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Cheese — Determination of rheological properties by uniaxial compression at constant displacement rate

Fromage — Détermination des propriétés rhéologiques par compression uniaxiale à vitesse constante de translation

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Contents

Page

Foreword

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ISO/TS 17996|IDF/RM 205 was prepared by Technical Committee ISO/TC 34, *Food products*, Subcommittee SC 5, *Milk and milk products*, and the International Dairy Federation (IDF). It is being published jointly by ISO and IDF.

Foreword

IDF (the International Dairy Federation) is a worldwide federation of the dairy sector with a National Committee in every member country. Every National Committee has the right to be represented on the IDF Standing Committees carrying out the technical work. IDF collaborates with ISO in the development of standard methods of analysis and sampling for milk and milk products.

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ISO/TS 17996|IDF/RM 205 was prepared by the International Dairy Federation (IDF) and Technical Committee ISO/TC 34, *Food products*, Subcommittee SC 5, *Milk and milk products.* It is being published jointly by IDF and ISO.

All work was carried out by the Joint ISO-IDF Action Team on *Physical properties and rheological tests*, of the Standing Committee on *Minor compounds and characterization of physical properties*, under the aegis of its former project leader, Mrs Y. Noël (FR), who performed most of the work, and its present project leader, Mr P. Watkinson (NZ).

Cheese — Determination of rheological properties by uniaxial compression at constant displacement rate

1 Scope

This Technical Specification describes a method for the determination of rheological properties by uniaxial compression at constant displacement rate in hard and semi-hard cheeses.

The method provides standard conditions for sampling and testing, for data representation and general principles of calculation.

NOTE Sampling might be difficult with some cheese varieties, for example caused by shortness, brittleness, stickiness and soft consistency. In these cases, reliable results cannot be achieved.

2 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

2.1
rheological properties
deformation under compression of the test sample by the procedure specified in this Technical Specification

3 Principle

A cylindrical test sample, of defined dimensions, is compressed at a constant crosshead speed with a compression tool up to a relative deformation sufficient to determine the apparent fracture point. The force, which is the resistance of the cheese sample during compression, is measured with a load cell. The displacement may be measured either from the position of the cross head or calculated from the elapsed time multiplied by the displacement rate.

A schematic representation of the principle of the test is given in Figure A.1.

4 Apparatus

Usual laboratory apparatus and, in particular, the following.

4.1 Cork-borer, such as that shown in Figure A.4 as an example.

It is recommended to mount the cork-borer on a drill-stand in order to drive it slowly and steadily through the test sample.

4.2 Parallel-wire cutting device, with a wire of diameter less than or equal to 0,4 mm and with a system to keep the two wires parallel to each other and perpendicular to the plug. It should also include a mechanically driven cutting system to cut the test sample to the required height.

4.3 Measuring cell, with a support and compression plate of the same stiff material, with smooth and parallel surfaces (e.g. stainless steel, aluminium or Teflon), of diameter larger (by 20 %) than that of the deformed test portion when at maximum compression. The load cell capacity shall have a reasonable relationship to the expected maximum force.

5 Sampling

A representative sample should have been sent to the laboratory. It should not have been damaged or changed during transport or storage.

Sampling is not part of the method specified in this Technical Specification. A recommended sampling method is given in ISO 707|IDF 50.

6 Procedure

6.1 Thermal equilibration of test samples

If the storage temperature of the loaf of cheese is above that of the measuring temperature, then the loaf of cheese shall be equilibrated at the measuring temperature for at least 50 h before further preparation of the test sample because of the slow crystallization of milk fat in the cheese.

If the storage temperature of the loaf of cheese is below that of the measuring temperature, before any preparation store the loaf of cheese at the measuring temperature for at least 12 h. If there are specific difficulties that can occur during the sample preparation at the measuring temperature, then sample at the lower storage temperature and then equilibrate the test samples to the measurement temperature. In this case, the sample thermal equilibration time may be less than 12 h.

NOTE Examples of specific sampling difficulties are that the cheese is hard to cut, or a heated loaf of cheese changes the storage regime and therefore stops the use of the unsampled portions of the loaf of cheese for future measurements.

The following shall be avoided:

a) dehydration of the test sample during the period of thermal equilibration;

b) deformation of the test sample due to its own mass.

6.2 Test portion

6.2.1 Location

Take the test portion from the loaf of the cheese with a plug about half a radius, either along a circle of a cylindrical cheese, or along one side of a rectangular cheese (see Figure A.2).

Cut the test portion in the plug in the area representing around half of the length (see Figure A.3, plug A). If the height of cheese is sufficient, two portions can be taken as shown in Figure A.3, plug B and plug C.

6.2.2 Direction

The standard direction for taking the test portion is parallel to the pressure axis in cheese making. See Annex B for non-standard sampling conditions.

6.2.3 Geometry

The shape of the test portion shall be a cylinder with initial height/diameter ratio (h_0/d_0) of between 1,1 and 1,5.

The initial height, h_0, of the test portion shall range from 12,5 mm to 25 mm. The diameter, d_0, for a given height follows the above-mentioned ratio.

6.2.4 Cutting

Remove the rind or the plastic cover. Take a test portion using a cork-borer (4.1) with shapes shown in Figure A.4. For sticky cheeses, samples are easier to take with corer A than corer B. For cheese varieties showing shortness or brittleness, form B shown in Figure A.4 is more appropriate than form A. It is recommended to use a cork-borer mounted on a drill-stand in order to drive it slowly and steadily through the test sample.

If it is difficult to obtain a good cylindrical form, it is recommended to use mineral oil of low viscosity (e.g. Vaseline oil) to lubricate the cork-borer. Do not test samples with cracks, holes or other visible defects.

Use a parallel-wire cutting device to cut the test sample to the required height. The wire diameter shall be less than or equal to 0,4 mm. It is essential to have a system that keeps the two wires parallel to each other and perpendicular to the plug. Preferably, use a mechanically driven cutting system. Taking these precautions into account reduces the lack in parallelism between the sample surface and the compression plate.

6.2.5 Delay

A delay between the taking of a test portion and its testing allows stress relaxation of the test portion. The recommended delay is between 10 min and 15 min. The upper limit is not strictly fixed but it should not exceed 2 h. This recommendation is not relevant when sampling is done at a lower temperature than the measuring temperature.

Store the test samples at the measuring temperature (6.3.5) and see Annex B for non-standard conditions. Store samples in a pill-box or wrapped in plastic film to avoid dehydration during the delay between sampling and testing.

6.3 Test conditions

6.3.1 Relative deformation

Perform the compression to just beyond the apparent fracture point (Figure A.5, curve 1) or to a predefined maximum deformation (Figure 5, curve 2).

6.3.2 Crosshead speed

The standard value of the crosshead speed or displacement rate is 50 mm/min (or 0,83 mm/s) for initial height 12,5 mm $\leqslant h_0 \leqslant$ 25 mm.

6.3.3 Number of compression cycles

Perform one compression cycle.

6.3.4 Number of test portions

Measure at least four test portions, but preferably carry out more than this.

6.3.5 Measuring temperature

Measure at the standardized measuring temperature of 15 °C ± 1 °C.

NOTE Although the chosen test temperature of 15 °C is a good compromise for a single temperature, the challenge remains that many studies will use other temperatures for good reasons, as outlined in Annex B.

See Annex B for non-standard conditions.

6.3.6 Nature of the interface between test portion and plates

Use a low viscosity mineral oil as lubricant between the test portion and the plates. Apply the oil as a very thin layer on the plates.

7 Analysis of the compression curves

7.1 Data representation and calculation

7.1.1 Data representation

Raw data files contain data pairs (s_i, F_i) with displacement data, s_i, of the compression plate and force data, F_i, in units depending on the system. If the displacement of the plate is recorded right from the beginning of the test (i.e. before the plate is in contact with the sample) then compute the absolute deformation data, $|\Delta h_i|$, of the sample before any other calculation is performed. Let $|s_0|$ be the absolute displacement of the plate when the force becomes significantly different from zero (indicating the start of the compression of the sample). Then calculate the absolute sample deformation data, $|\Delta h_i|$, using the following equation:

$$|\Delta h_i| = |s_i| - |s_0| \tag{1}$$

where

$|s_0|$ is the absolute displacement of the plate when the force becomes significantly different from zero;

$|s_i|$ is the absolute displacement of the plate.

The correct sample deformation data, Δh_i, is then found using the following equation:

$$\Delta h_i = -|\Delta h_i| \tag{2}$$

For further processing, it may be useful to remove data from the free precontact displacement of the compression plate.

If the measuring system automatically records the absolute deformation $|\Delta h|$ of the sample (starting with zero as soon as the contact force becomes significant), then $|s_i|$ is equal to $|\Delta h_i|$ and no correction has to be applied. However, the sign assignment $\Delta h_i = -|\Delta h_i|$ may still be necessary.

The deformation/force data (Δh, F) must be transformed to the normalized measures strain and stress in order to be comparable. Graphical representations, numerical values and computed parameters of compression curves are given in terms of strain and stress. Both of the following representations to evaluate the curves are recommended options. Either one or both of these options may be used:

a) engineering stress, σ_u, versus Cauchy strain, ε_C;

b) corrected stress, σ_c, versus Hencky strain, ε_H

Strain has no dimension; stress is given in pascals (Pa) or kilopascals (kPa).

Examples of compression curves from some cheese varieties are given in Annex C.

NOTE σ_u is also known as uncorrected stress; ε_C is the engineering strain or relative deformation. The terms ‘true stress’ and ‘true strain’ to denote corrected stress σ_c, and Hencky strain ε_H, respectively, are not recommended.

Stress correction is based on the cylindrical shape and volume constancy of the sample during the test, allowing the calculation of the cross section, A_t, at each time point.

This changing cross section, A_t, is calculated using the following equation:

$$A_t = A(\Delta h) = \frac{A_0 \cdot h_0}{h(t)} \tag{3}$$

where

h_0 is the initial sample height;

A_0 is the initial cross section;

$h(t)$ is the height at each time point is derived from h_0 and the deformation Δh, $h(t) = h_0 + \Delta h$. Under compression, Δh is a negative quantity ($\Delta h \leqslant 0$) because the deformation reduces the height of the sample, thus $h(t) = h_0 - |\Delta h|$ (see Reference [4]).

7.1.2 Calculation of stress and strain

The following equations assume that $\Delta h \leqslant 0$.

The transformations of force into stress and of deformation into strain are applied to all data points (Δh, F).

$$\sigma_\mathrm{u} = \frac{F_t}{A_0} \tag{4}$$

$$\varepsilon_\mathrm{C} = \frac{\Delta h}{h_0} \tag{5}$$

$$\sigma_\mathrm{C} = \frac{F_t}{A_t} = \frac{F_t}{A_0} \cdot \frac{h_t}{h_0} = \frac{F_t}{A_0} \cdot \left(1 + \varepsilon_\mathrm{C}\right) = \sigma_\mathrm{u} \cdot \left(1 + \varepsilon_\mathrm{C}\right) \tag{6}$$

$$\varepsilon_\mathrm{H} = \ln\left(\frac{h_t}{h_0}\right) = \ln\left(\frac{h_0 + \Delta h}{h_0}\right) = \ln\left(1 + \varepsilon_\mathrm{C}\right) \tag{7}$$

NOTE 1 According to Equations (5) and (6), compressive strain is a negative quantity (see Reference [4]). Although rheologically correct, it is not common practice to indicate the minus sign in graphical representations or in the computed parameters of the compression curves. The use of the sign may be useful if results from compression ($\varepsilon < 0$) and tension ($\varepsilon > 0$) need to be distinguished.

NOTE 2 The application of stress correction according to Equation (6) (see Reference [5]) induces a shift of the apparent fracture point towards lower absolute strain values (apart from decreasing the numerical stress values). This shift is a transformation property of the correction factor ($1 + \varepsilon_\mathrm{C}$), applied to curves with local maxima (see Reference [7]).

7.2 Parameters characterizing the compression curves

7.2.1 General

Descriptive mechanical parameters of the compression curves are evaluated from stress/strain data calculated according to Equations (4) to (7), respectively. The parameters characterize the first part of the curves and that part where fracture occurs. The apparent fracture work characterizes the curve up to the apparent fracture point.