

## STARCH TYPE EFFECT ON ORGANOLEPTIC, THERMOGRAVIMETRIC AND X-RAY DIFFRACTION INDICES OF EDIBLE FILMS AND COATINGS

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**Abstract.** The edible films from potato starch, chemically modified food starch refined from high amylose corn, carbohydrate polymer from a waxy corn and a special high-temperature dextrin refined from tapioca starch were studied according to organoleptic characteristics. The greatest elongation (108 %) and strength (47.6 MPa) were found to be with a film based on potato starch. The modified starches such as dextrin and tapioca starch retain amorphous of film structure better than others. The films from modified starch contain more crystallization moisture because there is no amylopectin in this starch. The obtained results are recommended to use for optimizing food production.

**Key words:** starch, thermogravimetric, X-ray diffraction, edible films and coatings.

### 1. Introduction

Edible films are packing materials that can solve several tasks: not to use synthetic polymer materials for bakery products; not to use sugar icing for gingerbreads and candy products and not to apply sugar on jelly products. The use of edible films for foods was proposed long ago [1–7], but they have not found massive applications in the manufacture of bakery and confectionery products yet. The natural polymers, like starch, pectin, gelatin and others are usually used for edible films production. It is most reasonable to use natural starch polymers as renewable raw materials from economic point of view. However, the question is what kind of starch is the most practical for making edible films, because nowadays there are a lot of modified starches as is stated in the European directive on food additives.

The literature provides data on study of films and coatings from starch [8], sago starch [9], pea starch [10],

potato starch [11], oatmeal starch [12], modified potato starch [13], native and modified yam starch [14], gelatin [15, 16], but there is no complex study of films from various modified and native corn and potato starches.

Edible films and coatings are investigated according to physical and mechanical parameters, as well as barrier properties (moisture and gas permeability) [17–19]. Thermal stability was also investigated in a stock of soy protein isolate-dodecyl sulfate sodium [20]. The influence of the whey protein solubility hydrolysis degree was explored in the film [21]. There is no data of complex analysis of edible films from the given raw materials and for the given indicators in the literature.

The purpose of the article is to study the edible films and coatings of various starch types according to organoleptic characteristics, to determine the amount of free and bound moisture by using thermogravimetric analysis, and changes in the amorphous structure of films using X-ray diffraction analysis.

### 2. Experimental

Edible films were made from such types of starch as corn starch (Vimal), potato starch (Vimal), chemically modified food starch refined from high amylose corn (E 1420, Ingredient), carbohydrate polymer of waxy corn (Ingredient), modified food starch (Ingredient), special dextrin refined from tapioca starch (Ingredient), modified starch from ordinary corn (Ingredient), special high-stable dextrin refined from tapioca starch (Ingredient), instant gelatin (E 441, Ingredient), urea (E 927b, Makrohim) or glycerol (E 422, Galette) were used as plasticizers.

The solution of edible coating was obtained by dissolving film formers in water at 348 K for 10 min under constant stirring till gelatin dissolved and starch gelatinised; then the plasticizer was added. After the film-forming solution was poured onto a Teflon surface (289 cm<sup>2</sup>) it was kept indoors until complete drying (24–36 h).

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All samples were made in the laboratory of the National University of Food Technologies (Kyiv, Ukraine).

A thermometer (TL-2, Standart-M Co) was used to determine the temperature of starch gelatinization during starch paste making process on a water bath in the ratio of 3 g of starch to 150 cm<sup>3</sup> of water.

Complex organoleptic index was calculated according to the methodology [22] and the characteristics [23]:

$$K_o = M_1 \frac{P_1}{P_1^b} + M_2 \frac{P_2}{P_2^b} + M_3 \frac{P_3}{P_3^b} + M_4 \frac{P_4}{P_4^b} + M_5 \frac{P_5}{P_5^b} \quad (1)$$

where  $P_1, P_2, P_3, P_4, P_5$  – indices that characterize organoleptic properties of the samples under study (taste, colour, smell, surface, appearance, shape), which vary from 0 to 5;  $P_1^b, P_2^b, P_3^b, P_4^b, P_5^b$  – the values of basic indices that characterize organoleptic properties of the samples ( $P_1^b = P_2^b = P_3^b = P_4^b = P_5^b = 5$ );  $M_1, M_2, M_3, M_4, M_5$  – the weight coefficients of corresponding organoleptic indices ( $M_1 + M_2 + M_3 + M_4 + M_5 = 1.0$ ).

Viscosity characteristics were determined using the “Reotest-2” rotary viscometer. The rheological properties of mixtures in volume of 10 cm<sup>3</sup> were investigated for different shear rates and temperature of (297±1 K) for a system of cylinders  $N$ . The measurement instrument error is ±4 %.

The viscosity ( $\eta$ , Pa) was determined according to the formula (2):

$$\eta = \frac{Z \cdot L \cdot 100}{D_r} \quad (2)$$

where  $Z$  – constant of a cylinder (tabular value);  $L$  – scale value of indicating device;  $D_r$  ( $\gamma$ ) – shear rate, s<sup>-1</sup>.

The shear rates  $D_r$  were taken from the reference table according to the number of rotations. The dynamic viscosity  $\eta$  for each measurement according to calculated share stress  $\tau$  and the corresponding value of  $D_r$  was calculated using the formula (3):

$$\eta = \frac{\tau}{D_r} \quad (3)$$

where  $\tau$  – shear stress, Pa;  $D_r$  – shear rate, s<sup>-1</sup>.

Tensile strength ( $TS$ , MPa) and elongation ( $E$ , %) in the control point was calculated (measured) by uniaxial stretching of a sample (10×2.5 cm) in one direction at 50 m/min on the F-1000 tensile testing machines. Films were analyzed in a climate room at a relative humidity of 53 % and temperature of 298 K. The sample length between the grips was 4 cm.  $TS$  was calculated by dividing the maximum load on the cross-sectional area of the film;  $E$  was expressed as a percentage of changes in the original length of the sample between the grips (4 cm), according to ASTM standard method D 882-88 [24].  $TS$  and  $E$  characteristics are given as an average value of 5 measurements.

X-ray diffraction analysis was carried out by the device DRON-3M with CuK $_{\alpha}$  radiation with Ni-filter;  $U = 35$  kV,  $I = 20$  mA; displacement angle of meter is  $\Delta 2\Theta = 0.04^\circ$ ; reading time of intensity is 3 s.

Thermogravimetric analysis was conducted by the Q-1500V unit, heating rate of samples was 10 K/min, sample volume was 80 mg, sensitivity was 100 mg,  $T_{room} = 293$  K,  $T = 1273$  K.

Statistical analysis. The experimental design was a completely randomized design with 11 treatments. All data were analyzed using SPSS version 17.0.0. for Windows (SPSS Inc., USA) with a one-way analysis of variance (ANOVA). Differences between means were compared by Duncan’s multiple range tests at a level of 0.05. Interaction between the composition of cheese milk and yield was studied using multiple regressions. All compositional analyses for each sample were carried out in triplicate.

## 3. Results and Discussion

### 3.1. Gelatinization Temperature

Film production involves starch gelatinization of a starch component as a film-former. It is known [25], that different types of starches have different gelatinization temperatures. The gelatinization temperature and time of studied starch types paste formation: corn starch (335–343 K), potato starch (331–339 K), chemically modified food starch, refined from high amylose corn (329–333 K), modified food starch (335–342 K), modified food starch refined from ordinary corn (333–341 K). The different starch gelatinization temperature is explained by the starch origin and kind of its modification.

The analysis of studied starch type gelatinization temperature allows selecting the mode of film matrix formation without excessive heat energy. The temperature of starch paste formation ranges from 331 to 343, depending on the type of starch. The data shows that a corn starch has the highest temperature of gelatinization.

### 3.2. Organoleptic Properties

The results of organoleptic quality evaluation of various starch types and dextrans edible films according to the complex quality index in arbitrary units from 0 to 1 are shown in Figs. 1 and 2.

In accordance with Fig. 1 the largest area has a profile film with a film forming agent – chemically modified food starch refined from high amylose corn. The smallest area has a profile film of modified edible starch.

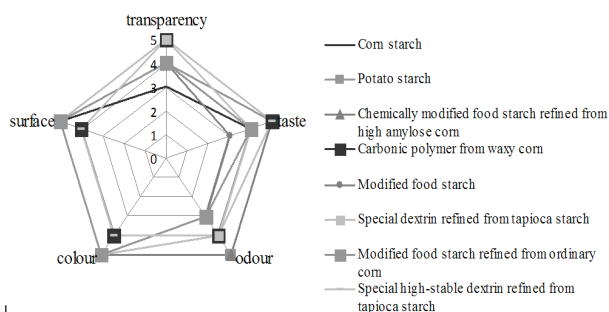


Fig. 1. Organoleptic evaluation of films quality based on different film-formers

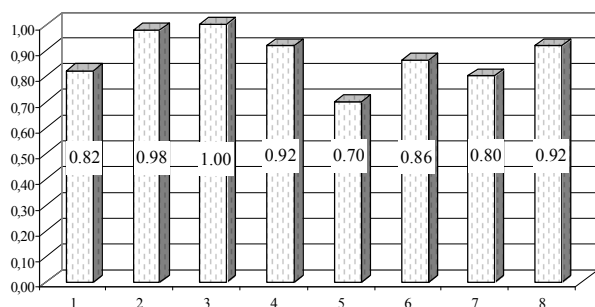


Fig. 2. Complex quality index of edible films: corn starch (1); potato starch (2); chemically modified food starch refined from high amylose corn (3); carbonic polymer from waxy corn (4); modified food starch (5); special dextrin refined from tapioca starch (6); modified from starch refined from ordinary corn (7) and special high-stable dextrin refined from tapioca starch (8)

According to the data obtained, the largest 1.0 points had the films based on chemically modified starch from high amylose corn. The potato starch films were slightly less significant – 0.98. The smallest value was obtained on the basis of modified edible starch, as there was an external taste and the smell. The intermediate results were obtained on the basis of other used film-forming agents, since not all of the films were transparent. In addition, some samples also showed an external smell and smell, which affected the number of points by individual indicators and, as a result, led to the indicated values of the complex quality index (Fig. 2). Therefore, it is most expedient to use films with a value of complex quality index of 0.9–1.0, since these values correspond to the quality of “excellent”. The films from potato starch, chemically modified food starch refined from high amylose corn, carbohydrate polymer from a waxy corn, a special high-temperature dextrin purified from tapioca starch are among them.

### 3.3. Viscosity Research

The nature of the rheological curve is influenced by such factors as amylose content in starch, types of starch, mineral additives content, pH of solution and temperature

of gelatinization [26]. The results of changes in the solution viscosity depending on the type of film-former (Pa·s): corn starch 0.009; potato starch 0.0247; chemically modified food starch refined from high amylose corn 0.0056; carbonic polymer from waxy corn 0.03; modified food starch 0.0067; special dextrin refined from tapioca starch 0.0032; modified from starch refined from ordinary corn 0.0056; special high-stable dextrin refined from tapioca starch 0.0034.

According to the obtained data the film solution with the use of potato starch has the highest viscosity (sample 1), because this starch has the highest amount of amylopectin – 77 %, while corn starch (sample 2) contains up to 72 % of amylopectin [27]. The tapioca starch doesn't contain amylose, but because dextrans from tapioca starch are used in the research, 1,6-glucosidic bond is broken and the length of chain is significantly shortened. As a result, the viscosity of such solutions is significantly lower than the viscosity of solutions with potato and corn starch (samples 6, 7).

### 3.4. Physical and Mechanical Properties

Physical and mechanical properties of films based on starch depend on the ratio of amorphous and crystalline structure. Characteristics of physical and mechanical properties of various film-formers films are shown in Fig. 3.

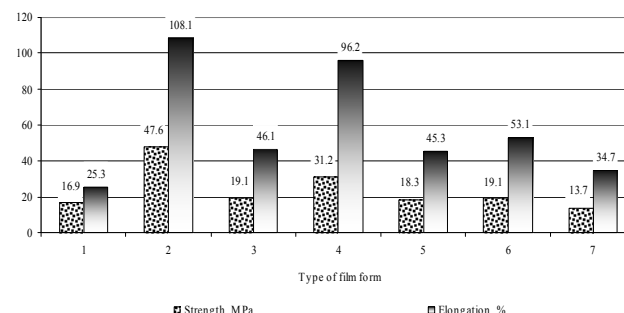


Fig. 3. Influence of a film-forming agent on strength and elongation of edible films: corn starch (1); potato starch (2); chemically modified food starch refined from high amylose corn (3); carbonic polymer from waxy corn (4); modified food starch (5); special dextrin refined from tapioca starch (6); modified from starch refined from ordinary corn (7) and special high-stable dextrin refined from tapioca starch (8)

According to the data obtained (Fig. 3), films based on chemically modified food starch from high amylose corn have a sufficient strength (31.2 MPa). When choosing the type of film-forming agent, a weighty indicator is the organoleptic properties of edible coating/film on their basis. The film on the basis of chemically modified food starch from high amylose corn varieties has the best complex quality index for organoleptic characteristics

(Fig. 3); that is why this starch is preferred as a film forming agent for the development of edible coatings. However, the cost of raw materials should also be taken into account. It is clear that modified starches are more expensive than native ones. Having all the above factors into considerations, the choice should be stopped on potato starch.

It is known that starch is subjected to retrogradation, the degree of which depends on the ratio of amylose to amylopectin in starch, which in its turn depends on the type and origin of starch [28].

Several authors [29, 30] state that starches with a high rate of crystallinity are inferior in physiological aspect to starches with amorphous structure, since they are less susceptible to the action of digestive enzymes.

### 3.5. X-Ray Analysis

Changes in the structure of starch during the storage period were determined using X-ray diffraction analysis. In our study various types of starches were used for the production of edible films of the following composition: starch 2 g, gelatin 2.4 g, urine or glycerol 1.6 g, water 94 cm<sup>3</sup>.

XRD pattern of corn starch film (1, Fig. 4a) consists of seven diffractions at values of  $2\theta = 7.5$ ; 12.1; 17.0; 20.1; 21.7; 25.0; 28.4. Starch has amorphous-crystalline structure with 17 % crystallinity. According to literature data [31, 32] starch has A-type packing.

XRD pattern of potato starch film (2, Fig. 4b) consists of nine diffractions at values of  $2\theta = 6.6$ ; 17.2; 20.1; 23.8; 25.0; 27.1; 31.9; 34.3; 38.0, that corresponds to the amorphous-crystalline phase content with 6 % crystallinity. According to literature data [33, 34] the starch has A-type packing.

XRD pattern of films obtained from chemically modified food starch refined from high amylose corns (3, Fig. 4c) consists of eight diffractions at values of  $2\theta = 6.6$ ; 12.3; 17.9; 21.0; 23.8; 25.0; 37.1; 38.1 that corresponds to amorphous-crystalline structure with 6 % crystallinity and A-type packing.

XRD pattern of films with carbohydrate polymer from waxy corn (4, Fig. 4d) consists of three diffractions at values of  $2\theta = 8.0$ ; 20.7; 25.0. These diffractions were seen at the same values in previous diffractograms, though diffraction at 8.0 value has a lower intensity 599.7 vs. 664.4, that points at partially crystalline structure of starch (5 %), but with a significant part of X-ray amorphous phase. It is proved by the fact that native starches are characterized by a crystalline structure, and after thermal treatment (gelatinization with further cooling), they have amorphous structure. It is known [33] that native starch is characterized by the crystalline structure, and after thermal treatment (cooking, drying, etc.) it becomes amorphous in structure. At the same time according to the literature data [34] carbohydrate polymer obtained from waxy corn is in an amorphous state which is also present in the film made of the polymer mentioned above.

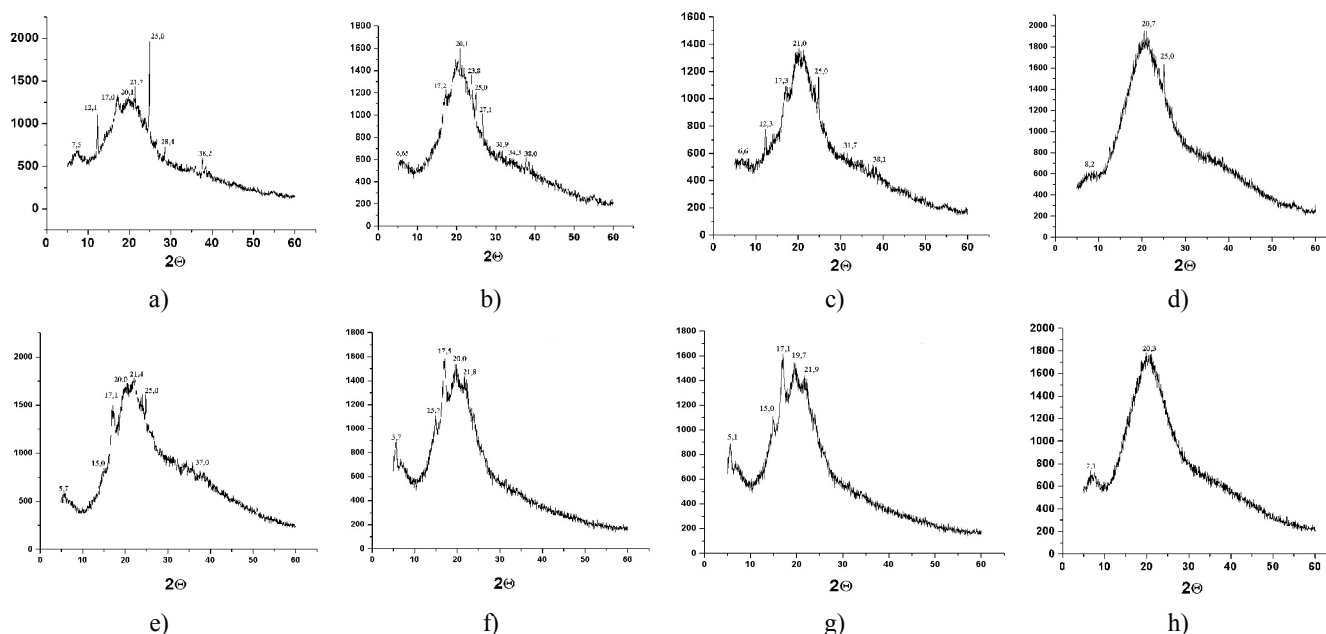


Fig. 4. XRD patterns of studied films

XRD pattern of film with modified food starch (5, Fig. 4e) consists of nine diffractions of values at  $2\theta = 5.7; 12.1; 15.0; 17.1; 20.0; 21.4; 23.8; 25.0; 37.0$ , so the phase structure is amorphous-crystalline with 10 % degree of crystallinity and according to the published data [31, 32] corresponds to A-type packing.

Pattern of the film with special dextrin from tapioca starch (6, Fig. 4f) consists of six diffractions at values of  $2\theta = 5.7; 15.0; 17.5; 20.0; 21.8$ , that corresponds to the structure of amorphous-crystalline starch of A-type packing with 12 % degree of crystallinity. It should be noted that there are no diffractions at values of  $2\theta = 25.0$  and  $37.0$  in this diffraction pattern.

Diffraction pattern of the film with a modified starch from ordinary corn (7, Fig. 4g) consists of five diffractions at  $2\theta = 5.1; 15.0; 17.1; 19.7; 21.9$  that corresponds to the structure of amorphous-crystalline starch with 12 % degree of crystallinity and A-type packing. It should be noted that there are no diffractions at values of  $2\theta = 25.0$  and  $37.0$  in this pattern.

Diffraction pattern of the special high-stable dextrin film obtained from tapioca starch (8, Fig. 4h) is a curve with two peaks, a small one at  $7.1^\circ$  and  $693.3$  intensity and a big one at  $20.3^\circ$  and  $1719.5$  intensity. These signals at values of  $2\theta$  are also observed in other diffraction patterns and confirm the presence of minimum content (2 %) of the crystalline structure of starch, but with a substantial content of X-ray amorphous phase.

From the above described data, we can state that special high-stable dextrin refined from tapioca starch (sample 8) and carbohydrate polymer from waxy corn (sample 4) retain the crystalline structure, as well as the amorphous one. Corn starch (sample 1), modified food starch (sample 5), dextrin refined from special tapioca starch (sample 6) and modified starch from ordinary corn (sample 7) better retain the crystalline structure.

### 3.6. Thermogravimetric Analysis

Various types of starch retain moisture differently. The thermogravimetric analysis was used to measure a moisture content depending on the starch type.

All DTA curves film thermograms have rather vague endo- and exo-effects that makes it impossible to identify the initial and end temperature of the process of thermal changes without the DTA curve that provides more complete information. In spite of the fact that there were different types of starch in the samples under study their thermograms are of the same type and have several thermolysis zones (Table 1).

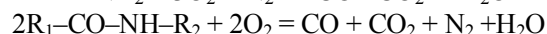
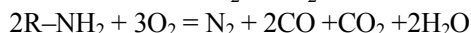
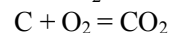
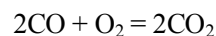
The first zone in the range of  $303\text{--}373$  K corresponds to evaporation and water boiling (minimum at TG curve at  $373$  K), dehydration of crystallization

water (at least at  $383, 393$  or  $413$  K for different samples) occurs at the temperatures above  $373$  K, Table 1).

The second thermolysis interval is within  $383\text{--}603$  K and includes two endo-effects with minimums at  $563$  K (broad and low intensity) and at  $603$  K (narrow and deep). According to the literature [35] a pure glycerol boils at  $563$  K and self-ignites at  $635$  K. This first endo-effect is connected with dehydration of residual bounded water and glycerol evaporation and boiling, the second endo-effect is connected with burning. Since intense formation and release of carbon dioxide and water occur during this process, the losses of samples film mass are the highest in this temperature range (Table 1).

The third temperature range (zone) lies in the range from  $623$  to  $823$  K (Table 1) and characterizes the process of starch (dextrin) and gelatin thermolysis to products of carbonization.

It can be assumed that the release of water, carbon dioxide, carbon monoxide, nitrogen (as a result of oxidation of amino groups of gelatin) is possible in this temperature zone:



Loss of mass is the least intensive in this zone.

The fourth thermolysis zone has temperatures of  $823$  K,  $828$  K (the lowest limit) and  $1053$  K (upper limit), here carbonized starch and gelatin products of the third thermolysis zone are burning and mostly carbon dioxide and some water are released.

Specified chemical composition of thermolysis products found in each zone can be determined by the method of qualitative and quantitative gas analysis.

As an example, the DTA curve of the sample 1 is studied (Table 1). Up to  $373$  K chemically unbound adsorbed water releases; this corresponds to 11.2 % of mass loss. Then crystallization water of OH groups releases, evaporates, boils and glycerol is burning with a total mass loss of 43.7 % (temperature  $393\text{--}623$  K). In the range of  $623\text{--}823$  K the thermolysis of gelatin and dextrin to the carbonized products occurs, the total mass loss is 15.0 %. At the temperatures of  $823\text{--}1073$  K carbonized products of gelatin and dextrin thermolysis 71.2 % are completely burned (mass loss is 27.5 %, and the total mass loss is 97.5 %).

The dry residue after calcination to  $1273$  K does not exceed 1.3 % and relates to oxide compounds of inorganic nature.

Table 2 shows data on the loss of adsorbed and crystallization water in the film samples of different composition.

Table 1

**Thermolysis zones of films depending on starch type**

Film sample	Thermolysis zones, K			
	I	II	III	IV
1	303–373	293–473, 473–563, 563–603	623–823	843–1053
2	303–373	293–483, 483–543, 563–593	623–833	833–1053
3	303–373	413–473, 473–543, 543–593	643–828	828–1073
4	303–373	413–473, 473–543	633–853	823–1043
5	303–373	393–503, 503–543, 543–593	633–853	853–1083
6	303–373	393–523, 523–543, 543–593	643–843	843–1033
7	303–373	383–483, 483–583, 543–593	623–843	843–1073
8	303–373	383–393, 423–543, 543–593	633–823	823–1053

Table 2

**Loss of adsorbed water and crystallization water depending on the temperature range**

Film sample	Release of adsorbed water		Release of crystallization water and constitutional water			
	Temperature range, K	$\Delta m_1$ , %	Temperature range, K	$\Delta m_2$ , %	Temperature range, K	$\Delta m_3$ , %
1	313–373	11.2	393–473	2.5	473–563	12.5
2	313–373	11.2	373–483	6.2	483–563	21.2
3	313–373	8.2	373–483	11.8	473–563	15.3
4	313–373	8.7	373–483	10.0	473–563	17.5
5	313–373	10.0	373–483	2.5	473–563	20.0
6	313–373	7.5	373–483	3.7	473–563	22.5
7	313–373	10.0	373–483	6.2	473–563	10.0
8	313–373	7.5	373–423	6.2	423–513 473–563	7.5 15.0

Table 2 shows that adsorbed water releases to 373 K and its loss in all samples does not exceed 11.2 %.

Table 2 also shows that films from corn starch (sample 1) and potato starch (sample 2) contain the highest amount of adsorbed water (11.2 %), which is typical for native starches. The films from modified food starch (sample 5) and modified starch from ordinary corn (sample 7) contain 10 % of adsorbed water. The chemically modified food starch films refined from high amylose corns (sample 3) and carbohydrate polymers films from waxy corn (sample 4) contain 8.2 and 8.7 % of adsorbed water, respectively. The special dextrin films refined from tapioca starch (sample 6) and special high-stable dextrin refined from tapioca starch (sample 8) contain only 7.5 % of adsorbed water. Thus, regularity is observed: the more starch is modified, the less is amount of adsorbed water in it. Modification destroys the native structure of starch, in particular, 1,6-glucosidic bond breaks, and it is well known that namely amylopectin absorbs water.

The other regularity is observed for crystallization water: in the temperature range of 393–473 K the films from corn starch and potato starch (Table 1) release 2.5 and 6.2 % water, it indicates that they are native starches. At the same time, chemically modified starch refined from high amylose corns and carbohydrate polymer (Table 1, samples 3 and 4) contain 11.8 and 10 % of water, respectively, because these starches are more capable to connect water. Chemically modified starches and carbohydrate polymer

contain a small amount of free carbonyl group, but the authors [34] consider that the carbonyl group in the acyclic form of carbohydrates is hydrated.

Crystallization water splits off at the temperatures above 373 K in the range to 473–513 K, after this temperature range water may release as a result of breaking down the skeleton of organic compounds that make up the film, so dextrin is formed from starch, *i.e.* a constitutional water releases. At 543–563 K a sharp decrease of mass begins, which is observed at the DTA curve, caused by boiling of glycerol or glycerol mixtures and accompanied by the intense evaporation. At 666 K glycerol self-ignites, then film components burn and carbonize. At the temperatures above 823–853 K the decomposition products of films are burnt completely. This process corresponds to the vague exo-effect at DTA curve and loss of mass at 823–1083 K.

Despite the similar thermograms of studied film samples, a detailed analysis revealed some differences. Sample 3 and sample 8 (Table 1) have additional endo-effects at the DTA curve recorded at 413 and 393 K, indicating dehydration of crystallization water. The effect corresponding to a glycerol boiling at 523–543 K has various manifestations, such as a shoulder, which in the case of sample 2 (Table 1) becomes a separate endo-effect with minimum at 543 K. Temperatures of complete burning of thermal degradation products of film samples also differ. For the sample 6 (Table 1) such temperature is 1033 K,

and for the sample 5 (Table 1) it is 1083 K, for other film samples is 1043–1073 K.

## 4. Conclusions

Thus, organoleptic studies demonstrated that it is most advisable to use potato starch, chemically modified food starch refined from high amylose corn, carbohydrate polymer from a waxy corn, and a special high-temperature dextrin purified from tapioca starch for making biodegradable edible coatings/films. The gelatinization temperature, viscosity and physico-mechanical properties of biodegradable films were investigated for different starch types. Thermogravimetric analysis allowed to establish the influence of starch modification on amount of adsorbed water in biodegradable films. The crystallinity degree of different starch types was determined.

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## ВПЛИВ РІЗНИХ ВИДІВ КРОХМАЛЮ НА ОРГАНОЛЕПТИЧНІ, ТЕРМОГРАВІМЕТРИЧНІ ТА РЕНТГЕНОФАЗОВІ ПОКАЗНИКИ ЇСТІВНИХ ПЛІВОК І ПОКРИТТІВ

**Анотація.** Проведено органолептичний аналіз їстівних плівок з картопляного крохмалю, хімічно модифікованого харчового крохмалю, виготовленого з високоамілозної кукурудзи, вуглеводного полімеру із воскової кукурудзи та спеціального високотемпературного декстрину, виготовленого з tapioca крохмалю. Встановлено, що найбільше подовження (108 %) та міцність (47,6 МПа) мають плівки на основі картопляного крохмалю. Показано, що модифіковані крохмалі, такі як декстрин та tapioca крохмаль, зберігають аморфну структуру плівки краще за інших. Плівки з модифікованого крохмалю містять більше кристалізаційної вологи, через відсутність амілопектину. Отримані результати рекомендуються для оптимізації виробництва харчових продуктів.

**Ключові слова:** крохмаль, термогравіметрія, рентгеноструктурний аналіз, їстівні плівки і покриття.