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Posted Date: 19 May 2026

doi: 10.20944/preprints202605.1214.v1

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Production of a Biodegradable Polymeric High Strength Material, Based on Xanthan Gum and Potato Starch, Modified by the Joint Addition of Plasticizers

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Abstract

Production of a biodegradable, environmentally friendly polymer film material, composed of potato starch (PS), xanthan gum (XG), and plasticizers: glycerin, sorbitol, and citric acid, was carried out. The effect of these components on the structural and biopolymer composite mechanical properties, including elasticity and tensile strength, was investigated. The addition of XG significantly reduces the hardness for the film forming materials, thereby lowering the difficulty of gelatinization. It was demonstrated that increasing the plasticizers mass during composite blend preparation improved elasticity but reduced the mechanical strength of the films. It is assumed that these additives in the biopolymer disrupted hydrogen bonds and other intermolecular contacts between starch and gum macro chains. Glycerol influences the elasticity of the bioplastic, while sorbitol influences its strength. Taking various factors into account, the optimal combined concentration of glycerol, sorbitol and citric acid was determined in composite during film preparation. Based on the results of the new polymeric films' flexibility study, it was concluded that they could be used as a replacement for traditional, non-biodegradable polymeric materials. At the optimal concentration of components, the strength of polymer films is 1.6 MPa, and the relative elongation is 45%.

Keywords: starch; xanthan gum (XG); biodegradable polymer; polymer film; plasticizer; glycerin; sorbitol; citric acid

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1. Introduction

The constant increase in plastic waste not only pollutes the environment and spoils the appearance of populated areas but also raises the question of eliminating plastic particle contamination of water and food products for society. This is why the production of environmentally friendly and biodegradable packaging has become one of the most pressing areas of scientific research today [1–14].

It's clear that food packaging materials play a crucial role in solving the problem of microplastic contamination. Biodegradable packaging materials, already used by modern industry, are believed to be capable of replacing synthetic ones. [2].

A key characteristic of films, both conventional and biodegradable, used for food packaging is their flexibility, which is improved by reducing the number of internal hydrogen bonds between polymer chains, leading to macromolecular mobility. To increase flexibility (plasticity), plasticizers are used substances that can reduce the viscosity, glass transition temperature, or elastic modulus of a polymer without damaging its macromolecules [4].

While for standard polymers such as polyethylene terephthalate (PET) the most common plasticizer is some phthalate ester [5], one of the most common plasticizers used in biodegradable film production technologies is glycerol [6]. Он характеризуется высокой температурой кипения, растворимостью в воде и нелетучестью. At the same time, other plasticizers, such as sorbitol and citric acid, are also highly soluble in water and easy to use [7]. However, in the practice of producing

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biodegradable films they are not used as often as glycerol. In this regard, in the presented work we used three of the most common and commercially available plasticizers to date: glycerin, sorbitol and citric acid - to obtain bioplastic with these substances in its composition, and to study their effect on the structural and mechanical properties of the resulting material.

The main goal of our research is to develop a packaging film that can become an alternative to those polymeric materials that are currently used in everyday life and industry, which slowly decompose in nature, and with gradual degradation form micro- and nanoplastics that are potentially dangerous to humans. [2,8].

We produced a biodegradable polymer film based on natural compounds such as starch and xanthan gum because these substances are the most environmentally friendly polymers, are of plant origin, and are extracted from renewable resources. It is especially important for us that we use only Russian-made potato starch in our work. In addition, we have previously studied the process of biological decomposition in soil of tapioca starch-based bioplastics [8], and the results of this study confirmed that such polymers do, indeed, dissolve without a trace in compost within 6 months, without forming any toxic waste.

Why did we choose xanthan gum as the key component of biodegradable polymer compositions? The fact is that this substance (food additive E415) is a polysaccharide of microbial origin, which provides gel-forming and barrier properties in the polymer composition, which often makes it a key component in the development of biopolymer coatings [10]. This component of the bioplastic is a heteropolysaccharide with unique structural and rheological properties, which is why it is widely used in the food, pharmaceutical and packaging industries as a stabilizer and thickener [11]. Xanthan gum dissolves readily in cold and hot water, forming pseudoplastic, highly viscous solutions even at concentrations less than 1%. These properties are particularly valuable in food and cosmetic formulations, as well as in the production of biopolymer films.

One of the key advantages of XG is its resistance to a wide range of temperatures (from 0 °C to 100 °C), pH levels (from 2 to 12) and the salt composition of the environment. Unlike many other hydrocolloids, XG maintains its viscosity during heat treatment, freezing and thawing.

Why is glycerol needed to produce biodegradable plastic? To ensure slippage between polymer chains and make the material sufficiently flexible. It acts as a lubricant in the structure of the resulting polymer material, making it soft and flexible [12].

The combined use of all three plasticizers (glycerin, sorbitol and citric acid) to produce films is also due to the possible synergistic effect that they can exhibit when used simultaneously [9–12]. Glycerin provides "lubrication" between macromolecules, sorbitol provides the hardness and rigidity of the composite, and citric acid provides an acidic environment, without which plasticization is generally impossible.

At present, there is already work on obtaining xanthan-starch biodegradable films [9–11], but there is no convenient and easily reproducible technology that could be scaled up to industrial application. For example, the method of forming films, proposed in the work [11], turned out to be non-reproducible: after 24 hours of keeping the initial solution with the composition proposed by the authors at room temperature, no film is formed from it. It also does not form when the resulting gel dries over the next 6 days. A thin film of 0.165 mm thickness, as we have established, is formed only after 7 days.

It is especially important to note that Russian-produced potato starch has never been used for these purposes before. For example, in the work of Chinese colleagues [9], the development of polymer films based on xanthan gum and starch from the tropical plant cassava, using a special hot-pressing press with a pressure of 6 MPa is described in detail. But not from Russian potato starch, which our country is so rich in and which we can share with the whole world. And such a press is not available in every laboratory or every production facility. Therefore, we will formulate another objective of our work: to develop a technology for producing mechanically strong and biodegradable films based on potato starch and other components produced in Russia.

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Furthermore, the effect of each of the substances we used (glycerin, citric acid, and sorbitol) on the film properties was previously studied individually. However, the question arises: how will these compounds behave when present together? Is it possible to expect a synergistic effect from their combined action on a biodegradable polymer?

2. Materials and Methods

2.1. Materials

Potato starch (PS) and xanthan gum (XG) were used to produce a biodegradable polymer film. Potato starch (PS) with a moisture content of 11 g/100 g starch, amylose content of 22 g/100 g starch, and mean particle size of 20 μm of the "Slavnaya Trapeza" brand was used (manufacturer: Slavnaya Trapeza Holding, Russia, Kaluga Region). XG was supplied by "Healthy Country", Russia, Moscow, ISO 22000.

The following plasticizers were used: food-grade distilled glycerol 99.7%, manufactured by "Delfin Industry", Pushkino, Moscow Region, Russia; sorbitol (manufacturer: "Sladkiy Mir", Moscow, Russia) and citric acid monohydrate. All of the above components are commercially available chemicals: sorbitol (E420) and citric acid (E330) are food additives, a sweetener and a preservative, respectively, and are therefore widely used in the food industry.

2.2. Preparation for the Starch Films

To obtain a polymer film, PS and XG solutions were prepared separately. Both components are powdery substances. But the solubility of these substances in water varies, therefore, to obtain solutions, the initial components weighing 2 g each were dissolved separately in 48 ml of distilled water while stirring on a magnetic stirrer at room temperature, thus obtaining 4% solutions. Xanthan gum dissolves worse than starch, but already at room temperature it forms a strong gel.

Plasticizers (solid in the case of sorbitol and citric acid, and liquid in the case of glycerin) were added to the starch solution one by one, without stopping stirring, in 4 different mass ratios: 0.5 g of each substance (starch-xanthan mixture was named PS/XG-0.5); 1.0 g (PS/XG-1); 2.0 g (PS/XG-2); and 3.0 g (PS/XG-3). The plasticizers were added in the following order: glycerin, sorbitol, and citric acid. Thus, a total of six types of films were obtained. Next, the solutions, without stopping stirring, were simultaneously heated to a temperature 80 °C, at which starch gelatinization begins. After this, the starch solution with plasticizers was added to the gum solution, mixed, and stirring and heating continued until the mixture turned into a gel. The resulting hot mass was evenly poured into Petri dishes, distributed over the surface and then kept for 3 days at room temperature (20–22 °C). During this process, drying and hardening of the final composite material was observed, and its transformation into a film was observed, which could be separated from the glass and used for further research.

In this way, several types of plastic and flexible transparent film were obtained, similar in appearance to polyethylene film (Figure 1):

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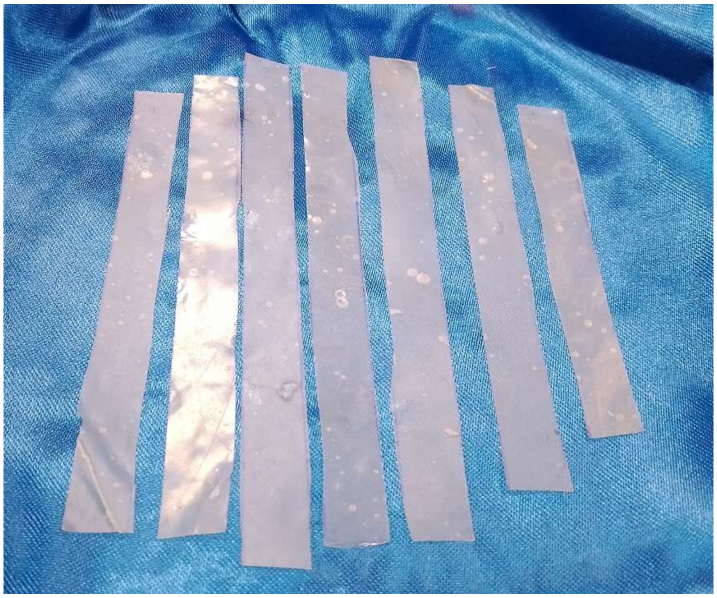

Figure 1. Appearance of the resulting polymer film PS/XG-1.

As a control sample, a biofilm was obtained without the addition of sorbitol and citric acid. However, without glycerin, the polymer is brittle, hard, and inelastic, making it unsuitable for practical applications today. Therefore, glycerin weighing 2 g was added to the control sample (PS/XG-0).

2.3. Characterization of Starch Films

2.3.1. Moisture Content

The moisture content was determined using equation 1 in three replicates for each type of film based on the difference in mass before and after drying a film weighing 0.3–0.6 g in a porcelain cup at 120 °C for 40 minutes.

$$\textbf{moisture content}\ (\%) = \mathbf{(m_0 - m)*100/m_0}\ (1),$$

where m_0 – film weight before drying, m – film weight after drying.

2.3.2. Degree of Solubility

Samples weighing 0.3–0.5 g (3 pieces for each type of film) were cut out and dried for an hour at 60 °C. The dry films were then immersed in 50 ml of distilled water and kept at room temperature for 24 hours. The films were then removed from the water and dried again for an hour at 60 °C. The degree of solubility of the polymers was calculated using equation 2:

$$\textbf{degree of solubility}\ (\%) = \mathbf{(m - m_0) \cdot 100/m_0}\ (2),$$

where m_0 – initial film weight before drying, m – final film weight.

2.3.3. Film Thickness

The thickness of each film was determined in accordance with ISO 4593 "Plastics — Film and sheeting — Determination of thickness by mechanical scanning" utilizing a digital caliper C640 (Labthink, China) equipped with a computer interface: for each sample, five films were selected, and for each film, six randomly chosen points were measured. The average of these measurements was subsequently reported.

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2.3.4. Mechanical Properties

The tensile strength (TS) and elongation at break (EB) of the films were evaluated using an Automatic tensile testing machine C610N (Labthink, China), equipped with a computer interface in compliance with the ASTM D882 standards. The permissible error limit for load measurement during forward motion did not exceed ±1% of the measured load. The specimen deformation rate was 100 mm/min. Each film sample was put through five tests to determine the average. For testing, rectangular strips 1 cm wide and 10 cm long were made from each type of film.

The calculations were performed using the following equations (3 and 4):

$$\textbf{TS}\ (\text{MPa}) = \textbf{Fmax/S}\ (3),$$

where F represents the maximal force at the film break (N), and S represents the initial cross-sectional area (m^2).

$$\textbf{EB}\ (\%) = \mathbf{(L/L_0)\cdot 100\%}\ (4),$$

where L_0 represents the original separation gap (mm), and L is the probe gap at the instant of rupture (mm).

2.3.5. Infrared Spectroscopy

IR-spectrum of polymer films with plasticizers were obtained by IR spectroscopy on an infrared FTIR spectrometer 990IN (Tianjin, China) (range 400–4000 cm^{-1}, permission 1 cm^{-1}).

3. Results and Discussion

Even before we began our research, it was clear that the technology for producing Xanthan-starch films should be as simple and easily reproducible as possible. If we look, for example, at the work [11], many questions arise regarding the process described there. The order in which the components are mixed is not specified. It's not even specified whether the gum and starch solutions are mixed first, followed by the addition of the plasticizers, or vice versa. The mixing process is described by a mysterious phrase «subsequently, the mixtures were combined in varying proportions and plasticizers were incorporated into the resultant blends. It is noted that after mixing, the solutions are poured into “glass dishes”, and the example shown is a Petri dish. All this makes the method completely non-reproducible and raises the question of developing a technology that is simple, understandable and accessible to all researchers and interested industrial manufacturers.

The method for producing films without starch and plasticizers is unclear. For example, in [13], the process is described as follows: "The films were prepared by mechanically mixing the starting reagents in distilled water." That's all.

In addition, work [11] indicates that the mass fraction of added plasticizers ranges from 0.5 to 3%. But it is completely unclear: is this a mass fraction of the original solution or of the film obtained after drying?

Furthermore, in [11], the authors write that a 1% concentration of gum and starch is 1 mg/ml, or 1 g/l. However, at this concentration, the mass fraction is not 1%, but 0.1%!

To test film production technology from [11], at the very beginning of our study, we precisely reproduced the sequence of actions described in that work: separately prepared 1% solution of starch and gum in distilled water. For this, 0.5 g of each substance was mixed with 49.5 g of water. Next, the resulting solutions were heated by stirring to 80 °C, poured together, and 1 g of glycerol was added to the resulting mixture. The mixture was stirred at the same temperature until gelatinization was achieved. The resulting viscous solution was poured into a Petri dish and dried for 8 days. The resulting thin film separated from the glass surface.

During the experiment, it was established that the method for forming films proposed in [11] was not reproducible: after 24 hours of holding the initial solution with the composition proposed by the authors at room temperature, no film formed from it. The solution still formed a viscous gel. We have already published photographs of this process in our work [15].

A film also doesn't form when the resulting gel dries within 6 days of production. We found that a thin film only forms after 7 days. At least, this is what happens when using Russian-made potato starch and xanthan gum.

In this regard, we were faced with the question: it is necessary to develop a technology for producing films based on Russian raw materials that would be simple and easily reproducible.

The biodegradable polymer films we obtained using our new method are formed in the form of a hydrogel by mixing aqueous solutions of xanthan gum and starch with the addition of various plasticizers: glycerin, sorbitol, and citric acid. Such films, even those formed without any plasticizers, are transparent, very brittle, and have cracks on the surface. Consequently, they cannot be used for packaging. We were also convinced of this fact after obtaining a similar film from starch and gum without plasticizers [15]. This phenomenon is because a dense network of intermolecular hydrogen bonds limits the mobility of the macromolecular chains of both polymers.

Biodegradable xanthan starch films formed from hydrogel in the presence of glycerin, sorbitol, and citric acid in varying ratios are virtually identical in appearance and color.

Adding glycerin to the starch-gum mixture at a rate of 1% of the hydrogel mass was also insufficient to create a flexible and durable film. This film was also rigid and cracked. This is why the control polymer PS/XG-0 contains 2 g of glycerin per 100 g of solution (2%).

A polymer composition containing 0.5% of each plasticizer (PS/XG-0.5) also forms a hard and inelastic film that shatters into fragments when attempting to separate it from the glass surface. Therefore, this bioplastic variant was excluded from further testing.

That is why, when forming films based on polysaccharides, it is useful to use plasticizers based on polyols precisely due to their hydrophilic nature [16], as was done in our work.

3.1. Degree of Solubility and Moisture Content

The solubility of biodegradable films in water is a key factor when using them as packaging materials. However, in some cases, films must be water-insoluble to maintain product integrity and the water-resistant properties of the packaging materials. In this regard, we studied the effect of plasticizers on the solubility of xanthan-starch films in water and on the moisture content in them (Figure 2). The solubility values for each plasticizer contained separately in the mixture were taken from [11] (as well as for the following Figures):

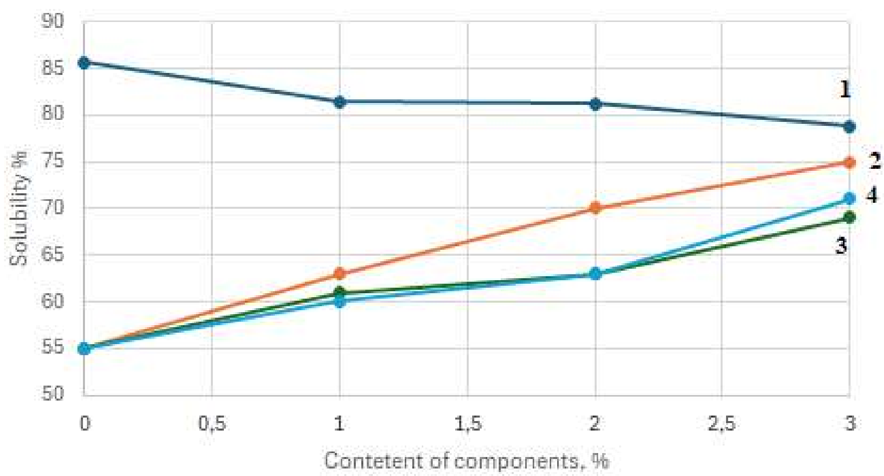

Figure 2. Effect of plasticizer content on the solubility of starch-xanthan polymer films (Row 1: combined content of three plasticizers, Row 2: glycerol, Row 3: citric acid, Row 4: sorbitol).

It turned out that the films we obtained were very soluble in water: they disintegrated almost completely, forming gel fragments that had to be filtered from the water and dried on filter paper.

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For films containing 2 grams or more of plasticizer, complete conversion to gel occurred within 4-6 hours, rather than within 24 hours.

As Figures 2 and 3 shows: the higher the plasticizer content in the films, the higher their water solubility and moisture content. This is likely due to the accumulation of hydrophilic substances between the polymer chains, which helps retain water molecules in the system. Films containing both plasticizers are no different in solubility than those containing them separately.

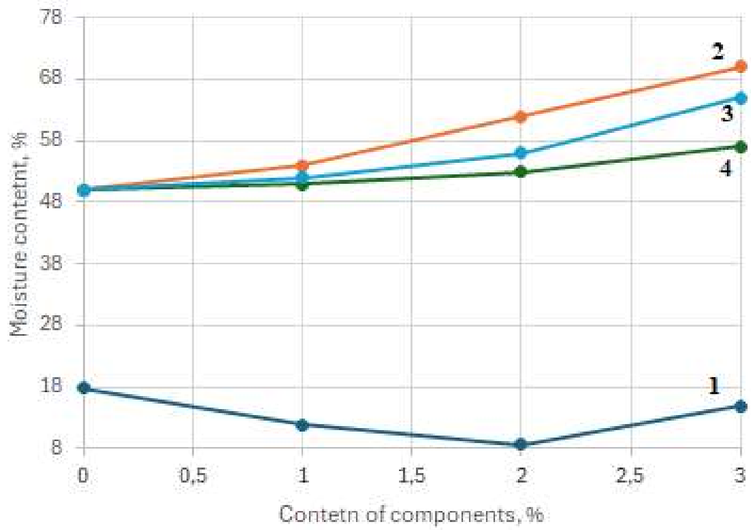

Figure 3. Effect of plasticizer content on the percentage of bound water (moisture) of starch-xanthan polymer films (Row 1: combined content of three plasticizers, Row 2: glycerol, Row 3: citric acid, Row 4: sorbitol).

However, increased moisture content can negatively impact the mechanical properties of films, so a "golden mean" must be found: this is plasticizer's ratio that simultaneously achieves good solubility and mechanical properties [17,18]. It's clear that our polymers contain 1.5 times less water than previously developed ones. This is a significant advantage.

3.2. Tensile Strength and Elongation at Break

Before studying the mechanical properties of films, it is imperative to determine their thickness. Without this parameter, the cross-sectional area cannot be calculated, and therefore, the tensile strength, which is the ratio of the tensile force (in N) divided by this area.

When producing a polymer composite using our method, the thickness of the same film can vary in different locations. Indeed, our measurements show thickness variations of ±50 μm for the same type of film (Table 1). However, this does not in any way worsen the mechanical properties of the films and can easily be corrected using industrial methods of film forming, for example, under the press.

Table 1. Results of measuring the thickness of starch-xanthan films of different compositions.

Смесь	d min	d max	d средний
PS/XG-0	309	392	350±41
PS/XG-1	331	424	365±42
PS/XG-2.0	691	774	724±33
PS/XG-3	877	980	917±41

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Plasticizers significantly influence the rheological properties of films, controlling their strength and relative elongation [19]. Figures 4 and 5 shows the dependence of tensile strength (TS) and elongation at break (EB) on the concentration of plasticizers in the mixture:

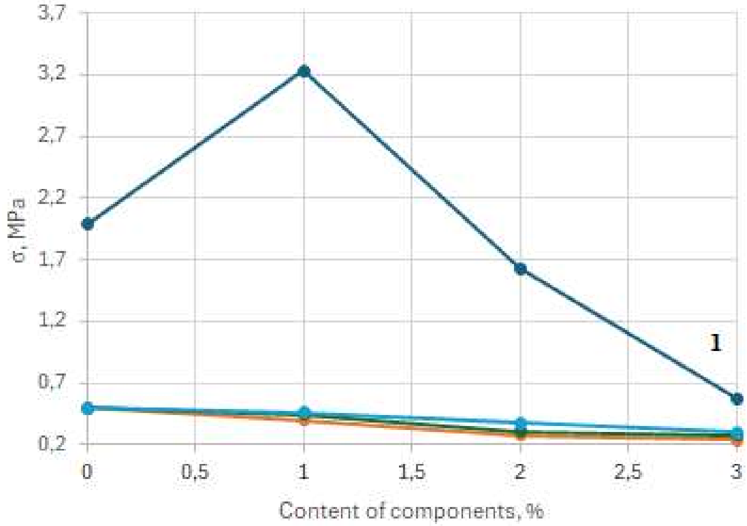

Figure 4. Effect of plasticizer content on tensile strength (TS) for starch-xanthan polymer films.(Row 1: combined content of three plasticizers, Row 2: glycerol, Row 3: citric acid, Row 4: sorbitol).

It is obvious that the polymer material we have developed is much stronger than previously known ones.

It should be noted that the result for a component content of 0%, proposed by the authors of the work [11], cannot be objective: a simple mixture of starch with gum forms a brittle and hard, completely inelastic film.

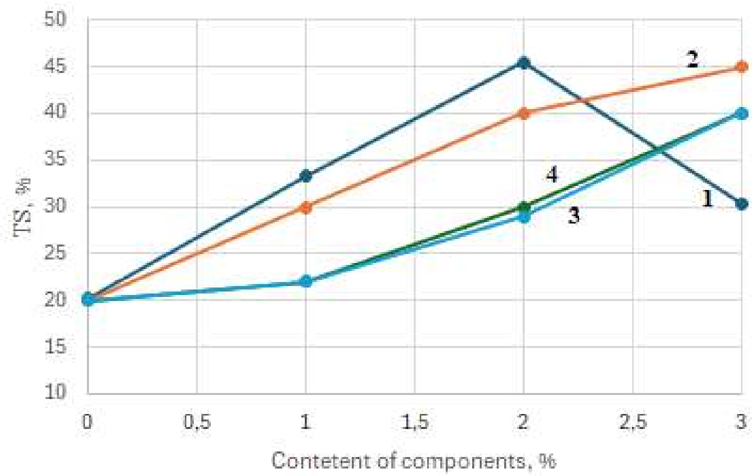

Figure 5. Effect of plasticizer content on elongation at break (EB) for starch-xanthan polymer films(Row 1: combined content of three plasticizers, Row 2: glycerol, Row 3: citric acid, Row 4: sorbitol).

As Figure 5 shows, increasing the plasticizer content increases the maximum film elongation before rupture. While an elongation of 20–45% is significantly lower than that of traditional polyethylene, which has an EB exceeding 250% [20], the value we achieved is sufficient for creating flexible packaging materials.

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It is obvious that the ability to accumulate in space between two macromolecules of biopolymers, destroying their intermolecular contacts, is most characteristic of glycerol, which has the lowest molecular weight and as many as 3 OH groups capable of forming hydrogen bonds. Therefore, it is glycerol that prevents the formation of such bonds between macromolecules and serves as a kind of “lubricant” for the entire polymer structure. But sorbitol and citric acid molecules, which have more developed hydrocarbon chains, also do their job in the mixture: they can have a cross-linking effect on macromolecules. And in an acidic environment, the bioplastic gels more easily. However, Figure 4 shows that with increasing plasticizer content, polymer strength gradually decreases. The main reason for this is the increased content of glycerol, which is a liquid and therefore negatively impacts strength.

Moreover, the tensile strength of 1.6–3.2 MPa for biofilms is significantly inferior to the strength of synthetic polymeric materials, in particular polyethylene (20–40 MPa).

If we compare the films we obtained with polymeric materials of similar chemical composition, for example, in work [11], we can see that previously known films had a maximum relative elongation of 20–45%, and a tensile strength of 0.2–0.5 MPa. As for the thickness of the films, it is not possible to compare them with the work [11] in terms of this parameter, since this work does not report a word about the thickness and methods of determining it.

Thus, when adding plasticizers, on the one hand, elasticity increases (Figure 5), but on the other hand, strength decreases (Figure 4). This means that, as a result of the work carried out, we have established optimal concentrations when using glycerin, sorbitol and citric acid in their simultaneous presence in the composition of a biodegradable xanthan-starch film. The optimal content can be 1–2 g per 100 g of solution (which corresponds to 1–2% by weight of the entire mixture) for each of the plasticizers. That is, both in the initial mixture and in the finished film, the mass of each plasticizer can be equal to the mass of starch and gum (PS/XG-1: in this case, a film with TS = 2.2 MPa and EB = 33% is obtained.

A xanthan-starch composite was studied using IR spectroscopy: without sorbitol and citric acid (film PS/XG-0) and with 2 g of each additive (film PS/XG-2). A comparison was also made with pure potato starch (Figure 6):

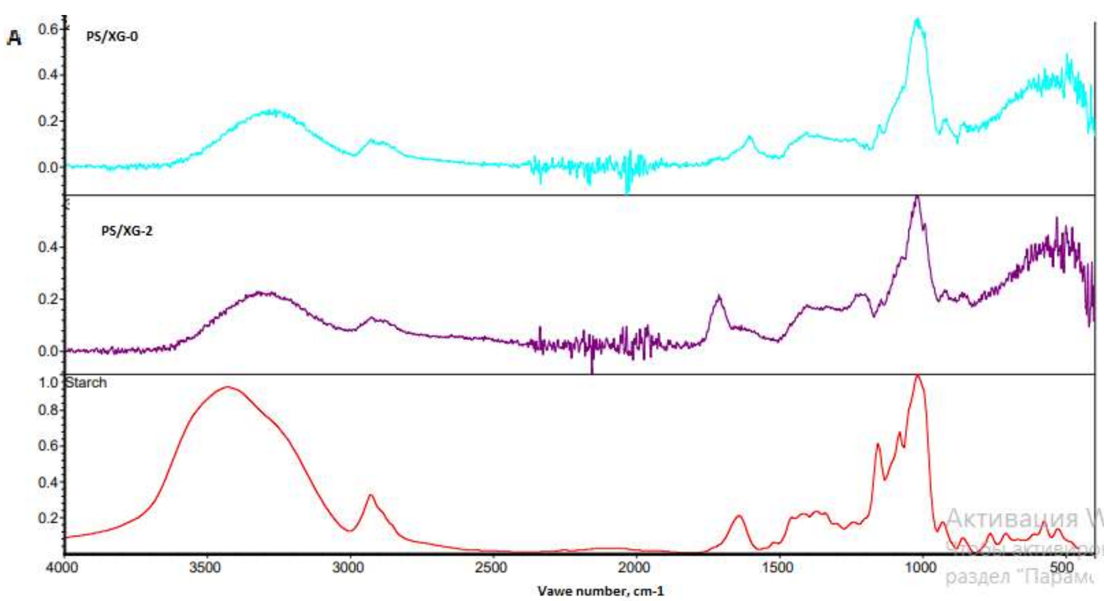

Figure 6. IR spectra of starch-xanthan polymer films in comparison with pure starch.

It is evident that the IR spectra of films with plasticizers differ from the IR spectrum of potato starch without additives only insignificantly: the presence of noise signals in the form of a “picket fence” and a change in the shape of the signal characteristic of the OH group.

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На спектрах видно:

1. the presence of characteristic vibration bands of C-H bonds: the most massive chemical bonds of hydrocarbon chains - in the range of vibration frequencies 2900 cm^{-1};
2. broadened absorption band 3000–3400 $см^{-1}$ — vibration region characteristic of OH groups linked by hydrogen bonds. Such bonds are quite typical for starch that has formed a secondary helical structure;
3. an absorption band centered at 1700 cm^{-1} (stretching vibrations of the C=O group) is present in the IR spectrum of the film with citric acid, but not in the spectrum with glycerol alone;
4. the most intense absorption band in the spectrum at 1050 cm^{-1} is characteristic of vibrations of the C—O bond of ethers or alcohols.

Thus, the use of glycerol, citric acid and sorbitol as plasticizers in the production of films from xanthan-starch biopolymers proved to be very effective. Glycerol molecules have the most significant effect on film flexibility, while the presence of sorbitol results in stronger films. It should be noted that all three plasticizers reduce the strength of the films with an increase in their content in the composite but increase elasticity.

The answer to the question: do three plasticizers - glycerin, citric acid and sorbitol - create a synergistic effect when used together becomes obvious looking at Figures 5 and 6: yes, they do.

Considering the influence of the plasticizer concentration on the structural and mechanical properties, it is possible to recommend a concentration of 1-2% of the initial gel for each of the substances: starch, gum, glycerin, sorbitol and citric acid together in the initial mixture to obtain a polymer film.

The polymer film samples we obtained consist of completely environmentally safe components. They are reliably biodegradable. This is demonstrated by our previous experiments studying similar materials [8] and by the fact that the films dissolve in water by 70–90% in less than 24 hours (Figure 3). They could become an alternative to traditional, non-biodegradable polymer packaging materials (polyethylene and polypropylene, as well as cellulose xanthate – cellophane).

Thus, to protect the environment from pollution by plastic waste, we currently offer not only a previously developed method for creating special sorbents for micro- and nanoplastics [17], but also the production of materials that, when destroyed in nature, do not release any plastic particles at all.

4. Conclusions

1. Samples of biopolymer material in the form of xanthan-starch films with the addition of plasticizers: glycerin, sorbitol and citric acid were obtained.
2. It has been established that an increase in the concentration of plasticizers leads to a gradual increase in the elasticity of films and a decrease in their strength.
3. The optimal concentration of plasticizers in the biodegradable polymer was determined to be 1-2% for each of the substances used based on the mass of the original mixture.
4. The resulting biopolymer films have a relative elongation at break of 33% and a tensile strength of 3.2 MPa, which makes them quite suitable for the industrial production of packaging materials.

Funding: This research received no external funding.

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: The original contributions presented in this study are included in the article. Further inquiries can be directed to the corresponding author(s).

Conflicts of Interest: The authors declare no conflict of interest.

 doi:10.20944/preprints202605.1214.v1

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