

## THE INFLUENCE OF THE MOISTURE CONTENT OF RAW MATERIALS ON THE STRUCTURING OF THE EXTRUDATES

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### ABSTRACT

The article presents the results of studies on the model systems of extrudates conducted with a view to determining the function of moisture during the process of forming the structure of starch pastes. There was studied the influence of the moisture content of raw materials on a starch gelatinization point. Studies showed that 15% moisture content in raw materials is sufficient for its constituent phase – starch gelatinization, as well as for the transition of the whole mass to a fluid-viscous state. Further increase in the moisture content is accompanied by a decrease in a gelatinization point. In order to study the influence of moisture on the formation of a porous structure of extrudates, we studied the relationship between the different-type starch pastes and the degree of its transparency and its embrittlement temperature. It has been found that during the process of thermal and mechanical impacts, there occurs the process of the formation of a structure of starch pastes, in particular, samples with the different moisture contents can have an amorphous or crystalline structure. There has been established the relationship between the moisture content of raw materials on the modulus of elasticity of starch pastes based on them. The modulus of elasticity of samples was determined one hour (cooling time to room temperature) and one week after obtaining the starch paste. The above studies showed that minimal physico-chemical and mechanical transformations occur in starch pastes, which are in an amorphous state, that is, in the conditions of a low moisture content. We have established that the moisture content of raw materials, on the one hand, ensures the transition of a high-dispersive phase to a fluid state, or implementing the ex process of extrusion, and on the other hand, influences on the formation of a porous structure in the extrudates.

**Keywords:** extrusion; moisture; gelatinization; structure; starch; paste

### INTRODUCTION

The process of thermoplastic extrusion in the food industry is a new innovative technology, which is distinguished by high productivity and efficiency. It provides complex processing of raw materials, i.e. the processes of mixing, hydrothermal processing and structure formation occur in one machine (Berk, 2012). It allows us for producing produce a wide selection of foods with high biological and nutritional values and balanced amino acid composition. The range of products obtained according to this progressive technology includes many commodities (Berk, 2012; Saravacos and Kostaropoulos, 2016; Martirosyan, 2013; Kraus, 2004), which in turn are classified into three groups:

- Porous breakfast cereals, dry concentrates, dried biscuits and other similar products;
- Anisotropic analogues of meat and fish products;
- Isotropic instant noodles.

In order to determine the optimum conditions for hydrothermo-mechanical processing of the mass undergoing treatment, as well as to produce high quality products, it is

necessary to study the natures of the change in physico-chemical properties of the main components of raw materials during the extrusion process.

The base raw materials that are subject to extrusive processing for the production of porous products are the starch-containing raw materials, such as corn flour, wheat flour, rice flour, oat flour, and so on.

The main components of raw materials listed are starch and protein, which have a particular impact on their processing conditions and the quality of extrudates. (Karpov, 2000; Alekseeva, 2007).

It is known that starch, whose content in raw material is 65 – 80%, is not a chemical individual, but rather two main fractions, the biopolymers of amylose and amylopectin. The physicochemical properties of the starch are associated with primarily with its polysaccharide composition, the sizes of the molecules, as well as firmness and the compact arrangement of its grains.

As we have mentioned, protein is the second main component of raw materials. During the extrusive processing of starch-containing raw materials, proteins that make up about 15 – 20% of their total mass undergo denaturation because of a

hydro-thermo-mechanical impact resulting in physico-chemical changes in them, so starch gelatinization and denaturation of proteins occur in extruding raw materials during the process of thermoplastic extrusion (Litviak, 2013; Karpov, 2000; Van Lengerich, Meuser and Pfaller, 1989).

The third main component of extruding raw materials is water - moisture. According to literature sources (Litviak, 2013; Karpov, 2000), the moisture content of extruding raw materials is 15 – 40%. This amount of moisture affects the gelatinization point of the mass undergoing treatment, viscosity of the fluid mass, the functional properties of extrudates, and their storage conditions. In other words, moisture affects both the technological parameters of thermoplastic extrusion and the consumer properties of product, however, it is important to distinguish moisture as a functional additive for raw materials and the moisture content of product during storage of extrudates.

The analysis of literary sources has shown that there are very few scientific studies in this area. Based on the above, the goal of research is, using an example of the model and highly concentrated systems (starch-water) of extrudates, to study the impact of the moisture content of raw materials on the formation of their structure.

To achieve this goal, the following objectives should be resolved:

- Studying the impact of moisture on the starch gelatinization point and on the properties of starch paste (gel);
- Studying the hydro-thermo-mechanical impact on the structure formation of starch paste (gel).

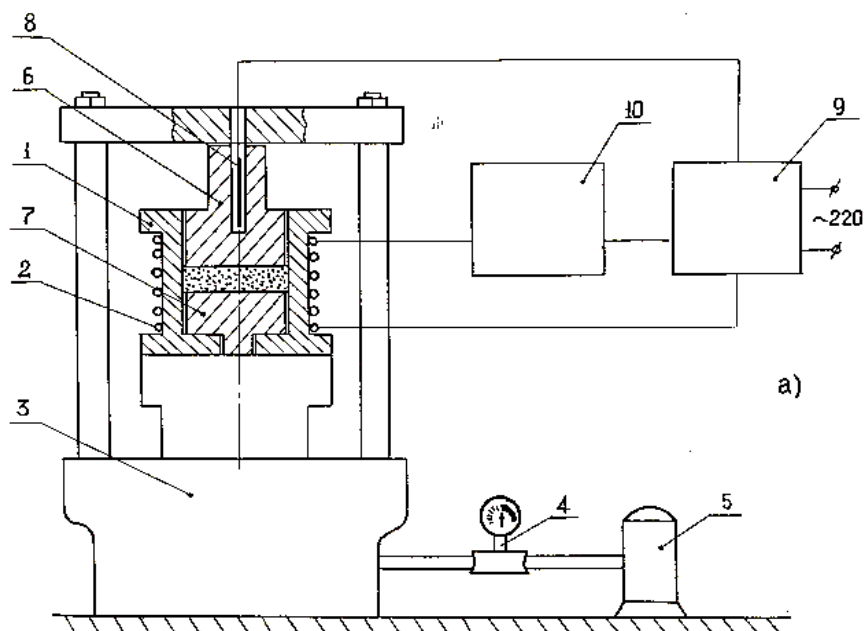
### SCIENTIFIC HYPOTHESIS

The process of obtaining extrudates by thermoplastic extrusion method can be seen as a thermotropic process of starch paste formation of biopolymers in the flow. It may be presumed that the study of a thermotropic process of starch paste formation in the highly concentrated systems containing 8 – 40% of moisture, and the study of the properties of obtained starch paste will provide information on the function of water in the process of thermoplastic extrusion.

### MATERIAL AND METHODOLOGY

For research, to produce starch paste, we used corn starch GOST 7697-82, potato starch GOST 7699-78 and drinking water GOST 2874-82. To conduct research on starch paste samples, we developed and created the test bench (Figure 1). The bench consists of a working chamber body 1, equipped with a spiral heating device 2, press 3, a temperature control unit 9 and the laboratory autotransformer 10.

The working chamber is a cylinder with an inner diameter of 26 mm. At the lower part of a cylinder, there is placed the lower fixed punch 7, on which the portion of test sample is sprinkled, and from above, the upper movable punch 6 is mounted. When current of a certain quantity passes through heating spiral, the working chamber is heated to the required temperature. We maintained the required temperature by means of temperature control unit, while the current strength required for the heating element, we generated by the laboratory autotransformer (LATR 1). The temperature inside the cell was monitored by a Ni-Cr thermistor with a multimeter DT9208A, and red the time with an electronic timer.



**Figure 1** The test bench scheme: 1 – a working chamber body; 2 – a spiral heating device; 3 – press hydrocylinder; 4 – pressure-measuring instrument; 5 – pump; 6 – upper punch; 7 – lower punch; 8 – thermal converter 9 – temperature control unit; 10 – autotransformer.

We counted the assembled chamber with the portion placed inside it, in a press, which by acting on the upper punch, creates and retains some pressure in a working chamber. Samples are obtained by the following method: starch samples with different moisture content were placed in a working chamber under pressure of 30 kg·cm<sup>-2</sup>, then we heated it to the temperature of 120 °C and retained for 5 minutes. After that, the system was cooled to room temperature and the obtained starch paste was taken from a working cylinder. The moisture content of starch paste was determined by drying method at the temperature of 105 °C until obtaining the constant mass. The mass was determined by means of analytical electronic scales.

We used the same test bench to determine the starch gelatinization point. We placed 10 g of starch sample with different moisture content in a working chamber between the punches under pressure of 30 kg·cm<sup>-2</sup> and heated it in a chamber until the pressure in a chamber is dropped. We recorded the temperature corresponding to this moment, which corresponds to the point of transition of a friable mass into a fluid mass. The transparency of starch paste of various origins was determined by means of a spectrometer „Specord UV-VIS“ (Karl-Zeiss, Jena, Germany). In a comparable cuvette filled with silicone oil, we placed 20 mm thick sample of test starch paste. The transparency percentage of test samples was determined according to wavelength of a beam spectrometer. In order to study the glass transition temperature or fragility of starch paste, we investigated the relationship between the relative elongation of samples and a temperature, under conditions of a scanning velocity of 5 °C·min<sup>-1</sup>. As the glass transition temperature, or the embrittlement temperature, we have taken the intersection point of the linear values of the relative elongation and the temperature of starch paste. The tests were carried out on a thermomechanical analyser "TMA - 973" (USA).

We studied modulus of elasticity of different starch paste samples on a universal test machine "Instron TM-SM-1" with a ball indenter by method of penetration. Modulus of elasticity of starch pastes was calculated according to the following formula:

$$G = \text{tg}\alpha \cdot (K \cdot \sqrt{R})^{-1} \quad (1)$$

where  $\alpha$  – angle of descent of a function  $P = f(h^{1.5})$ ;

$P$  – the axial load, N;

$h$  – insertion depth in the indenter, mm;

$K$  – non-dimensional coefficient,  $K = 10$ ;

$R$  – indenter radius, mm,  $R = 2.5$  mm.

We placed the starch paste samples with various moisture contents on the machine table. We chose the mode of operation of machine by the following parameters: self-recorder chart's velocity  $v_1 = 1000$  mm·min<sup>-1</sup> and the velocity of traverse  $v_{tr} = 5$  mm·min<sup>-1</sup>, to which the indenter is fastened. The self-recorder describes the function  $P = f(h)$ . The insertion depth of the indenter in a sample was calculated from a ratio  $v_{tr}/v_1 = h_{tr}/h = 1/200$ .

We moved the  $P = f(h)$  function into the relationship  $P = f(h^{1.5})$ , and then we determined the tangent of an angle of descent of this function. By inserting the obtained value into the formula (1), we determined elasticity modulus of samples. The starch deterioration temperature was determined using a derivatograph Q-1500D (Hungary).

## STATISTIC 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 is conducted a statistical analysis of the obtained data, and the reliability of the obtained data was evaluated by method of mathematical statistics T-test, using the Windows IBM SPSS Statistics software program (version 20.0). To describe the ordered sample, we used statistical functions of the average arithmetic value and the average standard error. Graphical interpretation of the results was made by using Microsoft Excel. In Figure 2, Figure 3A, Figure 3 B, Figure 4, Figure 5A and Figure 5B there are presented the data of typical tests, and each value is an average of at least ten determinations. We selected the value of reliability  $p = 0.05$ .

## RESULTS AND DISCUSSION

It is known that the maximum temperature required for the process of thermoplastic extrusion and the moisture content of processing raw materials have a particular impact on the quality of extrudates, as well as on the physicochemical and structural characteristics of starch paste.

The effect of hydration changes of snacks depended both on the hydration level and the extrusion process conditions. As a result of storage the shrinkage was more pronounced in case of extrudates obtained from raw material containing 16% of water regardless of the extrusion temperature. The hardness of extrudates depended both on the feed moisture and hydration. Samples obtained from the raw material containing 20% moisture were harder than those prepared at the feed moisture of 16%. Based on the results of molecular dynamics study by low field NMR technique, a model of hydration was developed and critical hydration value of extruded snacks was calculated. Higher values of the critical hydration were obtained for snacks extruded at temperature 135 °C than for those obtained at 175 °C (Makowska et al, 2017).

In the extrusion of sorghum flour increasing feed moisture increased the peak gelatinization temperature ( $T_p$ ), the degree of gelatinization (%) and starch crystallinity (%) while it decreased the gelatinization temperature ranges ( $T_c - T_o$ ), starch gelatinization enthalpy ( $\Delta H_G$ ) and amylose-lipid complex (%) formation. With increasing die temperature, the degree of gelatinization and amylose-lipid complex formation increased and the starch  $T_p$ ,  $T_c - T_o$ ,  $\Delta H_G$  and crystallinity decreased (Morteza, Koocheki and Milani, 2017).

When studying the effect of leavening agents on lysine loss, determined by the furosine content in corn extrudates, it was found that furosine levels decreased by about 20% when the moisture content in the feed stuff increased from 22% to 26% (Masatcioglu, Ng and Koksels, 2014).

Response surface methodology for evaluation of functional and physical properties of corn half products was developed. Response variables are: water absorption index (wai), water solubility index (wsi), degree of gelatinization (dg), water activity ( $a_w$ ), specific volume and color properties of extrudates. increased screw speed decreased wai and  $a_w$  values and increased wsi values significantly, it has been established that specific volume of half products

was low for all extrusion combinations and negative correlated with screw speed, feed moisture and xg level. lightness and yellowness of half products were increased with increases in screw speed, feed moisture and xg level (Gümüşay, Şeker and Sadıkoğlu, 2019).

There was studied the effect of flour moisture content during extrusion on in vitro fermentation properties of whole grain oats. Extrudates were processed at three moisture levels (15%, 18%, and 21%) at fixed screw speed (300 rpm) and temperature (130 °C). After 24 h of fermentation, samples processed at 15% moisture supported lower *Bifidobacterium* counts than those produced at other conditions, but had among the highest *Lactobacillus* counts. (Brahma, Weier and Rose, 2017).

So given the fact that behaviour of extruding raw materials in the process of hydro-thermo-mechanical treatment is due to the continuous phase - starch properties, that is why we studied the relationship between the gelatinization point of starch of different origins and their moisture contents (Figure 2). The results obtained are consistent with the findings of studies conducted by above mentioned authors.

It is known (Litviak, 2013; Blanshard, 1987) that the process of starch gelatinization is accompanied by two events: the first one - diffusion between water and starch molecules; the second one - solubility of starch polysaccharides. In other words, the process of starch gelatinization is the process of the transition of a dispersive system to a viscous-liquid state.

As Figure 2 shows, if the moisture content in starch is up to 15%, the gelatinization point is lower than its decomposition point. Increasing the amount of moisture in the system causes a further drop of the gelatinization point. At 60% moisture content, there is a little link between the gelatinization point and the moisture content of starch. The

results obtained are in compliance with the author's research (Blanshard, 1987; Van Lengerich, Meuser and Pfaller, 1989).

During the process of heat treatment of starch of various origins, there occurs its structural modification, as our earlier studies show, during the process of heat treatment, the starch pastes may have an amorphous or crystalline structure.

There was studied the low moisture extrusion of rice starch fortified with pea protein and pea fibre. The addition of protein and fibre resulted in increased specific mechanical energy inputs, bulk densities as well as air cell densities. Microstructural analyses revealed that at the highest protein contents (42%), protein and starch were distributed in thin layers within the extrudate which is proposed to cause the decreased sectional expansion indices (Beckab et al, 2018).

Figure 3A and Figure 3B illustrate the relationship between the transparency of the potato and corn starch pastes and their moisture contents. Figure shows that this relationship is of a "S"-like nature, however the change in the transparency of the aging process is observed only if the moisture content exceeds 15%.

It is noteworthy that the turbidity (the reciprocal of transparency) (Litviak, 2013) of both corn and potato starch pastes at a low moisture content, is one degree lower than the turbidity of samples with 25% or higher moisture content. Considering "S"- like nature of the relationship between the transparency and moisture content of starch paste, it can be assumed that the transition from a highly elastic state to a glass amorphous state occurs when the water content decreases. It is therefore natural that in a high-elastic state, there occurs the process of starch paste aging, since the movement of macromolecular chains leads to an increase in the turbidity of starch paste and in the number of crystals.

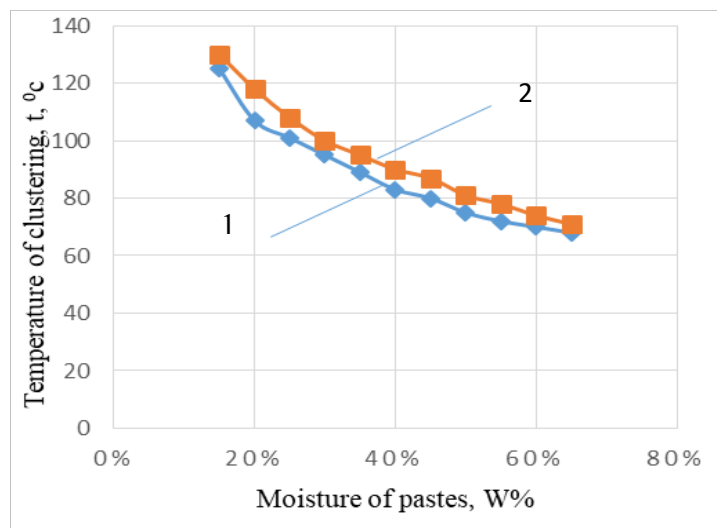
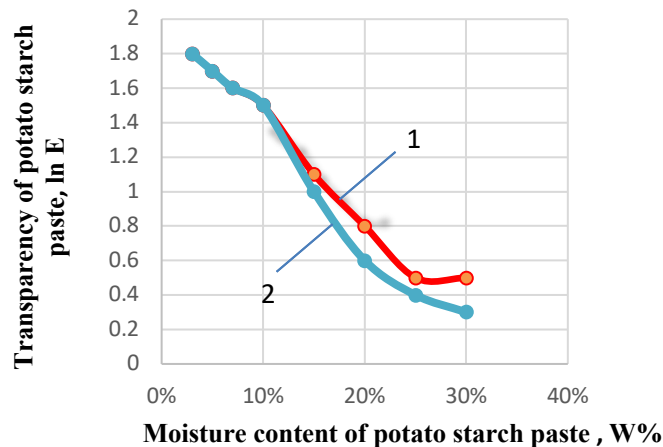
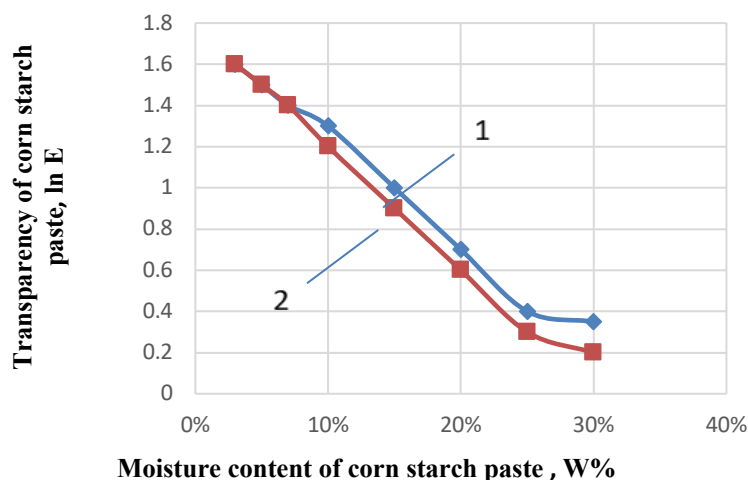


Figure 2 The relationship between the gelatinization point of the different-type starch pastes and the moisture content. 1 – potato starch; 2 – corn starch





**Figure 3A** The relationship of the transparency of the potato starch paste on their moisture contents. Note: 1 – potato starch paste one hour later; 2 – potato starch paste 14 days later.



**Figure 3B** The relationship of the transparency of the corn starch paste on their moisture contents. Note: 1 – corn starch paste one hour later; 2 – corn starch paste 14 days later.

Physical state and mechanical properties of extruded grain-based products were studied as a function of the sucrose content and relative humidity (RH) to evaluate how the presence of sucrose affects the glass transition temperature. The Young's modulus showed that water acts as an anti-plasticizer at low  $a_w$ , and a plasticizing effect at high  $a_w$ . The stability map may explain the brittle-dritile transition that occurred when it was lower (Masavang, Roudaut and Champion, 2019).

There was studied the effect of sucrose at concentrations ranging from 0 to 12.5 % wt. on the textural properties of extrudates of corn and wheat flours.

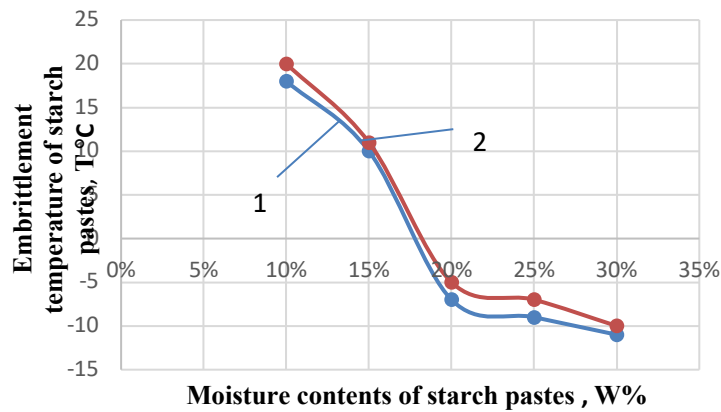
It has been established that (1) the internal structure of the extrudates evaluated by image analysis showed a reduction of the cell size with the sucrose whatever the flour; (2) the crispness of products evaluated by puncturing increased with the sucrose content until an optimum whatever the flour; (3) the addition of sucrose in corn and wheat mix reduced overall product expansion; and (4) increasing the amount of sucrose in the mix induced greater molecular degradation of corn starch than of wheat starch (Mezreb, et al, 2006).

Figure 4 illustrates the relationship between the embrittlement temperature of the potato and corn starch

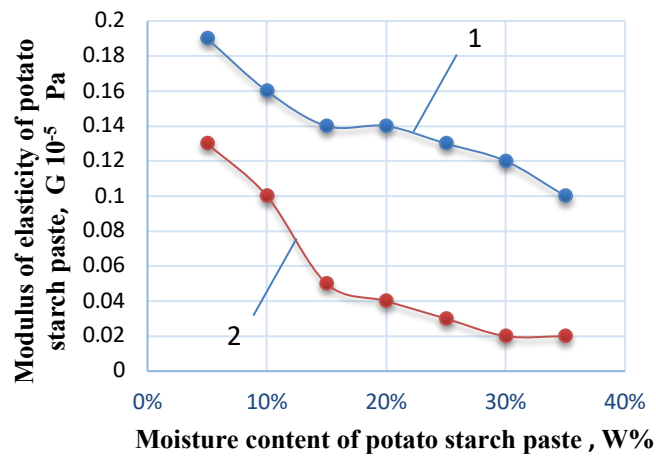
pastes and their moisture contents. Figure shows that in samples with a moisture content below 10%, the embrittlement temperature is higher than 20 °C, the increase in a moisture content of starch pastes results in a lower embrittlement temperature, for example in the case of 25 – 30% moisture content, the embrittlement temperature is -7 to (-11 °C), This means that moisture plays the role of a plasticizer in such a system, while the increase in its amount leads to increasing fluidity micromolecular chains. The difference in the embrittlement temperatures for the potato and corn starch pastes are due not only to their different natures, but also to production conditions.

The results obtained are consistent with the findings of studies conducted by above mentioned authors.

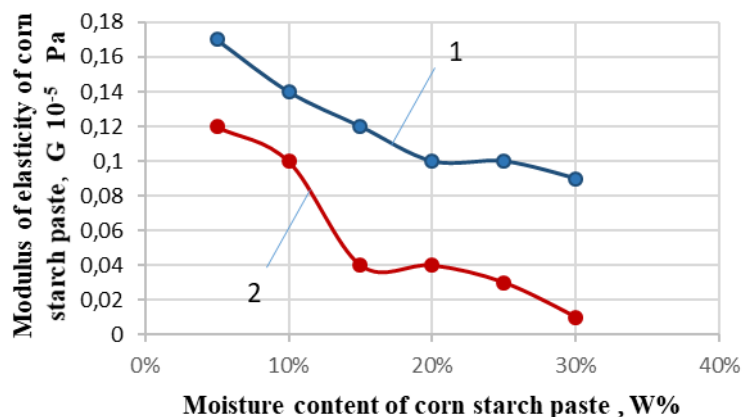
It has been established that grain refinement using a reduced-size aperture grid caused a narrowing of the particle size distribution. The smaller size of particles of the crushed extrudate resulted in obtaining the tablets with higher tensile strength. This, along with a narrowing of the particle size distribution, led to obtaining the more homogeneous tablets, therefore, to less weak places for crack propagation and to obtaining a more durable tablet (Hallam, Gabbott, 2019).



**Figure 4** The relationship between the embrittlement temperature of the different-type starch pastes and their moisture contents. Note: 1 – potato starch paste; 2 – corn starch paste.



**Figure 5A** The relationship between the modulus of elasticity of the potato starch pastes on their moisture contents. Note: 1 – potato starch paste 1 hour later; 2 – potato starch paste 14 days later.



**Figure 5B** The relationship between the modulus of elasticity of the corn starch pastes on their moisture contents. Note: 1 – corn starch paste 1 hour later; 2 – corn starch paste 14 days later.

There was studied the effect of feed moisture (12 – 16% wet basis), barrel temperature (90 – 110 °C) and screw speed (100 – 200 rpm) on the extrudates using a blend of oat, green pea and fenugreek seed flour and leaf powder properties like lateral expansion (LE), bulk density (BD), water absorption

index (WAI), water solubility index (WSI) and hardness was investigated. Desirable extruded products were obtained at moisture of 12%, 110 °C temperature and 200 rpm screw speed (Wani and Kumar, 2016).

Figure 5A and Figure 5 B illustrate the relationship between the modulus of elasticity of the potato and corn starch pastes and their moisture contents. This relationship is also a "S"-like nature, and the reduction in a moisture content increases the modulus of elasticity by one degree, while in the process of aging of the test starch pastes, the modulus of elasticity increases, regardless of the change in their moisture contents.

An adequate nature of the relationships between the transparency degree and the modulus elasticity of the test starch pastes, as well as the dependence on embrittlement temperature, indicate that in the starch pastes with modulus of elasticity is within the limits of  $10^4$  Pa, are in an amorphous state, while the starch pastes having the one degree lower modulus of elasticity, are in a high-elastic state (Litviak, 2013; Karpov, 2000; Abramov, 2011).

## CONCLUSION

Studies carried out in the highly concentrated systems "starch-moisture", which focused on the process of starch gelatinization, as well as the study of the properties and structure of starch pastes based on them, allow us for drawing conclusion on the function of moisture in the process of thermoplastic extrusion.

In the highly concentrated systems and starch pastes based on them, which represent the model of extrudates, moisture plays the role of a plasticizer, the increased number of which in a system leads to an increase in the movement and fluidity of the macromolecular segments and structural elements of starch pastes. This process is accompanied by the transition of starch pastes from an amorphous to a highly elastic state. It is noteworthy that in the aging process of starch pastes in a highly elastic state, their turbidity and elasticity limit are increasing and the process of crystallization is intensified, also, the increase in the moisture contents of porous extrudates affects the functional properties of product, in particular, unlike the highly-elastic state, the intensification of the crystallization process results in a decline in the strength of extrudates and disruption of a porous structure.

Thus, studies on the model systems allow us for drawing the following conclusions:

- Moisture, during the extrusion process, on the one hand, determines the temperature of the process of raw materials gelatinization, and on the other hand, influences on the formation of a porous structure in the extrudates.
- In the production of porous extrudates, the moisture content in raw material in the case of corn flour should be  $17 \pm 1\%$ , while the process temperature in the extruder should be  $140 \pm 5$  °C. In the case of potato flour, the moisture content should be  $15 \pm 1\%$  and the process temperature in the extruder should be  $130 \pm 5$  °C.
- Minimal physico-chemical transformations are observed in starch pastes, which are in an amorphous state, that is, when the the moisture content in starch paste is minimal, we can conclude that porous extrudates should be stored in the conditions of a low moisture content (4 – 7%).

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