

Investigation of mechanical properties the polylactide in function its degradation rate

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Abstract. Polylactide is compostable and biodegradable thermoplastic polymer. It is made from renewable sources like tapioca roots, corn starch or sugar cane. Polylactide can be used as a material in food and packaging industries. According to manufacturer's instruction biodegradable bioplastics can be used for disposables applications such as cups, cosmetic packaging etc. In some cases, the type of stored substances can affected on container stability. The aim of the study was an investigation of mechanical properties of PLA depend on its degradation rate. Obtained results show degradation process of injection moulded PLA in simulated physiological fluids. Results of the study can be helpful to prediction of the behaviour of products manufactured with biodegradable polylactide.

1. Introduction

Determining the possibility of using material in new application requires the assessment of a number of different parameters. On the base it can be predicted its suitability for a particular application. Currently, as a result of the trend of limiting the harmful impact on the environment of human activities biodegradable materials are becoming more and more important. Currently, many everyday objects are made of them, such as shopping bags or packages of various products [1], [2]. But also, these materials are increasingly used in more advanced applications, e.g. as medical materials [3-7].

Is very important especially for biodegradable materials that before applying in a new role, it should be assessed what is the impact of working conditions on changing of mechanical properties of the material, also whether the material properties not change significantly. Too fast loss of their original properties may result in non-fulfillment of requirements by the finished product.

The presence of water [8] or water vapor [9] is significantly changing the original properties of products from biodegradable materials. The aim of the study was to assess the impact simulated physiological fluid environment on the change of basic mechanical parameters of a biodegradable polymer. This evaluation is needed to get the initial answer or application of this type of polymer is possible for manufacturing of containers for the storage of liquids as blood or urine. Such containers could be used as disposable products in medical applications. Its degradation time after use is shorter compared to almost non-degradable polymers, e.g. polypropylene.

2. Material and methods

A biodegradable polymer Ingeo Biopolymer 3100HP for injection molding was used for the research. Standardized A1 shaped samples made in accordance with the PN-EN ISO 527-1: 2012 polish standard were used in the research.



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Due to the fact that the samples were subjected to degradation, the change in the mass of the samples as a result of exposure to fluids whose degradation was carried out was also evaluated.

150 samples were used for the tests. All samples were weighed prior to testing. The 30 samples were then subjected to a uniaxial tensile test to determine the initial properties of the material. The others were divided into three groups of 40 samples in each. Each of them was placed for degradation in the following fluids: distilled water, PBS solution and 0.9 % NaCl solution. Due to the potential use as a medical product, degradation was carried out at 37 °C. Of each group, 10 samples were tested after one week. The study was conducted for 4 weeks. Before the test, after degradation, the samples were re-weighed to determine their mass changes during degradation. The AS 220.X2 balance was used to measure the mass change (Radwag, Radom, Poland).

The tensile test was carried out on an Instron 8874 testing machine (Instron, High Wycombe, England). The static strain extensometer, about gauge length 12.5 mm, was used to measure strain. The deformation speed was 1 mm / min, the frequency of data recording was 100 Hz. Figure 1 shows the exemplary sample during the test.

Due to the number of samples used for research, the U Mann-Whitney test was used to assess the statistical significance of observed changes in mass and strength of samples. Test was conducted with the assumed $p = 0.05$ value.



Figure 1. Exemplary sample during the test.

3. Results

The results obtained are shown in figures 2-8. Figure 2 presents graphs of mass change of samples stored in H₂O for a period of 4 weeks. The increase in polymer mass as a result of liquid absorption was the highest during the first week. An increase in the mass of the sample about 0.8 % was observed in relation to the initial mass. Over the next three weeks, the further weight gain was about 0.2 %.

Figure 3 shows the average change in the mass of samples stored in various fluids for 1, 2, 3 and 4 weeks. With time, is visible an increase in the scatter of results obtained for individual samples. The change in the mass of samples stored in PBS and 0.9 NaCl solution has a similar course as in water, in the first week there is a fast increase in sample mass, which is then slower.

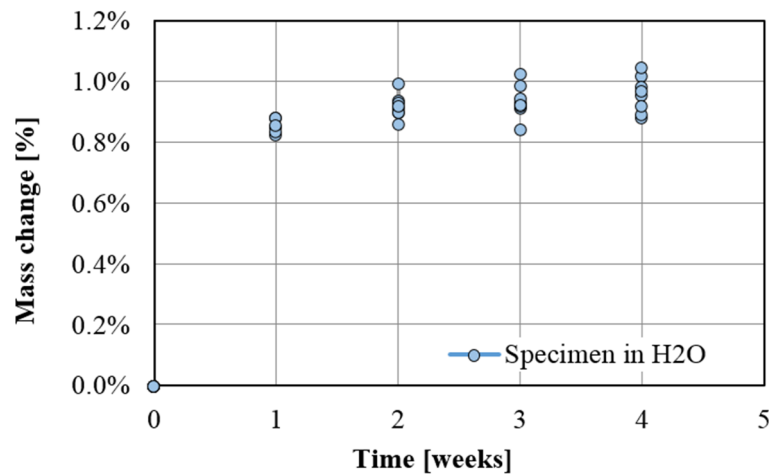


Figure 2. Mass change of samples stored in H₂O.

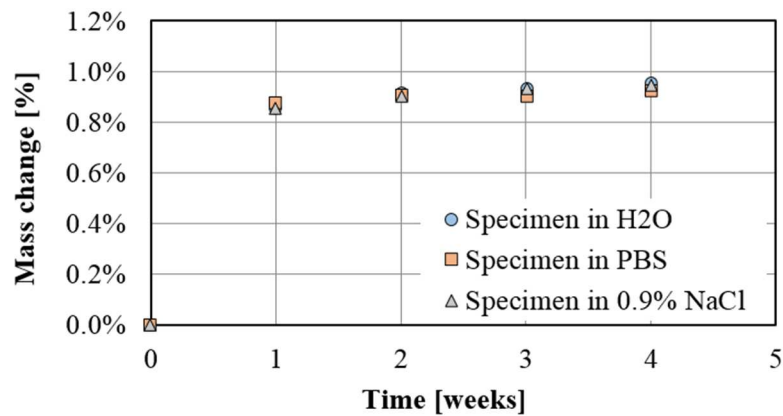


Figure 3. Average mass change of samples stored in various fluids.

Figures 4-6 show graphs from tensile test of samples degraded in various fluids for a period of 4 weeks. In this case, the similarity of biopolymer behavior can also be noticed. The initial value of tensile strength of polymer is about 58-59 MPa, the strain value for tensile strength is about $\epsilon = 0.022$.

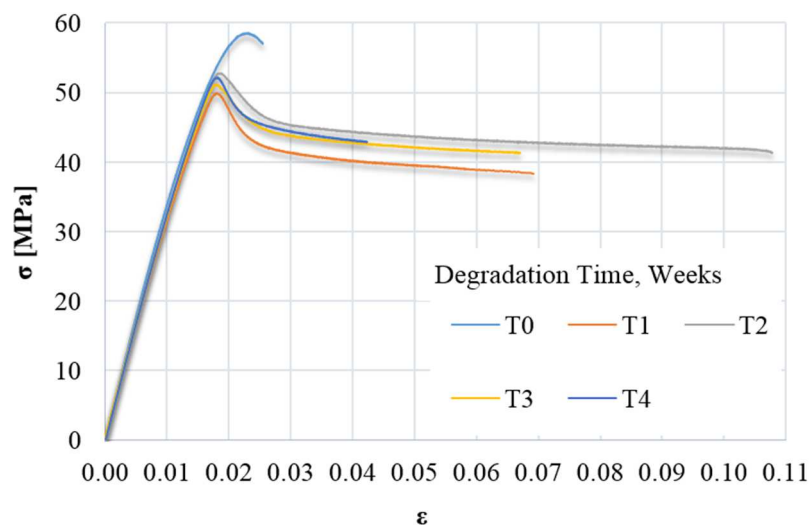


Figure 4. Tensile plots for samples degraded in H₂O.

The sample cracked at the value of ϵ about 0.026. After a week of storage, in all cases, a decrease in tensile strength up to about 50 MPa was observed. After 2 weeks, the strength value increased to around 53 MPa. The elongation value at which the sample was destroyed was about 0.08 - 0.11. The value of the elastic modulus was not calculated, but on the basis of comparison of shapes of curves on tensile plots is visible that the value have not significant changes during degradation.

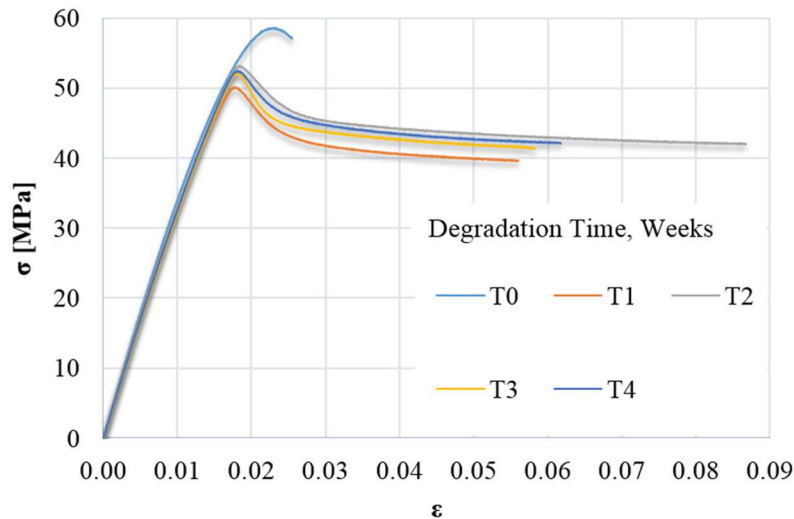


Figure 5. Tensile plots for samples degraded in PBS solution.

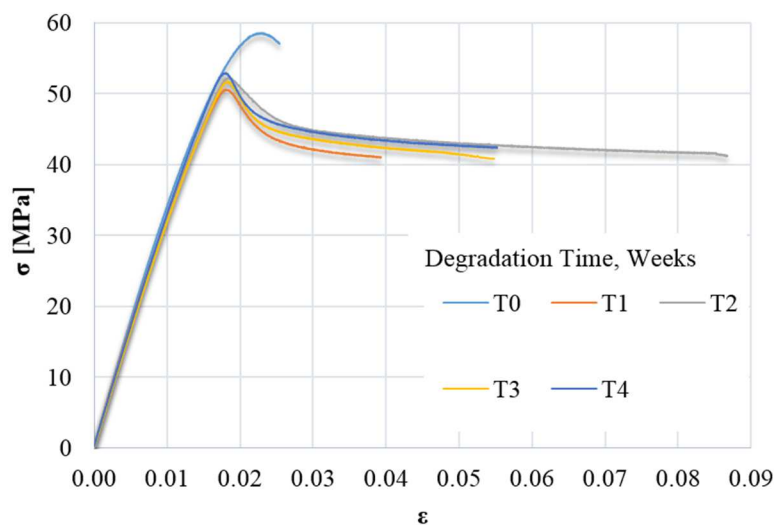


Figure 6. Tensile plots for samples degraded in 0.9 % NaCl solution.

Figure 7 shows changes in tensile strength over time for samples degraded in H_2O . Figure 8 shows the average value the changes obtained for samples degraded in various fluids. It can be noted changes in tensile strength for all groups have a similar character. Is visible a certain regularity of material behavior. After a strong decrease after the first week of degradation, the strength increased again. After 3 weeks, the drop-in strength was again observed, after a further increase of 4. With each subsequent week of degradation, the strength differences decreased.

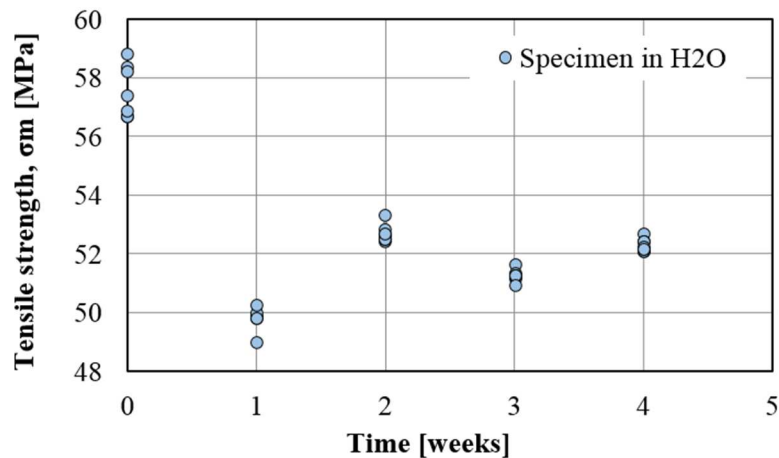


Figure 7. Changes in tensile strength for samples degraded in H₂O.

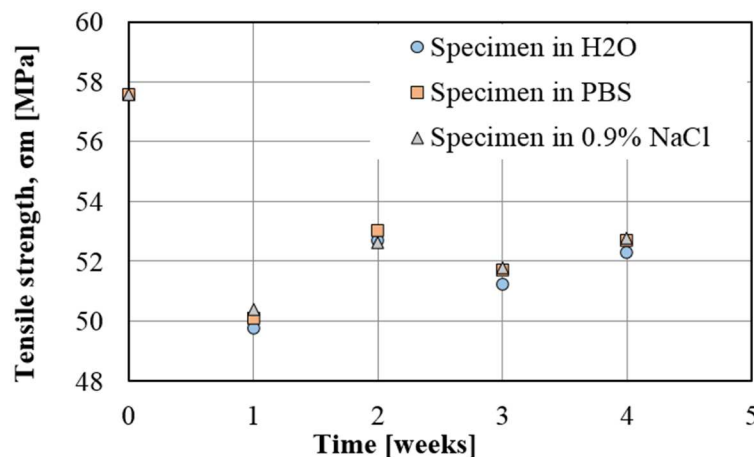


Figure 8. Changes in tensile strength for samples degraded in various fluids.

4. Discussion and conclusion

The aim of the work was to evaluate changes in tensile strength and biopolymer mass as a result of degradation in water, PBS and 0.9 % NaCl solution over a time of 4 weeks.

Based on the weight change study, it was found that the material being tested is a hygroscopic material. A statistically significant increase in the average mass of samples was obtained, regardless of the type of degradation medium. The increase is faster in the first degradation period and then slows down. Its character is similar regardless of the type of fluid in which the samples were degraded. It can therefore be concluded that the type of degradation fluid does not affect the rate of its absorption by the polymer.

Based on the obtained results, it was found that changes in strength and elongation of samples for strength are statistically significant over time. No statistically significant differences were found depending on the type of fluid in which the samples were degraded. Thus, just as in the case of mass changes, the chemical composition of the fluid did not affect the nature and magnitude of changes in the tensile strength of the polymer.

It has been noticed, however, that regardless of the type of degradation medium, the character of the σ - ϵ characteristic changes during the degradation. The material after absorbing the degradation fluid shows a behavior corresponding to the material with a pronounced yield stress. Nevertheless, no plastic deformation at the point of crack was observed on the samples destroyed during the tests.

It was found that the material does not undergo rapid degradation in the environment corresponding to the "in vivo" conditions. Assessing the nature of the changes in the examined mechanical properties, it was found that from this point of view there are no contraindications to the use of polymer in the planned applications of the tested time interval. In order to confirm this statement, additional tests on fatigue strength at constant-amplitude and with gradually increasing amplitude of loads [10] will also be performed.

The performed tests are preliminary tests aimed at assessing changes in mechanical properties of the polymer in various degradation fluids. Due to their positive result, they will be the starting point in further assessment of the material's suitability for the indicated applications. In subsequent stages, the impact of stored substances such as blood and urine on polymer will be made and vice versa. They will therefore be research in the field of chemical, biological and medical sciences. Such studies are already going beyond the field of tests used to study the mechanical properties of materials.

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