

Results of an international ring test for the determination of the rheological properties of wheat flour dough using the Haubelt Flourgraph E 7 (ICC standard no. 180)

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RESEARCH ARTICLE

Abstract

Measuring the tensile properties of dough is one of the most important techniques used to assess the quality of flours used for bread making. The introduction of ICC standard 180 Haubelt Flourgraph E 7 has the aim of introducing new equipment using a universal technique to quantify the values of measured variables that would characterise rheometric elements as: dough formation, properties of dough formation and its visco-elastic properties. The aim of this multinational collaborative study is to measure the performance of this equipment for the validation of the draft standard. The ring test for E 7 was organised and performed under the responsibility of Haubelt Laborgeräte GmbH. Ten laboratories participated in the ring, performing the test method on 5 flours of different rheological properties in addition to one sample investigated in duplicate (blind). Results were collected by the Haubelt company and the data forwarded to ICC's technical director for statistical evaluation of accuracy (trueness and precision) of measurement for energy, resistance to extension and extensibility at the intervals of 45, 90, and 135 minutes of resting time according to the requirements of ISO 5725 part 1, 2 and 6. The relationship between standard deviations and mean values can sufficiently be described by a linear regression. This means that for repeatability and reproducibility no fixed value can be derived. Calculation of the average repeatability and reproducibility as percentage of the mean may help to summarise the results of this ring test in a simple and condensed manner.

Keywords: dough tensile properties, dough quality, rheology, wheat flour

1. Introduction

In 1930 one of the first special instruments was designed for physical testing of wheat flour doughs, the so-called Brabender Extensograph (Bloksma and Bushuk, 1988; Kahraman *et al.*, 2008). All of the above mentioned equipment have been internationally validated as reference ICC standards since more than four decades (1972), and since basic rheological instruments are capable of providing the essential, or fundamental details of the material's rheological properties, ICC continuously struggles to study, validate and publish new standard methods for such equipment as the Haubelt Flourgraph E 7. In the aim of sustaining the development of apparatuses that helps in

improving the quality of bread and baked products by providing a comprehensive knowledge of dough rheology by delivering accurate measurement of the rheological data.

Thus, using the Flourgraph E 7, we can characterise the rheological properties of wheat flour dough which is essential to produce information concerning the quality of the raw material and the textural characteristics of the finished product.

Wheat dough is a unique material formed when wheat flour is mixed with water creating a viscoelastic dough that retains gas (Walker and Hazelton, 1996). Elongational flow is thought to be the predominant type of flow occurring in the dough surrounding the inflating gas bubbles during fermentation and baking (Van Vliet *et al.*, 1992, 1993). For this reason the Flourgraph E 7 works on the principle of uniaxial elongation or 'constant-strain extension test' which impose high strain levels to the dough via a travelling hook, deforming the sample until it is physically broken.

This procedure allows the measurement of specific rheological properties of wheat dough until the time of dough rupture, and the data is expressed as energy (cm²), maximum resistance to stretching and extensibility (E) expressed in arbitrary units, Haubelt units (HE).

The determination of the repeatability (r) and reproducibility (R) of the measured parameters is crucial because the quantification of the elongational properties of wheat flour dough in a reliable manner is an important step for the determination of the functionality of flours, and this step is recognised to be central to the successful manufacturing of bakery products. The method is applicable to dough made of bread wheat (*Triticum aestivum* L.) flour.

The aim of this study, performed by 10 laboratories on 5 samples of wheat flour under the responsibility of Haubelt Laborgeräte GmbH and the supervision of ICC's technical director, is to establish the accuracy of the results of the rheological parameters gathered through Haubelt Flourgraph E 7: energy, resistance to stretching at constant deformation and dough extensibility. Statistical results have been achieved according to ISO 5725-1, 5725-2 and 5725-6, respectively.

2. Materials and methods

Materials

The wheat flour material made available consisted of 5 samples of different baking performance with energetic values varying from 48.4 to 172.9 cm², to represent as much as possible the whole range encountered in practical application to bread wheat flour. 6 flour samples in total have been investigated each individual sample has been measured in duplicate. Flours no. 3 and 6 are the same (duplicate). Of the 5 samples, 2 flours are of type 550 from Germany and 3 flours from a Haubelt Cie partner in Turkey.

Ten laboratories participated in the ring test, 5 thereof being from Turkey, 2 from Germany and 1 from Austria, Bulgaria and Cyprus, respectively (Table 1). Thus the requirements of ISO, ICC and IUPAC for an international ring test are fulfilled.

Table 1. The laboratories participating in the ring test.

Company	Country	
Vatan	Turkey	
Detmold	Germany	
Boku	Austria	
IGV	Germany	
Alimet	Bulgaria	
Yavuzlar	Cyprus	
Begasan	Turkey	
Tuncoglu	Turkey	
Kalyon	Turkey	
Bedir Un	Turkey	

Dough preparation

For exercising the Flourgraph E 7 method correctly it is necessary to produce a dough with defined consistency. This comprises determination of the optimum water level necessary for the development of a cohesive and viscoelastic dough with optimum gluten strength (Abang Zaidel *et al.*, 2008; Faubion and Hoseney, 1989).

To assure the formation of a dough that can be referred to as 'developed dough' (Campos *et al.*, 1996, 1997; Schluentz *et al.*, 2000), the dough was made with the Haubelt Flourgraph E 6 according to ICC standard no. 179. Moreover, in this study, the water absorption values for the 6 samples were determined at Haubelt and provided to the participants, thus avoiding the contribution of the Flourgraph E 6 as a potential source of variation between laboratories.

Determination of viscoelastic properties

Sampling, sample preparation, chemical composition of flour and physical properties of dough were determined according to:

- ICC standard no. 130. Sampling of milling products (semolina, flours, agglomerated flours and by-products); 1980.
- ICC standard no. 110/1. Determination of the moisture content of cereals and cereal products (practical method); 1976.
- ICC standard no. 179. Determination of water absorption capacity of wheat flours and wheat meals and physical properties of wheat dough using the Haubelt Flourgraph E 6; 2012.
- ISO 3696. Water for analytical laboratory use specification and test method; 1987.

The dough was assessed with the Flourgraph E 7 (Figure 1) according to ICC standard no.180. Dough of flour, water and salt is mixed according to ICC standard no. 179. A test



Figure 1. Haubelt Flourgraph E 7.

piece of the dough is moulded into a standard shape using the balling unit and moulder of the Flourgraph E 7. After a fixed period of resting time the dough is stretched while recording the force required. After the first stretching the procedure of moulding, resting and stretching is repeated twice corresponding to the resting periods of 45, 90 and 135 minutes. Using window based software the stretching curves are monitored via a computer screen (Figure 2) located directly near the device.

The following parameters were determined with a Flourgraph E 7 (Figure 2):

- Resistance to stretching at constant deformation (R50), which is defined as the height of the recorded curves after 50 mm stretching expressed in Haubelt units.
- Extensibility is the distance recorded on the graph and correlates to the distance travelled by the hook from the moment it touches the dough until breaking of (one of the strings of) the sample.
- Energy E(a) is defined as the area in cm² under the recorded curve.

Statistical analysis

Statistical results have been achieved according to ISO 5725-1, 5725-2 and 5725-6, respectively, and the statistical analysis of the data was prepared using SPSS predictive analytics software for windows version 14.0 (SPSS Inc. Chicago, IL, USA) in order to determine the mean value and standard deviations for repeatability and reproducibility.

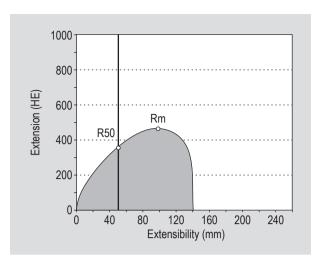


Figure 2. Dough parameters determined with a Haubelt Flourgraph E 7: resistance to stretching at 50 mm (R50); maximum resistance (Rm), energy needed to break the dough sample (grey area under the curve). HE = Haubelt unit.

3. Results and discussions

Flours 1 to 6 selected for this study have different stretching properties, regardless of their chemical composition, they were analysed in duplicate by the 10 participating laboratories and the results for the three main parameters (energy, R50 and extensibility) were statistically evaluated.

Energy

The energy E(a) is the energy needed to extend the dough till it reaches its breaking point. The estimated surface recorded under the curve is considered to be a measure of the stress placed upon the wheat flour dough.

The studied equipment expresses the energy as cm^2 . The mean value for the energy for the 6 samples after elimination of outliers varied after 45, 90 and 135 minutes from 39.1 to 150.8 cm^2 , 48.4 to 172.9 cm^2 and from 52.8 to 172.9 cm^2 , respectively. The statistical data of the three resting periods are detailed in Table 2, 3 and 4. and the standard deviation within each of the participating laboratories as well as between them is represented in Figure 3.

Resistance to stretching at constant deformation

R50, also known as resistance to extension, is conferred mainly to a wheat dough by its gluten content and depends specifically on the variation of the glutenin to gliadin ratio, as glutenin contributes to the elastic and gliadin to the viscous property of hydrated gluten (Janssen *et al.*, 1996; Khatkar *et al.*,1995). An increased level of glutenin increases the rupture viscosity but lowers the rupture strain (Uthayakumaran *et al.*, 2000) which leads to an increase in dough strength and resistance to extension.

Table 2. Precision data for the results of energy (cm²) on Flourgraph E 7 after 45 minutes.

	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5	Sample 6 ^a
L	10	10	10	10	8	10
Mean	150.8	75.0	106.2	86.8	39.1	103.2
s _r	14.0	4.5	5.0	4.6	1.7	7.5
RSD,	9.3%	6%	4.7%	5.3%	4.3%	7.3%
r	39.3	12.6	13.9	12.8	4.7	21.0
s_R	14.5	5.6	6.3	5.8	2.8	8.0
RSD _R	9.6%	7.4%	6%	6.7%	7.1%	7.7%
R	40.6	15.6	17.7	16.3	7.8	22.3

^a Sample 6 is identical to sample 3, i.e. is a blind sample.

Table 3. Precision data for the results of energy (cm²) on Flourgraph E 7 after 90 minutes.

	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5	Sample 6 ^a
L	10	10	10	10	10	9
Mean	172.9	102.8	110.4	90.1	48.4	115.7
S _r	10.4	6.3	8.2	6.1	2.7	6.0
RSD,	6%	6.1%	7.5%	6.8%	5.5%	5.2%
r	29.1	17.5	23.1	17.1	7.4	16.9
s_R	14.0	8.3	8.4	6.5	3.1	6.9
RSD _R	8.1%	8.1%	7.6%	7.2%	6.3%	6%
R	39.2	23.2	23.5	18.1	8.6	19.3

^a Sample 6 is identical to sample 3, i.e. is a blind sample.

Table 4. Precision data for the results of energy (cm²) on Flourgraph E 7after 135 minutes.

	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5	Sample 6 ^a
L	10	10	10	10	10	10
Mean	172.9	98.4	107.2	87.1	52.8	105.6
S _r	7.8	5.8	5.9	6.9	3.8	5.5
RSD,	4.5%	5.9%	5.5%	7.9%	7.3%	5.2%
r	21.8	16.1	16.6	19.2	10.7	15.5
s_R	9.2	5.9	5.9	6.9	3.8	6.4
RSD _R	5.3%	6%	5.5%	7.9%	7.3%	6.1%
R	25.6	16.4	16.6	19.2	10.7	18.0

^a Sample 6 is identical to sample 3, i.e. is a blind sample.

L = number of labs considered (according to Cochran and Grubbs test); s_r = standard deviation within the laboratory; RSD_r = relative standard deviation within labs; r = repeatability (r = 2.8 s_r); s_R = standard deviation between the different laboratories; RSD_R = relative SD between labs; R = reproducibility (R = 2.8 s_p)

L = number of labs considered (according to Cochran and Grubbs test); s_r = standard deviation within the laboratory; RSD_r = relative standard deviation within labs; r = repeatability (r = 2.8 s_r); s_R = standard deviation between the different laboratories; RSD_R = relative SD between labs; R = reproducibility (R = 2.8 s_R).

L = number of labs considered (according to Cochran and Grubbs test); s_r = standard deviation within the laboratory; RSD_r = relative standard deviation within labs; r = repeatability (r = 2.8 s_r); s_R = standard deviation between the different laboratories; RSD_R = relative SD between labs; R = reproducibility (R = 2.8 s_R).

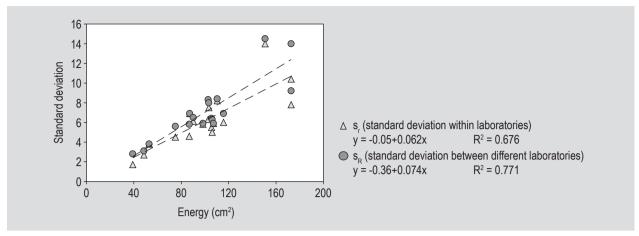


Figure 3. Standard deviation within and between laboratories for energy values after 45, 90 and 135 minutes.

The studied equipment expresses R50 in Haubelt units. The mean value for R50 for the 6 samples showed a good dispersal of results ranging from weak to strong wheat dough, and after elimination of outliers the values varied respectively after 45, 90 and 135 minutes from 111.3 to 526.9 HE, 155.0 to 828.9 HE and from 204.2 to 890.2 HE. The statistical data of the three resting periods are detailed in Table 5, 6 and 7. And the standard deviation within each of the participating laboratories as well as between them is represented in Figure 4.

Extensibility

Extensibility is the capacity of wheat dough to stretch under load, this rheological property is mainly conferred to the dough by the low molecular weight-glutenin subunits or gliadins which lowers the rupture viscosity but increases the rupture strain when present at an elevated level (Uthayakumaran *et al.*, 2000). It is well established that

gliadins acts like a plasticiser, promoting viscous behaviour and extensibility of gluten (Kuktaite, 2004).

The studied equipment expresses extensibility in mm. The mean value for extensibility for the 6 samples indicates various levels of viscosities ranging from low to highly extensible wheat dough, and after elimination of outliers the values varied respectively after 45, 90 and 135 minutes from 100.9 to 232.8 mm, 94.4 to 226.9 mm and from 89.5 to 226.4 mm. The statistical data of the three resting periods are detailed in Table 8, 9 and 10. and the standard deviation within each of the participating laboratories as well as between them is represented in Figure 5.

From the results shown in Figure 3, 4 and 5 the following can be deduced for the 3 parameters (energy, resistance to extension, extensibility):

• Generally the difference between s_R and s_r are small and in 17 to 22% of the results s_R is equal to s_r this is

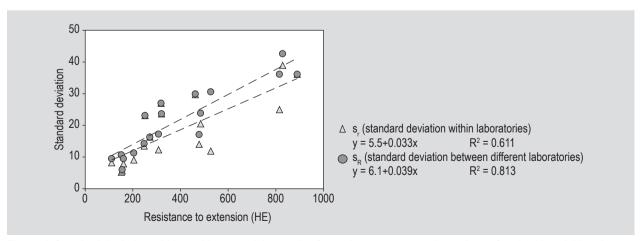


Figure 4. Standard deviation within and between laboratories for resistance to extension values after 45, 90 and 135 minutes. HE = Haubelt units.

Table 5. Precision data for the results of resistance to extension (HE) on Flourgraph E 7 after 45 minutes.

	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5	Sample 6 ^a
L	10	9	10	10	9	10
Mean	320.4	526.9	250.7	151.3	111.3	247.7
Sr	23.6	11.9	23.1	5.2	8.2	13.5
RSD,	7.4%	2.3%	9.2%	3.5%	7.4%	5.5%
r	66.0	33.4	64.7	14.6	23.1	37.8
s_R	23.6	30.6	23.1	10.7	9.5	14.2
RSD _R	7.4%	5.8%	9.2%	7.1%	8.6%	5.7%
R	66.2	85.7	64.7	30.1	26.7	39.8

^a Sample 6 is identical to sample 3, i.e. is a blind sample.

HE = Haubelt units; L = number of labs considered (according to Cochran and Grubbs test); s_r = standard deviation within the laboratory; RSD_r = relative standard deviation within labs; r = repeatability (r = 2.8 s_r); s_R = standard deviation between the different laboratories; RSD_R = relative SD between labs; R = reproducibility (R = 2.8 s_R).

Table 6. Precision data for the results of resistance to extension (HE) on Flourgraph E 7 after 90 minutes.

	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5	Sample 6 ^a
L	9	10	10	10	9	10
Mean	462.0	828.9	307.8	160.5	155.0	318.9
s _r	29.7	38.9	12.3	7.9	5.7	27.0
RSD,	6.4%	4.7%	4%	4.9%	3.7%	8.5%
r	83.1	108.9	34.5	22.0	16.1	75.7
s_R	29.9	42.6	17.2	9.5	6.0	27.0
RSD _R	6.5%	5.1%	5.6%	5.9%	3.9%	8.5%
R	83.8	119.2	48.3	26.6	16.8	75.7

^a Sample 6 is identical to sample 3, i.e. is a blind sample.

HE = Haubelt units; L = number of labs considered (according to Cochran and Grubbs test); s_r = standard deviation within the laboratory; RSD_r = relative standard deviation within labs; r = repeatability (r = 2.8 s_r); s_R = standard deviation between the different laboratories; RSD_R = relative SD between labs; R = reproducibility (R = 2.8 s_R).

Table 7. Precision data for the results of resistance to extension (HE) on Flourgraph E 7 after 135 minutes.

	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5	Sample 6 ^a
L	9	10	10	10	10	10
Mean	815.0	890.2	478.2	271.1	204.2	484.6
s _r	25.0	36.1	14.0	16.3	9.1	20.5
RSD,	3.1%	4.1%	2.9%	6%	4.5%	4.2%
r	70.0	100.9	39.1	45.7	25.5	57.3
s_R	36.1	36.1	17.1	16.3	11.3	23.8
RSD _R	4.4%	4.1%	3.6%	6%	5.5%	4.9%
R	101.2	100.9	47.8	45.7	31.7	66.7

^a Sample 6 is identical to sample 3, i.e. is a blind sample.

HE = Haubelt units; L = number of labs considered (according to Cochran and Grubbs test); sr = standard deviation within the laboratory; RSD_r = relative standard deviation within labs; r = repeatability (r = $2.8s_r$); s_R = standard deviation between the different laboratories; RSD_R = relative SD between labs; R = reproducibility (R = $2.8s_R$)

Table 8. Precision data for the extensibility results (mm) on Flourgraph E 7 after 45 minutes.

	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5	Sample 6 ^a
L	10	10	10	10	8	10
Mean	196.4	100.9	185.7	232.8	183.3	181.5
s _r	7.8	6.2	10.0	10.3	9.2	9.0
RSD,	3.9%	6.2%	5.4%	4.4%	5%	5%
r	21.7	17.5	28.1	29.0	25.8	25.3
s_R	8.9	7.1	10.7	10.6	9.7	10.0
RSD _R	4.5%	7.1%	5.7%	4.6%	5.3%	5.5%
R	25.0	19.9	29.9	29.7	27.1	28.0

^a Sample 6 is identical to sample 3, i.e. is a blind sample.

Table 9. Precision data for the extensibility results (mm) on Flourgraph E 7 after 90 minutes.

	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5	Sample 6 ^a
L	10	10	10	10	10	10
Mean	166.4	94.4	167.5	226.9	170.6	168.6
S _r	7.8	4.0	9.8	9.9	8.3	10.2
RSD,	4.7%	4.2%	5.8%	4.4%	4.8%	6.1%
r	21.8	11.1	27.4	27.8	23.1	28.6
s _R	9.8	6.1	10.0	10.5	10.9	10.2
RSD _R	5.9%	6.4%	6%	4.6%	6.4%	6.1%
R '`	27.4	17.0	28.1	29.5	30.5	28.6

^a Sample 6 is identical to sample 3, i.e. is a blind sample.

Table 10. Precision data for the extensibility results (mm) on Flourgraph E 7 after 135 minutes.

	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5	Sample 6 ^a
L	10	10	10	10	10	10
Mean	160.9	89.5	161.8	226.4	166.3	158.5
S _r	8.2	5.1	7.4	8.8	7.8	7.1
RSD,	5.1%	5.7%	4.6%	3.9%	4.7%	4.5%
r	23.1	14.2	20.8	24.8	21.9	19.9
s_R	8.7	5.1	7.9	9.7	8.9	7.1
RSD _R	5.4%	5.7%	4.9%	4.3%	5.3%	4.5%
R	24.4	14.2	22.1	27.1	24.8	19.9

^a Sample 6 is identical to sample 3, i.e. is a blind sample.

L = number of labs considered (according to Cochran and Grubbs test); s_r = standard deviation within the laboratory; RSD_r = relative standard deviation within labs; r = repeatability (r = 2.8 s_r); s_R = standard deviation between the different laboratories; RSD_R = relative SD between labs; R = reproducibility (R = 2.8 s_p).

L = number of labs considered (according to Cochran and Grubbs test); s_r = standard deviation within the laboratory; RSD_r = relative standard deviation within labs; r = repeatability (r = 2.8 s_r); s_R = standard deviation between the different laboratories; RSD_R = relative SD between labs; R = reproducibility (R = 2.8 s_R).

L = number of labs considered (according to Cochran and Grubbs test); s_r = standard deviation within the laboratory; RSD_r = relative standard deviation within labs; r = repeatability (r = 2.8s_r); s_R = standard deviation between the different laboratories; RSD_R = relative SD between labs; R = reproducibility (R = 2.8s_R).

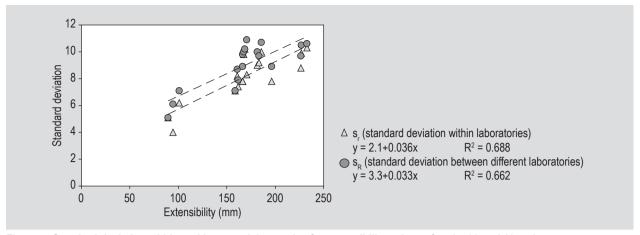


Figure 5. Standard deviation within and between laboratories for extensibility values after 45, 90 and 135 minutes.

due to the fact that 'water absorption' was fixed for the purpose of this study, avoiding one source of variation between laboratories and reducing standard deviation of reproducibility.

- The relationship between standard deviations (s_r, s_R) and mean values can sufficiently be described by a linear regression. This means that for repeatability (r) and reproducibility (R) no fixed value can be derived.
- Calculation of the measurement uncertainty at the 95% confidence level may help to summarise the results of this ring test in a simple and condensed manner, giving users an idea of the uncertainty generated by this equipment without considering the contribution of 'water absorption' to the uncertainty budgeting, as resumed in Table 11.

The uncertainty of measurement (expressed in %) was calculated from the relative standard deviation of reproducibility:

$$U = k \times RSD_R$$

Where U is uncertainty and k is coverage factor = 2.

Table 11. Uncertainty data (%) related to the measurements of energy, resistance to extension and extensibility values after 45, 90 and 135 minutes of resting.

	45 minutes	90 minutes	135 minutes
Energy	19%	16%	16%
Resistance to extension	18%	17%	12%
Extensibility	14%	13%	11%

4. Conclusions

The Haubelt Flourgraph E 7 uncertainty values for the three measured parameters (Table 11) are quite satisfactory when compared to similar other instruments that works on empirical methods using the same 'uniaxial elongation' principle. However, 20% of the statistical data showed similar values for repeatability and reproducibility do to the fact that 'water absorption' values were fixed in this study, artificially eliminating one source of variation between participating laboratories (reproducibility standard deviation) which also leads to an under-estimation of the uncertainty of measurement. A revision of the method based on a re-validation study might be required when a larger diversity of users may give a more realistic picture than the currently very limited prevalence of application of the method. For this reason a note was added at the end of ICC standard no. 180 citing:

In order to support the reliability of this ICC standard, a usual revision of the standard and its underlying data and figures will be performed after an adequate minimum time of the standard being in practical use (2 years), according to ICC rules and guidelines for standardisation.

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