fracture is often inside the test-piece. Besides, on dumb-bell shaped specimens it is possible to examine the effect of artificially made notches and so to determine the notch-sensitivity. Rheological parameters from tension test were the most sensitive to the effect of measurement temperature in a positive range from 0 to 40°C (Alvarez and Canet 1997). If dumb-bell shaped specimens are going to be treated thermally, it should be taken into account that because of their different shapes, the proportions of the tissues making up cylindrical and dumb-bell shaped specimens are likewise different as cited above. Dumb-bell shaped specimens contain a high percentage of inner phloem and, for instance, higher temperatures were required to attain a level of PME activity like that found in cylindrical specimens at the first step of blanching of frozen-thawed potato tissues (Alvarez and Canet 1999).

Shear test

The cell used to perform the shear tests is shown in **Fig. 5**. In shear testing it is necessary to cause the contiguous parts of the specimen to slide relative to each other in a direction parallel to the plane of contact under the influence of a force tangential to the section on which it acts (Bourne 1982). With the proposed method here, shearing of cylindrical specimens is carried out using a test cell in stainless steel (Canet 1980), consisting of two flat platforms with concentric holes of 9.525 ± 0.013 mm diameter (D). These are used in conjunction with a flat base finely ribbed with concentric rings 0.100 mm deep to ensure proper alignment of the flat-ended punch and the specimen and prevent their slipping during testing. Because the punch used is 9.450 ± 0.013 mm in diameter (d), tolerance ($T = D\text{-}d$) between the cell and the punch is 0.075 mm, so that the desired pure shear can be produced during testing. In this sense, the proposed method is more efficient that the existing ones. Because of the small tolerance, it is advisable to execute a blank run before testing in order to avoid any kind of friction between parts of the cell disturbing rheological parameter values. Shear testing was carried out at a high deformation rate, 400 mm min^{-1}, to avoid compression of the tissue. Force-deformation curves obtained by shearing fresh potato tissue are similar to the one shown in **Fig. 5**. The height of the first peak is a measure of the force required to shear the specimen and is designated the maximum shear force [F_s (N)]. Modulus of rigidity [G_s (MPa)] can be calculated from the relationship between shear stress ($F_G/\pi dL$, where πdL is the lateral area sheared by the punch, and L is the height of the specimen) and the unit deformation ($2\Delta L/D\text{-}d$, where ΔL is the deformation at a point in the linear zone of the curve corresponding to a force F_G). The area under a force-deformation curve is a measure of the energy required for shearing during a test, designated shear energy and expressed per unit of volume [U_s (μJ mm^{-3})].

For fresh and thermally- treated cylindrical specimens, average values of F_s, G_s, and U_s are shown in **Table 1**. Shear testing is often used for studying the small deformation behavior of foods. However, food technologists sometimes use shear to describe any cutting action that causes the product to be divided into two pieces. By utilizing pure shear action as described by physicists and engineers, parallel and opposite tangential deformations act on sections of the product with infinitesimally small gaps between them, and therefore this test is considered ideal for studying the microstructure of vegetables. Canet (1980) reported that maximum shear force F_s and modulus of rigidity G_s could be considered as indicators of the mechanical resistance to deformation and rupture of cell walls in potato tissues. Shear force was the most suitable rheological parameter for establishing the kinetics of softening of potato tissue by temperature fluctuations (Alvarez and Canet 2000a). By applying PCA analysis, maximum shear force has also been

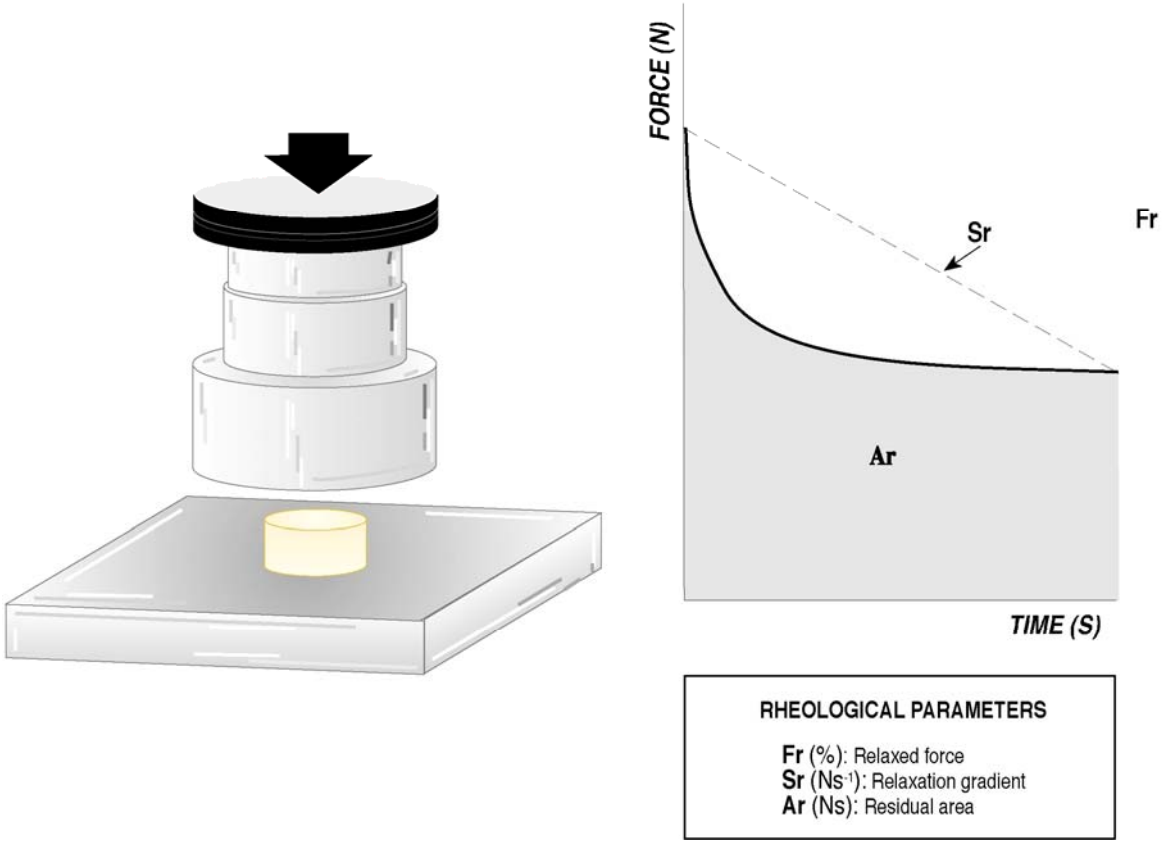

Fig. 6 Stress-relaxation test carried out on cylindrical potato specimens (diameter 25.4 mm, height 10 mm) using a 2.5 kN load cell and a 75 mm diameter flat compression plunger.

found to be a good rheological parameter for differentiation of the structural damage and softening produced in the tissue by temperature fluctuations during storage of frozen potato (Alvarez and Canet 2000b). The shear test has been found to be well suited to the study of freezing rate and programmed freezing effects on structure of potato (Alvarez *et al*. 1997). In this study, coefficients of softening per freeze/thaw cycle were determined, the highest value being given by the modulus of rigidity.

Stress-relaxation test

A typical force-time curve from stress-relaxation of a fresh potato specimen is shown in **Fig. 6**. Stress-relaxation in compression consists in the deformation of the specimen up to a fixed percentage, after which the specimen is allowed to relax for a given time (Canet 1980). The test procedure is similar to that of compression tests. A cylindrical specimen is placed on a flat platform and a compression platen (75 mm in diameter) is fitted to the load cell. During testing the specimen is compressed to a distance of 2 mm (20% strain based on original size) at a deformation rate of 400 mm min^{-1}. The deformation is then held constant and the specimen is allowed to relax for 1 min following deformation. Following previous studies (Canet 1988; Mouquet *et al*. 1992), the relaxed force [F_r (%)] is calculated as

$$F_r \text{ (1 min)} = (F_0 - F_i)/F_0 \quad (1)$$

In Eq. (1), F_0 is the maximum compression force for deformation of 2 mm and F_i is the force recorded after 1 min of relaxation. The relaxation gradient [S_r (N s^{-1})] is the slope of the straight line joining the maximum compression force and relaxed force points after 1 min. The relaxation residual area [A_r (N s)] is the area below the force-time curve.

For fresh, cooked and blanched-frozen-thawed potato tissues, average values obtained of F_r, S_r, and A_r corresponding to successive cycles of stress and relaxation are shown in **Table 1**. There are clear differences between the tissues as regards the behaviour of the rheological parameters and how they evolved with the number of cycles applied. For example, relaxation force in fresh tissue was very low and slow after the first compression when compared with that of cooked tissue. In fresh specimens, the relaxation gradient and residual area required to achieve 20% deformation of the specimen increased with the number of cycles. This phenomenon may be attributed to strain hardening caused by increased cohesion between walls and cell contents, and also to a cumulative increase of the internal cell pressure with each cyclic load. The relaxed force decreased with successive cycles in all cases. Relaxed force may be related to the response of the internal cell pressure and the elastic characteristics of the cell wall. In cooked tissue, this parameter was higher because of loss of turgor pressure and distension of the cell wall caused by gelatinization of the starch; this combined with solubilization of the pectin material in the cells to produce a decrease in their elastic properties. On the basis of relaxation tests several researchers have shown that fruits and vegetables exhibit viscoelastic behavior (Canet 1988; Mouquet *et al*. 1992). In fresh potato tissue, relaxed force F_r decreased consistently and linearly with time over a 140-day storage period, reflecting the decrease of cell turgor pressure resulting from the predominance of water loss through evaporation over water production (Alvarez and Canet 2000c). Using response surface methodology (RSM), relaxed force F_r has been found to be the most suitable parameter for detecting the firmness effect that the pectin methylesterase (PME) activity produces on frozen potato by stepwise blanching in the ranges of temperature (60-65°C) and time (25-35 min) (Alvarez and Canet 1999).

Creep compliance test

In creep compliance tests, when a constant stress is applied to the mobile plate, the specimens undergo deformation as a function of time and curves like the one in **Fig. 7** are plotted according to Sherman (1970). The figure also shows the

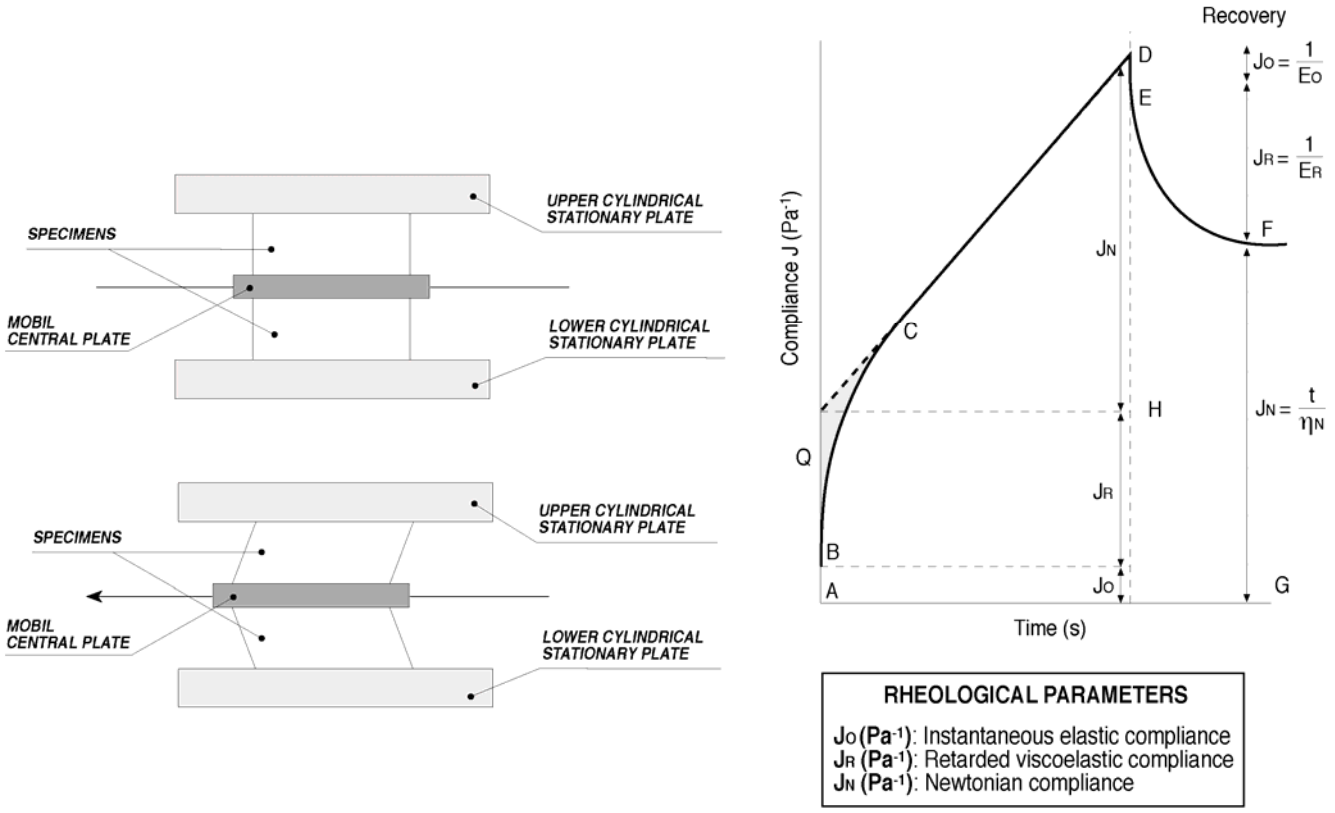

Fig. 7 Arrangement of potato specimens (diameter 19.05 mm, height 10 mm) and compliance-time curve derived from creep compliance test.

arrangement of specimens on the viscoelastometer and their shear strain during tests. The equipment used to measure creep compliance of potato tissues was a viscoelastometer with parallel-plate geometry based on the one described by Sherman (1966). A description of its principal components can be consulted (Alvarez *et al*. 1998). In these curves, compliance (*J*) represents the ratio between unit shear strain and the constantly applied shear stress. When this stress is applied to the mobile plate, the specimens undergo deformation as a function of time and compliance-time curves are plotted. Creep compliance [*J*] is defined as (Sherman 1970):

$$J(t, \sigma_0) = \gamma(t)/\sigma_0 \quad (2)$$

In Eq. (2), $\gamma(t)$ is the shear strain at time t and σ_0 is the constant applied shear stress $F_0/2A$, A being the cross-sectional area of the cylindrical specimen. The behaviour defined by this equation is delineated as one instantaneous elastic component [J_0 (Pa^{-1})]; two retarded elastic compliances [J_1 (Pa^{-1})] and [J_2 (Pa^{-1})] with their associated retardation times [τ_1 (s)] and [τ_2 (s)] and coefficients of viscosity [η_1 (Pa s)] and [η_2 (Pa s)] respectively; and one steady state viscous flow component where [η_N (Pa s)] is the coefficient of viscosity associated with Newtonian flow.

For fresh, cooked and blanched-frozen-steamed potato tissues, average values obtained of J_0, J_1, J_2, τ_1,τ_2, η_1, η_2 and η_N are shown in **Table 2**. It has been stated that creep compliance tests can provide more information than stress-relaxation procedures, because greater number of rheological parameters can be estimated and associated with discrete components of the product being tested (Mittal *et al*. 1987; Jackman and Stanley 1995). For fresh and cooked potato tissues, the instantaneous elastic modulus J_0 may be related to the internal cell pressure and gelatinization of starch, whereas the two viscoelastic units appeared to reflect viscoelastic properties of pectin substances and hemicelluloses respectively. It was also found that Burgers model parameters were the most appropriate for detecting the firmness effect caused by PME activity in freezing with

Table 2 Creep compliance rheological parameters and textural properties for fresh and thermally-treated potato tissues.

Reference	Thermal treatment	Creep compliance test									
		J_0 (Pa^{-1})	J_1 (Pa^{-1})	J_2 (Pa^{-1})	E_1 (Pa)	E_2 (Pa)	η_1 (Pa s)	η_2 (Pa s)	τ_1 (s)	τ_2 (s)	η_N (Pa s)
Alvarez and Canet 1998b	Fresh	1.98E-7	1.94E-7	2.23E-8	5.15E6	4.48E7	7.06E8	1.15E8	135.60	26.71	3.90E8
Alvarez and Canet 1998b	Cooking[a]	4.92E-7	2.44E-7	2.07E-7	4.02E6	4.82E6	2.24E8	0.85E8	52.20	18.77	1.14E8
Alvarez *et al*. 1999	Complete process of production of frozen potatoes[b]	3.63E-7	2.24E-7	1.20E-7	4.46E6	8.33E6	3.72E8	3.15E8	83.45	37.82	1.98E8
Reference	**Thermal treatment**	**Texture profile analysis (TPA)**									
		F (N)	H (N)	A_2/A_1 (-)	A_3 (J)	S (mm)	Ch (J)	-	-	-	-
Alvarez and Canet 1998b	Fresh	773.57	681.93	0.08	-0.13	0.56	31.64	-	-	-	-
Alvarez and Canet 1998b	Cooking[a]	16.97	24.35	0.05	-1.03	0.18	0.22	-	-	-	-
Alvarez *et al*. 1999	Complete process of production of frozen potatoes[b]	91.47	116.32	0.14	-0.48	0.51	8.45	-	-	-	-

[a]Cooking by immersion in water at 97°C for 15 min; [b]In the process of production of frozen potatoes, the first step of the stepwise blanching was performed at 65°C for 30 min, whereas the second step prior to freezing was performed at 97°C for 2 min. Freezing was carried out at 2°C min^{-1} and final cooking was carried out by pressure steaming at 117°C for 2 min.

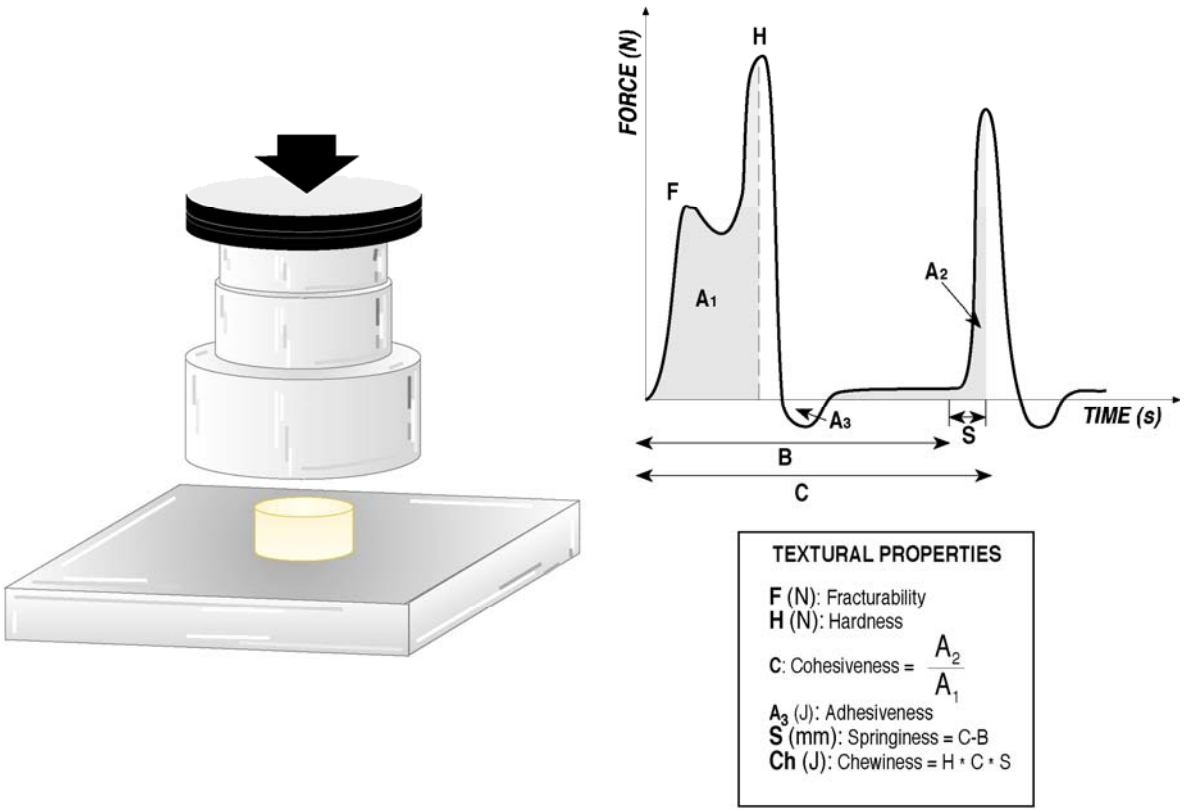

Fig. 8 Texture profile analysis (TPA) curve from the TA.HD texturometer for potato specimens (diameter 25.4 mm, height 10 mm) at 100 mm min^{-1} at degree of compression 70% of the initial height.

pressure steaming of potato tissue (Alvarez *et al*. 1999). In spite of the specimens were prepared under a strictly time-controlled regime, our own creep test results showed that the data are highly variable. Therefore, this is probably because the sample changes its turgor throughout the experiment, in part because the cells respond to stress by mobilizing starch into sugar and pulling the interstitial water into the cell, thus increasing turgor pressure (Alvarez *et al*. 2000). Further experiments are in progress to be able to estimate the rheological parameters corresponding to tissue recovery.

Texture profile analysis (TPA) test

A typical force-time curve derived from a TPA test is shown in **Fig. 8**. In TPA tests, cylindrical specimens are doubly compressed in a reciprocating motion that imitates the action of the jaw, and a number of textural parameters which correlate well with sensory evaluation of those parameters are extracted from the resulting force-time curve (Szczesniak *et al*. 1963; Bourne 1980, 1982). Again the test procedure is similar to that of compression and stress-relaxation tests. Although potatoes are not consumed freshly, TPA was also applied to fresh specimens and considered as a model vegetable for comparisons (**Table 2**). A cylindrical specimen is placed on a flat platform and a compression platen (75 mm in diameter) is fitted to the load cell. During testing the specimens were compressed to a distance of 7 mm (70% strain based on original size) at a deformation rate of 100 mm min^{-1}, which results in extensive breaking of the specimens. The influence of deformation rate and degree of compression on textural parameters of potato and apple tissues in TPA tests has been also studied (Alvarez *et al*. 2002). Textural properties were derived from the curve generated by such a test to give fracturability [F (N)] (the force at the first significant break in curve), hardness [H (N)] (the height of the peak force on the first compression), cohesiveness [A_2/A_1 (dimensionless)] (the ratio of the positive force areas under the first and second compressions], adhesiveness [A_3 (J)] (the negative force area of the first bite representing the work necessary to pull the compression platen away), springiness [S (mm)] (the distance that the food recovers its height during the time that elapses between the end of the first bite and the start of the second bite). Chewiness [Ch (J)] is derived by calculation from the measured parameters (**Fig. 8**). For fresh, cooked and blanched-frozen-steamed potato tissues, average values obtained of F, H, A_2/A_1, A_3, S and Ch are presented in **Table 2**. In fresh specimens, fracturability was greater than hardness; the opposite was the case in both processed tissues. Cohesiveness was greater in fresh than in cooked tissue, thus confirming that the thermal treatment in water reduced intercellular cohesion. By contrary, the highest cohesiveness produced by stepwise blanching prior to freezing and steaming under conditions indicated in **Table 2** was attributed to PME activity (Alvarez *et al*. 1999). As was to be expected, adhesiveness in fresh specimens was very small. Springiness was also greater in fresh than cooked specimens due to loss of the structure's elastic response. Chewiness in fresh has to be approached with caution, since it is a characteristic textural property of soft products (Nussinovich *et al*. 1990). In fresh tissue, fracturability could represent the mechanical response of the medial lamina, whose strength is greater than that of the cell walls (Jack *et al*. 1995). In cooked potato samples of different cultivars and specific gravities, Leung *et al*. (1983) found high correlations between the mealiness of the potato and the product of cohesiveness and adhesiveness. Jankowski (1992) used Texture Profile Analysis to study the influence of starch retrogradation on the texture of cooked potato. Compression parameters correlated with hardness from TPA in the optimization of freezing with pressure steaming of potato tissue (Alvarez *et al*. 1999).

CONCLUSIONS

The experimental results indicated that the objective methods used taken together constitute a complementary set of techniques that are useful in studying the rheological behaviour of either fresh or thermally treated potato tissue. No one technique is superior to the others (but each has advan-

tages and limitations). In fresh potato, maximum compression and tension forces (F_c, F_t), apparent modulus of elasticity from both tests (E_c, E_t), relaxed force (F_r), relaxation gradient (S_r) and instantaneous elastic compliance (J_0), reflect the mechanical response of the internal cell pressure and the elastic response capacity of the cell wall under deformation, compression or tension up to collapse and bursting of the cell. Maximum shear force (F_s) reflects the mechanical response of the tissue to deformation and cell wall rupture and modulus of rigidity (G_s) can be considered as indicator of the elastic response capacity of the cell wall under shear. In thermally- treated potato tissue these same parameters reflect mechanical response of intercellular cohesion and the elastic response of the cell walls disrupted by starch gelatinization. In fresh and treated potato, the two retarded elastic compliances (J_1, J_2) appeared to reflect viscoelastic properties of pectin substances and hemicelluloses, respectively.

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