EFFECT OF PROCESS PARAMETERS ON THE FUNCTIONAL AND PHYSICOCHEMICAL PROPERTIES OF EXTRUDATES ENRICHED WITH STARCH-BASED NUT FLOUR

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ABSTRACT
Widening the range of products produced on the basis of agricultural raw materials and improving the quality of these products and increasing their nutritional value represent urgent challenges. Therefore, the production of new mass consumption products with high nutritional and biological value brings to the fore the use of local nut flour as an enriching supplement in innovative technological processes. The high nutritional value of nuts (nuts, walnuts, and peanuts) is due to their chemical composition, including lipids, a large amount of soluble proteins that are well absorbed by the human body, sufficiently large quantities of vitamin B1 and a small amount of vitamins PP and E. It is known that in peanut grains, lipids have a balanced composition of fats and acids, as well as sufficiently large amounts of essential amino acids, which makes their protein composition closer to that of animal proteins. This study considers the influence of thermoplastic extrusion parameters on the functional and physicochemical properties of extrudates in their formation process. The technological and design parameters of the process and their variation ranges are based on studies conducted on model systems. The ratio of the extrusion mixture components (formulation) is also developed. Based on the methodology for multifactorial experimental design, the variation of the volume weights, expansion rates, and mechanical specific energy expenditure of porous extrudates enriched with starch-based nut flour is studied. It has been established that the best quality indicators of the products are achieved with the minimum volume weight and the maximum expansion rate.

Keywords: extrudate; process; starch; walnut; parameter

INTRODUCTION
Extruded products with different compositions and functional properties are produced on the basis of starch-containing raw materials using various plant and animal supplements. We have experimentally substantiated the use of non-traditional raw materials, namely, nut flours, as enriching supplements. This will expand not only the range of these products, but will also improve their quality and nutritional value. (Sesikashvili, Zverev and Berulava, 2018).

Based on previous studies, we have investigated the influence of raw materials in shaping the structure of extrudates and conducted studies on extrudate model systems, using starch gel from various origins. This article describes the process of producing extrudates by the method of thermoplastic extrusion as a thermotropic process for the formation of biopolymer gels in the stream (Tsagareishvili et al., 2019). The study of the properties of the obtained gels allows us to identify the moisture function in the process of thermoplastic extrusion, as well as during the storage of the obtained product. It is established that:

• During the process of extrusion, moisture determines the gelatinization point of the processed raw materials, whilst also influencing the shaping of the structure of extrudates (Tsagareishvili et al., 2019).

• Minimal physicochemical transformations occur in gels in an amorphous state, that is, when their moisture content is minimal. Therefore, the storage of porous extrudates is recommended at low (4–7%) moisture contents (Tsagareishvili et al., 2019).
Based on a literature review, it can be concluded that in addition to the composition (formulation) and technological parameters (moisture content, temperature and processing time), the shaping of the structure of the extrudates is also influenced by the parameters of the thermoplastic extrusion process (technological and design parameters).

Currently, the development of new formulations for products manufactured by the method of thermoplastic extrusion is mostly based on the empirical selection of the components of processed raw materials and the process operating and design parameters. In the best-case scenario, the systems analysis method is used for the multifactorial process (Van Lengerich, Meuser and Pfaller, 1989; Meuser, 1984).

(Meuser, 1984) showed that the functional properties of the product depend on the system parameters of the process, with the latter being a function of the process parameters (moisture content, temperature, auger speed, die hole diameter and pressure on the auger).

Therefore, the task of studying the structure of extrudates is based on studying the forms of the isotropic and anisotropic microstructural forms of products based on starch, proteins or their mixtures. As shown in the thermoplastic extrusion process scheme (Figure 1), the raw materials to be processed in the mixing zone are moistened, mixed and by means of the auger, they move into the channel and in the melt zone transform into a plastic state. Studies by (Van Lengerich, Meuser and Pfaller, 1989; Meuser, 1984) note that during the process of biopolymer flow movement, under the action of shear deformation at the end of the cylinder and in the forming die, there occurs the process of especially intensive structuring of the melt. After leaving the forming die, the product is finally formed.

In order to explain the mechanism for shaping the anisotropic structure of extrudates (Harper, 1986), a concept was proposed where macromolecules change their position under the action of shear deformation when raw materials transform into a plastic state during the gelatinization of starch and the denaturation of proteins. During the melt cooling process, the macromolecules bind and link together.

Based on the above, the purpose of this study is to investigate the influence of the extrusion process parameters on the functional characteristics of the extrudates (volume weight – \( \rho \) and expansion rate - \( \text{Exp} \) and physicochemical properties (mechanical specific energy - \( E \)).

**SCIENTIFIC HYPOTHESIS**

Based on the studies conducted, the influence of thermoplastic extrusion process parameters (\( W, T, n, s \) and \( d \)) on the formation of the functional (\( \rho \) and \( \text{Exp} \)) and physicochemical parameters (\( E \)) of extrudates will be determined. The advantage of using the systems analysis method for solving the optimization problem to produce functional products with useful properties will be shown.

**MATERIAL AND METHODOLOGY**

For the purpose of carrying out experimental studies, in accordance with the formulations, we selected the following materials: cornflakes – GOST 6002-69; corn starch - GOST 7697-82; walnut, peanut and table salt - GOST 13830-84.

For the extrudates, we used an extruder K - 30 (Ukraine), composed of an extrusion chamber, an auger kit, a forming die with different matrix diameters and a control panel.

The extruder chamber is a hollow cylinder with a 400 mm length and a 19 mm inner diameter of the auger, with six longitudinal channels designed to transport the mass processed when using raw materials with the flouiry structure. On the outer surface of the cylinder, there are two mounted heating elements. In general, the extruder has three zones: mixing, plasticizing and charging. From the top of the cylinder, there is a vertical single-screw proportioning feeder secured with a pyramid hopper. Inside the chamber, a single-thread variable-pitch auger is placed 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 different hole diameters. We varied the auger speed from 150 to 230 min\(^{-1}\) and measured the rotary speed by means of a tachometer. The temperature in the cylinder was measured using a thermocouple.

The testing methodology used in the experiments is as follows. In the preliminary studies, the ratio of the extrusion mixture components was determined by a sensory analysis of taste, color and rigidity:
- Cornflakes – 56.8%;
- Corn starch – 10%;
- Walnut – 12%;
- Peanut – 4.5%;
- Table salt – 0.7%;
- Moisture content of mixture with added water – 16%.

During the experiments, the moisture content of the mixture varied between 16% and 35%. The mixture components were hydrated before the extrusion process and settled at a temperature of 5 °C for 24 h. We extruded the mixture for the determined values of moisture content, temperature, auger speed, auger types and matrix size. A temperature of 70 °C was maintained in the feed zone of the extruder.

After the extruder was operated in a stable mode, we took samples and determined their volume weights and expansion rates, and in parallel, we calculated the mechanical specific energy expenditure.

The volume weights of the extrudates were determined using vessels of a previously-known capacity of \(0.5 \cdot 10^{-3}\) dm\(^3\) that were filled with extrudates and then weighed on an analytical balance. The volume weight was calculated by the following formula (1):

\[
\rho = \frac{G}{V} \quad (1)
\]
where G is the weight of the extrudates, kg, and V is the volume occupied by the extrudates, m³.

The expansion rate of the extrudates was calculated as follows (2):

\[ \text{Exp} = \frac{D}{d} \]  

(2)

where D is the extrudate diameter, mm, and d is the matrix die hole diameter, mm.

The mechanical specific energy was calculated using the formula (3):

\[ E = \frac{M \cdot n}{Q} \]  

(3)

where M is the auger torque, nm, N is the auger speed, min⁻¹, and Q is the extruder capacity, kg/h.

To further optimize the process of obtaining the base product, we used a mathematical method multifactorial experimental design (Grachev, 1979). The main factors for obtaining the base product were the moisture content of the extrudate mixture (W), the temperature in the forming die of the extruder (T), the auger’s number of rotations (n), the charging degree of the extruder (S) and the matrix diameter (d). According to (Grachev, 1979), in order to further optimize the process in the case of five-factorial experiments, it is necessary to conduct 25 experiments and vary each factor at five levels. The factor variation range was chosen on the basis of literature reviews and studies that we conducted previously on model systems. The factor variation range is given in Table 1.

The experiment performance grid is shown in Table 2.

### STATISTICAL ANALYSIS

To analyze the test parameters (the moisture content of the starch paste, gelatinization point, starch paste transparency, starch paste embrittlement temperature and starch paste modulus of elasticity) of the extrusion products, a statistical analysis of the data was carried out and the reliability of the data was evaluated by a T-test using the Windows IBM SPSS Statistics program (version 20.0). To describe the ordered sample, we used the statistical functions of the average arithmetic value and the average standard error. A graphical interpretation of the results was made using Microsoft Excel. Figure 2, Figure 3 and Figure 4 illustrate the data of typical tests and each value is an average of at least ten determinations. We selected the value of reliability as \( \rho < 0.05 \).

### RESULTS AND DISCUSSION

As is known, any production technology requires establishing the process parameters. The extrusion process parameters are as follows: the moisture content of raw materials, the temperature in the extruder’s cylinder, the auger speed, and the extruder design parameters (the auger type and a matrix die hole diameter).

Based on the experimental studies, we have determined the relationship between the process parameters and the functional and physical characteristics of extrudates. The results of the studies are presented in Table 3.

Based on the experimental results, we have constructed the experimental curves for the relationship between the process parameters and the functional and physical characteristics of extrudates.

Figure 2 illustrates the influence of the process parameters on the volume weight of extrudates. As the graphs show, the moisture content of the mixture has a particular influence on the volume weight. The best indicator of this functional characteristic in the 15-20% moisture content range. Further increase in the moisture content results in an increase in the volume weight, which worsens the quality of extrudates - the extrudate becomes more rigid. All other parameters in the same moisture content range do not adversely affect the volume weight variation. The end product is more airy and tender.

Enrichment of rice-based extrudates with the salts of calcium reduces the volume weight from 0.224 to 0.126 g·cm⁻³, and the expansion index from 3.21 to 2.93 (Janye and Singhal, 2018). Sugar crystallization during the thermo-hydro-mechanical processing of extrudates based on corn starch with sugar-containing supplements reduces the degree of expansion of extrudate (Wang, Gu and Ganjyal, 2019). Infusion of nitrogen injection during extrusion significantly increases the degree of expansion of extrudate and its porous structure (Li, Masatcioglu and Koksela, 2019).

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**Table 3** The influence of the process parameters on the functional and physical characteristics of extrudates

| #  | Functions | 1     | 2     | 3     | 4     | 5     |
|----|-----------|-------|-------|-------|-------|-------|
| 1  | ρ (W) 10⁻³ | 0.160 | 0.144 | 0.225 | 0.311 | 0.360 |
| 2  | ρ (T) 10⁻³ | 0.234 | 0.214 | 0.218 | 0.234 | 0.300 |
| 3  | ρ (n) 10⁻³ | 0.222 | 0.236 | 0.260 | 0.272 | 0.210 |
| 4  | ρ (S) 10⁻³ | 0.262 | 0.244 | 0.210 | 0.234 | 0.250 |
| 5  | ρ (d) 10⁻³ | 0.228 | 0.218 | 0.241 | 0.245 | 0.268 |
| 6  | Exp (W)    | 1.72  | 2.52  | 2.2   | 2.0   | 1.56  |
| 7  | Exp (T)    | 1.68  | 1.88  | 1.92  | 2.01  | 2.51  |
| 8  | Exp (n)    | 1.95  | 1.78  | 1.85  | 1.95  | 2.47  |
| 9  | Exp (S)    | 1.34  | 1.96  | 2.14  | 2.22  | 2.34  |
| 10 | Exp (d)    | 2.25  | 2.43  | 2.11  | 1.90  | 1.31  |
| 11 | E(W) 10⁻³  | 7.725 | 7.197 | 6.801 | 6.450 | 4.332 |
| 12 | E(T) 10⁻³  | 7.235 | 7.568 | 6.930 | 6.720 | 4.052 |
| 13 | E(n) 10⁻³  | 4.950 | 5.212 | 6.803 | 7.015 | 8.525 |
| 14 | E(S) 10⁻³  | 4.514 | 5.520 | 6.529 | 7.502 | 8.440 |
| 15 | E(d) 10⁻³  | 8.110 | 8.052 | 7.603 | 5.224 | 3.516 |
The addition of coconut to extrudates produced from corn and rice flour reduces the degree of expansion, increases the volume weight and is characterized by relatively dark color, but they were rich in protein, minerals, and have antioxidant properties (Arivalagan et al., 2018).

Many studies (Skamniotis et al., 2018; Beck et al., 2018; Do Carmo et al., 2019; Da Silva Teba et al., 2017; Singh et al., 2019) have reported similar functional characteristics of extrudates enriched with various supplements and their results concur with ours.

Figure 3 illustrates the influence of the process parameters on the expansion rate of extrudates.

It is known that the greater the expansion rate of the extrudates, the greater the number of porous masses and the better their visual side and organoleptic characteristics. Compared to other parameters, the moisture content of the raw materials and the matrix die hole diameter have a greater influence on the expansion rate of the extruder. The maximum expansion rate is observed in the case of a 20% moisture content and a matrix die hole diameter of 3 mm.

In this case, the relatively low values of the volume weights and expansion rates of the extrudates, compared to the similar data of extrudates based on pure starch or corn flour, is explained by the supplement of nut flour, due to the fat content, which partially impedes the extrusion process.

Enrichment of extrudates based on corn flour with barley, fondant apple and sugar-beet reduces the degree of their expansion, deteriorates their structure, increases their density and firmness, while addition of 1% pectin improves the degree of their expansion (Ačkar et al., 2018). When studying the physical properties of extrudates produced corn and yellow chickpea, it has been established that the best results were achieved by thermoplastic extrusion process parameters as follows: cell temperature - 158.64 °C, auger speed - 372 m\(^{-1}\), moisture content - 18.4% (Jacques-Fajardo et al., 2017). Regression analysis of the production of functional extrudates with high nutritional value based on corn flour by adding of dry broccoli or olive paste extrudates revealed that the increase in the concentration of broccoli or olive paste, as well as reducing the temperature and auger speed led to obtaining more dense extrudates with lower density.

At a moisture content of 14% and a 4%-concentration of product and the, extrudates had the highest expansion ratio at an auger speed of 250 m\(^{-1}\) (Bisharat et al., 2013).

The relationship between the expansion rate of extrudates and the process parameters has been extensively studied (Arivalagan et al., 2018; Sharma, Srivastava and Saxena, 2019; Carvalho et al., 2010; Zhang et al., 2020; Wang, Gu and Ganjyal, 2019). These studies confirm the accuracy of the tests that we conducted.

Figure 4 illustrates the influence of the thermoplastic extrusion process parameters on the mechanical specific energy. The plots clearly show the influence of each process parameter on mechanical specific energy expenditure, in particular, the increase in the rotary speed and in the charging rate of extruder results in increasing the mechanical specific energy, while the increase in the moisture content, temperature and matrix die hole diameter leads to a downward trend in the share of the mechanical specific energy. The results obtained concur with the previous studies (Jacques-Fajardo et al., 2017; Sharma, Srivastava and Saxena, 2019; Carvalho et al., 2010; Beck et al., 2018).
CONCLUSION

This work allows us to understand the physical nature of the influence of each factor affecting the thermoplastic extrusion process on the functional and physical characteristics of extrudates enriched with nut flour. By varying the process parameters, studies of the volume weights and the expansion rates show that they are inversely proportional to each other. In particular, the greater the extrudates expansion rates, the less their volume weights. In addition, the obtained products are airier, more tender and have better nutritional and consumer values. Therefore, in order to optimize the process of producing extrudates with a porous structure, it is necessary to solve the optimization problem: \( \rho(W; T; n; s; d) = \min; \exp(W; T; n; s; d) = \max \).

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