Estonian Journal of Ecology, 2014, **63**, 1, 15–27 doi: 10.3176/eco.2014.1.02

Properties and behaviour of starch and rapeseed cake based composites in horticultural applications

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Received 14 October 2013, revised 23 November 2013, accepted 26 November 2013

Abstract. The application of composites consisting of starch, rapeseed cake, crude glycerol, and urea for the fabrication of disposable biodegradable nursery pots is estimated. Mechanical properties of composite films, their hygroscopicity, solubility in water, and water vapour transmission rate were studied. The evaporation of water rate from the plant pots prepared from the composites was found to be twice lower than that observed for the commercial peat plant pots. The fluctuations of the soil temperature were also lower than in the commercial peat pots. The average time of the biodegradation of the composites was about one month. The rate of biodegradation depended on the species of microorganisms and on their physiological properties.

Key words: biodegradable plant pots, composites, starch, crude glycerol, rapeseed cake.

INTRODUCTION

Plastic plant pots, containers, and trays are widely used in industrial greenhouses and in private farms. After use they end up on scrapheaps or are dumped in landfills where they degrade very slowly. In the total flow of agricultural plastic waste, which reaches ca 400 000 tonnes per year, the plastic pots and trays constitute ca 16 000 tonnes (www.greenfacts.org...). Biodegradable items represent a good alternative to plastic articles (Evans and Karcher, 2004; Nechita et al., 2010).

Among biodegradable goods, disposable plant pots made from peat or from a mixture of peat and wood fibre are most widely used. They can be either embedded into soil together with plants or digested. However, the peat plant pots have some drawbacks. They are mechanically unstable and have a high permeability to water vapour. The soil often dries out in such pots and the nutrient matter crystallizes in the walls of the pots. The salts concentrated in the walls can later harm the plants. The use of peat compost and other peat products leads to much larger discharge of carbon dioxide than that observed during the cultivation of soil (Maljanen et al., 2010; Verhoeven and Setter, 2010). For these reasons gardeners are not encouraged to use peat products. Moreover, peat digging is an environmentally problematic process. It is usually accompanied by the damage of vegetation and destruction of the fauna habitat.

The plant pots prepared from coconut fibre or from bird feathers are mechanically stable and retain moisture well (Evans et al., 2010). However, they can not be embedded into soil together with plants and can only be digested. In addition, the plant pots prepared from coconut fibre or from feathers are rather expensive. Therefore they can only be used when the exploitation time is relatively long. For short-time usages, such as cultivation of plants before planting into soil or into larger plant pots, cheaper biodegradable materials are required.

Plastic plant pots are light, cheap, and durable. Their walls are relatively impermeable. Plants cultivated in such pots require less watering; salts do not concentrate in their walls. However, the recycling of the waste plastic plant pots is still an unsolved problem. Therefore development of cheap biodegradable plastic plant pots with properties comparable to those of non-biodegradable plastic pots remains an urgent task.

Composites containing components of natural orgin are usually hygroscopic, and their films are permeable to water vapour. Their mechanical properties are dependent on the ambient moisture content. They can change with the migration of the plasticizer from the composites (Orliac et al., 2003; Ma and Yu, 2004). The water vapour permeability rate of the plant pots prepared from such composites and their ability to absorb and retain moisture can influence the temperature and moisture content of the substrate in the pots. These are important factors strongly affecting plant growth. In addition, entry of the components of the pot composites into the soil can change its agrochemical properties.

Properties of starch-based biocomposites depend to a large extent on the nature and extent of the plasticizer (Tudorachi et al., 2000; Bourtoom, 2008). Different plasticizers, such as amino acids, polyols, and compounds containing amido moieties are used for the improvement of their elasticity (Stein and Griene, 1997; Pushpadass et al., 2008; Muscat et al., 2012). However, an increase of the concentration of the plasticizer leads to an increase of the hygroscopicity of the composites and to a decrease of their tensile strength. The waterproofness of the composites can be enhanced by the cross-linking of the polymer, or by adding water-resistant fillers (Gáspár et al., 2005; Kumar and Singh, 2008). Thus, by specific variation of fillers, plasticizers, or their mixtures, composites with a desirable combination of mechanical properties, sorption capacity, and water vapour permeability can be obtained.

Biocomposites intended for the fabrication of disposable plant pots for short-time use have to be cheap and mechanically strong. Prepared from such biocomposites, they ensure the optimum water–oxygen regime and good plant growth. When embedded into soil together with plants such pots have to degrade quickly in order not to hinder the growth of roots. Fillers and plasticizers present in preferable biocomposite pots have to be environmentally friendly. It is desirable that the components of biocomposites intended for the fabrication of disposable plant pots for short-time use could fulfill some additional functions such as plant nutrition.

The aim of this study was to investigate the mechanical properties, sorption capacity, water vapour transmission rate, and biodegradability of composites prepared from starch, urea, and by-products of biodiesel production (rapeseed cake, crude glycerol). Finally, the performance of such composite plant pots was evaluated in plant growth experiments.

MATERIAL AND METHODS

Materials

Reagent grade soluble starch (from potatoes) and urea were purchased from Lach-Ner (Chech Republic). Crude glycerol and rapeseed cake were obtained from the biodiesel company ‘Mestilla’ (Lithuania). Their characteristics are given in Table 1. The rapeseed cake was ground and screened before use with a sieve of the mesh diameter of 0.16 mm.

Peat substrate (IST 4492998-01:1999) was purchased from JSC ‘Rekyva’ (Lithuania). Peat plant pots were purchased from JSC ‘Emolus’ (Lithuania).

Table 1. Characteristics of the used crude glycerol and rapeseed cake

Substance	Amount, %
Crude glycerol	
Glycerol	76.2
Moisture	16.5
Sodium phosphate	4.3
Methanol	Trace
Fatty acids	2.1
pH	6.25
Rapeseed cake	
Water	1.9
Proteins	46.2
Fats	0.9
Fibres	29.7
Soluble carbohydrates	13.6
Ash	7.9

Sample preparation

A total of 13 mixtures with different proportions of the components were prepared for making films. The compositions of the mixtures are given in Table 2. The mixtures were heated while stirring up to 85 °C during 15–20 min and stirred at this temperature for further 30 min. The films were cast from hot composite mixtures into polypropylene Petri dishes and dried in a ventilated oven at 25 °C for 48 h. The dried films were removed from the casting surface and stored at the ambient temperature of 23 ± 2 °C and 50% relative humidity (RH) in a desiccator containing a saturated solution of calcium nitrate.

Mechanical testing

Tensile tests were performed using a ZWICK/ROELL BDO-FBO.5TH testing machine with a crosshead speed of 10 mm min^{-1}. The test samples had a length of 45 mm and a width of 5 mm. Their thickness was measured with a digital micrometer (MEGA Dory Wybor, Poland). The presented values are the average of at least five measurements.

Solubility

Film samples (2 cm × 2 cm) were dried during 1 week in a desiccator with silica gel and weighed with the accuracy of ±0.1 mg (m_0). For the estimation of

Table 2. Composition of the mixtures used for composite preparation

No.	Components, g*			
	Starch	Rapeseed cake	Glycerol	Urea
1	3	5	2	0.2
2	3	5	4	0.6
3	3	7	2	0.6
4	3	7	4	0.2
5	4	6	3	0.4
6	5	5	2	0.6
7	5	5	4	0.2
8	10	0	3	0
9	8	2	3	0
10	6	4	3	0
11	5	5	3	0
12	4	6	3	0
13	3	7	3	0

* Water up to 100 g.

solubility, the samples were placed into a glass vessel with distilled water and kept at 20 ± 2 °C for 24 h. Then the samples were taken out and dried first at room temperature and later at 105 °C. After drying, the samples were cooled down in the desiccator and weighed (m). The solubility in water S (%) was calculated by the equation: $S = [(m_0 - m)/m_0] \cdot 100$.

Moisture absorption

To determine moisture absorption, the samples were dried in a desiccator with silica gel at 20 ± 1 °C for 1 week and weighed with the accuracy of ±0.1 mg (m_0). Then they were conditioned in a container at 54% and 100% RH at 20 ± 1 °C and were weighed at certain time intervals (m_1). The moisture absorption MA (%) was calculated by the equation: $MA = [(m_1 - m_0)/m_0] \cdot 100$.

Water vapour transmission rate

Flasks of 50 mL with srew-caps containing holes whose diameters were equivalent to the diameters of the necks of the flasks were filled with dry silica gel particles to maintain RH of 0% inside the flasks. The samples were placed on the necks of the flasks and srewed with the caps. The flasks were put into an environmental chamber at 20 °C and RH of 50 ± 5%. The flasks were weighed every 24 h with the accuracy of ±0.1 mg. The dependences $\Delta m = f(t)$ were plotted. Water vapour transmission rates (g m^{-2} day^{-1}) of the films were estimated from the linear parts of the plots.

Biodegradability

Samples of biocomposite films with a size of 30 mm × 30 mm were used for the biodegradability tests. A carbonless sample consisting of NaCl (3 g), NaH_2PO_4 (2 g), agar (18 g), and distilled water (1000 mL) was inoculated with microscopic fungi (*Paecilomyces variotii*, *Penicillium* spp., or *Trichoderma virens*). The suspension obtained was poured into Petri dishes with diameters of 110 mm. The control samples were placed into media containing no microscopic fungi. The samples were incubated for 28 days in a thermostat at 24 ± 2 °C.

The biodegradability of the test samples was estimated by evaluating their overgrowing with microsopic fungi using a five-point scale. The samples in which fungus growth was observed under a microscope were marked by zero points. The samples in which spores of germinated fungi developing unbranched hyphae were observed under a microscope were marked by one point. The samples with the mycelium consisting of branched hyphae were marked by two points.

The samples in which fungus growth could hardly be observed with the naked eye but was well visible under a microscope were marked by three points. The samples with fungus growth visible to the naked eye and the mycelium covering less than 25% of the surface were marked by four points. The samples with fungus growth visible to the naked eye and the mycelium covering more than 25% of the surface were marked by five points.

RESULTS AND DISCUSSION

Composites consisting of starch, rapeseed cake, crude glycerol, and urea were prepared as materials for making plant pots, and their properties were studied. Rapeseed cake was used as the filler, and crude glycerol and urea were used for the plastification of the composites. Phosphorus contained in the crude glycerol and nitrogen in the composition of rapeseed cake and urea are potential nutrients for plants.

Sorption properties

The sorption properties of a composite depend on the ability of its components to absorb water vapour. Of the components of the composites described in this paper, at RH of 100% glycerol showed the highest hygroscopicity (Fig. 1). For glycerol the absorption of water vapour reached 65% after 10 days.

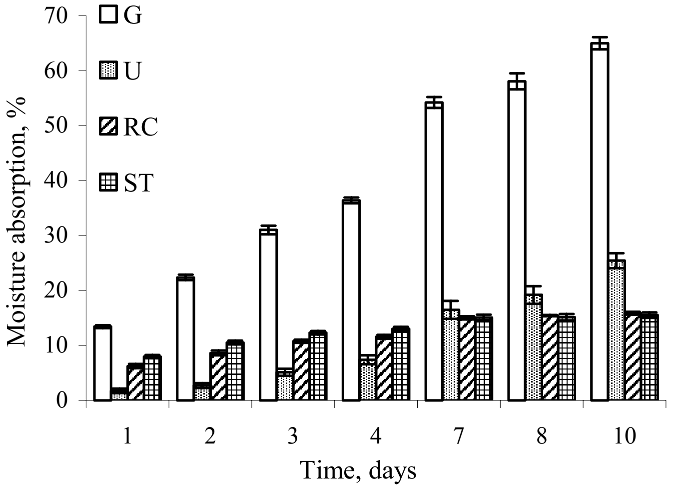

Fig. 1. Moisture absorption by starch (ST), rapeseed cake (RC), glycerol (G), and urea (U) as a function of storage time at relative humidity of 100%.

The water vapour absorption of urea strongly depended on the time of exposition in the air saturated with water vapour (Fig. 1). The degree of absorption increased from 1.7% after one day to 25% after 10 days.

The water vapour absorption of starch and that of rapeseed cake were found to be comparable. The absorption degree rose from 6.3% after one day to 15.8% after 10 days for rapeseed cake and from 8.3% to 15.5% for starch.

These results allowed us to predict that the absorption properties of the composites would depend on the amount of these components. Figure 2 shows the influence of the mass ratio of starch and rapeseed cake on the absorption properties of the composites versus a fixed amount of glycerol (Table 2, Nos 8–13). Since the hygroscopicities of rapeseed cake and starch are comparable, the change of the mass ratio of these components had no significant influence on the absorption properties of the composite films.

The moisture absorption of the composite films with different mass ratios of starch, rapeseed cake, glycerol, and urea obtained at RH of 54% and 100% is shown in Fig. 3. The saturation degree of the films with water vapour at 54% RH of air ranged from 25% to 30%. The moisture absorption of the films with a higher content of glycerol and urea (No. 2 and No. 4 in Table 3) was by 5–7% higher than that of the films with a lower content of the plasticizer. This difference was much more pronounced after storage of the films for 6–8 days at RH of 100%. The composite films of the composites with a higher content of glycerol markedly softened after a longer exposure to moisture. The degree of saturation of the composite films with moisture at 100% RH of the environment ranged from 50% to 65% depending on their composition. The saturation degree with water vapour of the peat plant pots at RH of 54% and 100% was 8.09% and 19.9%, repectively.

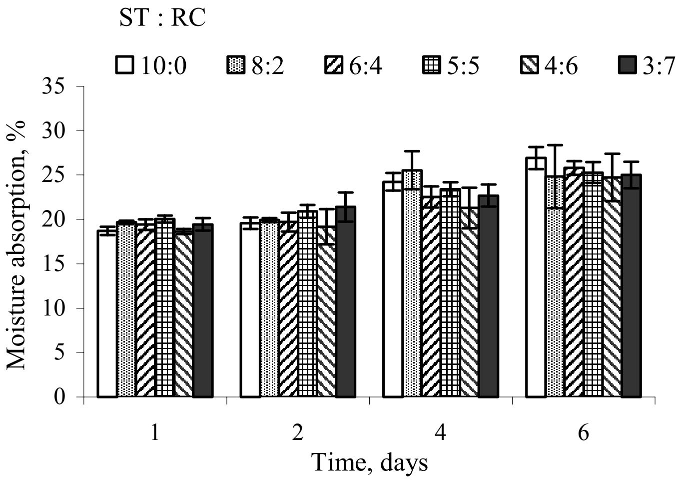

Fig. 2. Moisture absorption of films prepared with different starch and rapeseed cake mass ratios (ST : RC) at relative humidity of 54%.

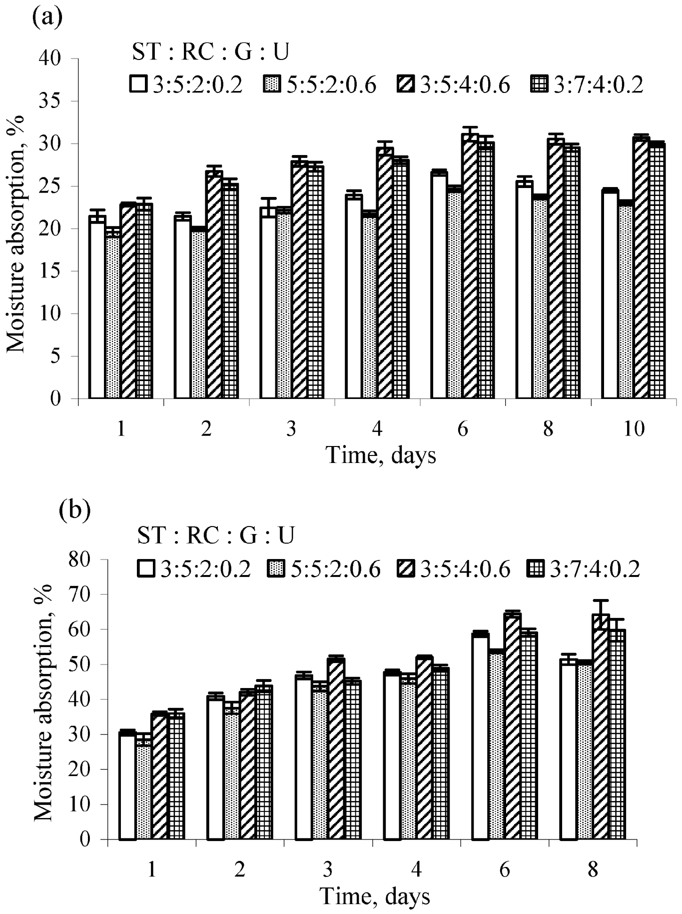

Fig. 3. Moisture absorption of films prepared with different starch, rapeseed cake, glycerol, and urea mass ratios (ST : RC : G : U) as a function of storage time at relative humidity of 54% (a) and 100% (b).

Table 3. Solubility and water vapour transmission rate of composite films based on starch and rapeseed cake and of a commercial peat pot

No.	ST : RC : G : U*	Thickness, mm	G/G + U, %	Solubility, %	Water vapour transmission rate, g m^{-2} day^{-1}
1	3 : 5 : 2 : 0.2	0.17	19.6/21.6	36.3 ± 2.5	133 ± 14.5
2	3 : 5 : 4 : 0.6	0.18	31.7/36.5	49.2 ± 0.75	192 ± 15.3
3	3 : 7 : 2 : 0.6	0.18	15.9/20.7	35.6 ± 0.96	125 ± 14.2
4	3 : 7 : 4 : 0.2	0.18	28.2/29.6	46.4 ± 1.32	182 ± 15.6
5	4 : 6 : 3 : 0.4	0.18	22.4/25.4	44.2 ± 1.75	154 ± 13.2
6	5 : 5 : 2 : 0.6	0.19	15.9/20.7	39.1 ± 1.79	121 ± 11.4
7	5 : 5 : 4 : 0.2	0.19	28.2/29.6	43.2 ± 1.29	151 ± 16.5
Peat plant pot		1.00	0	6.7 ± 0.23	293 ± 20.3

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* ST – starch, RC – rapeseed cake, G – glycerol, U – urea.

Mechanical properties, solubility, and water vapour transmission rate

Our study of the mechanical properties, solubility, and water vapour permeability rate of the composite films containing different amounts of starch, rapeseed cake, glycerol, and urea showed that depending on the amounts of components and their ratios, the mechanical properties of the composites varied in a wide range (Table 4). The tensile strength ranged from 0.7 to 7.7 MPa, the elongation at break from 5.5% to 12.5%, and the tensile modulus from 5.9 to 207 MPa. The main factors that determined the mechanical properties of the composites were the amount of glycerol and the ratio of rapeseed cake and starch. Films No. 2 and No. 4 (Table 4), which contained the largest amount of glycerol, showed the lowest tensile modulus and the highest elongation at break. The ratio of the amounts of rapeseed cake and starch in films No. 2 and No. 4 was respectively 1.67 and 2.33.

The mass ratio of the components in the composites strongly influenced also their solubility and water vapour transmission rate. The solubility of the composites was mainly influenced by the amounts of the water-soluble components, i.e. glycerol and urea (Table 3). The films containing smaller amounts of glycerol had a lower water vapour transmission rate.

Operational properties

For the investigation of the operational properties plant pots with a diameter of 6 cm were prepared from a composite film (Fig. 4). The mass ratio of starch, rapeseed cake, technical glycerol, and urea in the composite film was 4 : 6 : 3 : 0.4. Their characteristics in the cultivation of tomato sprouts were studied. Tests with commercial peat pots were performed for comparison. A special peat substrate (30 g) was placed into each pot and tomato sprouts were planted. The duration of the cultivation test was 14 days.

Table 4. Mechanical properties of composite films based on starch and rapeseed cake and of a commercial peat plant pot

No.	ST : RC : G : U*	RC/ST ratio	Tensile strength, MPa	Elongation at break, %	Tensile modulus, MPa
1	3 : 5 : 2 : 0.2	1.67	4.7 ± 1.05	8.7 ± 0.10	124 ± 6.03
2	3 : 5 : 4 : 0.6	1.67	0.74 ± 0.02	12.4 ± 1.48	5.9 ± 0.91
3	3 : 7 : 2 : 0.6	2.33	4.9 ± 0.05	7.3 ± 0.04	120 ± 2.65
4	3 : 7 : 4 : 0.2	2.33	1.9 ± 0.37	11.3 ± 1.04	14.3 ± 2.18
5	4 : 6 : 3 : 0.4	1.5	3.2 ± 0.46	5.5 ± 0.17	72.3 ± 6.32
6	5 : 5 : 2 : 0.6	1.0	7.7 ± 0.59	10.6 ± 1.18	207 ± 23.7
7	5 : 5 : 4 : 0.2	1.0	3.4 ± 0.74	11.6 ± 1.72	29.3 ± 6.67
Peat plant pot			1.5 ± 0.19	3.6 ± 0.12	24.8 ± 2.41

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* ST – starch, RC – rapeseed cake, G – glycerol, U – urea.

Fig. 4. Plant pots prepared from a composite based on starch and rapeseed cake.

The tests were performed in six peat plant pots and in six pots prepared from the composites. The volumes of the pots were equal. In order to estimate the moisture ratio, 20 mL of water was poured into each nursery pot. The pots were weighed after certain periods of time. Water fully evaporated from the peat plant pots within 38 h, while the evaporation from the pots made of the composite took 63 h. Since evaporation is an endothermal process, the different evaporation rates could be due to the different temperatures of the substrates in the plant pots (Fig. 5). The soil temperature in the peat pots was on average ca 4 °C lower than the ambient temperature, while that recorded in the pots made of the composite was 1.1 °C higher than the ambient temperature. Thus, under the same conditions

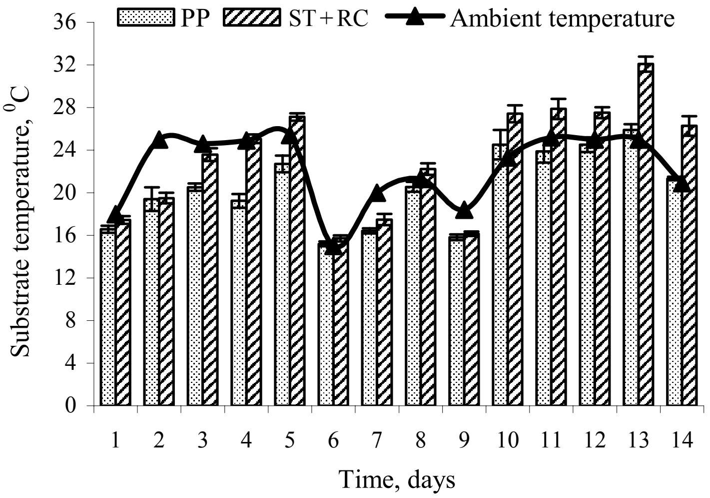

Fig. 5. Soil temperature versus time curves for peat plant pots (PP) and for the plant pots prepared from the composites based on starch and rapeseed cake (ST + RC).

the soil temperature and humidity were higher in the composite plant pots than in the commercial peat pots.

The differences in soil humidity and temperature did influence the vegetation of tomato sprouts. The tomato sprouts in the peat pots grew slower than those in the starch–rapeseed pots. After 14 days of cultivation the mass of the aboveground part of the plants in the composite pots was twice as high as that of the plants cultivated in the peat pots.

After 14 days, the plants were removed from the pots, and the pots were dried and weighed. The mass of the peat plant pots had increased by 7.3%, while the mass of the starch–rapeseed pots had decreased by 18.5%. This indicates that the peat pots had absorbed soluble substances from the substrate while the composite pots had released soluble substances, including nutrients, into the substrate.

Biodegradability

The only source of carbon in the biodegradability tests was the organic compounds of the composite films. Therefore, the overgrowing of the biocomposite samples with microscopic fungi indicated digestion of this element from the films and their biodegradation. Dependence of biodegradability on the film composition was observed even in the case of the control samples, in which background infectious microorganisms (*Aspergillus niger*, *Penicillium* spp., and bacteria) were present in the raw materials. The microorganisms grew the slowest on films No. 2 and No. 6 (Table 5), which contained the largest amount of urea. The biodegradability of these samples was evaluated as 3.7 and 3.3 points, respectively.

For a more precise estimation of biodegradability we used an additional contamination with microscopic fungi, i.e. *Penicillium* spp. and *Trichoderma virens*. After seeding with these fungi, the biodegradability of film No. 6 increased compared to that of the control sample and reached 4.3 points. Application of *Paecilomyces variotii* did not show such effect. Contamination with microscopic fungi of the film of sample No. 2 did not lead to an increase of its biodegradability. Moreover, the degree of biodegradation of film No. 2, contaminated with microscopic fungi, was lower than that of the control sample. Apparently the members of the community of microorganisms suppressed the activity of each other. The additional contamination with microscopic fungi did not enhance the biodegradation in the case of film No. 1 either: the overgrowing of the contaminated sample with microscopic fungi was lower than that of the control sample. In the case of the contamination with *Penicillium* spp. of sample No. 4, which contained a larger amount of rapeseed cake, the degree of biodegradation was comparable with that of the control sample.

For comparison we studied the biodegradability of commercial peat plant pots. The degree of their biodegradation was estimated as considerable. However, the shape of the samples remained unchanged. This observation can apparently be explained by the higher thickness of the walls of the peat pots, so the mass of the samples was larger and they required more time for the full degradation.

Table 5. Overgrowth of the samples with microscopic fungi after the incubation time of 28 days

No.	ST : RC : G : U*	Microscopic fungi	Overgrowth, points
1	3 : 5 : 2 : 0.2	*Paecilomyces variotii*	4.3
		Penicillium spp.	4.3
		Trichoderma virens	2.7
		Control sample	5.0
2	3 : 5 : 4 : 0.6	*Paecilomyces variotii*	1.3
		Penicillium spp.	1.3
		Trichoderma virens	2.3
		Control sample	3.7
4	3 : 7 : 4 : 0.2	*Paecilomyces variotii*	3.0
		Penicillium spp.	5.0
		Trichoderma virens	4.7
		Control sample	5.0
6	5 : 5 : 2 : 0.6	*Paecilomyces variotii*	3.0
		Penicillium spp.	4.3
		Trichoderma virens	4.3
		Control sample	3.3
Peat plant pot		*Paecilomyces variotii*	5.0
		Penicillium spp.	3.7
		Trichoderma virens	4.7
		Control sample	5.0

* ST – starch, RC – rapeseed cake, G – glycerol, U – urea.

Concluding the discussion of the results of the biodegradability study, we can state that all the studied composite samples were biodegradable: after 28 days all the films were degraded. The film biodegradation depended on the species of microorganisms and on their physiological properties, i.e. on their ability to assimilate particular organic compounds.

CONCLUSIONS

We estimated the possible application of composites consisting of starch, rapeseed cake, glycerol, and urea for the fabrication of disposable plant pots. We compared the properties of the pots with those of commercial peat plant pots. The pots made of films prepared from starch and rapeseed cake based composites showed an about two times lower transmission rate of water vapour and five times higher moisture absorption than the commercial peat plant pots. The moisture absorption ability of the composites depended on the mass ratio of glycerol and urea in their composition. This did not significantly affect the hygroscopicity of the composites. The duration of the evaporation of irrigation water from soil was longer than that observed for the peat plant pots made of composites under the same conditions. Fluctuations in the soil temperature recorded in the starch–rapeseed pots were smaller than those observed for the peat pots. The slower evaporation of irrigation water and smaller fluctuation in the temperature of the soil fostered faster vegetation of plants. The biodegradability of the composites depended on the species of microorganisms used and on their physiological properties. After 28 days, all the studied samples of the composite based on starch and rapeseed cake were fully degraded under the action of microorganisms.

ACKNOWLEDGEMENT

Financial support of this research by the Research Council of Lithuania (project No. MIP-057/2012) is gratefully acknowledged.

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