Investigation of the influence of the composition on mechanical properties of polylactide

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Abstract. In this paper we describe the creation of films from polylactide. Studied the mechanical properties of developed polymer films of polylactide. The effect of the molecular weight of polylactide on the mechanical properties of the resulting polymer films is shown. The dependence of the mechanical properties of polylactide films on the polymer concentration in chloroform was studied. The possibility of creating biodegradable films with specified mechanical properties is shown.

1. Introduction
Treatment of many diseases is very often inextricably linked with the negative impact on healthy human organs. Modern medicine has long sought to minimize such possible side effects, including the use of various medications [1-3].

One of the variants of solving this problem is the scientific society sees in the creation of systems of controlled drug delivery. Such systems provide therapeutic effects directly in the desired area, reducing or completely eliminating the effect on the rest of the body [4-6].

The promising basis for the creation of these systems are biodegradable polymeric materials of synthetic and natural origin, among which polyesters: polylactides, polylactide glycolides, polyethylene glycol, polyanhydrides, polyorthoesters, polysaccharides (starch, dextran, chitosan) are the most developed [7-8].

Films based on polylactide of different molecular weights are considered in this paper. Polylactide is an aliphatic polyester whose monomer is lactic acid. It is biodegradable, biocompatible and thermoplastic. An obvious advantage is the fact that the raw materials for the production of polylactide are renewable resources, namely silo-forming agricultural crops. Polylactide is already used in medicine, for the manufacture of surgical threads and the processing of pins. [9].

Thus, polymers can be obtained with different molecular weights and structures, and can also encapsulate molecules of almost any size. Using the proposed molecular weight of the polymer, different concentrations of polymer solutions, it is possible to provide a wide range of coatings with different properties, with further selection of the required characteristics. [10]

Polylactide is soluble in most solvents, including chlorinated solvents, tetrahydrofuran, acetone, ethyl acetate. In the work as a solvent, chloroform was selected. [11]
2. Materials and methods

To create the films, we prepared samples of polymers of mass 2; 6 and 10 g (± 0.01 g).

Chloroform, 200 ml in volume, was placed in a 500 ml flask and heated to 80° C with a magnetic stirrer.

Further, the polymer samples weighed were dissolved to a homogeneous state in chloroform at 80° C for 1 hour with constant stirring by means of an electronic overhead stirrer.

At the end of dissolution of the polymer, the volume of the solution was adjusted with chloroform to 200 ml. The resulting solution was aged for 5 minutes at 80° C and spread over glass pallets.

Drying was carried out for 2 days in air at 37° C in a thermostat.

At the end of the drying, the resulting films were removed.

The obtained samples were assigned ciphers used for independent research in the future (Table 1):

| Code | Polymer   | Molecular weight, kDa | Concentration, g per 100 ml of chloroform |
|------|-----------|-----------------------|------------------------------------------|
| 1.1  | Polylactide | 45                    | 1                                        |
| 1.2  | Polylactide | 45                    | 3                                        |
| 1.3  | Polylactide | 45                    | 5                                        |
| 2.1  | Polylactide | 90                    | 1                                        |
| 2.2  | Polylactide | 90                    | 3                                        |
| 2.3  | Polylactide | 90                    | 5                                        |
| 3.1  | Polylactide | 180                   | 1                                        |
| 3.2  | Polylactide | 180                   | 3                                        |
| 3.3  | Polylactide | 180                   | 5                                        |

Tensile strength studies of polymeric films from polylactide were carried out on a universal test machine INSTRON 3382 at a loading rate of 10 mm / min. Samples of polymer films for testing were made in accordance with GOST 14236-81, in the form of a double blade. The sample was fixed in the grippers of the testing machine, which was tightened evenly to ensure that the sample did not slip during the test. Tests of polymer films with determination of the relative elongation, yield strength and tensile strength were carried out in accordance with GOST 14236-81. The processing of the test results in determining the characteristics of mechanical properties was carried out using the software INSTRON Bluehill 2.0. The measurement error of the test machine is less than 1%. Five samples were tested for one experimental point. The values of yield strength $\sigma_y$, tensile strength $\sigma_b$ and deformation $\delta$ were determined.

3. Results and discussion

Table 2 presents the averaged results of mechanical testing of polymer films for each composition based on polylactide (PLA) with different molecular weights with different dissolution concentrations in chloroform.

From the results obtained, graphs of the dependence were plotted, along which it is possible to trace the tendency of the change in mechanical characteristics (Figures 1-3).

As can be seen from the graphs shown in Figures 1-3, the thickness of polylactide films, their plasticity and strength is directly related to the concentration of the solution and the molecular mass of
the substance. Increasing the concentration of the polymer in the solution promotes thickening of the polymer layer, thereby increasing plasticity, but decreasing the strength of the polymer film.

The change in the molecular weight of the polylactide showed that films based on polylactide 90 kDa and a strength of 45 kDa have a better plasticity. For lower concentrations in this study (1 and 3 g per 100 ml of chloroform), the change in the molecular weight of the polymer does not significantly affect the thickness of the resulting film. For a concentration of 5 g per 100 ml, the film thickness is reduced to 45% with an increase in molecular weight from 45 kDa to 180 kDa.

Table 2. Mechanical properties of polymer films.

| Code | Polymer and concentration | Deformation, % | Yield strength, MPa | Ultimate Strength, MPa | Thickness, μm |
|------|---------------------------|---------------|--------------------|------------------------|--------------|
| 1.1  | PLA 45kDa 1x100           | 7.08          | 22.90              | 35.63                  | 42           |
| 1.2  | PLA 45kDa 3x100           | 60.21         | 17.39              | 21.68                  | 107          |
| 1.3  | PLA 45kDa 5x100           | 209.69        | 2.86               | 5.73                   | 339          |
| 2.1  | PLA 90kDa 1x100           | 58.09         | 17.27              | 20.12                  | 38           |
| 2.2  | PLA 90kDa 3x100           | 225.95        | 3.10               | 4.41                   | 125          |
| 2.3  | PLA 90kDa 5x100           | 305.37        | 0.63               | 2.05                   | 262          |
| 3.1  | PLA 180kDa 1x100          | 43.86         | 16.77              | 19.63                  | 39           |
| 3.2  | PLA 180kDa 3x100          | 140.88        | 5.37               | 6.64                   | 82           |
| 3.3  | PLA 180kDa 5x100          | 304.71        | 1.87               | 3.28                   | 152          |

Figure 1. Diagram of deformation depending on the concentration of the solution for PLA.
Figure 2. Diagram of the dependence of tensile strength on the concentration of solution for PLA

Figure 3. The diagram of the dependence of the thickness of polymer films on the concentration of solution for PLA
4. Conclusions
Investigations of the mechanical properties of the developed polymer films of polylactide were carried out and the possibility of creating biodegradable films with specified mechanical properties was demonstrated.

Acknowledgements
The work was supported by the Ministry of Education and Science of Russia (grant identifier RFMEFI60417X0196).

References
[1] Giessen WV, Lincoff A, Schwartz R, Beusekom HV, Serruys P, Holmes D, et al. Marked inflammatory sequelae to implantation of biodegradable and nonbiodegradable polymers in porcine coronary arteries. Circulation 1996;94(7):1690–7.
[2] Nasakina E.O., Baikin A.S., Sevost’yanov M.A., Kolmakov A.G., Zabolotnyi V.T., Solntsev K.A. Properties of nanostructured titanium nickelide and composite based on it. Theoretical Foundations of Chemical Engineering, 2014. - V.48. №.4. - P.477–486.
[3] Nasakina E.O., Sevost’yanov M.A., Mikhailova A.B., Gol’dberg M.A., Demin K.Yu., Kolmakov A.G., Zabolotnyi V.T. Preparation of a nanostructured shape memory composite material for biomedical applications. Inorganic Materials, 2015, Vol. 51, No. 4, pp. 400-404
[4] Cooper SL, Visser SA, Hergenrother RW, Lamba NMK. Polymers.In: Ratner BD, Hoffman AS, Schoen FJ, Lemons JE, editors. Biomaterials science an introduction to materials in medicine. 2nd ed. San Diego: Elsevier Academic Press; 2004. p. 67–79.
[5] Stack R, Califf R, Phillips H, Pryor D, Quigley P, Bauman R, et al. Interventional cardiac catheterization at Duke medical center. Am J Cardiol 1988;62:3F–24F.
[6] Lincoff AM, Schwartz RS, Giessen WJVD, Beusekom HMMV, Serruys PW, Holmes DR, et al. Biodegradable polymers can evoke a unique inflammatory response when implanted in the coronary artery. Circulation 1992;86(Suppl.4):
[7] Supper S., Anton N., Riemenschnitter M., Curdy C., Vandamme T. Thermosensitive chitosan/glycerophosphate-based hydrogel and its derivatives in pharmaceutical and biomedical applications. Expert Opin. Drug Deliv. 2014;11:249–267. doi: 10.1517/17425247.2014.867326.
[8] Sevost’yanov M.A., Fedotov A.Yu., Nasakina E.O., Teterina A.Yu., Baikin A.S., Sergienko K.V., Kolmakov A.G., Komlev V.S., Ivanov V.E., Karp O.E., Gudkov S.V., Barinov S.M. Kinetics of the release of antibiotics from chitosan-based biodegradable biopolymer membranes. Doklady Chemistry, 2015. V.465. Part.1. P.278-280.
[9] Timchenko TV, Shcherbakova LI, Kompanstev VA Poly-D. L-lactide-co-glycolide: methods of preparation, properties and use for the development of drugs with micro- and nanodelivery means. Modern problems of science and education. 2015. № 4. P.559-570 (in russian)
[10] Kedik SA, Zhavoronok ES, Sedishev IP, Panov AV, Polymers for delayed drug delivery systems (review) (May 3, 2013) (in russian)
[11] Glotova VN, Novikov VT, Izhembina TN, Titova NG Solubility of lactide and glycolide in organic solvents. Polzunovskii vestnik. 2014. No. 3. P. 145-147. (in russian)