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The influence of enzymatic treatment on the modification of polylactide fibres surface properties

D. Wojciechowska, B. Lipp-Symonowicz, S. Kowalska, and I. Krucińska

Lodz University of Technology, Poland
Department of Material and Commodity Sciences and Textile Metrology
Centre of Advanced Technologies of Human-Friendly Textiles "Pro Humano Tex"
90-924 Lodz, 166 Zeromskiego
dorota.wojciechowska@p.lodz.pl

Introduction

Enzymatic treatment of polylactide fibres can be an effective, ecological and safe way of changing fibres' surface properties and preparing it for further processing by creation of active centres. As the enzyme acts only on the fibre's surface, the treatment doesn't affect the inner structure of fibre, preserving its initial properties.

For the study an enzyme Esterase was chosen as an hydrolising agent for polymer's ester bonds. Esterase was successfully applied as modifier of aromatic polyester PET fibres' surface by Kardas, et. al. [1]. Achieved surface development for esterase treatment was highest and the most uniform of all enzymes used. Also obtained increase in crystallinity degree and hygroscopicity was the most propitious for this enzyme.

Materials and methods

For the modification, commercial PLA SLN2660D, 6den/64mm polylactide fibres from Far Eastern Textiles Ltd. were chosen. Enzyme used and modification conditions are presented in Table 1.

Table 1. Modifying agent and conditions of its applications

Enzyme	Activity	Manufacturer	pH	Temperature of application	Time of treatment	Enzyme concentration	Sample weight	Liquor ratio
Esterase from *Bacillus stearothe-rmophilus*	≥0,2 units/ mg	Sigma Aldrich	7,2	60°C	30, 60, 120 min	1, 2 %	0,5000 g	1:200

Temperature of polylactide fibres treatment was just below the polymer's glass transition temperature. In first test enzyme was inactivated using pH 10 NaOH water solution. After the treatment samples were thoroughly washed with distilled water and dried in room temperature. Following, the weight loss was assessed.

Properties of modified fibres were evaluated with use of following methods:

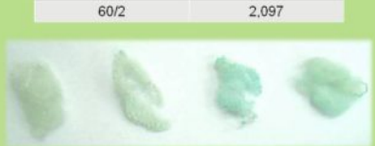
- Tensile strength - measured on Instron 5944 tensile testing machine equipped with the 250N grips. The test was performed in normal conditions, with the grips velocity of 5mm/min.
- Water sorption - was evaluated in Petri dishes filled with water to cover the samples. Samples weight was 0,1 g, temp. 37°C, time – 30, 90,150 minutes.
- Test dyeing - was carried out with use of basic dye – malachite green
- ATR spectroscopy - was performed on Thermo Scientific Nicolet 6700 FT-IR spectrophotometer. The results were prepared with use of ACD/SpecManager.
- SEM analysis - was carried out with use of FEI NOVA NanoSEM 230 microscope
- AFM analysis - was performed on Nanonics MultiView 1000 microscope.

Results

Basing on the measurements, the samples with most significant changes compared to reference samples were chosen. After the treatment tensile strength (tenacity) of fibres did not decrease significantly compared to the reference samples. Other results of conduced research are presented below.

Table 2. Weight loss of selected modified fibres and reference samples (%).

Sample - time of treatment min/concentration of enzyme %	Weight loss %
0/0	1,738
120/0	1,978
120/1	2,372
60/2	2,097



0min/0% 120min/0% 120min/1% 60min/2%

Figure 1. Results of the test dyeing of selected samples with malachite green dye.

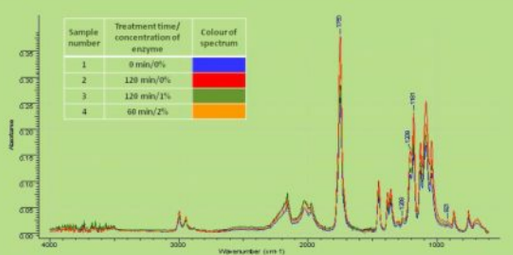


Figure 2. ATR spectroscopy spectra with the characteristic IR absorption bands listed.

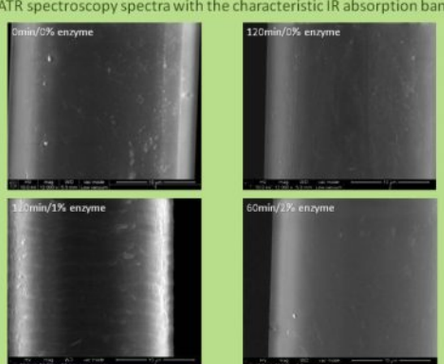
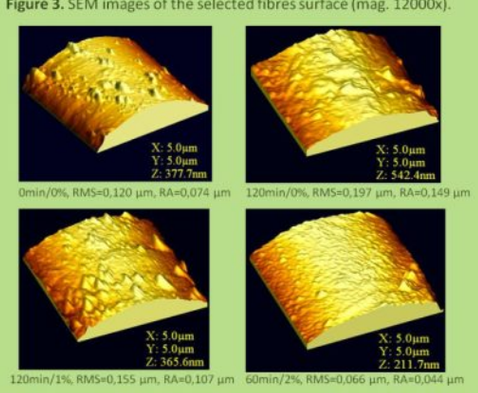


Figure 3. SEM images of the selected fibres surface (mag. 12000x).



0min/0%, RMS=0,120 µm, RA=0,074 µm 120min/0%, RMS=0,197 µm, RA=0,149 µm

120min/1%, RMS=0,155 µm, RA=0,107 µm 60min/2%, RMS=0,066 µm, RA=0,044 µm

Figure 4. AFM images of the selected fibres surface with the RMS and RA values.

Conclusions

The unchanged tenacity index after the enzymatic treatment indicates that the hydrolysing enzyme's activity only affects the fibre's surface. As a result of hydrolysing activity of enzyme on the fibre's surface layers, an increase in amount of carboxylic groups occurs. It is confirmed by dyeing test with use of basic dye but not enough clearly by spectroscopic measurement's results. As a result of hydrolysing activity of enzyme on the fibre's surface layers, AFM analysis demonstrates high surface development of treated fibres in comparison to other variants. Basing on the obtained results it can be seen, that the increase of enzyme's concentration affects the surface changes in a more drastic way. It decreases selectivity of enzyme's activity (surface smoothing), what is demonstrated by removal of surface layers causing higher weight loss for modified samples.

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References

[1] Kardas I.; Lipp-Symonowicz B. & Sztajnowski S.: The influence of enzymatic treatment on the surface modification of PET fibers, *Journal of Applied Polymer Science*, **Vol. 119** (2011), No 6, pp. 3117–3126, ISSN 0021-8995.