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Physical and mechanical properties of nonwoven materials for medical purposes based on polyhydroxybutyrate

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Abstract. Ultrathin fibers based on biopolymer poly-3-hydroxybutyrate were obtained by electrospinning method. Using the methods of scanning electron and optical microscopy, macrophysical characteristics of the fibrous layer were established and classified. Physical and mechanical properties of materials and their changes under the influence of the ozone gas as a sterilizing agent were also determined. The paper shows the principal features of nonwoven materials based on poly-3-hydroxybutyrate obtained by electrospinning method, which contribute to their use as effective medical materials.

1. Introduction

Currently, the development and research of nonwoven fibrous materials for medical purposes based on biopolymers is an area of a great practical interest. One of the most promising methods for producing nonwoven materials with a highly developed surface is electrospinning. The purpose of this research was to consider the features of the structure of ultrathin fibers based on poly-3-hydroxybutyrate (PHB), which form a layer of material, and to establish the laws of their influence on the physical and mechanical properties. The study of nonwoven electrospun materials allowed to generalize several key factors that determine the structural organization in the material at such levels as macrostructure, which includes arranging and relative position in space of the elements of non-woven fabric (fiber) [1], and microstructure, which includes orientation of polymer molecules in the material [2]. Macrophysical characteristics of materials have a high value, as they allow to describe in details the features of the fiber layer and to establish the relationship between the process of fiber formation and a number of properties due to the parameters of both individual fibers and the entire material. Among the basic indicators of the structural organization in the material were identified as determining such characteristics as relative density of the structure, γ ; index of orientation of the fibers, ϕ ; materials intensity expressed through average surface density, δ . Another important characteristic is the average diameter of the fibers. These characteristics have a significant impact on the physical and mechanical properties of the material. The vast majority of polymer materials obtained by the electrospinning method consists of sufficiently dry fibers that are practically incapable of reversible elastic deformations [3]. One of the most important parameters in assessing the mechanical properties of such materials is the resistance to monoaxial



extension. Of great interest is the study of the ozoning effects on materials and medical products, since the ozonation method is one of the most effective methods of sterilization and disinfection of medical devices [4]. The assessment of changes in the mechanical properties of the material under the influence of ozone has especially high importance.

2. Experimental setup

In this work, we used a natural biodegradable polymer poly-3-hydroxybutyrate (PHB) 16F series obtained by microbiological synthesis of the company “BIOMER” (Germany). Viscosity-average molecular weight of PHB was 2.06×10^5 . The fibers were obtained by the electrospinning method using a single-capillary laboratory unit with a capillary diameter 0.1 mm, the electrical voltage was 12 kV, the distance between electrodes was 180 mm and the electroconductibility of the solution was 10 $\mu\text{S}/\text{cm}$. Electrospinning process conditions largely affect the nature and distribution of the fibers in the material structure. It is important to note that the structure of the material as a whole is irregular and the fibers are oriented randomly. Fiber distribution was investigated by a set of methods of optical and scanning electron microscopy. Mechanical properties were estimated by the method of monoaxial stretching on a tensile testing machine “DEVOTRANS” (Turkey), in accordance with GOST R 53226-2008 “Nonwoven Fabrics. Methods for determining strength”. Ozonation of materials was carried out using a discharge ozonizer in the laboratory of Emanuel Institute of Biochemical Physics RAS. The principle of operation of the ozonizer was to obtain ozone from oxygen by an electric discharge, where the increase in voltage increased the concentration of gas. The experiment was carried out at the working ozone concentration 5.5×10^{-5} mol/L. The ozone concentration was determined in the UV range at a wave length of 254 nm. The gas flow rate was 100 mL/min, the ozonation time of material samples ranged from 3 to 5 minutes.

3. Results and discussion

In this paper, three main types of fiber distribution were identified: uniform (regular), medium (average) and chaotic (random). Figure 1 shows the microphotographs of different types of fiber placement in the material, which were obtained on a polarizing transmission microscope Polar-3 “Micromed” (Russia).

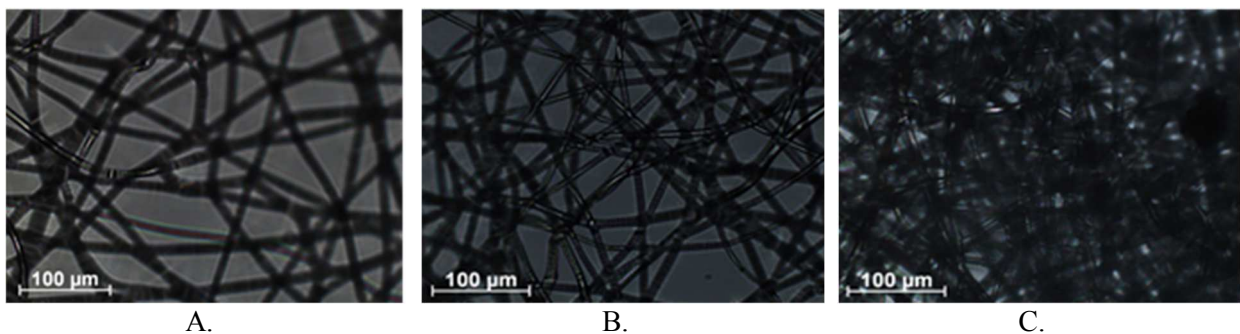


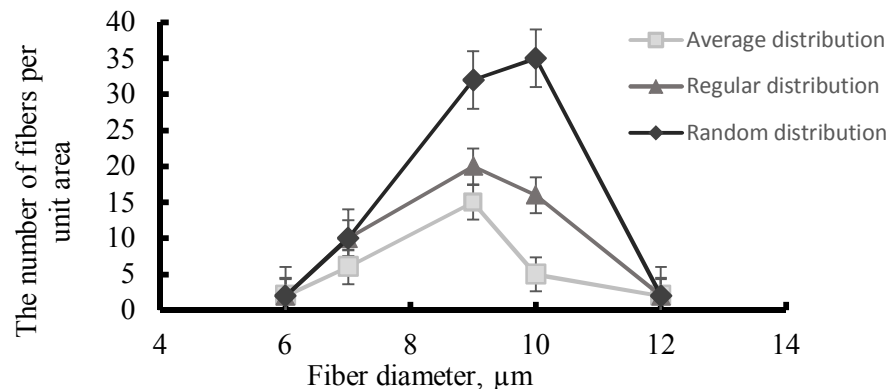
Figure 1. Microphotography of nonwoven fibrous material based on PHB: A – average distribution of fibres; B – regular distribution; C – random distribution.

A number of macrophysical characteristics that distinguish these materials and characterize the morphology of the fibrous layer are given in Table 1. The relative density of the structure expresses the proportion of fiber-free volume of the material and is related to the packing density of fibers in the porous layer of the material. Mainly, relative density varies in the range from 80 to 90 % for the materials obtained by the electrospinning method. The specific mass of the fibers in the material is described through the surface density of the layer. The orientation index of the fibers distinguishes the direction and specific character of their crimpiness per unit area with the established thickness of the fibrous layer.

Table 1. Main macrophysical characteristics of nonwoven fiber material based on PHB.

The main parameters of the structure	The type of fiber distribution		
	Regular	Average	Random
The index of orientation of the fibers, φ	0.74	0.67	0.36
Surface density, mg/m ²	36	26	16
Average diameter of fibers d, μm	8.6	8.1	9.2
Relative density γ , %	98	89	84

These characteristics together allow us to evaluate the efficiency of the electrospinning process, to prevent many defects on the fiber surface, elastic shrinkage and bonding of fibers during the curing of the jet on the electrode, as well as to affect the process of formation of functional properties, including physical and mechanical. The distribution of fiber diameters is also an important characteristic, as it allows to evaluate the uniformity and degree of variation of the characteristics of individual elements in the structure of the material. The distribution was estimated by a series of microphotographs, where the number of fibers and their average diameters per unit area (400 per 300 μm) were determined using direct methods of optical and scanning microscopy (Figure 2).

**Figure 2.** Dependency diagrams of fiber diameters depending on their number per unit area of the fiber layer.

In the course of the study of structural features of these materials, it was also found that with a decrease in the average fiber diameter increases the wryness, degree of tortuosity and density of fiber packaging, which leads to an increase in mechanical characteristics such as the breaking length of the material, little effect on the elongation at break. These characteristics of the macrostructure of nonwoven materials as a whole allow us to estimate accurately the average distance between the fibers, the density and type of their laying, the average diameters, deviations from the average values, the variation per unit area and the presence of defects. The irregularity of the materials obtained in this work did not exceed 10 %. Depending on the fiber distribution, the intensity of breaking varied from 1.4 to 2.2 N; and the relative deformation varied from 1.1 to 4 %. Based on the medical purpose of the materials, the regular distribution was chosen as the most optimal for evaluating the mechanical properties of non-woven fibrous cloth based on PHB, as well as their changes in the case of sterilization treatment with ozone. Moreover, the volume of gas absorbed during ozonation depended on the type of fiber distribution in the material structure and was 300-330 mole/m² for regular distribution; 400-440 mole/m² for average distribution and 450-480 mole/m² for random distribution. As a result of a series of experiments, it was found that under the influence of ozone the breaking load of nonwoven fibrous materials based on PHB increases by about 2 times. The results are shown in Table 2. Moreover, mechanical properties such as

modulus of elasticity, relative deformation and maximum elongation of the material up to the breaking point also increase markedly.

Table 2. Physical and mechanical properties of nonwoven fiber material based on PHB before and after ozone influence.

Type of the material	Intensity of breaking, N ($\Delta \pm 0.02$)	Modulus of elasticity, MPa ($\Delta \pm 2$)	Relative deformation, % ($\Delta \pm 0.2$)
PHB	1.7	39.8	3.4
PHB after ozon	3.5	58.8	7.6

PHB fibers are characterized by nonequilibrium structure. This is typical for materials obtained by extrusion, casting and electrospinning, when the fiber is extracted from solutions and melts. This causes the formation of overstressed areas of engagement and interlacing of macromolecules. These sites do not allow passing the complete relaxation and crystallization processes in polymeric material. During oxidation, these areas are torn in the first place [6]. Due to this, the PHB crystallizes, the number of defective zones in it decreases, and the degree of crystallinity grows. This leads to an increase in mechanical performance. Oxidation of PGB macromolecules can also be noted as an important reason for increasing the strength of the material after ozonation. The mechanism of ozone oxidation is characterized by an incensement of the number of functional groups. This causes an increase in the polarity of molecules due to the accumulation of oxygen-containing functional groups and, consequently, an increase in strength. Another possible reason for the increase in strength is the cross-linking of macromolecules after ozonation.

4. Conclusion

Based on the experimental data obtained, all samples of nonwoven electrospun fabric based on PHB in the established range of technological parameters can be divided into three groups that reliably describe the properties of the material structure: regular fiber distribution, average and random. The obtained data show a great potential of application of nonwovens based on PHB, obtained by the electrospinning method, for medical purposes, confirm the effectiveness of ozone sterilization of products based on these materials without compromising mechanical properties.

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