TOXICITY TESTING OF NONWOVENS USED FOR PRODUCTION OF RESPIRATORY PROTECTIVE EQUIPMENT

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SUMMARY

Objectives: During the covid-19 pandemic, protective equipment such as respirators and masks were widely used to protect respiratory tract. This disposable protective equipment is usually made from plastic fibre-based nonwoven fabrics. If used masks and respirators are improperly discarded, they pollute the environment by becoming a source of micro and nanoplastics. The aim of the study was to find out how stable the materials of protective equipment are and how released nano and microplastics can affect aquatic and soil organisms.

Materials: The input materials used to produce respirators and masks were tested for their thermal stability and resistance to the release of plastic particles into the environment. To determine the thermal stability of the materials, a simultaneous thermal analysis – thermogravimetry (TGA) and differential scanning calorimetry (DSC) were performed.

Results: Materials of masks and respirators are stable at temperatures common to temperate climate zone. However, the possible effects of chemical reactions of the materials with the environment were not considered during the measurement. The materials were also subjected to ecotoxicity tests according to European standards.

Conclusion: While the leachate obtained by shaking the materials in water did not show acute toxicity to the selected aquatic organisms, the material itself had a significant effect on selected soil organisms (springtails).

Key words: protective equipment, nonwoven fabrics, covid-19, ecotoxicity, environment

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INTRODUCTION

Papers dealing with the environmental impacts of micro and nanoplastics on individual components of the environment have been extensively published in scientific and popular literature in recent years. Micro and nanoplastics are ubiquitous – they are found in the atmosphere, waterways, seas, oceans, and soil. Similarly, they have been found to be very stable particles (1–3). Therefore, fine plastic particles can be identified as a new driver of global change with potential ecological impacts (4).

Following studies on the effects of micro and nanoplastics on aquatic organisms, scientific teams are now focusing on the interactions of plastic particles with plants, especially agricultural crops (5, 6); on the effects of plastic particles on soil organisms and soil enzyme activities; on the reduction of stability of soil aggregates; and on the effects on soil density and porosity (5, 7–11). In addition to toxicity to microorganisms and invertebrates (12, 13), attention is also paid to toxicity in mammals, including humans (14).

The toxicity of micro and nanoplastics depends on a number of factors, such as the mode of fragmentation of the raw material (mechanical action, biological degradation, photochemical oxidation), particle shape and size, concentration (quantity), exposure time, polymer composition and its stability to depolymerisation, surface character (hydrophobic, hydrophilic), adsorption of pollutants and surface, environmental conditions (pH, temperature, sanitation) (1, 15, 16).

In their critical review on the environmental impacts of microfibres on various environmental matrices Kwak et al. stated that there is still a lack of evidence of the toxic effects of microfibres at the level of primary producers in food chains (e.g. phytoplankton) although previous studies reported adverse effects of microfibres on organisms living in various ecosystems (17). Therefore, it is essential to continue to study the effects of micro and nanofibres on ecosystems as a whole, as well as on their individual components.

Our study focuses on the expected negative impacts of the covid-19 pandemic on ecosystems, in particular the increase of micro and nanoplastics in the environment. The pandemic has triggered a growing demand for personal protective equipment and protective gear. Nonwovens, including nanotextiles, are the basic material for the production of a range of disposable protective equipment. If the used protective equipment is improperly discarded, textiles consisting of plastic fibres enter the environment and become a source of micro and nanoplastics (18).

Nonwovens are produced in various ways from a range of plastic polymers. Production technology affects the properties of nonwovens. Spunbond (S) and melt blown (M) technologies are usually used to produce nonwovens, while nanotextiles are produced through electrospinning. Conventional spunbond textiles are made up of coarser fibres with larger diameters, while melt blown textiles have smaller diameters, including submicron filaments. Melt blown textiles feature a greater variability in fibre diameters than spunbond textiles. Spunbond fabrics have a much higher tensile strength and less pressure drop, while melt blown fabrics have excellent filtration properties. Layered fabrics are generally used in the production of protective equipment, where spunbond and melt blown fabrics are combined in various ways, e.g., SM or SMS fabrics. To achieve better filtration properties, melt blown fabrics are combined with nanotextiles (19).

MATERIALS AND METHODS

Protective Equipment Material Used in Tests

The fabric samples were provided by the respirator manufacturer, NAFIGATE Corporation, Ltd. Two layered nonwoven fabrics were tested. The internal structures of the fabrics are shown in Figure 1. The base of both samples consists of polypropylene (PP) fibres produced by S and M technologies. Subsequently, the electrospinning method was used to apply nanofibres of polyvinylidene difluoride (PVDF) from dimethylacetamide solution on the M layer.

The tests were performed on samples with the nanolayer exposed and on laminated samples where the nanolayer was covered with an additional layer of PP fabric produced by the S technology. Microscopic images of the surface of the tested nonwovens are shown in Figure 2. The cross-sections of the used textiles are presented in Figure 3.

Methods Used to Test Physicochemical Properties of the Material

While previous tests focused on the release of micro and nanoplastics from the test material into the atmosphere during squeezing, wiping, tearing, cutting, and exposure to air pressure (20), the focus of this study was on the stability of the materials in aqueous environment. Samples of both the materials of approximately 3×3 cm in size were shaken (120 rpm) in 100 ml of demineralized water at laboratory temperature for 24 hours. The

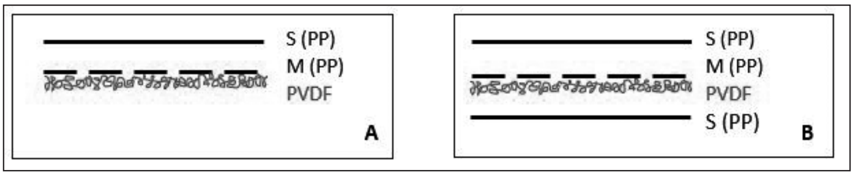

Fig. 1. *Simplified diagram of internal structure of tested layered textiles.*

A – textiles with nanofibre layer exposed; B – textiles with laminated nanofibre layer

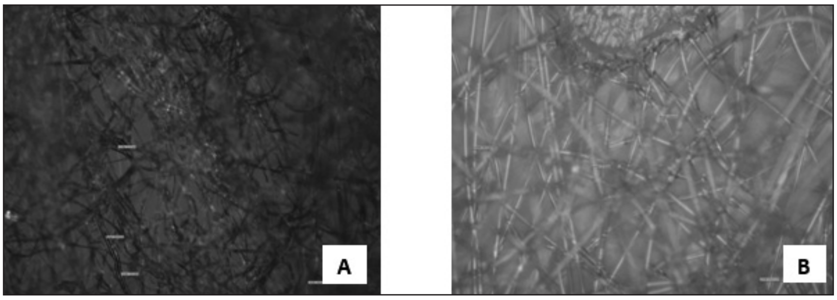

Fig. 2. *Microscopic images of tested nonwovens surface.*

A – view of exposed PVDF nanolayer; B – view of PP fibre layer produced with spunbond technology (magnified 20x)

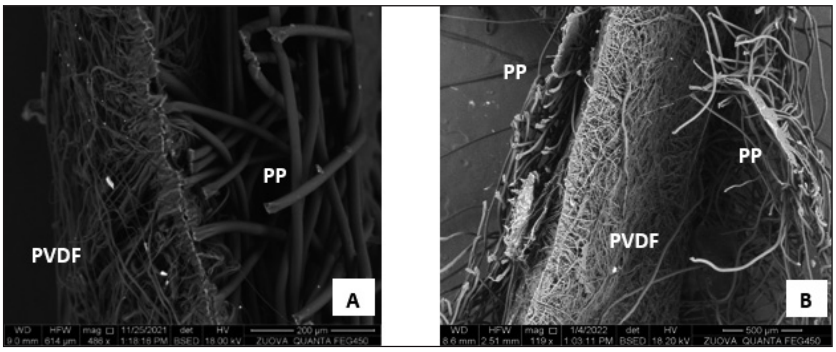

Fig. 3. *Cross-section of layered nonwoven fabric.*

A – non-laminated fabric composed of S(PP) layer and PVDF nanolayer (magnified 485x); B – laminated fabric where PVDF nanofibre layer is enclosed between two PP layers (magnified 120x) obtained leachate (aqueous suspension) was filtered through a membrane filter (Pragopor 0.4 μm). The filtrate was then analysed for its total and non-purgeable organic carbon content (Skalar, Formacs^{HT-I}, The Netherlands). The filter membrane was analysed with scanning electron microscopy (Quanta 450 FEG) to detect the presence of potentially released micro and nanoplastics. In view of the achieved results, the experiment was then repeated, while the shaking took place for twice as long – 48 hours. The obtained leachates were firstly concentrated by classic centrifugation, then by ultracentrifugation directly on the TEM grid. The prepared samples were analysed using transmission electron microscopy (TEM, Hitachi HT7800, accelerating voltage 100 kV) (the analysis was performed at the electron microscopy workplace of the National Institute of Public Health).

To determine the thermal stability of the material, a simultaneous thermal analysis – thermogravimetry (TGA) and differential scanning calorimetry (DSC) (Mettler Toledo, USA; experimental range 25–750°C, 20K.min^{-1}, Air 50.min^{-1}) were performed on the laminated material. Simultaneously, the thermal analysis was also performed on the nanofibre layer alone, which was obtained for this measurement from the non-laminated material by stripping the PVDF layer from the supporting PP fabric.

For the laminated textile, whose properties are the closest to protective equipment, the BET method was applied to determine the specific area (Sorptomatic 1990, Thermo Finnigan, Italy).

Ecotoxicity Testing Methods

Ecotoxicity of the selected nonwovens was verified at the workplace of the accredited testing laboratory of ABITEC, Ltd. The tests were carried out according to European standards (Table 1).

The tests carried out on aquatic organisms used leachates obtained by shaking 10×10 mm squares of nonwoven fabric. The water leachate was prepared according to EN 12457-4 (21); the leaching time was 24 hours and the liquid and solid phases were separated on a 4 mm mesh sieve.

The tests of inhibition/stimulation of nitrification activity and the tests on soil organisms used fabric squares of approximately 5×5 mm mixed with artificial soil in a specified weight ratio.

All ecotoxicity tests performed were always carried out strictly according to the stated procedure and conditions, which are defined in the above-mentioned EN ISO standards. Both parallel determinations were carried out, in which the resulting values were arithmetically averaged, and the samples were subsequently compared with control determinations so that the resulting values were valid and their validity is guaranteed by the accredited laboratory ABITEC, Ltd.

RESULTS

Tests on Physicochemical Properties of the Material

After filtration of the leachate obtained by shaking the laminated (LAM) and non-laminated (NONLAM) materials, no organic polymer nanofibres were found on the filter membrane by scanning electron microscopy (SEM). For the NONLAM material, some fine spherical particles of organic origin were detected on the membrane (Fig. 4A), which were also identified on the PVDF fibres of the initial material (Fig. 4B). It can be assumed that this is an unfibrillated PVDF polymer.

Surprised by the minimal quantity of plastic particles released into the aqueous leachate, we repeated the experiment with approximately 9 cm^2 of LAM and NONLAM material samples in 80 ml of distilled water for 48 hours. The analysis of these longer prepared leachates using TEM also provided similar results to SEM. In the case of LAM textile, fragments of nanofibres were occasionally found in the samples (samples processed by classic centrifugation and ultracentrifugation did not show any differences). Spherical particles with a size of 1.5–2 μm and a fibre fraction were recorded in the NONLAM leachate after classical centrifugation, as in the case of SEM. After ultracentrifugation, several clusters of nanofibres (diameter around 15 nm, length from 100 nm to several μm) and occasional micrometer-long fibres were found (Fig. 4C and 4D).

The course of the simultaneous thermal analysis for the laminated material is shown in Fig. 5 and for the nanofibre PVDF layer alone in Fig. 6. The curves show that at usual temperatures of the environment both of the tested materials are stable and they are not thermally decomposed.

For the LAM material, an endothermic effect was first observed without weight loss in the range of 120–215 °C (change of structure, melting of the material), followed by an exothermic effect in the range of 215–425 °C (maximum at 412 °C), which was gradually accompanied by a complete weight loss (decomposition of the material) and was terminated at 550 °C.

The thermal analysis of the PVDF nanotextile layer alone followed a similar course. Initially, an endothermic effect without weight loss was observed in the range of 135–210 °C (change in structure, melting of the material). This was followed by an exothermic effect, which was accompanied by a weight loss of

Table 1. *Overview of standards used for ecotoxicity tests of nonwovens*

	European Standard designation	European Standard name
Water quality	EN ISO 8692:2012	Water quality – Fresh water algal growth inhibition test with unicellular green algae
	EN ISO 11348-2:2009	Water quality – Determination of the inhibitory effect of water samples on the light emission of *Vibrio fischeri* (Luminescent bacteria test) – Part 2: Method using liquid-dried bacteria
	EN ISO 6341:2012	Water quality – Determination of the inhibition of the mobility of *Daphnia magna* Straus (*Cladocera*, *Crustacea*) – Acute toxicity test
Soil quality	EN ISO 15685:2012	Soil quality – Determination of potential nitrification and inhibition of nitrification – Rapid test by ammonium oxidation
	EN ISO 11267:2014	Soil quality – Inhibition of reproduction of Collembola (*Folsomia candida*) by soil contaminants

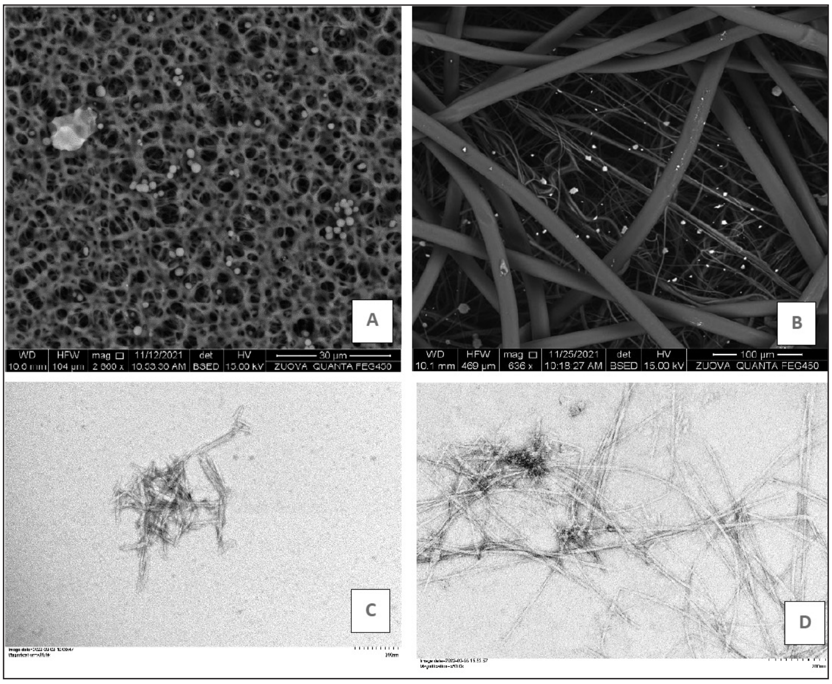

Fig. 4. Representative image.

A – SEM of leachate filter membrane (magnified 2,860x); B – SEM of initial non-laminated material (magnified 635x);
C – TEM of leachate from initial laminated material (magnified 40,000x); D – TEM of leachate from initial non-laminated material (magnified 50,000x)

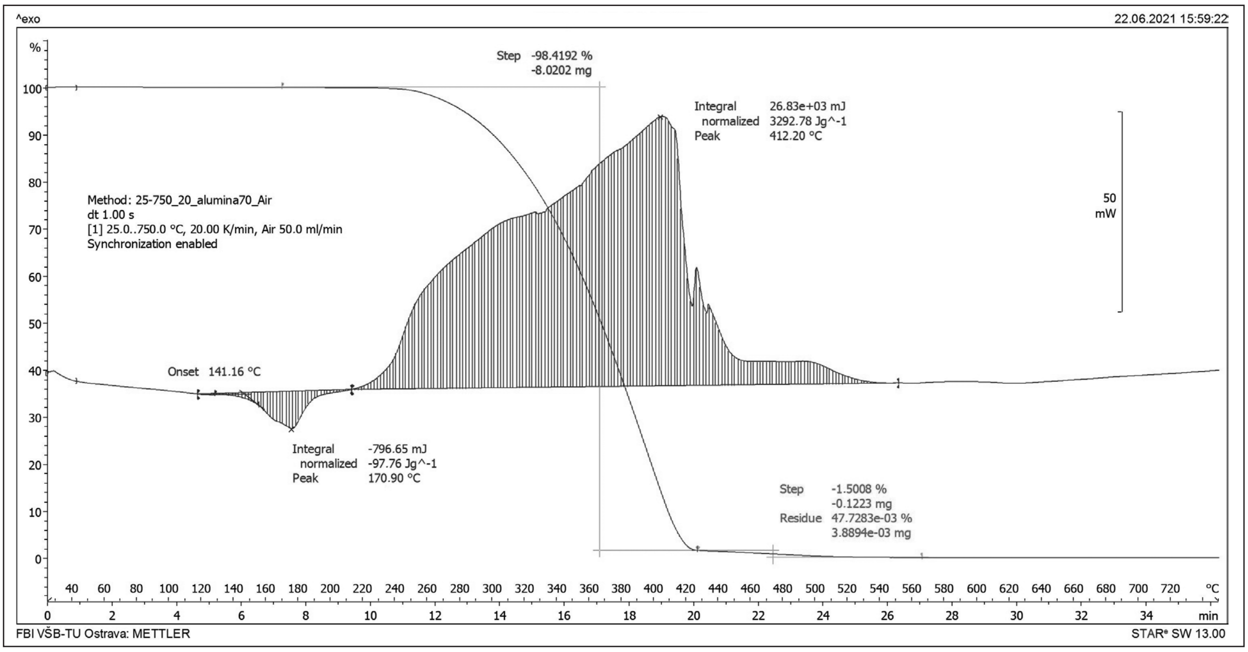

Fig. 5. Graphical representation of simultaneous thermal analysis.

Thermogravimetry (TGA) and differential scanning calorimetry (DSC) for laminated material (LAM); sample: PVDF – laminated, 8.1490 mg and silk TGADSC

96% in the range of 210–440 °C. The exothermic effect continued with two peaks at 450 °C and 509.7 °C. The decomposition of PVDF was terminated at 640 °C. The difference compared to LAM was 110 °C.

Laminated Product PP-PVDF-PP

The controlled sorption method (BET analysis on Sorptomatic 1990, Thermo Finnigan, Italy) provided the so-called adsorption isotherm (Fig. 7) for the laminated material, indicating the dependence of the adsorbed gas on the pressure at a constant temperature. The specific surface area of LAM was determined to be 4.29 m^2/g.

The monomolecular layer volume was 0.9850 ml/g. The volume of micropores and mesopores was not detected while macropores dominated with VMA = 0.253 ml/g.

Ecotoxicity Results

Results of tests carried out on aquatic organisms (according to the standards listed in Table 1) are provided in Table 2.

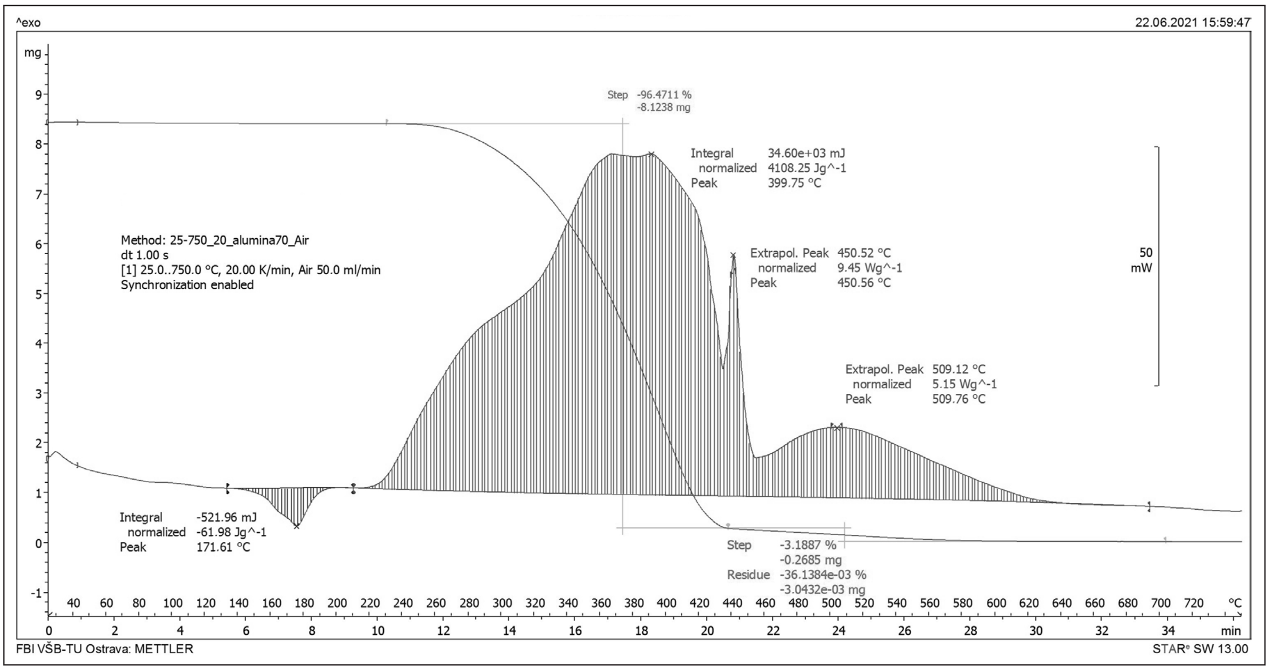

Fig. 6. *Graphical representation of simultaneous thermal analysis.*

Thermogravimetry (TGA) and differential scanning calorimetry (DSC) for PVDF nanofibre layer alone; sample: KK PVDF – nanofibre, 8.4210 mg

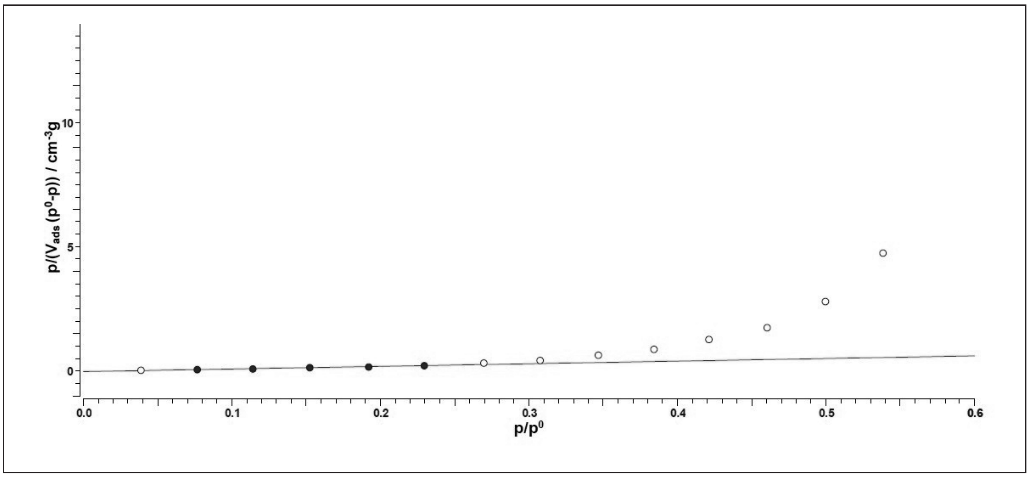

Fig. 7. *BET isotherm of laminated product (PP-PVDF-PP).*

The rapid ammonium ion oxidation test (incubation time 6 h) showed an increased nitrification activity compared to the control sample. For the NONLAM sample, the value of inhibition of nitrification activity was I = −14.8%, and for the laminated sample the value was I = −24.6%.

Springtails (*Folsomia candida*), tiny arthropods living in soil, were exposed to artificial soil with fabric cuttings for 28 days (Fig. 8). Results of the test are shown in Table 3. For the sample containing laminated fabric there was a statistically significant difference in inhibition of springtail reproduction (I = 57.9%) in comparison with the control sample.

Table 2. *Results of tests carried out on freshwater microorganisms*

Material used for leaching	**Inhibition of light emission by *Aliivibrio fischeri* (%)**		**Inhibition of mobility *Daphnia magna* Straus (%)**	**Growth rate inhibition of *Desmodesmus subspicatus* (%)**
	Exposure 15 minutes	Exposure 30 minutes	Exposure 48 hours	Exposure 72 hours
LAM (PP-PVDF-PP)	5.6	1.5	0	−0.7
NELAM (PP-PVDF)	0.8	3.2	0	3.3

Fig. 8. *Control sample (left) and a sample prepared by mixing of artificial soil with nonwoven fabric cuttings.*

Table 3. *Results of springtail reproduction inhibition test (exposure period 28 days)*

Sample	Sample No.	c (g/kg)	n (pcs)	CV (%)	I (%)	p-value
Control (artificial soil)	–	0	731	17.6	–	
LAM (PP-PVDF-PP)	17043	166.7	308	19.9	57.9	0.0035
NELAM (PP-PVDF)	17044	166.7	652	2.0	10.8	0.3502

c – concentration of test sample; n – average number of juvenile springtails in the control sample or the test sample at the end of the test (pieces); CV – coefficient of variation of parallel determinations; I – inhibition of springtail reproduction compared to the control sample

DISCUSSION

The simultaneous thermal analysis curves show that at normal ambient temperatures the material used for the production of face masks and respirators is stable and does not undergo thermal decomposition. However, in addition to thermal resistance, mechanical and chemical resistance must also be considered. The selected properties influencing the release of plastic particles into the environment (e.g. tensile strength) are discussed in the article of Roupcová et al. (20). Stability of the material in the environment is also influenced by exposure to solar radiation, oxygen and other gases, pH (rainwater, surface water, soil), as well as temperature fluctuations between day and night.

Although a number of studies have highlighted the increasing presence of micro and nanofibres in the environment (1–3), when textiles (LAM and NONLAM) were shaken with demineralized water for short periods of time, no evidence of polymer fibres was found in the resulting leachate. This result does not correspond to the results of measurements performed on textiles in dry environments, where significant quantities of particles were released from the textiles by the action of airflow (20). This is probably because of the fact that the nonwoven fabrics used for the tests were non-wetting and non-polar. The completed BET analysis has shown the prevalence of macropores in the LAM textile that further increase its hydrophobicity (22).

The absence of nanofibres in the aqueous leachate may have affected the results of short-term ecotoxicity tests carried out with various aquatic microorganisms. The tests indicate that the leachates from nonwoven textiles do not show acute toxicity in the selected organisms. However, based on results obtained in experiments with *Daphnia magna*, it can be concluded that even if PP particles were released into the leachate, they would not be acutely toxic for the crustaceans used in our tests (23).

The medium-term tests (28 days) carried out with *F. candida* in artificial soil with the addition of fabric cuttings, however, provided different results. A statistically significant inhibition of reproduction was found in springtails. This finding corresponds with the results obtained in experiments where springtails were chronically exposed to polyethylene microparticles in various concentrations, resulting in a severe reduction in their reproductive capacity (up to 70%) (24). This may be due to the toxicity of unfibrillated PVDF particles, as well as the toxicity of textile fibres released when mixing the textile cuttings with artificial soil. In addition to soil microorganisms, the presence of micro and nanoplastics in the soil also affects plant germination and root growth (5, 20). From the above, it follows that soil ecosystems can be affected by the presence of micro and nanoplastics at least in the same way as was demonstrated in relation to aquatic ecosystems (3, 25).

CONCLUSION

We can conclude that in medium-term tests we have demonstrated the ecotoxic effects of the test materials in contact with soil and soil organisms. This confirms certain concerns and raises questions about the effect of micro and nanoplastics in soil, as demonstrated above.

Due to the evidence of toxicity in soil organisms and its impact on soil processes, we highly appreciate the European project

Macro and Microplastic in Agricultural Soil Systems (SOPLAS) funded by the European Commission. The objective of the project is to bring together workplaces studying the behaviour of microplastics in soil, including their movement in the soil profile together with water. The project links 14 sub-projects and 20 research sites in total. The Czech Republic is represented by the Faculty of Civil Engineering of the Czech Technical University in Prague.

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Conflict of Interests

None declared

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