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Original scientific paper

CHANGES OF THE OPTICAL PROPERTIES OF TOP-GRADE FLOUR (SEMOLINA) FROM DURUM WHEAT DURING ITS RIPENING

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KEY WORDS:
durum wheat flour, optical properties, color characteristics, semolina ripening, carotenoids, traceability, group a pasta, digital image

ABSTRACT

Using the rapid method of digital image analysis by the developed scanning flour analyzer the optical properties of flour (semolina) obtained in laboratory and production conditions from durum wheat of three years of harvest (2017–2019), namely the «yellowness» indicator and the color characteristic in the blue part of the spectrum, were determined. The semolina color was also evaluated by the Konica Minolta CR-410 colorimeter. It is established that the «yellowness» indicator and the color characteristic in the blue part of the spectrum did not change within the first 5 to 6 days after grinding. The change of these indicators for all the samples is observed in the period from 6 to 20 days after grinding, there with the «yellowness» indicator decreased by 25 to 40 relative units, the color characteristic in the blue part of the spectrum increased on average by 133,75 relative units. Over the next three months, there was no change of color (by both indicators). In the course of experimental work the optical properties change depending upon carotenoids content of flour (semolina) during its ripening was shown. The correlation dependence between the «yellowness» indicator of flour (semolina) and its carotenoids content is characterized by a high approximation coefficient. The dependence of the color characteristic of flour in the blue part of the spectrum on the content of carotenoids is characterized by an approximation coefficient equal to 0.9358, and is described as a polynomial equation. It shows, that with a low carotenoid content, the considered indicator is higher by an average of 1100 relative units compared to the color of samples with a carotenoid content from 0.70 to 1.21 mcg/g. At that during storage the optical properties of flour variety with the lowest carotenoids content remained practically the same. During 78 days of storage, there was no significant change of color characteristics of the industrial flour samples, studied from the eleventh day after grinding — 5–8 times higher than the average repeatability of the measurement results.

1. Introduction

The requirements for the design and implementation of a traceability system in the feed and food production chain are regulated by GOST R ISO 22005–2009 [1], which came into force in January 2011. The quality and safety of food products [2] is one of the main tasks of implementing a traceability system. Standards for implementing the system in the production of certain types of food products, have already been developed, for example, in the chain of confectionery, fish production. At KorolevPharm LLC, the introduction of traceability contributes not only to the release of high quality biological food additives, but also to their safety. One of the goals of implementing traceability in this company is to meet customer requirements.

The development and implementation of a traceability system is relevant in the production of group A pasta, as it meets the priority areas for the development of science in terms of the introduction of digital control methods. It is aimed at the production of high-quality pasta that meets the concept of state policy in the field of healthy nutrition of the population, and the exclusion of counterfeit products. The insufficient volume of durum wheat production and its price [3,4,5] is the reason for the falsification.

The main requirement for creating a traceability system is the ability to receive data quickly, accurately throughout the supply chain. The express method of product quality control, which is based on digital technologies, will provide a solution of the problem of supplying the population with healthy food products.

In order to maintain the quality of group A pasta that meets the requirements of consumers and to exclude adulterated products, an instrumental method of monitoring durum wheat flour for the presence of soft wheat flour impurities by the optical properties of flour has been developed. The method is based on obtaining optical characteristics from the results of mathematical analysis of a digital image of the studied flour and comparing them with the optical characteristics of standards with a fixed content of soft wheat flour. A patent was received for the method [6,7,8]. Analysis time is 3 to 4 minutes. There are no analogues [9,10,11]. A method for analyzing the image of durum wheat grain (Triticum durum) without destroying its structure was described in [11]. Used to assess the morphological properties of grain.

As a result of studies conducted in 2018 [12] to determine the feasibility and effectiveness of introducing optical properties (color characteristics) determined by the digital image method, when evaluating products by individual elements of the traceability chain in the production of group A pasta in three regions of Russia, it was found: a confounding factor for the introduction of an effective system for assessing the quality of group A pasta by its optical properties is the process of semolina ripening, affecting the change in its quality by its optical properties — by the «yellowness» indicator and by the color characteristic in the blue part of the spectrum.

The goal is to study the change in the optical properties of flour (semolina) obtained during laboratory grinding of durum wheat, as well as produced during industrial production, in the process of its ripening. According to the literature, it is known that during the ripening of flour during storage, its color becomes lighter [13]. The reason for the color change of the flour is the oxidation of the carotenoids contained in it. Carotenoids are the substances painted in yellow or orange color that belong to the «pigments» group [14,15]. These pigments oxidize with a large amount of oxygen and transform into oxidized, colorless forms. According to the literature [15, 16], the duration of flour ripening depends on storage conditions, on the quality of the grain itself. The ripening

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of flour occurs the faster, the longer the period between harvesting and grinding of grain is. According to L. Ya. Auerman [17], wheat flour at a temperature of (20 ± 5) °C ripens within 1.5–2.0 months.

2. Objects and methods

The object of study is flour (semolina) obtained in laboratory conditions during the grinding of durum wheat grain of three years of harvest (2017–2019), as well as during production grinding (2018). Durum wheat flour (semolina) for pasta obtained during laboratory grinding, according to physico-chemical parameters, meets the requirements for the characteristics of durum wheat flour, regulated by the standard. The grindings were carried out on durum wheat, the quality indicators of which are presented in Table 1. According to the main quality indicators, the grain met the requirements established by the standards.

Optical properties (color characteristics) of flour (semolina) — the «yellowness» index calculated from the basic colors and the color characteristic in the blue part of the spectrum are determined from the digital image of the flour. The measurements were carried out on an experimental sample of a scanning analyzer (CAM), designed to obtain a digital image of the studied sample [6,7,8]. A standard flatbed scanner of the Epson Perfection type with a CCD type sensor was used as the main unit. The experimental sample was developed at the VNIIZ in conjunction with the Scientific Research Center «Intelligent Scanning Systems».

The preparation of flour samples for measuring it on the CAM was carried out according to a special method using an original design cuvette. The measurement is carried out in this way: a cuvette with flour is mounted on a template — a latch placed on the surface of the scanner's exposure glass, a digital image is created and then is transmitted to the computer. A digital image of the flour is processed using the created special software (SSW) for calculating color characteristics.

The determination of the main indicators of the quality of grain and semolina, as well as the content of carotenoid pigments of semolina, was carried out according to the methods regulated by the standards. Laboratory grinding of durum wheat grain was carried out at the VNIIZ laboratory stand according to an extensive technological scheme in accordance with the «Rules for the organization and conduct of the technological process».

3. Results and discussion

As mentioned above, the duration of the ripening process depends on many factors: this is the period between harvesting and grinding of grain, the content of carotenoids, storage conditions, grain quality. These factors served as the basis for planning and conducting an experiment to study the influence of ripening of top-grade flour (semolina) on its optical properties.

The patterns of the optical properties change (the «yellowness» index and the color index in the blue part of the spectrum) of durum wheat flour (semolina) obtained by laboratory grinding of the 1st class durum wheat grain, harvest of 2017, carried out in 2018, are shown in Figure 1 and Figure 2.

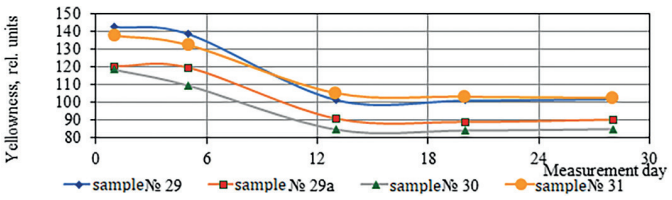

Figure 1. Change of the «yellowness» indicator of flour (semolina) in the process of ripening

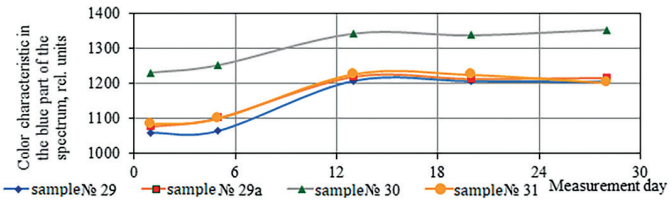

Figure 2. Change of the color characteristics in the blue part of the spectrum during the ripening of flour (semolina)

Analysis of the curves showed:

- the color during the first 5–6 days remains practically unchanged (indicators within the measurement error);
- in the next 6 to 12 days, a sharp decrease of the «yellowness» indicator is observed for all analyzed samples i. e. the color has become less yellow. The change of color characteristic in

Table 1

Quality indicators of durum wheat

Quality indicators	Durum wheat grain of the crop							
	2017				2019			
	Sample Number				Sample Number			
	29	29a	30	31	1z	2z	6z	7z
Vitreousness %	80	98	98	99	96	97	86	97
Test weight, g/l	820	806	833	826	818	817	831	800
Moisture, %	11.4	10.2	11.6	10.8	9.5	11.5	10.8	11.0
Gluten quantity, %	27.0	31.0	30.0	28.0	25.7	29.8	28.2	30.0
Gluten quality, IDK, group	82	72, I good	75, I good	75, I good	63, I good	68, I good	76, I good	91, II satisfactory weak
Protein, %	14,9	15,9	14,2	14,7	—	—	—	—
Falling-number value, s	269	422	345	363	244	256	437	344
Ash content, %	1.71	1.81	1.49	1.68	1.80	1.84	1.75	1.70
Grain admixture, %	3.9	1.67	1.14	2.8	1.80 including 0.48 — barley 0.74 — beaten 0.66 — germinating	1.79 including 0.68 — barley 1,11 — beaten	2.40 including 0.06 — barley 2.20 — beaten 0,14 — rye	2.34 including 0.22 — barley 2.12 — beaten
Weed admixture, %	0.9	0.15	0.02	0.1	0.80	0.30	0.10	0.10
Grain with a darkened germ, %	—	—	—	—	8.7	3.7	1.2	1.3

the blue part of the spectrum for all 4 samples was an average of 133.75 rel. units — it increased, i. e. it is the very characteristic by which the enlightenment of samples, the transition of carotenoid pigments to oxidized colorless forms can be detected;

- for three months of further observation, a color change (according to both characteristics) is within the established measurement errors.

It was these time frames that served as a guide for the experiment in 2019 on top-grade flour (semolina), formed from grains of the first and second quality grades obtained by laboratory grinding of grains, the characteristics of which are presented in Table 1. Color characteristics of top-grade flour (semolina) are given in Table 2.

Table 2

Color characteristics of the top-grade flour (semolina) obtained by laboratory grinding of grain from commercial batches of durum wheat

Product name	Sample No.	Yield, %	Ash content, %	Color characteristics	
				B	Y
Top-grade flour (semolina)	M — 1z — 19	63.2	0.79	1210	112.48
	M — 2z — 19	57.0	0.78	2368	92.11
	M — 6z — 19	61.1	0.71	1257	114.93
	M — 7z — 19	59.3	0.83	1237	122.57

The semolina obtained by grinding of the grain from sample № 2z at the lowest yield among the studied samples of semolina has the lowest «yellowness» indicator. Analyzing the data in the table, we see that the flour is different in yield and ash content, despite the fact that durum wheat grain is almost the same in terms of quality (vitreousness, test weight), and the process of grain samples grinding is carried out according to one scheme.

For the proper analysis of the ripening process for each grind of durum wheat, the top-grade flour (semolina) with a yield of 60% was formed. The formation of flour varieties (semolina) with a yield of 60% and the measurement of color characteristics by digital flour image analysis was carried out on the 6th day after grinding of each grain sample. The carotenoids content of flour (semolina) was determined on the 10th day. Carotenoid pigments contained in flour (semolina) impart to pasta the desired amber-yellow color. That is why the research of individual breeding institutes is devoted to the selection of durum wheat for a high content of carotenoid pigments in grain [19, 20].

The dependence of the «yellowness» indicator of flour (semolina) on its carotenoids content is presented in Figure 3.

The interrelation between the «yellowness» indicator of top-grade flour (semolina) with a yield of 60% and the content of carotenoid pigments in it is characterized by a high approximation coefficient.

After 10 days of storage, both color and pigment content were measured on the same day. The results of the experiment are presented in Table 3.

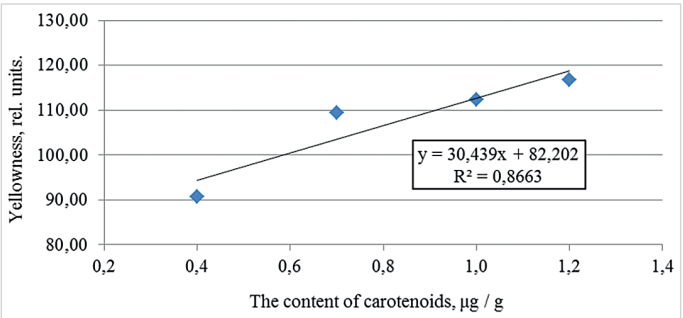

Figure 3. The «yellowness» indicator of top-grade flour (semolina), depending on the content of carotenoids

The table shows that:

- the carotenoid content of the studied samples decreased on average by 0.14 µg / g. (with repeatability of the measurement results — 0.03 µg / g);
- the color characteristics of the flour (semolina) grade with the lowest carotenoid content during storage remained almost the same (within the repeatability range);
- the decrease of the «yellowness» indicator for 10 days of storage is insignifican and lies in the range from 7.5 to 10.8 relative units;
- an increase in the value of the color characteristic in the blue part of the spectrum for samples with a carotenoid content of 1.0 to 1.2 µg / g averages 38.50 relative units.

The monitoring of the change in color characteristics of flour samples (semolina) shown in table 3 was continued for another 4 months until October 21. The «yellowness» indicator of these samples was determined monthly. It was found that for the samples of flour (semolina) obtained from 1z and 2z grain no further color change was observed, and a significant change was noted in terms of «yellowness» indicator for the M- 7z-19 and M — 6z — 19 samples — it reached a final value of 74.28 rel. units, and 83.33 rel. units respectively. Thus, during the summer storage time of semolina samples — M — 6z-19 and M — 7z –19, the carotenoid content of which was 0.9 to 1.0 µg / g, the ripening process continued and the change of the «yellowness» indicator was 18.30 rel. units and 33.60 rel. units from the values reached on 06.27.2019 respectively for semolina obtained from 6z and 7z grain.

In 2018, an additional experiment was conducted to identify changes in the color characteristics of top-grade flour (semolina) provided to us by individual enterprises with an exact date of flour production. Samples of flour were received on day 10 after production. By the presence of soft wheat flour, all 4 samples met the requirements regulated by GOST 9353–2016, according to which up to 15% of soft wheat is allowed at durum wheat processing. Using an instrumental determination method (patent), 10% of soft wheat flour was determined in the samples (based on comparison with flour standards with different soft wheat contents). The studied durum wheat flour (semolina) for pasta in all respects meets the requirements for the characteristics of durum wheat flour, regulated by the standard (Table 4).

Table 3

Change of color characteristics and carotenoid content of top-grade flour (semolina) during its ripening

Product name	Sample No.	The content of carotenoids, µg / g			Color characteristics, rel. units					
		18.06	27.06	difference	14.06.2019		27.06.2019		difference	
					B	Y	B	Y	B	Y
Flour (semolina)	M-1z –19	0.70	0.60	minus 0.10	1210	109.44	1225	101.95	15	minus 7.49
	M-2z –19	0.41	0.38	minus 0.03	2328	90.74	2332	89.91	4	minus 0.83
	M- 6z –19	1.00	0.87	minus 0.13	1266	112.37	1299	101.63	33	minus10.74
	M-7z-19	1.20	0.99	minus 0.21	1248	116.71	1292	107.81	44	minus 8.90

Table 4

Physico-chemical characteristics of durum wheat flour for pasta

Sample number	Humidity, %	Crude gluten content, %	Gluten quality, units IDK	Drop number, s	Ash content, %	Fineness, %	
						Residue on sieve No. 12.5	Passage through sieve No. 24.
2kr — 18	12.6	30.7	71	492	0.83	–	37.0
3kr — 18	12.6	28.5	58	435	0.71	–	30.4
18kr — 18	13.0	29.1	69	426	0.86	–	42.4
19kr — 18	13.7	29.0	69	443	0.82	–	38.2

On the 11th day after production all 4 samples were checked for color characteristics. It was found that 3 samples from the studied did not meet the standards for color (at least 47.0 rel. units) developed by VNIIZ [21] for top-grade flour (semolina) of industrial grinding. The calculation is based on the interrelation between the readings of the CR-410 colorimeter and the scanning flour analyzer, which we use to obtain a digital image of the flour, described by the equation y = 3.378x-55.78, where y is the «yellowness» indicator for the scanning analyzer, x — for CR-410 [21]. At present, some Russian enterprises producing flour from durum wheat use devices of foreign manufacture — the CR-410 Konica Minolta colorimeter — for the flour color evaluation. The colorimeter is manufactured by Konica Minolta Sensing, a Japanese company that is a leader in the development and manufacture of a precision measuring equipment for the color determination and control. In 2008 the CR-400, CR-410 colorimeters were included in the State register of measuring equipment of the Russian Federation (Registration No. 0 5 Э 5 ~ 0 9). When using the CR-410 Konica Minolta colorimeter user-defined formulas entering is provided for evaluating and calculating the color of any object. For the purpose of evaluating the color of flour from durum wheat for pasta production, flour manufacturers were suggested to determine this color by the «yellowness» indicator. For this indicator on the colorimeter VNIIZ has developed the color standards (project) for top-grade flour (semolina) obtained from the durum wheat grain (durum), and intended for pasta production.

The results of changes of the optical properties (color characteristics) of flour produced at flour mills for three months are shown in Table 5.

Statistical processing of the results presented in table 4 showed: the color characteristics of flour (semolina) for 78 days of storage is insignificant for both the «yellowness» indicator and the color characteristic in the blue part of the spectrum. The maximum variation value is 5 times higher than the average repeatability value of the measurement results (7.5) in the blue part, and 8 times higher than the average repeatability value of the measurements results for the «yellowness» indicator, which is 1.13 rel. units. The results of an experiment conducted on industrial flour samples, the color characteristics of which were measured only on the 11th day after production, confirmed that flour ripening affected the color in the first 6–12 days (Figures 1, 2).

4. Conclusions

As a result of studies conducted to reveal the influence of the ripening process on the optical properties of top-grade flour (semolina) obtained in laboratory and industrial conditions from the durum wheat grain of various quality of three years of harvest the following data were collected:

- experimental data on the physicochemical properties of the durum wheat grain the analysis of which showed correspondence of the studied grain to the requirements regulated by the standard;
- experimental data on the optical properties («yellowness» indicator and color characteristic in the blue part of the spectrum) of the formed varieties of top-grade flour (semolina) with the yield of 60% and also of the industrial flour, that were determined by the rapid method of digital image analysis using the developed scanning analyzer and by the Konica Minolta CR-410 colorimeter;
- experimental data on the carotenoid pigments content of the formed varieties of top-grade flour (semolina) with the yield of 60%;
- patterns of the flour (semolina) optical properties change depending on its ripening process.

As a result of the obtained data analysis the following conclusions can be drawn:

The change of the optical properties of top-grade flour (semolina) obtained by grinding the grain of the 2017 harvest starts on the 6th day and lasts till the 20th day. There was no change in the next three months;

During the simulation of the flour ripening process conducted with the formed varieties of top-grade flour (semolina) with the yield of 60% obtained by laboratory grinding of the 2019 harvest grain it was found:

- dependencies between the flour (semolina) color characteristics and its carotenoids content are characterized by high approximation coefficients — with that in terms of «yellowness» indicator the dependence is linear and in terms of color characteristic in the blue part of the spectrum it is described by polynomial equation;
- after 10 days of storage the color characteristics of flour (semolina) with the lowest carotenoids content remained practically the same;
- after 10 days of storage the magnitude of the carotenoids content decrease in the studied samples in 5 times exceeded the repeatability of the results of carotenoids content determination;
- monitoring of the color characteristics change of the studied samples with the carotenoids content of 0,9–1,0 μg/g for four months detected that the ripening process had been lasting;
- the color characteristics change of the industrial flour samples during 78 days of storage starting from the 11th day after grinding is insignificant — 5–8 times higher than the average repeatability of the measurement results.

Table 5

Color characteristics of flour (semolina) of industrial production

Date of measurement	Sample No. 2kr-18		Sample No. 18 kr-18		Sample No. 19 kr-18		Sample No. 3kr –18	
	In the blue part of the spectrum (B)	Yellowness (Y)	In the blue part of the spectrum (B)	Yellowness (Y)	In the blue part of the spectrum (B)	Yellowness (Y)	In the blue part of the spectrum (B)	Yellowness (Y)
13.06.18	1347	90.84	1190	94.11	1197	92.63	1314	105.65
05.09.18	1366	85.42	1210	86.24	1236	83.15	1333	99.51
Change in readings for the entire period	19	minus 5.42	20	minus 7.86	39	minus 9.45	19	minus 6.14

The optical properties change of top-grade flour (semolina) from durum wheat during the ripening process significantly depends on the initial grain quality, including the carotenoids content, the year of grain harvest, the color of the flour, i. e. on the duration of the flour ripening process itself.

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