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Research Article

Moisture Sorption by Extrusion Products Enriched with Nut Flour

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Abstract

The article describes the change in the moisture content of the extrusion products over time, as well as the influence of ambient relative humidity on the equilibrium moisture content of extrudates with a porous structure. Based on the experimental data, the moisture sorption isotherms were constructed and the ranges of adsorption, water saturation and capillary condensation were determined. Based on the data obtained from the experiment, the amount of moisture retention energy of extrudates was calculated, and the nature of its change was assessed with respect for ambient relative humidity and the equilibrium moisture content of the product.

Studies have shown that the process of moisture sorption in extrudates is reflected in the interaction of water molecules with the biopolymer binding centers, as well as in changes related to the structure of extrudates. In addition, it should be noted that change in the moisture content in the samples depends on ambient relative humidity. Thus, due to the fact that the minimal sorption of moisture occurs at 10% ambient relative humidity, when the equilibrium moisture content reaches 5,2% these data represent the storage conditions for the products with the mentioned structure and chemical compositions.

Keywords: Extrudate; Moisture; Sorbtion Process; Nut Flour; Structure

Introduction

Production of any items is aimed not only at correct selection of the machines and equipment and compliance with operating modes and technology, but also at storing the resulting products in an appropriate environmentally-safe facility. This process requires a holistic approach for selected products in order to choose the individual microclimate required for product storage (in warehouses or packaging) to retain its high quality and consumer characteristics.

Previous studies conducted by the authors have examined the influence of raw material moisture content on the starch gelatinization point. Studies show that the 15% - moisture content in the processed raw material is sufficient for translation for its component phase, starch gelatinization and translation of the whole mass into the fluid-viscous state. Subsequent increase in moisture content is accompanied by lowering in the gelatinization temperature. It has been established that the minimum temperature for the thermoplastic extrusion process is the starch gelatinization point, while the raw material's minimal moisture content is 15 - 20% [1].

In order to study the influence of moisture on the formation of the porous structure of extrudates, we have investigated the relationship between the moisture content of different types of starch gels and the degree of its transparency and the embrittlement temperature. It has been established the process of thermal and mechanical action brings about the formation the structure of the gels, in particular the same samples of different humidity can obtain an amorphous or crystalline structure.

The authors of the article have also investigated the dependence of the moisture content of raw materials on the modulus of elasticity of gels. The modulus of elasticity of the samples was determined one hour (cooling time to room temperature) after gel was produced and two weeks after as well. The above studies have shown that minimal physicochemical and mechanical transformations take place in gels that are in an amorphous glassy state, i.e. under low humidity conditions [1].

The authors of the article have found that the moisture content of raw materials on the one hand ensures the transition of the high-dispersion phase into the fluid state, that is conducts the extrusion process, and on the other hand, it affects the formation of the porous structure of extrudates [1].

Earlier studies on the model systems of extrudates such as starch gels, have shown that, on the one hand, the moisture in the extrusion process affects the gelatinization point of processed raw materials, and on the other hand, it serves as steam generator in the extrusion process [2].

The hygroscopic properties of extrudates, as well as the patterns of heat and mass transfer are mostly dependent on and determined by their capillary-porous structure, so of particular interest is to determine the values of the characteristic parameters of the porous structure, in order to solve many theoretical or practical issues in the future.

The structure of the material or product is mainly characterized by the general porosity, the specific surface area of the pores, the dimensions of the pores and the integral and differential curves of the distribution along the radius (volume) of the pores [3-7]. In order to determine all these values, it is necessary to study the process of moisture sorption of extrudates with porous structure, which provides a proper understanding of the mechanism of moisture retention in the product and the transition from one form to another.

It is known that the process of moisture sorption consists of the processes of adsorption of water molecules, water saturation of extrudates, and the process of capillary condensation. In general, this process is clearly depicted in the sorption curves, in the form of different sections on isotherms [8].

Given that the properties of extrudates are determined mainly by the nature of its component continuous phase - the starch, extrudates with a porous structure enriched with nut flour should be stored in conditions of low moisture content of the product. Therefore, the aim of the study is to investigate the process of moisture sorption by new, extrusion starch-based products enriched with nut, as well as to determine the optimal parameters for their storage.

Scientific hypothesis

Based on our earlier studies [2], the minimal physicochemical transformations in starch gels take place at 5-7% of their moisture content, and the equilibrium moisture content of extrudates depends on ambient relative humidity, so we can assume that extrudates with the porous structure should be stored at less than 10% relative humidity, that is, in a packaged airtight environment, where a special microclimate will be created.

Materials and Methods

The authors of the article were taking samples of extrudates for the studies on an extruder K - 30 (Kiiko, Dnepropetrovsk, Ukraine) composed of the following main units and parts, such as: extrusion chamber, auger kit, forming die with different matrix diameters and the control panel. The extruder chamber is a hollow cylinder of 400 mm length and 19 mm inner diameter of the auger, with 6 longitudinal channels designed to transport the mass processed when using raw materials with the floury structure. On the outer surface of the cylinder, there are mounted two heating elements. In general, the extruder has three zones: mixing, plasticizing and charging zones. From the top of the cylinder, there is secured a vertical single-screw proportioning feeder with a pyramid hopper. Inside the chamber, there is placed a single-thread variable-pitch auger with an outer diameter of 19 mm. During the studies, we used the auger kit with varying values of charging. At the end of the extruder chamber, the forming die with a matrix is connected by a threaded connection. We used matrices with the different hole diameters. The authors of the article varied the auger speed from

150 min^{-1} to 230 min^{-1}, and measured the rotary speed by means of a tachometer, and the temperature in the cylinder was measured using a thermocouple.

For the purpose of carrying out experimental studies in accordance with the formulations, the authors of the article have selected materials as follows: corn grits, corn starch according to ISO 11085:2008 [9], walnut according to DDP-02 2016 [10]; peanut according to ISO 6478:1990 [11] and table salt according to CODEX STAN 150-1985 [12].

The authors of the article took the flour from walnuts, peanuts, almonds and hazelnuts in the following way: the authors cleared the fruits from damaged grains and other impurities, washed and dried it to a moisture content of not more than 14%. The dried grains were crushed on a laboratory grain crushing machine (Retsch, Kiev, Ukraine) and passed through a laboratory sieve No. 08 (Retsch, Kiev, Ukraine). Qualitative indicators of the obtained samples of flour from walnuts, peanuts, almonds and hazelnuts are given in table 1.

Nut samples	Flour color	Grinding thickness - the mass remained on the sieve No. 8, [%]	Taste	Smell
Walnut flour	Honey color with membrane particles	1.3	Nice walnut taste with characteristic bitterness	Normal, characteristic of walnut
Peanut flour	Yellow	1.3	Specific, characteristic of peanut	Normal, characteristic of peanut
Almond flour	Dark cream-colored	1.2	Nice taste characteristic of almond	Good smell characteristic of almond
Hazelnut flour	Cream-colored	1,2	Nice nutty taste	Good smell characteristic of nut

Table 1: Qualitative indicators of nut flour.

Based on the analysis of all the available data, by applying the equity proportion of starch, proteins and other components in the extrusion raw materials, taking into account the conditions for the smooth implementation of the extrusion process, the authors of the article have developed the mixture's formula compositions.

The first extrusion mixrture:

- Corn grits with a 70% starch content - 56.8%
- Corn starch - 10%
- Walnut - 12%
- Peanut- 4.5%
- Table salt - 0.7%
- Mixture's moisture content taking into account the added water - 16%.

The second extrusion mixrture:

- Corn grits with a 70% starch content - 57.8%
- Wheat grits with a 75% starch content - 10%
- Hazelnut - 4.5%
- Table salt - 0.7%
- Mixture's moisture content taking into account the added water - 17%.

The second extrusion mixrture:

- Corn grits with a 70% starch content - 57.8%
- Wheat grits with a 75% starch content - 10%
- Almond - 10%
- Hazelnut - 5.5%
- Table salt - 0.7%
- Mixture's moisture content taking into account the added water - 16%.

The optimal parameters of the extrusion process are given in table 2.

Process parameters	Functional properties of the base product	
	The mass density, ρ = 113 kg · m^{-3}	Expansion ratio, Exp = 3
The die hole diameter, d [m]	0.003	0.003
Auger pressurization degree, S	4 : 1	5 : 1
Temperature in extruder cylinder, T [°C]	190	188
Extrusion mass moisture content, W [%]	20	20

Table 2: The optimal parameters of the extrusion process.

Moisture determination technique

The relationship between the equilibrium moisture content (w) and the relative air humidity (w_0) at a constant temperature was determined by means of statistical strain-gauge method [13].

The experiment used the desiccators with the prone-leak corks, and the glass weighting bottles with a diameter of 40 mm. The weighed sample in the amount of 4 g was transferred to the glass weighting bottle and reduced to constant weight by drying at a temperature of 105°C, then these bottles were placed in the desiccators to moisten the product. To maintain the required relative humidity of the air, the authors put 450 mL of a sulfuric acid solution of 80% concentration in the desiccators and tightly put the cork on them.

The hygroscopic properties of extrudates were studied within the limits from 10% to 100% of the air relative humidity and at a temperature of 20°C in the desiccators. The authors weighed the glass weighting bottles with samples using an electronic digital analytical balance SF-400C model (Toms, Qilin, China) with a weighing accuracy of 0.01 g.

The authors of the article took the weighting bottles out of the desiccators every 24 hours, put down the cover on them for a while and weighed on an electronic digital analytical balance model SF-400C and then placed it back in the desiccators. Each weighing allowed us to determine the amount of moisture absorbed by 1 g of extrudate. For each sample, the authors studied the moisture sorption process until reaching the constant mass. The constant mass of the samples, however, indicated the establishment of a equilibrium state of humidity in the sample.

For the accuracy and statistical evaluation of the results obtained, the authors took three samples of extrudates for each value of relative air humidity. The initial moisture content of the samples was determined by the drying method, which involves determining the difference between the masses of the samples with the initial moisture content and the samples dried to a constant mass.

Statistical analysis

To analyse the test parameters (the moisture content of starch paste, gelatinization point, starch paste transparency, starch paste embrittlement temperature, starch paste modulus of elasticity) of extrusion products, there was carried out a statistical analysis of the data obtained, and the reliability of these data was evaluated by method of mathematical statistics T-test, using the Windows IBM SPSS Statistics software program version 20.0. (IBM Co., Armonk, New York, 2015). To describe the ordered sample, we used statistical functions of the average arithmetic value and the average standard error. We selected the value of reliability p <0.05.

Results and Discussion

It is known that foods contain large amounts of moisture. Moisture in foods is present in the bound and free states. The bound state is a condition in which water molecules are closely bound to the solvent, or to individual parts of its molecules. The bound water cannot be a solvent of some other substance, it is considered that it does not freeze at minus 20 degrees. The mass content of moisture of extrudates produced from starch-based raw materials varies due to many factors affecting it, such as the type of raw material, the properties of extrudates, term and conditions of storage, and so on [4,14-17].

In previous studies, the authors have established that moisture plays an important role in the production of extrudates of a porous structure produced from starch-containing raw materials. In particular, on the one hand, during the extrusion process, it serves as a plasticizer, adding of which affects the gelatinization point of the processed raw materials. On the other hand, upon the hydro-thermo-mechanical impact, while exiting the molding head, the plastic serves as a steam generator, ensuring the formation of a porous structure. The structure in turn affects the physicochemical properties of the product, in particular its ability to absorb moisture during its preparation or storage [1].

Based on experimental studies, Table 3 shows the change in moisture (moisture content) of extrudates over time at different ambient relative humidity. As shown in table, the process of moisture absorption by extrudates is intensive during the first two days, with a further increase in storage time leading to slight changes in the moisture content of extrudates. In six days, the moisture content practically remains unchanged, except for of extrudates which are stored at 100% relative humidity.

Storage time [days]	Ambient relative humidity [%]									
	10	20	30	40	50	60	70	80	90	100
2	5.0	8.5	10.7	10.0	12.5	12.8	11.3	12.1	14.2	17.5
4	5.2	9.2	11.2	12.0	12.6	13.2	13.0	14.2	16.5	21.4
6	5.2	9.8	11.6	12.4	12.8	13.4	13.8	14.8	17.8	24.0
8	5.2	9.8	11.6	12.4	12.8	13.4	13.8	15.0	18.5	26.6
10	5.2	9.8	11.6	12.4	12.8	13.4	13.8	15.0	18.5	28.0
12	5.2	9.8	11.6	12.4	12.8	13.4	13.8	15.0	18.5	29.3

Table 3: Change in the moisture content of extrudates over time during storage at different ambient relative humidity.

Figure 1 illustrates the influence curve of the equilibrium moisture content and ambient relative humidity of extrudates enriched with nut flour, which was constructed based on the experimental data from table 4. The dependence obtained is a typical isotherm of the moisture sorption process by biopolymers [15,16,18-20].

As shown in figure, with relative humidity between 0 and 35%, the equilibrium humidity curve has a small curvature with respect to the axis of ordinates. In this section, the isotherm reflects the adsorption process. In the range of 35% to 70% of ambient relative humidity, an almost linear relationship is observed between the equilibrium and relative humidity, at which time the moisture absorption occurs due to the adsorption and water saturation processes. A further increase in ambient relative humidity is characterized by a sharp increase in the equilibrium moisture content of

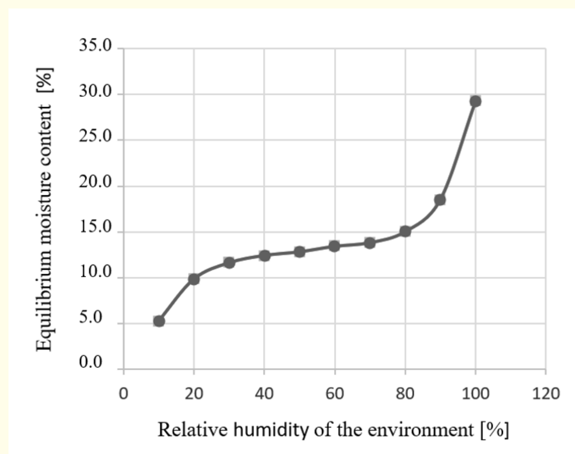

Figure 1: The dependence of the equilibrium moisture content of extrudates on ambient relative humidity.

Ambient relative humidity [%]	10	20	30	40	50	60	70	80	90	100
Equilibrium moisture content [%]	5.20	9.80	11.60	12.40	12.80	13.40	13.80	15.00	18.50	29.30

Table 4: The dependence of the equilibrium moisture content of extrudates on ambient relative humidity.

extrudates, with the isothermal branch ascending along the axis of ordinates, indicating the capillary condensation process of water vapor [4,6-8,20].

For a clearer idea and description of the transition of moisture retention in products from one form to another, Egorov [Moscow, Kolos, 1984] proposed the following relationship

$$W = a + b \cdot \left[\lg \frac{1}{1-\frac{P}{P_0}}\right]^{1/2} \text{-------- (1)}$$

Where W - equilibrium moisture content of product, (in percent); $\frac{P}{P_0} = \varphi$ - ambient relative humidity (in percent); a and b - constants depending on the sorption process (Table 5).

Ambient relative humidity φ [%]	Coefficients a, b		Correlation factor r
	a	b	
0.10 - 0.35	0.360	-0.021	0.98
0.35 - 0.75	0.060	0.035	0.96
0.75 - 1.00	0.213	-0.028	0.99

Table 5: The values of the coefficients a and b for the Egorov's formula.

Figure 2 illustrates the graph of the relationship between the equilibrium moisture content of extrudates and ambient relative humidity along the Egorov's coordinates, which was constructed on the basis of data given in table 6. The graph consists of three linear sections, which depict the areas of adsorption, saturation and capillary condensation processes, respectively. The results obtained are close to the results obtained by the authors, who studied the issues of moisture sorption by the rice- and oat-based extrudates [6,8,16,20].

It is known that the moisture retention energy in the product (extrudates) E [J·kg^{-1}], at a constant temperature, is numerically equal to the work required to remove one mole of moisture from extrudates

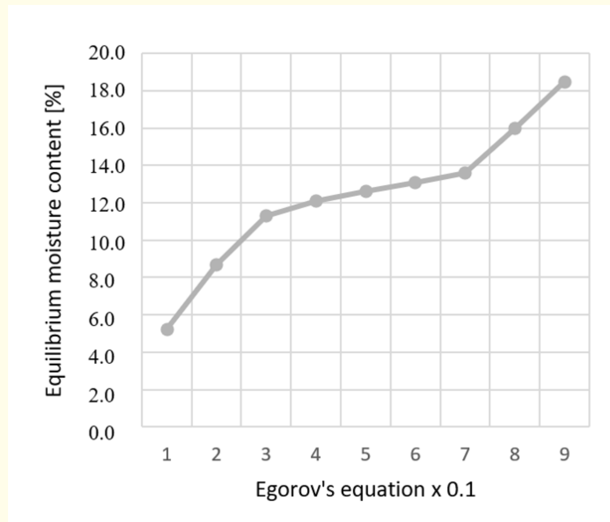

Figure 2: Sorption isotherm in the coordinates of the Egorov's equation.

Ambient relative humidity [%] $\varphi=P/P_0$ [%]	0.10	0.20	0.30	0.40	0.50	0.60	0.70	0.80	0.90
$\lg \frac{1}{1-\frac{P}{P_0}}$	0.214	0.351	0.394	0.47	0.549	0.631	0.723	0.836	1.00
The equilibrium moisture content of extrudates [%]	5.2	8.7	11.3	12.1	12.6	13.1	13.6	16.0	18.5

Table 6: The dependence of the equilibrium moisture content of extrudates on ambient relative humidity in the Egorov's coordinates.

E = R · T · ln φ -------- (2)

Where E - the moisture retention energy in extrudate (Joule in kilograms); T - an ambient temperature T = 193 K, R - universal constant, R = 8,314 (Joule in (mole Kelvin)); φ - ambient relative humidity (in percent).

Taking into account the experimentally obtained values of the equilibrium moisture content of extrudates, in the formula (2), the authors of the article calculate the values of the moisture retention energy in the product, on the basis of which we construct the corresponding relationship graph (Figure 3). As can be seen from the graph, with increasing equilibrium moisture content (Figure 3-a), the amount of moisture retention energy in the extrudates decreases, in particular from $33.11 \cdot 10^5$ $J \cdot kg^{-1}$ to $0.1 \cdot 10^5$ $J \cdot kg^{-1}$ (Figure 3-b).

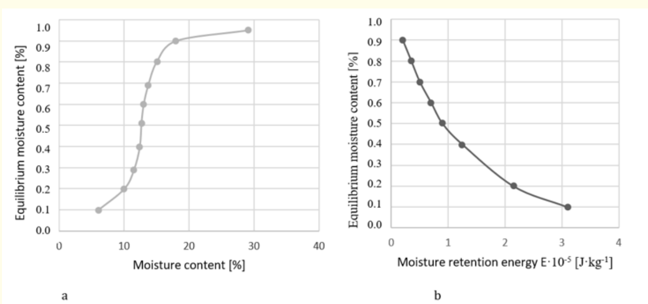

Figure 3: Dependence of equilibrium moisture content (a) and moisture bond energy (b) on the relative moisture content.

Studies have shown that the process of moisture sorption in extrudates is reflected in the interaction of water molecules with the biopolymer binding centers, as well as in changes related to the structure of the extrudates, and these processes are in line with the findings of the authors of [5,7]. It should be noted that change in moisture content in the samples depends on ambient relative humidity, with the minimum moisture absorption by extrudates occurring at 10% relative humidity, when the equilibrium moisture content reaches 5.2%. Thus, since the minimal physicochemical transformations in the samples the authors of the article studied (in starch gels) take place under conditions of humidity less than 5-7%, it can be concluded that extrudates with porous macrostructures, in particular, the extrusion products based on corn grits and enriched with nut flour should be stored at ambient relative humidity less than 10%.

Conclusion

The analysis of the conducted studies allows us to conclude that at 60 - 80% of ambient relative humidity, which is typical for food storage facilities, the equilibrium moisture content of extrudates enriched with nut flour is 13 - 15% (more than 7%), making it necessary to store these products in tight packing, where the microclimate of less than 10% relative humidity is created and maintained, under which the equilibrium moisture content of the product does not exceed 5.2% (in this case, the physico-mechanical characteristics are preserved which, as shown by previous studies, do not change and ensure the quality of the product), since high humidity leads to a deterioration of the quality of products with the same structure, in particular, changes in color and taste, as well as reduction in crispness.

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