SENSORIAL AND PHYSICAL-CHEMICAL ANALYSIS OF A "000" WHEAT FLOUR TYPE OBTAINED INTO A MID CAPACITY MILL

ANALIZA SENZORIALĂ ŞI FIZICO-CHIMICĂ A FĂINII DE GRÂU TIP "000" PRODUSĂ ÎNTR-O MOARĂ DE CAPACITATE MEDIE

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Abstract. *The aim of the paper was to effectuate a sensorial and physical-chemical analyse for a "000" wheat flour type which was obtained into a mid capacity mill. Sensorial analyse of wheat flour targeted on appreciation of colour, taste, smell as well as on flour infestation degree. From physical-chemical analysis view point, the aims of the current paper were focused on the following parameters: moisture, wet gluten, ash, acidity, granulosity and falling index.*
Key words: "000" wheat flour type, sensorial characteristics, physical-chemical properties.

Rezumat. *Scopul prezentei lucrări este de a efectua o analiză senzorială şi fizico-chimică pentru făina de grâu de tip "000" care este produsă într-o moară cu o capacitate medie. Analizele senzoriale au vizat aprecierea culorii, gustului, mirosului precum şi gradul de infestare a făinii. Din punct de vedere a analizelor fizico-chimice lucrarea de faţă s-a axat pe studierea următorilor parametri: umiditate, conţinutul de gluten umed, conţinutul de cenuşă, aciditate, granulozitate şi indicele de cădere.*
Cuvinte cheie: făină de grâu tip "000", caracteristici senzoriale, proprietăți fizico-chimice

INTRODUCTION

From the ancient times cereals represented one of the main food source of mankind, fact which lead to interweaving of their processing history with mankind history and thus with material cultural history of humanity (Zaharia, 2011).

During development of civilisation and human society, cereal production as well as the specific technologies for their processing known a permanent improvement and were always adapted to food demands and needs of mankind (Avarvarei, 1999).

Cereals belong to category of agricultural plants which are cultivated for seed obtaining. In Romania, the main cultivated cereal plants are: wheat, corn, rye, barley, oat, millet, sorghum.

Due to the fact that cereal seeds have a high content in starch are also called

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starchy agricultural products. Cereal seeds have in their composition sugars, proteins, vitamins, mineral salts and fats, substances which are very necessary both for human nourishment as well as for animal's one (Mogârzan and Robu, 2005; Roman *et al.,* 2011).

Having in view the role and importance of wheat in human nourishment we could affirm that in parallel with evolution of human civilisation took place an evolution, respectively an improvement of its processing technologies.

The obtaining of wheat flour was at beginning a household occupation, in the way that each family had the necessary tools for its grinding. Some of the most primitive tools for flour obtaining were grindstones (fig. 1) (Alban and Chester, 2005).

Grinding of cereals using grindstones was realized by their rubbing between a larger stone, which have a plain surface and a smaller stone which was hand moved.

Fig. 1 Ancient grindstones (Alban and Chester, 2005)

a b

Fig. 2 Roman grindstone a) manual drive; b) animal drive

In ancient Egypt were founded hand carved stones which were formed from a stone provided with a central hollow, where cereals were introduced and grinded using an elongated stone, with repeated moves of arms.

Grinding of cereals in ancient Rome, was effectuated with stone mills (fig. 2), where milling process was realized by their friction between a mobile stone and a fixed one (Alban and Chester, 2005).

Initially stone drive was manually realized, using arms' force through some handles.

Then it was a gradually passed to driving of those mills by using animal power, water power as well as by using wind power (Alban and Chester, 2005).

In milling industry the technological flow for wheat flour have the following stages: reception (quantitative, qualitative) – storage – cleaning of impurities – conditioning – peeling – grinding – sifting – storage for maturation – packing – storage – delivery.

The aim of the paper was to effectuate a sensorial and physical-chemical analyse for a "000" wheat flour type which was obtained into a mid capacity mill.

Sensorial analyse of wheat flour targeted on appreciation of colour, taste, smell as well as on flour infestation degree.

From physical-chemical analysis view point, the aims of the current paper were focused on the following parameters: moisture, wet gluten, ash, acidity, granulosity and falling index.

MATERIAL AND METHOD

Sensorial characteristics of wheat flour were determined in according with Romanian national standard SR EN ISO 90:2007 (Sensorial characteristics of wheat flour).

Determination of flours' colour is based on comparison of analysed sample's colour with colour of some well established flour benchmarks. It is a quality index which is often used in practice for establishing, with some relativity, of extraction degree for certain flour. Particles which come from endosperm have a white-yellowish colour, due to yellow pigments (xanthophylls and its esters, carotene and oxidation products resulted due to chlorophyll cleaving). Parts which came from coating have a dark colour, due to flavonic pigments. By grinding a part of yellow pigments are eliminated together with bran and the most of it remain in endosperm particles which form the flour, thus determining its colour (Bordei, 2004).

To establish the colour of flour will be weighted around 50 grams of flour, which will be stretched into a rectangular layer with dimensions of 4 x 5 cm, on a wooden shovel; the layer's thickness must be around 0.5 cm. Near this layer will be stretched an equal quantity of benchmark flour, this layer must have the same dimensions. Flour will be pressed using a glass plate, and after pressing bran particles as well as other particles which could be contained by flour, will be obviously observed at surface. After that the shovel with samples, will be introduced in water into a slightly oblique position. Flour layers will be compared in dry state as well as in wet state. Moistening is realised in the following way: shovel with pressed flour sample is introduced slightly oblique into a recipient with cold water, maintaining duration is around 1 minute. The shovel with wet flour it will come out of the water and will be placed from drying, at room temperature for 5-10 minutes. After that will be examined the analysed samples in comparison with standard sample, both in diffuse light and also in direct light. During observations shovel must be placed in such a way in which the light fascicle to be perpendicular on its surface. Analyse is realised in wet state because by wetting colours became more evident and the presence of bran could be better observed.

For *determination of flavour* into a Berzelius glass will be introduced a quantity of 5 grams of flour sample and after that will be added 25 cm^3 of distillate water which was previously heated at 60-65°C. Content will be homogenized for around 1 minute, and after that glass will be covered with a laboratory watch glass and will be placed for resting for about 4-5 minutes. After that the watch glass is removed and the formed suspension will be smelled.

Flour must have a pleasant smell specific to wheat. Flours which are not suitable will present unpleasant smells. If flour presents a honey flavour this means that flour was contaminated by mites, and the presence of altered fish odour is the proof that during milling process were also grinded seeds which were attacked by blight (Mogârzan and Robu, 2005). Another way for determination of flour flavour is to take in hand a quantity at around 5 grams from flour sample and after was slightly rubbed with the other hand, it is smelled.

For *taste determination* will be taken 1 gram from analyzed flour sample, will be chewed, appreciating in this way the taste and the possible presence of some mineral impurities (soil, sand) due to characteristic noise which those one produce during mastication process. Normally flour has a sweetish taste, pleasant, characteristic to wheat. The presence of strange tastes could be due to unsuitable storage conditions or flour's infestation. Altered flour, due to rancid fats, has a bitter taste (Banu *et al.*, 1998).

Determination of infestation is based on sifting of a flour sample (0.5 kg) using a silk sieve or a number 4XX synthetic fibre fabric sieve; the remained residuum will be examined with a 5x magnifying glass, for observing the possible presence of insects or mites (live, dead or part of them). This determination is realised in according with Romanian standard demands SR ISO 6639-1/2/3/4 (Cereals and leguminous. Determination of hidden infestation with insects).

Infestation with mites could be also checked by:

- strong honey flavour of flour;
- crumbling after around one hour of a cone realised from 100 flour grams (built through a conic funnel);
- presence of some characteristic tracks on plain surface of flour (Mogârzan and Robu, 2005).

The realised physical-chemical analyses were: moisture, wet gluten, ash, acidity, granulosity and falling index.

Determination of moisture (M%) was realised by drying at oven, in according with the demands impose by standard SR EN ISO 712:2010 (Cereals and products from cereals. Moisture determination. Reference method).

Method is based on determination of mass loosening due to heating into an oven at a temperature of 130±2°C, for 60 minutes. Into a weighting ampoule, with low form and lid, which was previously heated at 130±2°C, will weighted a quantity of 5 flour grams, with a 0.001 g precision. Ampoule with flour will be placed (with the lid near it) into an oven which was previously heated at 130±2°C. It will be kept into oven at 130±2°C, for 60 minutes. Ampoule will be extracted from oven, will be covered with the lid and will be placed, for chilling till temperature of environment, into desiccators with anhydride $CaCl_2$. After chilling (30-60 minutes), ampoule will be weighted with the same 0.001 precision. It will be realised two subjected to analysis determinations for the same sample.

Moisture (%) will be calculated using formula [1] and the result will be expressed with one decimal:

$$M\ (\%) = [(m_1-m_2)/(m_1-m)] \times 100 \quad [1]$$

in which:

m is mass of empty ampoule (g);

m_1 is mass of ampoule with analyzed flour sample, before drying (g);

m_2 is mass of ampoule with analyzed flour sample, after drying (g).

As final result, will be used the arithmetic mean of those two determinations, if repeatability conditions are fulfilled. That means that the differences between two determinations effectuated in parallel, in the same laboratory, by the same operator and from the same sample aren't allowed to exceed 0.3%.

Determination of wet gluten was realized in according with Romanian standard SR EN ISO 21415-1/2/3/4/2007 (Wheat and wheat flour. Gluten content).

Protein substances under the form of gluten are separated, due to washing of batter prepared from flour sample with a NaCl solution and airing of obtained gluten.

Into porcelain mortar will be placed 25 sample grams, which were weighted with a precision of 0.01 g. It will be added 12.5 cm^3 of NaCl solution and after that, by using a pestle, will be kneaded for 3-4 minutes, till will be obtained homogenous dough. The obtained dough will be washed just after knead, manually or mechanically, with a NaCl solution, above a silk sieve.

Wet gluten content (%) will be calculated using formula [2] and the result will be expressed with one decimal.

$$\text{Wet gluten}\ (\%) = (m_1/m) \times 100 \quad [2]$$

in which:
m is mass of analyzed flour sample (g);
m_1 is mass of gluten which remained after drying (g).

As final result, will be used the arithmetic mean of those two determinations, if repeatability conditions are fulfilled. That means that the differences between two determinations effectuated in parallel, in the same laboratory, by the same operator and from the same sample aren't allowed to exceed 2% (which means 2 grams of wet gluten for 100 grams of sample).

Determination of ash was effectuated in conformity with Romanian standard SR EN ISO 2171:2010 (Determination of ash from cereals and from products obtained from it).

The standard stipulates that total mineral substances represent the residuum which is obtained after sample calcinations at a temperature of 525±25°C till a constant weight is obtained. So, into clean and dried porcelain melting pot which was previously calibrated will be weighted, using an analytical balance, 5 grams from analyzed product. The moisture from oven will be eliminated by its adjustment at a temperature of 125°C, after that being subjected to burning till carbonization at the flame of a gas burner for 10-15 minutes. At the end of carbonization operation, melting pots will be introduced, using some pliers with long arms, into calcinations oven which was adjusted at a temperature of 525±25°C where will be kept uninterrupted for 16-18 hours. Calcinations operation will be repeated by 1-2 exposures in oven for around 1 hour, till a constant weight if reached.

Ash content (%) will be calculated in according with formula [3].

$$\text{Ash (\%)} = (m_1/m) \times 100 \quad [3]$$

where:
m is quantity of analyzed product, calculated as difference between mass of melting pot with sample before drying and its calibration (g);
m_1 is ash quantity, (g), which is calculated as being the difference between mass of melting pot with ash and mass of empty pot.

Determination of falling index was realized in conformity with standard SR EN ISO 3093:2010 (Wheat, rye and flours, durum wheat and durum wheat flour. Determination of Hagberg–Perten falling index).

This index measures indirectly the amylase activity, due to fast gelling of an aqueous suspension of integral grist from wheat of flour, into a boiling water bath as well as by measurement of liquefaction produced by α-amylase to starch gel which contained into analyzed sample. Hagberg–Perten falling index is expressed in seconds and optimal values are in interval 220-280 seconds. Values over 280 seconds indicate that flours have a low amylase activity and the ones under 220 seconds show that flours have an intense amylase activity.

Determination of flour's acidity was realized in concordance with the demands imposed by standard SR 877-1996 (Wheat flour. Determination of flour's acidity).

Was utilized the method of suspension in water, so into an Erlenmeyer balloon are placed 5 grams of flour weighted with a precision of 0.01 g. Is added 50 cm^3 distillate water and the content is homogenized for 5-10 minutes, avoiding formation of lumps. At the end of homogenization will be added 3 drops of phenolphthalein and will be titrated with NaOH 0.1 n till apparition of a pink colour, which must persist for one minute. Will be realized two determinations for the same sample which was subjected to analysis.

Commonly acidity is expressed in acidity degrees, 1 acidity degree representing acidity for 100 g product, which will be neutralized by 1 cm^3 NaOH 0.1 n solution.

Acidity (degrees) will be calculated by using formula [4] and the result will be expressed with one decimal.

$$\text{Acidity (degrees)} = [(V \times 0.1)/m] \times 100 \quad [4]$$

where:
V is volume of NaOH 0.1 n solution used at titration (cm^3);
0.1 is normality of sodium hydroxide solution;
m is mass of analyzed sample (g).

As final result, will be utilised the arithmetic mean of those two determinations, if repeatability conditions are fulfilled. This refers that difference between two determinations effectuated in parallel, in the same laboratory, by the same operator and from the same sample aren't allowed to exceed 0.2 acidity degrees.

Determination of flour's granulosity, have as principle the sifting of flour through a specific sieve for the type which will be analyzed whereupon is weighted the residuum on the rarer sieve and what's going on the dense sieve.

It will be weighted with a precision of 0.01 g, 100 grams from analyzed sample that will sift through sieve, manually or mechanically. If it is used a manually sifting its duration will be 6 minutes, with application of 80-100 movements come and go/minute. For realizing an intensification of sifting, together with flour sample, on sieve will be placed rubber balls or rings, which will be removed at sifting finalization. In case in which the analyzed sample present a moisture above 16% these one will be placed on a sheet of paper into a thin layer whereupon will be let for drying for 2-3 hours, at environment temperature, till moisture will decrease under the value of 15%, after which will proceed to sifting. Separate will be weighted, with a precision of 0.01 g, residuum remained on rarer sieve, obtaining directly the final result.

RESULTS AND DISCUSSIONS

Sensorial appreciation of wheat flour

Sensorial appreciation of wheat flour presumed effectuation of the following analysis: determination of flour's colour, taste, smell and its infestation degree.

It was utilised method of sensorial qualities appreciation through score method. Analyze was effectuated by a team of 6 specialists whom at the end of tasting give ratings for each of those 4 analyzed properties (colour, taste, smell, infestation degree).

Scoring scale was from 0 to 4; in case of flour's colour, taste and smell mark 0 was minimum and mark 4 was represented the maximum value which a sensorial feature was able to obtain. Regarding infestation degree, mark 4 was attributed to the flour which wasn't infested while mark 0 is given to a very infested flour by mites and which is unsuitable in the process of obtaining bakery, pastry and confectionery products.

In table 1 are presented the results of sensorial analysis at the end of examination of "000" wheat flour type which was obtained into a mid capacity mill.

The maximum score which could be obtained by "000" wheat flour type for each of those four analyzed features was 24 points and the minimum score which could be achieved was 0 points (tab. 1).

Table 1

Sensorial analysis of "000" wheat flour type

No.	Colour (points)	Taste (points)	Smell (points)	Infestation degree (points)	TOTAL (points)
Specialist 1	4	3	3	4	14
Specialist 2	4	3	3	4	14
Specialist 3	4	3	4	4	15
Specialist 4	4	4	4	4	16
Specialist 5	4	4	4	4	16
Specialist 6	4	4	4	4	16
TOTAL	24	21	22	24	

As we can observe from table 1 regarding colour, assortment "000" wheat flour recorded a total score of 24 points which means that specialists gave maximum marks for this characteristic.

Referring to the taste of "000" wheat flour type (tab. 1) we remark that the total score was 21, three from specialists noting this characteristic with 3 points while 3 of them gave the maximum score of 4 points.

Analyzing the obtained score by "000" wheat flour type regarding smell (tab. 1) could be observed that two specialists gave the mark 3, while the other four members of tasting team gave the maximum score of 4 points. Totally for smell feature "000" wheat flour type achieved a number of 22 points.

Regarding infestation degree of "000" wheat flour type (tab. 1) could be observed that this one had a score of 24 points, which means that none of those specialists didn't determine that analyzed product was infested by mites, all of them giving the maximum rating.

From analyse of data presented in table 1 could be observed that the total score given by those six specialists was situated in interval 14 points – 16 points (which is the maxim value which could be reached by "000" wheat flour type). Two of the team members evaluated globally the sensorial characteristics of studied product with a number of 14 points, one gave 15 points for the studied product, and three specialists evaluated the sensorial characteristics with maximum total score of 16 points.

A possible explication of those small fluctuations in appreciation of sensorial features of studied product could be that each specialist has its own sensations regarding taste and smell.

Physical-chemical appreciation of wheat flour

For a physical-chemical appreciation of "000" wheat flour type was analyzed the following parameters: moisture, wet gluten content, ash content, falling index (Hagberg-Perten), acidity, granulosity. The obtained results at the end of laboratory determinations were compared with the values imposed by national standard (tab. 2).

Determination of moisture

Regarding moisture of "000" wheat flour type was obtained a mean value,

for this feature, of 14.29±0.028%. For moisture the minimum obtained value was 14.2% while the maximum founded value was 14.4%. Studied parameter had slightly low values than value of 14.5% which is imposed by national standard (tab. 2). Variation coefficient which was calculated had a value of 0.613% which shown a very good homogeneity for the studied character (tab. 2).

Table 2

Physical-chemical appreciation of wheat flour

Physical-chemical characteristic	Standard	Results (n=10)			
		Minimum	Maximum	$\overline{X} \pm s_{\overline{X}}$	V (%)
Moisture (%)	14.5	14.2	14.4	14.29±0.028	0.613
Wet gluten (%)	min. 30	30.1	30.5	30.29±0.038	0.395
Ash (%)	max. 0.48	0.45	0.47	0.46±0.003	1.775
Falling index (sec.)	220-280	256	262	259.2±0.573	0.700
Acidity (grade)	2.2	2.0	2.1	2.09±0.010	1.513
Granulosity (%)	8	7.6	7.9	7.78±0.33	1.328

Determination of wet gluten content

Regarding content of wet gluten from "000" wheat flour type, could be observed that obtained values oscillated between a minimum value of 30.1% and a maximum value of 30.5%. Mean value obtained for wet gluten content was 30.29±0.038%, in conditions in which national standard impose a minimum value of 30% for wet gluten (tab. 2). Variation coefficient, for this feature, had the value of 0.395% fact which shown that also this studied character had a very good homogeneity inside lot (tab. 2).

Determination of ash content

"000" wheat flour type had a mean ash content of 0.46±0.003%, in conditions in which minimum was situated at a value of 0.45% and maximum was 0.47% (tab. 2). For ash content national standard impose a maximum value of 0.48%, so we could affirm that studied product "000" wheat flour type meets the imposed requirements. For this feature, variation coefficient, recorded the value of 1.775% (tab. 2) which allow us to affirm that studied parameter presented a very good homogeneity inside lot.

Determination of Hagberg-Perten falling index

Analyzing Hagberg-Perten falling index for "000" wheat flour type, could be observe that obtained values oscillated in interval 256 seconds (minimum value) and 262 seconds (maximum value). National standard impose that this parameter to fit into interval 220 seconds and 280 seconds, from which result that mean value (259.2±0.573 seconds) obtained by us is in according with those demands (tab. 2). Variation coefficient (0.700%) obtained for Hagberg-Perten falling index shows the fact that inside lot homogeneity was very good (tab. 2).

Determination of acidity

Regarding acidity of "000" wheat flour type could be observed that was obtained a mean value of 2.09±0.010 acidity degrees in conditions in which

standard provides a value of maximum 2.2 acidity degrees (tab. 2). The obtained values at the end of effectuated experiments were situated into interval between 2.0 acidity degrees (minimum value) and 2.1 acidity degrees (maximum value). Variability coefficient obtained a value of 1.513%, so this fact allows us to affirm that inside studied lot homogeneity for this parameter was very good (tab. 2).

Determination of granulosity

Granulosity or finesse of "000" wheat flour type, recorded a minimum value of 7.6% while maximum value was 7.9%. Mean value obtained for this parameter was 7.78±0.33% being slightly inferior to standard which impose a value of 8%. For variability coefficient was obtained a value of 1.328%. This fact allows us to say that inside studied lot also the homogeneity for this parameter was a very good one (tab. 2).

CONCLUSIONS

Based on sensorial and physical-chemical analysis effectuated on "000" wheat flour type a series of conclusions could be drawn.

1. Regarding colour "000" wheat flour type assortment recorded a total score of 24 points which means that specialist gave maximum marks for this feature.

2. Regarding taste of "000" wheat flour type it is observed that total score was 21 points, three from specialists noting this characteristic with 3 points, while other 3 of them gave the maximum score of 4 points.

3. Regarding smell of "000" wheat flour type, this parameter accumulated a number of 22 points. Two specialists according mark 3, while the other four members of tasting team accorded maxim score of 4 points.

4. Regarding infestation degree of "000" wheat flour type it could be observed that this one had a score of 24 points, which means that all specialists gave the maximum mark.

5. Total score given by those six specialists was situated in interval 14 points – 16 points (which is the maxim value which could be reached by "000" wheat flour type). Two of the team members evaluated globally the sensorial characteristics of studied product with a number of 14 points, one gave 15 points for the studied product, and three specialists evaluated the sensorial characteristics with maximum total score of 16 points.

6. So we could affirm that from sensorial point of view "000" wheat flour type is in according with the demands imposed by the nowadays legislation.

7. Moisture of "000" wheat flour type had a mean value of 14.29±0.028%. Minimum obtained value was 14.2% while founded maximum value was 14.4%. Variation coefficient had a value of 0.613% which shown a very good homogeneity for the studied character.

8. Wet gluten content from "000" wheat flour type recorded values which oscillated between a minimum value of 30.1% and a maximum value of 30.5%. Mean value obtained for wet gluten content was 30.29±0.038%; variation

coefficient, for this feature, had the value of 0.395% fact which shown that also this studied character had a very good homogeneity inside lot.

9. "000" wheat flour type, had a mean ash content of 0.46±0.003%, in conditions in which minimum was situated at a value of 0.45% and maximum was 0.47%. Variation coefficient, recorded the value of 1.775%, so studied parameter presented a very good homogeneity inside lot.

10. Hagberg-Perten falling index for "000" wheat flour type, had values which oscillated into interval 256 seconds (minimum value) and 262 seconds (maximum value). Variation coefficient (0.700%) obtained for Hagberg-Perten falling index shows the fact that inside lot homogeneity was very good.

11. Acidity of "000" wheat flour type recorded a mean value of 2.09±0.010 acidity degrees in conditions in which standard provides a value of maximum 2.2 acidity degrees. The obtained values were situated into interval between 2.0 acidity degrees (minimum value) and 2.1 acidity degrees (maximum value). For variability coefficient was obtained a value of 1.513%, so homogeneity for this parameter was a very good one.

12. Granulosity of "000" wheat flour type, recorded a minimum value of 7.6% while maximum value was 7.9%; mean value obtained for this parameter was 7.78±0.33%. For variability coefficient was obtained a value of 1.328% fact which show us that inside studied lot homogeneity for this parameter was a very good one.

13. We consider that physical-chemical speaking "000" wheat flour type fulfils all the demands imposed by nowadays legislation.

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