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Effect of biodegradation conditions on morphology of ternary compositions of low density polyethylene with poly(lactic acid) and starch

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Experimental

Materials

Low density polyethylene (LDPE-15803-020) (GOST (State Standard) 16337-77; m.p. = 107–110°C, M_n = 2.3 x 10^4 g/mol, MFI 0.2 g/10 min (190°C, 2.16 kg)), potato starch (OOO "Kholding "Slavnaya Trapeza", Russia) and PLA 4042D ("Nature works", USA) (M_w = 1.3 x 10^5 g/mol; dispersity index 1.8; m.p. = 155–170°C, transparency 2.1%) were the objects of the research.

Mixing

The polymers were mixed in a rotor disperser at 130°C under conditions of shear deformations according to a technique described in [S1]. The joint action of temperature and shear deformations is resulted in formation of the polymer powders. The polymers were loaded in certain time intervals and its residence time at every pass was about 5 min.

Pressing

The samples were pressed as films with a thickness of 0.18 – 0.25 mm on a CARVER press (USA) at 160°C and a pressure of 10 MPa for 10 min, followed by cooling under pressure at a rate of ~ 15 °C/min.

Biodegradability under environmental conditions

To study the biodegradation of the polymer blends under conditions modeling the environmental processes (ASTM D5988-12), the samples were placed into containers with wet soil (pH = 6.5) meant for plants ("GreenUP" trademark, "TD"Agroopttorg", Russia), and kept in a thermostat at

30°C. The biodegradation rate was controlled by the mass loss of the samples, the measurements were carried out in certain time intervals.

Tests on fungus resistance

The laboratory tests on fungus resistance were carried out according to GOST (State Standard) 9.049-91. The materials were treated with mold fungus spores from the All-Russian Collection of Microorganisms (VKM) under optimal conditions for their growth. Then the fungus resistance was estimated by the degree of the fungus growth. The evaluation of the fungus resistance by the intensity of the fungus development on the samples was carried out according to a six-point scale.

SEM imaging

The samples were cut as 5x5 mm sheets and were placed onto electro-conductive carbon tape adhered to SEM sample holder. Gold layer of 10 nm thickness was deposited on the sample surface inside spatter coater Q 150R ES (Quorum) followed by sample loading inside a chamber of Prisma E (Thermo Fisher) scanning electron microscope. SEM imaging was preferably performed at low voltage settings to demonstrate surface features of each sample.

FTIR-spectroscopy

The film sample was placed onto the stage of Fourier-transform infrared spectroscopy microscope (FTIR) Lumos II (Bruker) and was analyzed in attenuated total reflection mode (ATR). The probe was placed in 8 points over 1mm^2 surface of the sample and averaged signal was obtained. Each of the analyzed points has ~20μm diameter and penetration depth <5 μm associated with ATR mode.

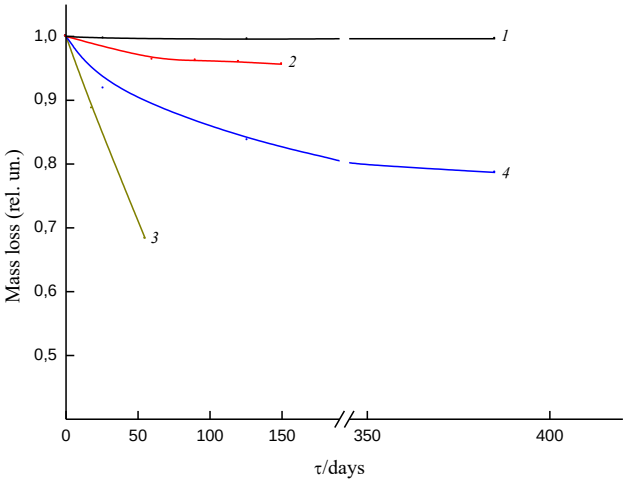
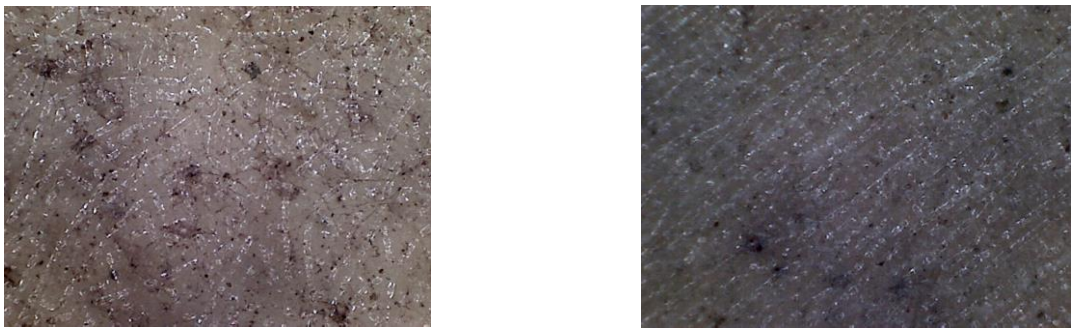


Figure S1 Mass loss curves of LDPE–PLA (60:40 wt %) (*1*), LDPE–starch (50:50 wt %) (*2*), PLA–starch (60:40 wt %) (*3*), LDPE–PLA–starch (40:20:40 wt %) (*4*) films after exposure in soil [S1–S4].

Here are presented the optical micrographs of the sample surface obtained after tests on fungus resistance. The whole surface is covered by peaks of a dark color, indicating the formation of the fungus fruiting bodies. In this case, the intensity of fungus development was rated with the highest point of 5, that is, the fungus growth covering more than 25% of surface is clearly seen by the naked eye [S2].



a *b*

Figure S2 Optical micrographs of film surface of LDPE–PLA–starch blends (50:30:20 wt%) (*a*) and (40:20:40 wt%) (*b*) after action of mold fungus spores in 84 days (x200) [S2].

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