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JUSTIFICATION OF SAFE SHELF LIFE OF WHOLE WHEAT FLOUR OF VARIOUS SIZES, DEPENDING ON THE PROCESSING METHOD

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The objects of this study were wheat grain (Shortandinskaya variety, harvest 2021) and wheat grain grinding products – whole grain (whole-ground) wheat flour of coarse, medium, and fine grinds.

Studies have been carried out to establish the terms of safe storage of whole-grain (whole-ground) wheat flour of various sizes. Samples of whole-grain flour of coarse, medium, and fine grinding were obtained by grinding wheat grain in a finger-type single-rotor eight-row disintegrator.

The resistance to storage of samples of whole-grain wheat flour of various sizes, as well as treated with gases (nitrogen, carbon dioxide) with a concentration of 2.0 mg/l, at a pressure of P=2.0 atm for 10 minutes, was investigated.

Based on the indicators of QMAFAnM, the acid number of fat, and the acidity of gas-processed and unprocessed whole-grain wheat flour of various sizes, the terms of its safe storage were established. All safety indicators were examined within three months with a frequency of every 10 days. The relationship between the size of the flour product and its stability during storage has been established. It is recommended to store unprocessed whole grain wheat flour of coarse grinding up to 50 days, medium grinding – up to 40 days, fine grinding – up to 30 days.

The treatment of whole-grain flour before storage with carbon dioxide has made it possible to prolong the period of safe storage of coarse flour to 70 days, medium grinding – up to 50 days, fine grinding – up to 40 days.

The best results in the preservation of whole-grain flour, depending on the size of the grind, were shown by its treatment before storage with nitrogen in comparison with similar treatment with carbon dioxide. Nitrogen treatment has made it possible to recommend the duration of safe storage of coarse flour up to 90 days, medium – up to 60 days, small – up to 50 days

Keywords: whole grain flour, gas treatment, grinding size, safe storage, quality indicators

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1. Introduction

The production of whole-grain flour and its use in the bakery industry are becoming increasingly relevant. Whole grain flour is the most important source of dietary fiber, antioxidants, vitamins, polyunsaturated fatty acids, phytoestrogens, and other useful components in comparison with varietal flour. In addition, the process of producing such flour is more economical and makes it possible to ensure savings and more efficient use of grain reserves.

Flour as a product of intensive grinding of grain is a less stable object of storage compared to other products (grain, cereals) as it has a more developed surface of contact with air oxygen and microflora. Thus, the attackability and availability of flour nutrients to the action of external factors increases significantly. An important role in accelerating the processes of spoilage of flour products during storage is played by the temperature and humidity of the air, its gas composition, the presence and species composition of microflora. It has been established that dry varietal flour obtained from healthy wheat grain is stored at a temperature of 20±5 °C up to 6–8 months. However, the duration of safe storage of whole-grain wheat flour has not yet been sufficiently studied.

However, with proper storage and processing before the start of storage, the development of microorganisms present in the flour does not occur or is significantly inhibited. With an increase in the relative humidity of the air, microorganisms that have fallen into the flour and are in an inactive state in it begin to develop. Molds develop first, which grow at minimal humidity. Most often, molds of the genera *Penicillium* and *Aspergillus* are found in flour, which synthesize mycotoxins. Many of them are heat-resistant and can be stored in bread. The baking properties of flour during mold are sharply reduced, flour acquires an unpleasant musty smell and its use in bread baking is limited.

In addition to microbiological processes, during the storage of grain processing products, various chemical processes can occur. Under adverse storage conditions, these processes develop at a high rate, which leads to an undesirable deterioration in product quality.

One of the main reasons for the decrease in the quality of flour is the presence of the accumulation of free fatty acids, and with their further oxidation - peroxides and hydroperoxides, which leads to the development of the process of rancidity. The processes of hydrolytic decomposition that develop in grain and flour during storage lead to an increase in the content of acidic phosphates, organic and free fatty acids in them, which increases the acidity of stored products. The level of acidity reflects the degree of freshness of grain and products of its processing; the acid number of fat is an indicator of safe storage.

In this regard, the search for new ways of processing flour, ensuring long-term storage of whole-grain flour with high nutritional value seems relevant. It is necessary to establish the terms of safe storage, at which there is no significant increase in microorganisms, acidity, and acid number of fat, as the main criteria for preserving the technological, baking advantages of whole-grain wheat flour as a raw material for bread baking.

The use of gaseous substances to treat flour and other products of grain processing is relevant in order to regulate the functional and technological properties of flour raw materials and finished products. This method improves the sanitary condition and food safety indicators.

2. Literature review and problem statement

Paper [1] reports the results of studies info the ability of cold plasma of atmospheric pressure to change the biochemical and physical-chemical properties of flour products. It has been shown that treating wheat flour with a low level of cold plasma (air, 15 V and 20 V) for 60 or 120 s does not result in a change in the total amount of aerobic bacteria or molds. At the same time, an increase in oxidation markers (hydroperoxide number and free space above n-hexane) with the use of such treatment confirmed the acceleration of lipid oxidation. The described results give grounds for the authors to talk about the potential of cold plasma to change the functionality of flour.

All this suggests that it is expedient to conduct a study on the treatment of flour products with gaseous ozone. The established data confirm the feasibility of using this gas not only as a fumigant in the storage of grain and flour but also as a regulator of the functional, technological, and baking properties of flour. The effectiveness of ozone from positive to negative is manifested with an increase in the dose, determined by the concentration and time of exposure of the gas to the product. The authors note that flour functionality decreased when ozone exposure exceeded 9 min and the concentration of ozonated flour exceeded 200 $g/kg^{(-1)}$ [2].

An option to overcome the relevant difficulties may be the search for alternative methods that can reduce or inhibit the growth of microbes in flour and extend the shelf life of products of its processing; it is expressed in recommendations for the use of chlorine dioxide. The ability of chlorine dioxide to effectively inactivate insects, bacteria, fungi, viruses, spores, and toxigenic forms is interrelated with the above fact. Treatment with chlorine dioxide significantly reduces the total flavonoids in the flour, increasing whiteness and stability time. When stored, the growth of microorganisms in products made from composite flour based on buckwheat flour treated with chlorine dioxide slows down [3]. This is a strong and highly effective oxidizing agent, the suitability of which for use in gaseous formulations for disinfection and preservation of green coffee beans is confirmed by the authors of [4]. The use of gaseous chlorine dioxide for the effective processing and storage of vegetables and fruits is confirmed in work [5]. The use of an aqueous solution of ClO_2 makes it possible to control the growth of microflora on contaminated wheat grains [6].

It is necessary to emphasize the direction of application of inert gases for the processing of food raw materials to improve its microbiological purity. The term “inert” is justifiably used to describe substances that do not enter into chemical reactions. Historically, noble gases of helium, neon, argon, krypton, xenon, and radon have been referred to as inert gases. However, reactivity depends on environmental conditions, on the situation.

For example, nitrogen (N_2) and carbon dioxide (CO_2) are used as inert gases in the wine industry because they have little to no reaction with wine [7]. They can be used alone or as a mixture in varying proportions to protect wines from oxygen exposure, which includes oxidation and an increased risk of microbial contamination.

The use of inert gas is expressed in the fact that it does not enter into chemical reactions either with the manufactured product or with the human body. All this allows us to assert that it is advisable to conduct a study on the use of these gases in storage as follows: various negative processes of vital activity in the grain and flour itself are slowed down, which accordingly prolongs the duration of the shelf life [8].

A scientifically based and stable view has been formed that the use of inert gases as an alternative treatment for pest control in food products is a growing need of the food industry. This approach satisfies the needs of consumers with regard to reducing or eliminating the use of pesticides and protecting the environment [9].

Nevertheless, the results of scientific research and practical search for the effectiveness of the use of inert gases to improve the conditions for safe storage of food products are rather ambiguous. Thus, the authors of [10] investigated the effect of argon on the quality of wheat flour under storage conditions and the possibility of preventing quantitative and qualitative losses. Grain samples were stored in sealed plastic containers, in which argon was used as an air medium. Storage was carried out at a temperature of 12 to 23 °C for 22 weeks. The authors did not establish a significant difference between the indicators of water absorption capacity, pharynograms of dough kneading, the number of falls and the parameters of the alveogram in samples stored in normal air and in an argon environment. Thus, the authors did not recommend the use of wheat grain storage in an argon inert gas environment since the cost of such storage exceeds the expected improvement in the quality of wheat.

A different approach to the use of inert gases to inhibit microflora during food storage was demonstrated by the authors of [11]. This study was aimed at assessing the effects of compressed CO_2, N_2 and atmospheric air on immature stages and adults of secondary insect species *Oryzaephilus surinamensis* (L.) and *Stegobium paniceum* (L.). Insects were exposed to 5 levels (1–5 bar) of compressed gases in a metal gas cylinder for one and two days of exposure at an average temperature of 20 °C. Increased sensitivity of insects to pressure (the higher the larger) and reduced, to the type of gas, was established. Overall, the results confirmed that the use of CO_2 under high pressure is an effective and quick way to control most pests affecting food products during storage.

The data obtained are in good agreement with the results on treatment with a modified atmosphere at atmospheric pressure, presented in study [12], as well as with the proposal to apply high pressure, which provides for a much faster action [9]. The efficiency of using CO_2 under high pressure, which depends on the type of pest, the stage of development of insects, pressure, exposure time and temperature, is noted in work [13].

Thus, the above allows us to assert the feasibility of conducting a study on improving the quality of flour and grain raw materials, increasing the period of its safe storage. Researchers recommend various methods, among which the leading position is occupied by the modification of the gas composition of the air at storage facilities. Recommendations also include the use of complex effects – increased pressure, original equipment design, etc.

It should be noted that the scientific search is carried out not only in the direction of improving the conditions for the safe storage of flour raw materials but also in terms of expanding the range of flour products.

With the growth of the well-being of the population and the development of the global information field in society, the need for healthy and individualized nutrition began to form, which could not but affect the flour market. This new trend is reflected in the development of legal regulation of food markets and has set a trend in the direction of healthy eating.

This industrial revolution and the use of high-purity and processed flour as a raw material for food products led to the loss of their previously inherent useful properties. Such flour contains only the endosperm of the grain while a significant part of the shells and embryo are lost in the process of grinding. However, it is these parts of the grain that contain fiber and healthy micronutrients. As a result, interest in products derived from whole-grain flour increased.

Whole grain products are the most important source of most essential nutrients: dietary fiber, antioxidants, vitamins, polyunsaturated fatty acids, phytoestrogens, and other components that have preventive properties in relation to the development of most chronic diseases. For example, whole grain flour contains 10.7 % of dietary fiber, 61.8 μg/100 g of selenium, 0.5 mg of thiamine, 72.8 mg of betaine, 1.9 mg of γ-tocopherol. Whole grain wheat flour is richer (by 1.48–3.75 times) in vitamins B_1, B_2, B6, B9, E, PP than wheat flour of the highest grade. In this regard, it is advisable to use it for the production of preventive food products. Most of these components are found in the germ and bran, which are removed during the process of varietal grinding of grain.

Unresolved issues related to the technological properties of grain products correlating with the content of these substances in them remained unresolved. It is known that dietary fiber increases the time of hydrothermal treatment, and the color of the product changes. At the same time, lipids become more resistant to oxidation due to their antioxidant content. Therefore, researchers and manufacturers are faced with the task of increasing the consumption of whole grain products. This is ensured by the preservation of their organoleptic and technological properties.

A draft standard for bakery products with the addition of grain and products of its processing has been developed [14]. The forms of inclusion of whole grain products in the diet are diverse, for example, products from whole-grain flour (pizza base, pasta, bread) [15]. At the same time, recommendations of micronization modes of wheat grain for the production of whole grain products are presented in [16]. The authors of [17] substantiate the effectiveness of the use of whole-ground flour from sprouted wheat grain in the technology of a wide range of food products. Scientifically substantiated ion-ozone technology of processing grain and its products is presented in work [18]. Study [19] is aimed at determining the influence of various parameters of ion-ozone treatment on the quality and safety of whole grain products from millet, buckwheat, chickpeas, and lentils. The effectiveness of grain processing using it as grain bases of food products is shown in [20].

Most of the research work carried out, as can be seen from the above information, is aimed at solving the problem of processing food products with gaseous agents in order to improve their quality indicators, as well as inhibiting the microflora of the product. At the same time, insufficient attention was paid to justifying the shelf life of food products on the basis of their special technological properties and safety indicators.

The problem of scientifically based terms of safe storage is especially acute for new grain products. Among the possible reasons, it is necessary to indicate the complexity of the equipment for processing flour products with gases, which is often stored in a bulk way and in large volumes.

It should be clarified that according to the Codex Alimentarius, the term “whole grain” refers to whole grains found in bread or bread crust, squeezed or turned into flakes, or to “whole-grain” flour used to make “whole-grain” bread. Simultaneously with this term, which is more understandable to consumers, there is the term “whole ground” flour. Perhaps it is in it that there is a technological component.

Obtaining whole-grain flour and its use in the bakery industry is a fairly promising and relevant direction that makes it possible to ensure savings and effective use of huge material and financial resources in the field of the Agro-Industrial Complex (AIC).

The main components of whole wheat flour are the shell, endosperm, bran, and germ. They contain large volumes of vitamins, proteins, and minerals. The shell contains 80 % of all micro- and macro elements and is a source of dietary fiber. Germ is the main concentrate of vitamins, amino acids, and polyunsaturated fatty acids. The endosperm (mealy nucleus), which occupies most of the grain, is rich in carbohydrates, proteins, and contains nicotinic acid and iron, as well as unique water-soluble dietary fiber “beta”-glucans. Therefore, such flour is a product much more useful than traditional flour of the highest, first, and second grades.

One of the main reasons for the decrease in the quality of whole-grain flour is the presence in it of a crushed germ, an aleurone layer with high fat content. It accumulates free fatty acids, and with their further oxidation – peroxides and hydroperoxides, which leads to the development of the process of rancidity. The processes of hydrolytic decomposition that develop in such flour during storage lead to an increase in the content of acid phosphates, organic and free fatty acids in them, which increases the acidity of stored products.

In this regard, ensuring the safety of whole-grain flour, which has a high nutritional value, is an extremely urgent task.

A promising area of research is the use of gases (carbon dioxide, nitrogen). These gases do not enter into chemical reactions either with the manufactured product or with the human body. They are able to slow down various negative processes in flour, which increases the time of its safe storage.

3. The aim and objectives of the study

The purpose of our research is to scientifically substantiate the safe shelf life of whole-grain wheat flour. This will provide an opportunity to expand the range of functional grain products to improve the quality of life of the population.

To accomplish the aim, the following tasks have been set:

– to establish initial indicators of the quality of flour products after grinding;

– to determine the effect of gases on increasing the safety of whole-grain wheat flour.

4. The study materials and methods

The objects of this study were wheat grain (Shortandinskaya variety of the 2021 harvest), as well as wheat grain grinding products – whole grain (whole-ground) wheat flour (WGWF) of coarse, medium, and fine grinding. At the same time, the size of the grinding was determined by the intensity of rotation of the rotor of the laboratory mill after grinding and with subsequent storage of flour for 10–90 days.

The grain cleaned of impurities was subjected to washing, conditioning, and drying for 24 hours (AASS 26–10.02,1999). Then grinding was carried out at the laboratory mill Bühler MLU (Germany). Grain grinding took place in a finger-type single-rotor eight-row disintegrator DPO-320-7.5, designed for grinding cereals.

The size of the crushed wheat grain particles, which was regulated by the change in rotor speed, ranged from the largest (more than 1000 μm) to the smallest (5–10 μm) particles. Finely ground WGWF (whole wheat flour) was produced at a rotor rotation speed of 3000 rpm, medium grinding WGWF – at a rotor rotation speed of 2100 rpm, and coarse – at a rotor rotation speed at 1200 rpm.

Based on the results of the studies proposed by the authors of [21], there is a scheme for obtaining oat flour. A similar experiment was conducted to obtain whole-grain wheat flour of various sizes, the scheme of which is shown in Fig. 1.

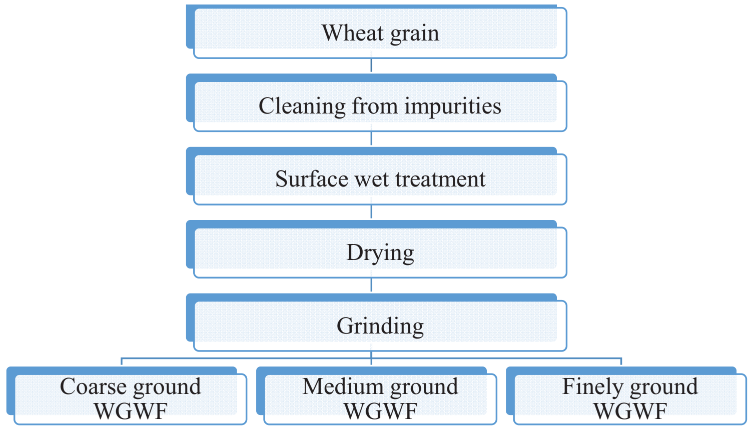

Fig. 1. Generalized scheme of obtaining whole-grain wheat flour

In order to increase the safe shelf life of WGWF of various grinding sizes, the studied flour samples were treated with nitrogen (N_2) and carbon dioxide (CO_2). The gas concentration (both N_2 and CO_2) was 2.0 mg/l; pressure, P=2.0 atm; processing duration, 10 min [24–26].

Processing of the studied samples of flour with gases was carried out after 5 days of resting after grinding. As control samples, WGWF of various grinding sizes obtained in the same laboratory mill were used, without the use of gas treatment.

Treatment of flour with gases was carried out in laboratory silo-type containers made of plexiglass (Fig. 2). Storage of processed (experimental) and raw (control) samples of WGWF was carried out at a temperature of 20–25 °C in these containers.

To assess the safety and quality of flour products, microbiological, chemical, and technological indicators were investigated.

QMAFAnM (total microbial number) was determined as the number of mesophilic aerobic and facultative-anaerobic microorganisms. This is an extensive group of different microorganisms that can contaminate a food product. We determined QMAFAnM according to GOST 10444.15-94 – "Methods for determining the number of mesophilic aerobic and facultative-anaerobic microorganisms" by seeding on Petri dishes with a nutrient medium (meat-peptic agar). The number of microorganisms was determined in 1 cm^3 of the product of liquid consistency. We determined the titrated acidity of flour, which is an important criterion for its freshness, according to GOST 27493-87. This indicator characterizes the total volume of free acids and acidic salts. The degree of acidity was understood as the amount of 1 n. sodium hydroxide solution required to neutralize acids and acidic salts contained in 100 g of flour. The method used to determine acidity according to GOST 27493-87 provides for determining the titrated acidity by a water-flour suspension.

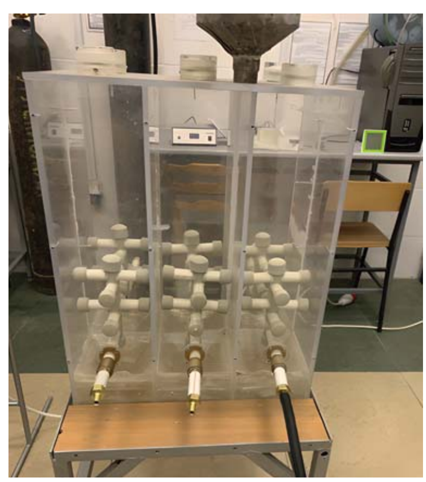

Fig. 2. Laboratory silo-type installation for processing and storage of flour

Determining the acid number of fat (ANF) was carried out according to GOST 31700 "Grain and products of its processing. Method for determining the acid number of fat"; humidity – according to GOST 9404; the volume of crude gluten and the compressibility of gluten – at the GDM device (gluten deformation meter) – according to GOST 27839.

The general scheme of experimental studies is shown in Fig. 3.

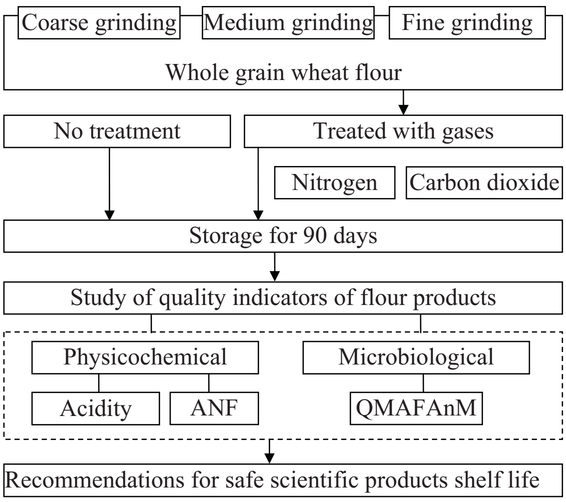

Fig. 3. General scheme of experimental studies on the storage of whole-ground wheat flour in the laboratory

For the value of the safe shelf life, the duration of storage in days preceding the growth of colonies of microorganisms on the flour by 10 times was taken, while the acidity and ANF of the flour were also monitored.

The above diagram (Fig. 3) shows the sequence of storage of whole-ground flour in comparison with the control sample.

5. Results of investigating the storage of whole-ground wheat flour

5. 1. Establishment of quality indicators of flour products after grinding

In accordance with the scheme for obtaining flour products in the laboratory (Fig. 1), depending on the intensity of grinding, samples of whole-grain wheat flour of various sizes were obtained. The indicators of the quality of flour products immediately after grinding are given in Table 1.

Table 1

Quality indicators of flour products after grinding (n=5, p<0,05)

Indicators of flour quality	WGWF grinding type		
	coarse	medium	fine
Humidity, %	13.0	13.6	14.0
Acidity, degree	6.0	7.1	8.5
Acid number of fat, mg. KOH per 1 g of fat	12.8	13.1	13.5
Amount of raw gluten, %	31.0	32.9	33.4
Gluten quality, GDM units	63.0	65.9	70.0
QMAFAnM, KOE/g	$8\cdot10^3$	$8\cdot10^3$	$8\cdot10^3$

The qualitative composition of whole-grain wheat flour, studied depending on the coarseness of grinding, is given in Table 1. Humidity ranged from 13 to 14 **%**. Acidity – from 6 to 8.5 degrees. In terms of the quantity and quality of gluten, there is an increase in values from coarse to fine grinding.

5. 2. Determining the effect of gases (nitrogen, carbon dioxide) on increasing the storage safety of whole wheat flour

Our studies have shown that treatment with gases (both nitrogen and carbon dioxide) before storage led to a noticeable change in the microbiological indicators of the quality of experimental samples compared to control ones (without processing), and during storage the differences were preserved (Tables 2–4).

Table 2

Change in the microbiological contamination of coarse-ground WGWF during storage (n=5, p<0,05)

Treatment type	QMAFAnM, CFU/g, when stored for, *day*								
	0	10	20	30	40	50	60	70	90
No treatment (control)	$8\cdot10^3$	$8\cdot10^3$	$8\cdot10^3$	$9\cdot10^3$	$6\cdot10^3$	$8\cdot10^4$	$9\cdot10^5$	$9\cdot10^6$	$8\cdot10^8$
Treatment with nitrogen	$2\cdot10^3$	$2\cdot10^3$	$3\cdot10^3$	$4\cdot10^3$	$5\cdot10^3$	$6\cdot10^3$	$7\cdot10^3$	$8\cdot10^3$	$4\cdot10^4$
Treatment with carbon dioxide	$5\cdot10^3$	$6\cdot10^3$	$7\cdot10^3$	$8\cdot10^3$	$8\cdot10^3$	$9\cdot10^3$	$9\cdot10^3$	$6\cdot10^4$	$8\cdot10^5$

Table 3

Change in the microbiological contamination of medium-ground WGWF during storage (n=5, p<0,05)

Treatment type	QMAFAnM, CFU/g, when stored for, *day*								
	0	10	20	30	40	50	60	70	90
No treatment (control)	$8\cdot10^3$	$4\cdot10^3$	$8\cdot10^3$	$9\cdot10^3$	$6\cdot10^4$	$7\cdot10^5$	$9\cdot10^5$	$9\cdot10^6$	Not studied
Treatment with nitrogen	$2\cdot10^3$	$4\cdot10^3$	$6\cdot10^3$	$7\cdot10^3$	$8\cdot10^3$	$8\cdot10^3$	$9\cdot10^4$	$2\cdot10^5$	Not studied
Treatment with carbon dioxide	$3\cdot10^3$	$3\cdot10^3$	$5\cdot10^3$	$6\cdot10^3$	$9\cdot10^3$	$9\cdot10^4$	$3\cdot10^5$	$2\cdot10^6$	Not studied

Table 4

Change in the microbiological contamination of fine-ground WGWF during storage (n=5, p<0,05)

Treatment type	QMAFAnM, CFU/g, when stored for, *day*								
	0	10	20	30	40	50	60	70	90
No treatment (control)	$8\cdot10^3$	$6\cdot10^3$	$8\cdot10^3$	$6\cdot10^4$	$7\cdot10^4$	$8\cdot10^5$	$9\cdot10^6$	$9\cdot10^7$	Not studied
Treatment with nitrogen	$2\cdot10^3$	$2\cdot10^3$	$3\cdot10^3$	$4\cdot10^3$	$5\cdot10^3$	$6\cdot10^3$	$7\cdot10^3$	$8\cdot10^3$	Not studied
Treatment with carbon dioxide	$5\cdot10^3$	$6\cdot10^3$	$7\cdot10^3$	$8\cdot10^3$	$8\cdot10^3$	$9\cdot10^3$	$9\cdot10^3$	$6\cdot10^4$	Not studied

Similar trends are reported in the study of the acidity of prototype and control samples of flour of coarse grinding in Fig. 4–6.

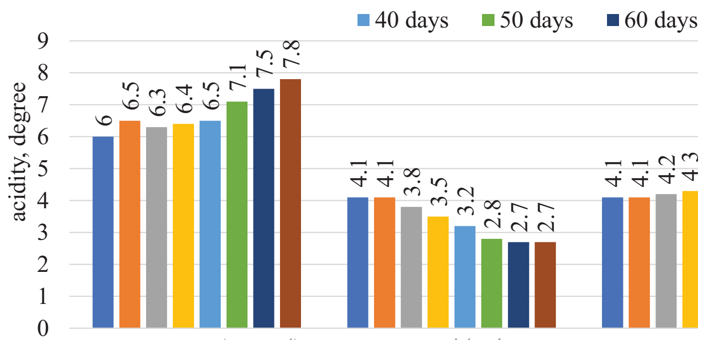

Fig. 4. Acidity of whole-ground wheat flour of coarse grinding during storage

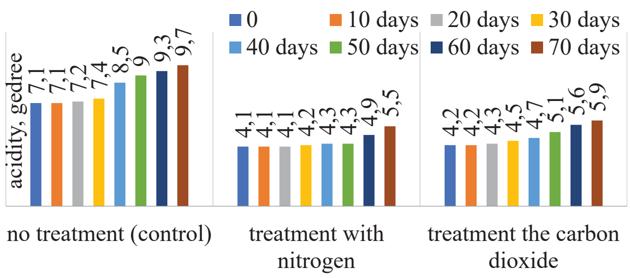

Fig. 5. Acidity of whole-ground wheat flour of medium grinding during storage

Fig. 7–9 present changes in the ANF of unprocessed whole-grain wheat flour depending on the duration of storage and the change in the acid number of fat during storage of whole-ground wheat flour of various grinds after treatment with carbon dioxide and nitrogen.

Fig. 7–9 give grounds to recommend the storage of whole-ground wheat flour with treatment with carbon dioxide and nitrogen. More efficient storage is in a nitrogen environment.

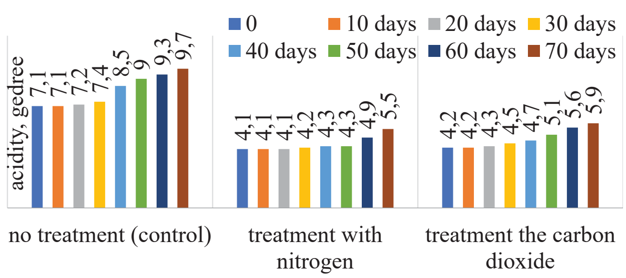

Fig. 6. Acidity of whole-ground wheat flour of fine grinding during storage

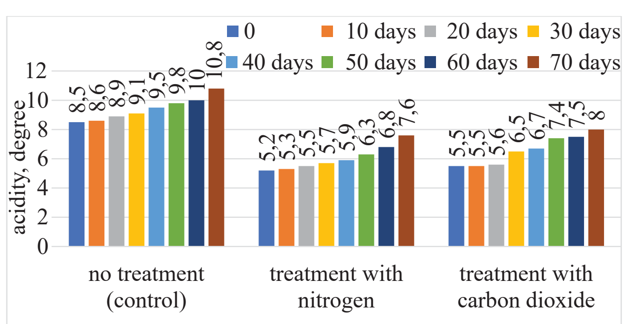

Fig. 7. Change in the acid number of fat during storage of unprocessed whole-ground wheat flour of various grinds

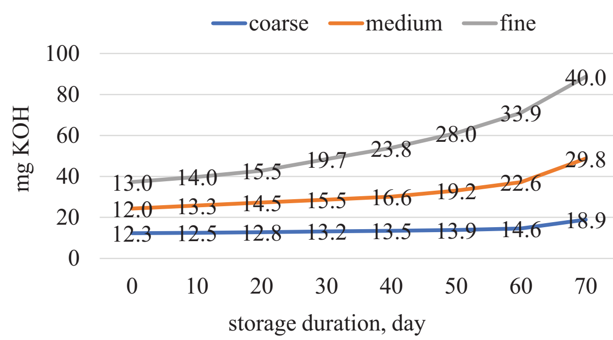

Fig. 8. Change in the acid number of fat during storage of whole-ground wheat flour of various grinds after treatment with carbon dioxide

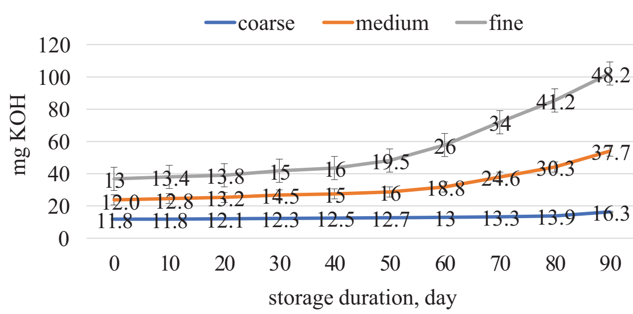

Fig. 9. Change in the acid number of fat during storage of whole-ground wheat flour of various grinds after nitrogen treatment

6. Discussion of results of investigating the effect of gas treatment on safety in the storage of whole wheat flour

The production of whole grain wheat flour increases annually. This is due to the fact that it is advisable to use it for the production of preventive nutrition products because whole grain flour is rich in dietary fiber, vitamins, polyunsaturated fatty acids, phytoestrogens, and other components that have preventive properties in relation to the development of most chronic diseases. Most of these useful components are found in the germ and bran, which are removed during the process of varietal grinding of grain.

Whole grain wheat flour containing bran and germ is less stable in storage compared to white refined flour. Storing whole-grain wheat flour for only 30 days at 75 °F can result in an undesirable odor and taste in products derived from whole-grain flour. Simultaneously with the appearance of the aftertaste, the volume of free fatty acids, correlated with the increase in oxygen present in the flour, and the formation of oxidative components of rancidity increases. Reducing the size of the particles increases the rate and degree of spoilage of the grain components. Wet and heat treatment is typically used to inactivate the enzymes responsible for flour spoilage, although it has recently been found to contribute to rancidity caused by oxidative processes measured by the formation of hexanal, a well-known marker used to determine rancidity caused by oxidative processes in oatmeal. It is believed that the increase in rancidity caused by oxidative processes is due to the destruction of cellular structures that stabilize lipids, or due to the inactivation of unstable antioxidants [22].

It is known that the shelf life of flour is set by the manufacturer of products at an ambient temperature not exceeding 25 °C (for rye flour, not higher than 20 °C) and a relative humidity not higher than 70 %, provided that the acid number of fat is not more than 60 mg KOH per 1 g of fat for wheat flour, including pasta, and not more than 80 mg KOH – for rye bakery. Under such conditions, varietal wheat flour is usually stored for 6–8 months, durum and soft wheat flour for pasta and rye flour – for no more than 6 months. Storage of flour at low temperatures (about 0 °C and below) extends the shelf life of flour to two years or more [23].

Given that storage in production conditions takes place at a warehouse temperature not higher than 25 degrees, it is important to compare the results obtained with production storage. In this regard, gas treatment and storage of unprocessed and processed flour was carried out in laboratory silo-type containers (made of plexiglass) at a temperature of 20–25 °C in these containers (Fig. 2).

In accordance with the scheme of production of flour products in the laboratory (Fig. 1), depending on the intensity of grinding, samples of wheat whole-grain flour of various sizes were obtained. Indicators of the quality of flour products immediately after grinding are given in Table 1. Analysis of Table 1. shows that with a decrease in the coarseness of flour, the indicators of humidity, acidity, ANF increased. In contrast, the volume of crude gluten increased from 31.0 % in coarse flour to 32.9 % and 33.4 % for medium and fine grinding, respectively.

The terms of safe storage in the nitrogen medium, both for medium and fine grinding, have increased significantly in comparison with the treatment with carbon dioxide (Tables 3, 4). Microbiological indicators were in the range of $9*10^4$. The same growth of microorganisms was established on day 50 when treating with chloride dioxide, and when treating with nitrogen – on day 60, which confirms the effectiveness of nitrogen use during storage.

Treatment with gas with (both nitrogen and carbon dioxide) before storage led to a noticeable change in the microbiological indicators of the quality of experimental samples compared to control ones (without processing), and, during storage, the differences persisted (Tables 2–4). Similar trends were observed in the study of the acidity of experimental and control samples of flour (Fig. 4–6) and the acid number of fat (Fig. 7–9).

Analysis of microbiological indicators of the quality of flour products showed that the most effective disinfection occurred during nitrogen treatment. Thus, QMAFAnM in the WGWF of coarse, medium, and fine grinds without processing (control) and without storage was at the same level and amounted to $8{\cdot}10^3$ CFU/g. When storing unprocessed flour of coarse grinding for 50 days, the growth of microorganisms increased by one order of magnitude (up to $8{\cdot}10^4$ CFU/g), and the titrated acidity – up to 7.1 degrees, which corresponds to the period of safe storage (Table 2, Fig. 4).

The values of ANF (the acid number of fat) laid down for the storage of whole-grain wheat flour of various sizes, which has not been processed and processed, differ significantly. During the storage of unprocessed whole-grain wheat flour, for samples of coarse, medium, and fine grinds, different terms of safe storage were established. Thus, unprocessed coarse flour with an initial humidity of 13.0 %, an acidity of 6.0 degrees, and ANF of 12.8 mg KOH per 1 g of fat (Table 1) was stored practically without changing the initial values for 45...50 days. In coarse flour treated with nitrogen, the duration of storage increased to 90 days and the growth of QMAFAnM began only on day 90. Treatment with carbon dioxide made it possible to extend the shelf life of such flour by 20 days (in comparison with an unprocessed sample), namely up to 70 days (Table 2).

In unprocessed flour of medium grinding, a sharp increase in the acid number of fat (by 50 %) was observed on day 40 (Fig. 5) while the volume of QMAFAnM corresponded to $6{\cdot}10^4$ CFU/g (Table 3). Also, with an increase in the acid number of fat and microorganisms, the acidity increased by 20 % and amounted to 8.5 degrees.

When storing unprocessed flour of fine grinding (Fig. 6), a significant increase in the acid number of fat was established on day 30, from 13.5 to 20.0 mg KOH per 1 g of fat. This corresponded to a 10-fold increase in the volume of QMAFAnM (Table 4), from $8{\cdot}10^3$ to $6{\cdot}10^4$ CFU/g, while the value of flour acidity also increased by 19 %.

Thus, in the course of experimental studies, a significant change in the indicators of safe storage of flour products was established. Depending on the size of the flour (coarse, medium, and fine grinds), the presence and type of gas for processing the flour before laying for storage (nitrogen, carbon dioxide).

It should be noted that in the case of treatment of WGWF with nitrogen during further storage in the flour of coarse grinding, the increase in acidity and QMAFAnM was practically not observed. Only on day 70, there began a slight (by 10 %) increase in ANF, from 11.8 to 13.3 mg of KOH per 1 g of fat. This indicates that ANF is a more significant indicator of the safety of flour and with an increase in the duration of storage increases earlier and more intensively than other indicators characterizing the freshness of flour. However, after 60 days of storage in medium-milled flour treated with nitrogen, ANF increased significantly and amounted to 18.8 mg of KOH per 1 g of fat, an increase of about 50 % compared to the initial data. A significant increase in ANF during the storage of finely ground WGWF treated with nitrogen began on the 50th day of storage and amounted to 19.5 mg of KOH per 1 g of fat (Fig. 8). The number of QMAFAnM increased by an order of magnitude from $7{\cdot}10^3$ to $9{\cdot}10^4$ CFU/g, the acidity increased by 10 %, that is, from 5.2 to 6.3 degrees.

Fig. 7 shows the modification of ANF of the untreated WGWF depending on the duration of storage. The growth of ANF was observed in all samples (control) without processing in the following order: WGWF of fine grinding – on day 30, medium – on day 40, large – on day 50. In all samples, the growth of ANF was about 48...51 % compared to the initial values when laying for storage.

When treating WGWF with carbon dioxide (Fig. 8), the safe storage time of flour of various grinds increased significantly in comparison with unprocessed samples. The maximum period of safe storage was 60 days for coarse-ground WGWF, medium – 50, small – 40 days.

Treatment of WGWF of various grindings with nitrogen showed the best results, which make it possible to recommend longer periods of safe storage. As can be seen from Fig. 9, nitrogen treatment has increased the safe shelf life to varying degrees depending on the coarseness of the grind. It can be recommended to increase the safe shelf life of whole-ground wheat flour of coarse grinding to 90 days, medium – up to 60 days, small – up to 50 days.

Based on the results of the above studies (Fig. 7–9), it should be noted that in the established terms of safe storage according to the growth trend, QMAFAnM and ANF (Tables 4–6) have a positive correlation. The effectiveness of nitrogen treatment of whole-grain flour of various grinds was confirmed.

The obtained results of microbiology, CPH, acidity can exist within the use of certain concentrations of gases, storage conditions, and depending on the purpose of use of the studied products.

The peculiarity of the method is the fact that to increase the shelf life of the product, methods of processing whole-grain wheat flour with gases were used.

The lack of industrial equipment for pumping gas media into the stored mass of the product. There are no express devices for designing quality products.

The development of this study can lead to an increase in the range of useful products, an increase in the quality of products consumed for a healthy lifestyle. It has been established that an increase in the consumption of food products from whole wheat, rye, oats, barley, spelt implies the prevention of cardiovascular diseases, diabetes, obesity, beriberi. Whole grain wheat flour has 25–30 % more protein, almost 4 times more wheat fiber, and less carbohydrates. Depending on the type of product, ordinary flour can be replaced with whole-grain flour either partially or completely. Whole grain flour enriches products with dietary fiber, while reducing caloric content. Since whole grain flour contains small fractions of bran particles, the dough from such flour is easy to mold. Whole grain flour can be used in the production of butter, oatmeal, sugar, puffed cookies, crackers and biscuits, muffins, etc.

7. Conclusions

1. It has been established that the coarseness of grinding is reflected in the technological indicators of the quality of whole grain wheat flour. With a decrease in the coarseness of flour, the indicators of humidity, acidity, and ANF increased. The volume of raw gluten in whole grain in coarse flour was 31 %. In the medium and fine ground flour, the volume of raw gluten corresponded to 32.9 % and 33.4 %.

2. When flour is treated with carbon dioxide, the terms of safe storage of flour of various grinds have increased significantly in comparison with untreated samples. The maximum period of safe storage was, for coarse flour – 60, medium – 50, small – 40 days.

According to the results of changes in microbiological parameters, the acid number of fat and the acidity of whole grain wheat flour of different sizes, it was shown that more effective disinfection occurred during nitrogen treatment (concentration 2.0 mg/l; processing duration, 10 min). The terms of safe storage of wheat whole grain coarse flour can be increased to 90 days, medium – up to 60, small – to 50 days.

Conflict of interests

The authors declare that they have no conflict of interest in relation to this research, whether financial, personal, authorship or otherwise, that could affect the research and its results presented in this paper.

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