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Wholemeal and flour from wheat (*Triticum aestivum* L.) — Determination of rheological behaviour as a function of mixing and temperature increase

*Farine et mouture complète de blé tendre (*Triticum aestivum* L.) — Détermination du comportement rhéologique en fonction du pétrissage et de l'augmentation de la température*

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 17718 was prepared by Technical Committee ISO/TC 34, *Food products*, Subcommittee SC 4, *Cereals and pulses*.

Introduction

The behaviour of dough is dependent on numerous parameters. Some of these parameters, such as water absorption, dough development time, and kneading stability, are linked to the quality and quantity of the proteins, while other parameters, such as gelatinization, gelling stability, and retrogradation, are linked to the properties of the starch.

The Mixolab®[1)] measures the torque between two mixing arms during kneading while varying the in-bowl temperature, making it possible to gain in-depth information on samples and thus gain a better understanding of the characteristics of tested wheat or flours.

Wholemeal and flour from wheat (*Triticum aestivum* L.) — Determination of rheological behaviour as a function of mixing and temperature increase

1 Scope

This International Standard specifies the determination of rheological behaviour as a function of mixing and temperature increase. It is applicable to all wholemeal and flour samples from industrial or laboratory milling of wheat (*Triticum aestivum* L.).

NOTE Wheat can be milled in the laboratory according to the methods described in ISO 27971[5] or in BIPEA guidance document BY.102.D.9302.[7]

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 712, *Cereals and cereal products — Determination of moisture content — Reference method*

3 Terms and definitions

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

3.1
water absorption
volume of water required to obtain a dough with a maximum consistency of (1,10 ± 0,05) Nm

Note 1 to entry: Water absorption is expressed in millilitres per 100 g of flour with a moisture content of 14 % mass fraction.

3.2
time T1
time required for the dough to reach the target consistency C1 of (1,10 ± 0,05) Nm

Note 1 to entry: Development time is expressed in minutes.

3.3
stability
calculated time during which the dough achieves a consistency higher than C1 – 11 % × C1

4 Principle

Dough behaviour is determined as it is subjected to a combined kneading and temperature treatment during a constant temperature phase, followed by a heating phase, then held at high temperature, and subsequently cooled. Water is added to flour to achieve a maximum dough consistency of (1,10 ± 0,05) Nm during the first constant temperature phase.

The dough is first kneaded between two mixing arms rotating in opposite directions at 80 r/min, at a starting temperature of 30 °C. The torque that the dough creates between the two mixing arms is recorded. Kneading then continues while temperature is increased to 90 °C at a rate of 4 °C/min. The temperature is then held at a controlled 90 °C for 15 min. The dough mixer is then cooled at a rate of 4 °C/min to a temperature of 50 °C.

Dough consistency, like temperature, is recorded throughout the test. The results provide information on gluten strength, starch gelatinization and retrogradation, enzyme activity, and all the interactions taking place between dough components throughout the process.

5 Reagent

Use only distilled or demineralized water or water of equivalent purity.

6 Equipment

Usual laboratory apparatus and, in particular, the following.

6.1 Chopin Mixolab®,[1)] including the following components.

6.1.1 Drive motor, capable of delivering a mixing arm rotation speed of 80 r/min.

6.1.2 Water tank, containing water (Clause 5) thermostatically maintained at 30 °C.

6.1.3 Dough mixer, comprising a bowl, two hub-flanges, and two detachable mixing arms.

6.1.4 Removable cover, for positioning the water injection nozzle.

6.1.5 Water injection nozzle, fitted with four delivery channels.

6.1.6 Software, for programming the test conditions and measuring and recording the test results.

6.2 Laboratory scale, capable of weighing to ±0,1 g.

6.3 Laboratory mill, hammermill model,[2)]

fitted with a 0,8 mm mesh sieve and able to provide a wholemeal flour of specified homogeneous particle size.

Mill performance should be re-checked at regular intervals on a sample of milled grain. The milled sample shall meet the specifications given in Table 1.

Table 1

Sieve mesh size	Proportion of wheat wholemeal passing through the sieve
µm	%
710	100
500	95 to 100
210 to 200	≤80

1) Chopin Mixolab® is the trade name of a product supplied by Chopin Technologies. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

2) LM 3100 and LM 120 mills are examples of suitable products available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of these products.

7 Sampling

Sampling is not part of the method specified in this International Standard. A recommended sampling method is given in ISO 24333.[4]

It is important the laboratory receive a truly represeantative sample which has not been damaged or changed during transport or storage.

8 Procedure

8.1 General

To implement this International Standard on wholemeal flour requires a milling step.

If the sample is delivered as flour, go to 8.3.

8.2 Sample milling

Grind (6.3) 200 g to 300 g of cleaned grains as specified in the mill manufacturer's instructions.

8.3 Determination of flour moisture content

Determine the moisture content of flour or milled sample as specified in ISO 712.

8.4 Preparation of the device

Make sure that the water-injection nozzle is set above the water tank.

Power up the Mixolab®1) (6.1) 30 min ahead of the first test.

Use the level gauge to check there is a sufficient quantity of water (Clause 5) in the tank (6.1.2).

Make sure the bowl, hub flange, and mixing arms assembly is well connected (6.1.3), and install this dough mixer assembly.

Close the removable cover (6.1.4).

8.5 Preparation of the test

8.5.1 General

There are two steps:

a) determination of the water absorption of flour or wholemeal;

b) determination of the rheological characteristics of the flour or wholemeal.

8.5.2 Determination of water absorption

8.5.2.1 Use the program menu (6.1.6) to define the protocol to perform test: Chopin+ for flour and Chopin Wheat+ for wholemeal.

8.5.2.2 Use the program menu (6.1.6) to set the test conditions, specifying flour moisture content (8.3) and setting a 55 % hydration (the baseline 14 % is selected automatically).

8.5.2.3 Use the laboratory scale (6.2) to weigh out the quantity of flour stipulated by the device (6.1.6).

8.5.2.4 Boot the test, and introduce the weighed flour into the dough-mixer (6.1.3).

8.5.2.5 Position the water injection nozzle (6.1.5) in its place above the cover. It is important to wait for the software (6.1.6) prompt before positioning the nozzle.

8.5.2.6 When the curve has reached its peak and a downcurve sign is indicated, stop the test and record the C_{max} read-out value.

If the maximum torque, C_{max}, reaches (1,10 ± 0,05) Nm, allow the curve complete development for 45 min.

8.5.2.7 Remove the injection nozzle from the removable cover (6.1.4) and position it above the water tank (6.1.2).

8.5.2.8 Remove the dough-mixer bowl and mixing arms (6.1.3), and clean.

8.5.2.9 Re-assemble the dough-mixer and reposition it on the Mixolab®.1)

8.5.3 Full test

8.5.3.1 Use the program menu (6.1.6) to define the protocol to perform test: Chopin+ for flour and Chopin Wheat+ for wholemeal.

8.5.3.2 Use the program menu (6.1.6) to set the test conditions, specifying the hydration figure used in the previous test (8.5.2), the sample moisture content determined in 8.3, and the C_{max} torque obtained in the previous test (8.5.2).

8.5.3.3 Use the laboratory scale (6.2) to weigh out the quantity of flour stipulated by the device (6.1.6).

8.5.3.4 Boot the test, and introduce the weighed flour into the dough-mixer (6.1.3).

8.5.3.5 Position the water-injection nozzle (6.1.5) in its place above the cover. It is important to wait for the software (6.1.6) prompt before positioning the nozzle.

8.5.3.6 Monitor the dough behaviour during the first few minutes of the test.

If the maximum torque, C_{max}, reaches (1,10 ± 0,05) Nm, allow the curve complete development for 45 min, if C_{max} does not reach (1,10 ± 0,05) Nm, stop the test, record the C_{max} read-out value, and go back to 8.5.2.6.

8.5.3.7 Remove the injection nozzle from the removable cover (6.1.4) and position it above the water tank (6.1.2).

8.5.3.8 Remove the dough-mixer bowl and mixing arms (6.1.3), and clean.

8.5.4 Parameter recordings

8.5.4.1 Measuring points for water absorption

C1 is the first maximum consistency of the dough and corresponds to the target consistency that needs to be achieved. It shall be in the range 1,05 Nm to 1,15 Nm.

8.5.4.2 Parameters giving information on dough behaviour

C2 is the minimum consistency obtained during curve development and corresponds to the lowest point on the curve after C1, when the temperature applied to the bowl is on the increase.

C3 is the maximum consistency obtained after starch gelatinization and corresponds to the peak consistency value obtained after C2, or consistency value at the end of 90 °C phase if there is no peak. C3 is related to starch granules swelling under the effect of the heat.

C4 is the minimum consistency obtained after starch gelatinization and corresponds to the drop in consistency after C3 during the kneading period at 90 °C. This drop can only be calculated if it reaches 89 % of the C3 value.

C5 is the final consistency of the dough and corresponds to the dough consistency at the end of the test, after the cooling phase.

Stability corresponds to the calculated time during the dough maintains a consistency higher than C1 − 11 % × C1.

Time T1 which corresponds to the time needed to reach torque C1.

Temperatures D1, D2, D3, D4, and D5, which correspond to an estimation of dough temperatures at test-point characteristics C1, C2, C3, C4, and C5, respectively.

9 Expression of results

Express the results for torque values C1, C2, C3, C4, and C5 to the nearest 0,01 Nm.

Express stability, Ts, and time T1 to the nearest 0,01 min.

Record dough temperature estimations (D1, D2, D3, D4, and D5) to the nearest 0,1 °C.

See Figures A.1 and A.2.

10 Precision

10.1 Interlaboratory tests

See Annex B. The values derived from this interlaboratory test campaign may not be applicable to other Mixolab®1) parameters, concentration ranges or wholemeals and flours from grains other than wheat (*Triticum aestivum* L.).

10.2 Repeatability limits, *r*

The repeatability limit is the value below which the absolute difference between two single test results obtained under repeatability conditions may be expected to lie with a probability of 95 %.

Water absorption, μ_{H_2O}: $r = 0,29 \times 2,8 = 0,8$

C2: $r = 0,013 \times 2,8 = 0,04$

C3: $r = 0,019 \times 2,8 = 0,05$

C4: $r = 0,029 \times 2,8 = 0,08$

C5: $r = 0,078 \times 2,8 = 0,22$

Stability, Ts: r = (– 0,090 2 × Ts + 1,276 2) ×2,8

Time T1: r = (0,081 4 × T1 + 0,125 2) × 2,8

D1: r = 0,567 × 2,8 = 1,6

D2: r = 0,651 × 2,8 = 1,8

D3: r = 0,781 × 2,8 = 2,2

D4: r = 0,767 × 2,8 = 2,1

D5: r = 0,741 × 2,8 = 2,1

10.3 Reproducibility limits, R

The reproducibility limit is the value below which the absolute difference between two test results obtained under reproducibility conditions may be expected to lie with a probability of 95 %.

Water absorption, μ_{H_2O} : R = 0,75 × 2,8 = 2,1

C2: R = 0,027 × 2,8 = 0,08

C3: R = 0,076 × 2,8 = 0,21

C4: R = 0,090 × 2,8 = 0,25

C5: R = 0,190 × 2,8 = 0,53

Stability, Ts: R = (–0,151 3 × Ts + 2,201 4) × 2,8

Time T1: R = (0,171 6 × T1 + 0,114 7) × 2,8

D1: R = 0,970 × 2,8 = 2,7

D2: R = 1,585 × 2,8 = 4,4

D3: R = 1,691 × 2,8 = 4,7

D4: R = (– 0,3798 × D4 + 33,649) × 2,8

D5: R = 2,724 × 2,8 = 7,6

10.4 Critical difference, d_C

10.4.1 General

The critical difference is the deviation between two values obtained from two test results under repeatability conditions.

10.4.2 Comparison of two measurement groups in the same laboratory

The critical difference for comparing two average values obtained from two test results in the same laboratory, under repeatability conditions is given by:

$$d_{C,r} = 2{,}8\, s_r \sqrt{\frac{1}{2n_1} + \frac{1}{2n_2}} = 2{,}8\, s_r \sqrt{\frac{1}{2}} = 1{,}98\, s_r$$

where

s_r is the repeatability standard deviation;

n_1, n_2 are the number of test results for each of the average values — here, n_1 and n_2 equal 2.

10.4.3 Comparison of two measurement groups in two different laboratories

The critical difference for comparing two values obtained from two test results in two different laboratories, under repeatability conditions is given by:

$$d_{C,R} = 2{,}8 \sqrt{s_R^2 - s_r^2 \left(1 - \frac{1}{2n_1} - \frac{1}{2n_2}\right)} = 2{,}8 \sqrt{s_R^2 - 0{,}5\, s_r^2}$$

where

s_r is the repeatability standard deviation;

s_R is the reproducibility standard deviation;

n_1, n_2 are the number of test results for each of the average values — here, n_1 and n_2 equal 2.

10.5 Uncertainty, u

Uncertainty, u, is a parameter characterizing the dispersion of values that may reasonably be attributed to the result. The uncertainty figure is established based on a statistical distribution of results derived from the interlaboratory test, and is characterized by the experimental standard deviation.

For each parameter, uncertainty can be considered as $\pm 2s_R$, where s_R is the standard deviation of reproducibility stated in this International Standard.

11 Test report

The test report shall contain at least the following information:

a) all information necessary for the complete identification of the sample;

b) the sampling method used, if known;

c) the test method used, with reference to this International Standard (ISO 17718:2013);