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Nanotextiles – materials suitable for respiratory tract protection but a source of nano- and microplastic particles in the environment

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Abstract: The paper deals with the formation of nanomaterials (nanoparticles and nanofibers) in the manufacture and use of respiratory protective equipment. It focuses mainly on processes leading to the release of nanoplastics into the workplace and the environment. Based on selected properties of materials used for the manufacture of protective equipment, their stability in the environment is revealed. The paper demonstrates the impact on the environment considering semichronic phytotoxicity of nanoplastics.

Keywords: nanofiber, electrospinning, nonwovens, nanoparticles, respirator

Introduction

Numerous studies (Waymana and Niemann, 2021; Gerritse et al., 2020; Brewer et al., 2021; Pirsaheb et al., 2020; Pivokonský et al., 2020; Novotná et al., 2019) have confirmed the occurrence of nano- and microplastics in the waters of the world's oceans and fresh waters, including drinking water. Similarly, the presence of nano- and microplastics has been confirmed in soil (Brewer et al., 2021; Wahl et al., 2021). Primary source of nanoplastics in nature is the production and processing of plastics while secondary source of nanoplastics is the fragmentation of plastic products (Barnes et al., 2009). Fragmentation of plastics is the result of a number of processes which depend on both the composition of plastics and environmental conditions. Fragmentation involves physical, physico-chemical, and biological processes (cracking, friction, photo oxidation, hydrolysis, biodegradation) (Gerritse et al., 2020; Barnes et al., 2009; Enfrin et al., 2020).
Nano- and microplastics released into the environment affect organisms present in the environment. The best studied problem (although still not sufficiently) is currently the effect of nano- and microplastics on aquatic organisms. The effect on living organisms depends on the concentration of plastic particles, their size, and shape, the material from which they originate, and the time of exposure (Kögel et al., 2020; Barboza et al., 2018). Toxic effects of nano- and micro-plastics are therefore influenced, for example, by flame retardants present in polymers, organic pollutants adsorbed on the plastic surface, pesticides, heavy metals (Brennecke et al., 2016), or antibiotics (Guo and Wang, 2019). Plastic particles affect the behavior and neurological functions of fish, intestinal permeability, metabolism, and diversity of their intestinal microbiomes (Barboza et al., 2018; Jacob et al., 2020). At the same time, they affect the immune system of fish (Barboza et al., 2019), their hormonal regulation and reproduction, they alter fat metabolism, and cause oxidative stress in cells (Kögel et al., 2020). The number of negative effects observed in fish increases as the size of the plastic particles decreases (Jacob et al., 2020; Lee et al., 2013).
Nano- and microplastics enter the soil in various ways. One of them is pollution from water and wastewater treatment plants (especially particles and fibers released during the laundering of clothes). Contamination from water treatment plants reaches higher levels in soils than in water (Wahl et al., 2021; Nizzetto et al., 2016). Nano- and microplastics in soils affect soil micro- and macroorganisms and their condition, the soil properties and thereby even the cycles of substances in the soil (Wahl et al., 2021; Maity and Pramanick, 2020).
The current pandemic of the COVID-19 respiratory disease has increased the demand for respiratory protective equipment, especially masks and respirators and those including nanotextiles made of plastic nanofibers are especially popular and widely used. Due to the number of nanotextile-based masks and respirators used daily, their production, use and improper and unregulated disposal contribute to and will continue to contribute to increasing environmental pollution by nanoplastics (Sullivan et al., 2021). Therefore, we decided to identify processes that can be considered as sources of nanoparticles in the life cycle of protective devices containing nanofibers, and to further investigate them.
The following were specified as processes that lead to the release of nanoparticles into the work and natural environment:

1. Production of nanofibers by electrostatic spinning (leakage into the working environment, exposure of employees),
2. Production of nanofiber respirators and nanofiber masks by processing textiles containing nanofibers (leakage into the working environment, exposure of employees),
3. Use of nanofiber respirators and nanofiber masks, especially the mechanical stress of nanofiber textiles that are part of them (leakage into the environment, exposure of people in the vicinity),
4. Improper disposal and removal of used nanofiber respirators and nanofiber masks (disposal into elements of the environment – water, soil); effect of pollutant particles adsorbed on nanotextiles or absorbed (intercalated) undesirable substances between layers of nonwoven fabric and nanotextile changing their toxicological properties may be present.

Assessment of protective equipment as a possible source of nanoplastic particles

Production of nanofibers by electrostatic spinning

Nanotextile-based protective devices usually consist of non-woven fabric onto which nanofibers formed by electrospinning have been applied. Nanoparticle concentration released into the working environment was measured at three workplaces where continuous production of nanofiber textiles takes place – SPUR a.s., NAFIGATE Corporation a.s., and Nano Medical s.r.o. At these companies, different technological arrangement is used for the production of nanotextiles. However, the space where the actual spinning of the input material takes place is always separated from the surrounding working environment. Air from the spinning chamber is removed and filtered through HEPA filters. A testo DiSCmini 133 instrument was used for the measurement, enabling the measurement of nanoparticle concentrations in the range of 10–700 nm. At SPUR a.s., SpurTex material is produced on a production line for the preparation of nanofibers of their own design – SPIN Line. The measured nanoparticle concentrations in the working environment where an operator is present ranged from 30,000 to 40,000 $\# \cdot cm^{-3}$, and the mean diameter of the nanoparticles was about 40 nm.

At NAFIGATE Corporation, a.s. and Nano Medical s.r.o., nanotextiles are produced on lines manufactured by ELMARCO s.r.o. These electrospinning lines comprise two spinning segments with string electrodes. During our measurements, polyvinylidene difluoride (PVDF) was spun from a dimethylacetamide solution at both companies. The resulting PVDF nanotextile was immediately laminated between two layers of spunbond nonwoven fabric. The measured course of nanoparticle concentrations had a pulsating character at both workplaces. In the area for the line operator, values in the interval 4,000–7,000 $\# \cdot cm^{-3}$ with more pronounced peaks at 9,000 $\# \cdot cm^{-3}$ and 16,000 $\# \cdot cm^{-3}$ were measured at Nano Medical s.r.o. (Fig. 1). At NAFIGATE Corporation a.s., nanoparticle concentrations in the range of 2,000–8,000 $\# \cdot cm^{-3}$ with an average of 4,000 $\# \cdot cm^{-3}$ were measured, the mean diameter of the nanoparticles was about 30 nm. Fluctuations at low concentration were measured, increasing the average to 60; 100; 180 nm.

Processing of textiles made of nanofibers into a product

One processor of nanotextiles, PARDAM NANO-4FIBERS s.r.o., is a manufacturer of nanofiber respirators. At this company, measurements were performed during two different processing steps which take place in the same production space

Fig. 1. Large-capacity production line for continuous production of nanofiber textiles at Nano Medical s.r.o. Letter A indicates the location of the spinning units with containers of polymeric material.

Fig. 2. Automated extrusion line for nanotextile respirators.

Fig. 3. Automated line for nanofiber masks Nanovia Mask 99,97 production.

(production hall). The first place was the place designated for cutting nanotextiles from coil into strips which are then used in an automatic line for extruding respirators. The testo DiSCmini 133 instrument was used again for the measurement. Near the cutting machine, when cutting the PA-6 nylon nanotextile on a polypropylene (PP) substrate, nanoparticle concentrations of approx. 37,000 $\# \cdot cm^{-3}$ were measured (cutting process is not separated from the surrounding working environment in any way). The second place was the space around the automatic line for extruding respirators, see Fig. 2 (the space where the actual extrusion takes place is separated from the working environment in the hall). Nanoparticle concentration in the space where the line operator is present reached the values of 15,000–25,000 $\# \cdot cm^{-3}$.

The next measurement was done at the nanofiber mask manufacturer Nanovia Mask 99,97 (name omitted at the request of the manufacturer). A three-layer sandwich is used for the mask production, the middle layer of which has a nanofiber structure (PVDF). The production of nanofiber masks takes place in a hall (190 m^3) where the production line is located (Fig. 3). No part of the production line is separated from the working environment of the hall, and the hall is not equipped with exhaust ventilation. This fact is most likely the reason why higher nanoparticle concentrations were measured in the working environment of the hall ranging from 40 000–60 000 $\# \cdot cm^{-3}$.

Mechanical stress of nanofiber materials

Mechanical stress during the use of products that include nanotextiles was simulated by squeezing and kneading nanotextile samples. Pilot measurements were performed with about 2 m^2 of a three-layer sandwich with an inner layer formed

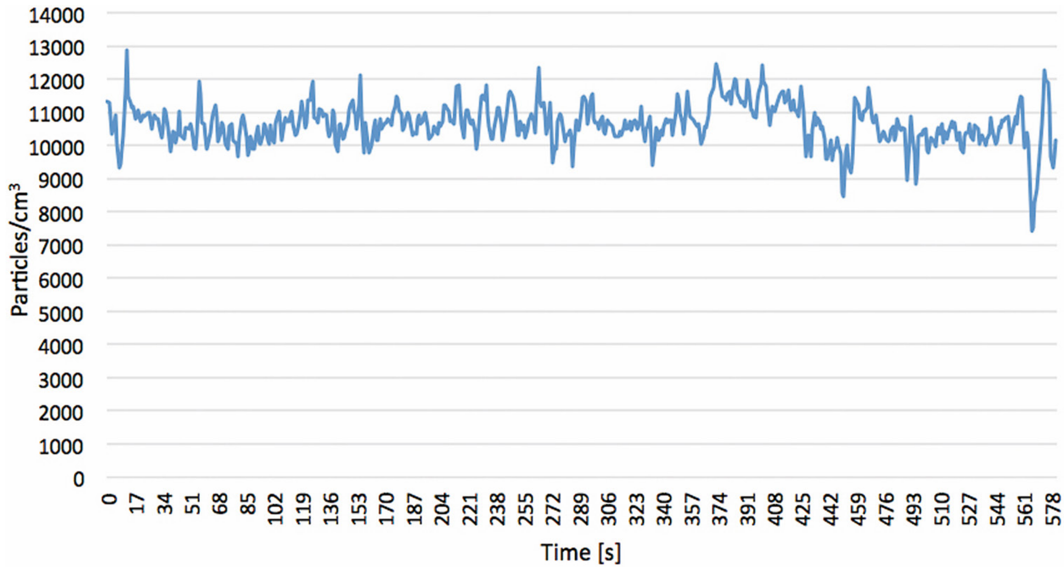


Fig. 4. Nanoparticle concentration measured during slight kneading of nanotextiles (PP + PVDF).

by PVDF nanofibers. A sample of the nanotextile was kneaded on a desk in the office for 10 minutes, with the testo DiSCmini 133 measuring instrument placed 0.5 m above the tabletop. The course of the nanoparticle concentration had a pulsating character in the range of 10,000–12,500 $\# \cdot cm^{-3}$ (Fig. 4) with a mean diameter of nanoparticles of 75–80 nm.

Testing of selected material properties for nanofiber masks and respirators production

Because various physical, physico-chemical, but also biological processes are involved in the fragmentation of plastics, including textile fibers used in the manufacture of respiratory protective devices (Gerritse et al., 2020; Barnes et al., 2009; Enfrin et al., 2020), several experiments during which the related release of nanoparticles into the environment was monitored were conducted. Materials that are either semi-finished products or are directly used to produce nanofiber masks and nanofiber respirators of the FFP-2 class were tested. These are layered fabrics composed of non-woven PP fabrics created by Spunbond (S) and Meltblown (M) technologies, on which PVDF nanofiber is applied by electrospinning. The nanotextile layer is then laminated with a non-woven PP fabric (S), creating a sandwich textile triple layer (PP fabric created by S and M technologies behaves as a single layer). Samples for testing were obtained from the manufacturer NAFIGATE Corporation a.s., and both the fabric with nanolayer laminated layer of PP (S) and the fabric with exposed nanolayer – without lamination with the last PP (S) layer, were tested.

Adhesion of nanofibers to carrier fabric

For adhesion testing, i.e., the cohesion of nanofibers with a carrier nonwoven fabric, a device allowing unfixed nanofibers blowing was built. The device contains two fans – one blows air into a slot nozzle located 5 mm above the tested surface. The second directional fan discharges air with loose nanofibers outside the device to the testo DiSCmini 133 measuring instrument, see Fig. 5. Data were obtained by moving the device over the tested fabric.

Measured values of nanoparticle concentrations released from both types of tested samples were surprising. While 8,000–10,000 $\# \cdot cm^{-3}$ with a stable mean particle diameter of about 54 nm is released from the laminated fabric, significantly fewer particles with the size in the range of 10–700 nm are released from the non-laminated sample (measuring instrument range). The obtained nanoparticle concentrations for the non-laminated sample reached values in the range of approx. 6000–7000 $\# \cdot cm^{-3}$.

This is probably due to the nanotextile layer serving also as a filter layer in this case. From the microscopic image of the exposed (non-laminated) nanofiber layer of PVDF (Fig. 6), significantly higher fiber density can be seen in this layer than in the top layer of the nonwoven PP fabric (Fig. 7).

Thermal stability and microscopic analysis of input materials

Using thermogravimetric analysis, a sample of the laminated material and a sample of the nanofiber layer itself formed of PVDF nanofibers withdrawn from the carrier nonwoven fabric, were analyzed. In the laminated product analysis (Fig. 8), endo effect

Fig. 5. Experimental equipment for blowing fibers (nanofibers).

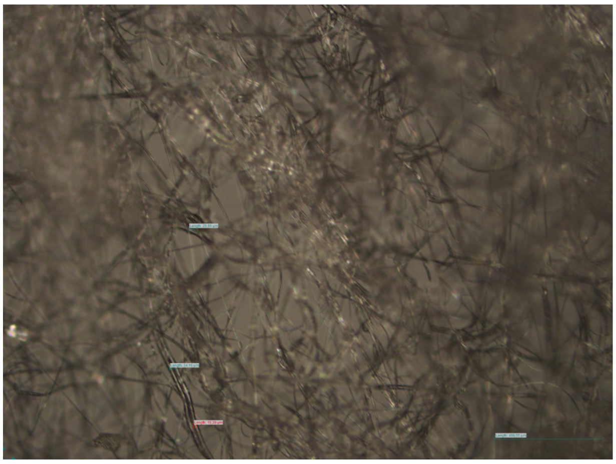


Fig. 6. Microscopic image of exposed (non-laminated) PVDF layer (magnification 20×).

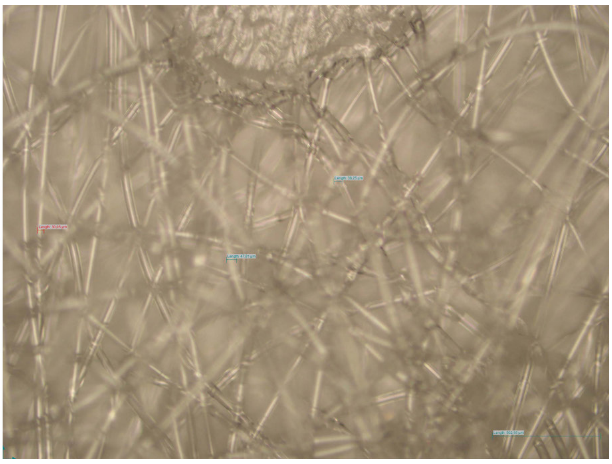


Fig. 7. Microscopic image of upper layer of PP unwoven textile (magnification 20×).

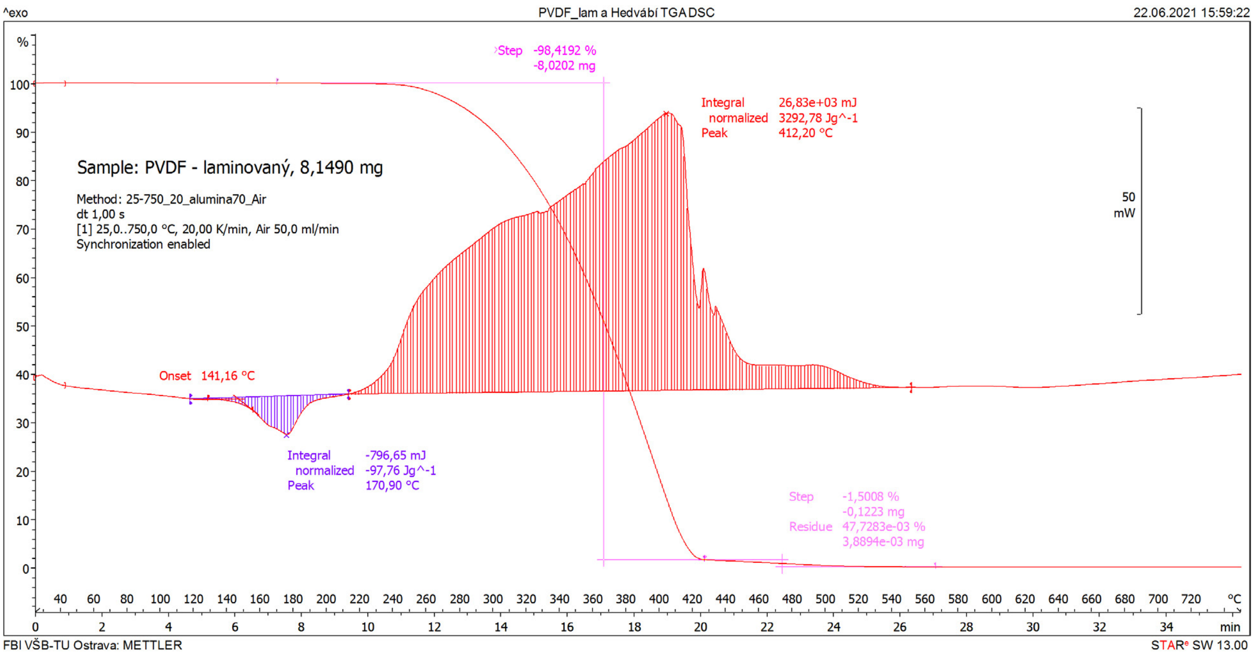


Fig. 8. Thermogravimetric analysis of laminated material.

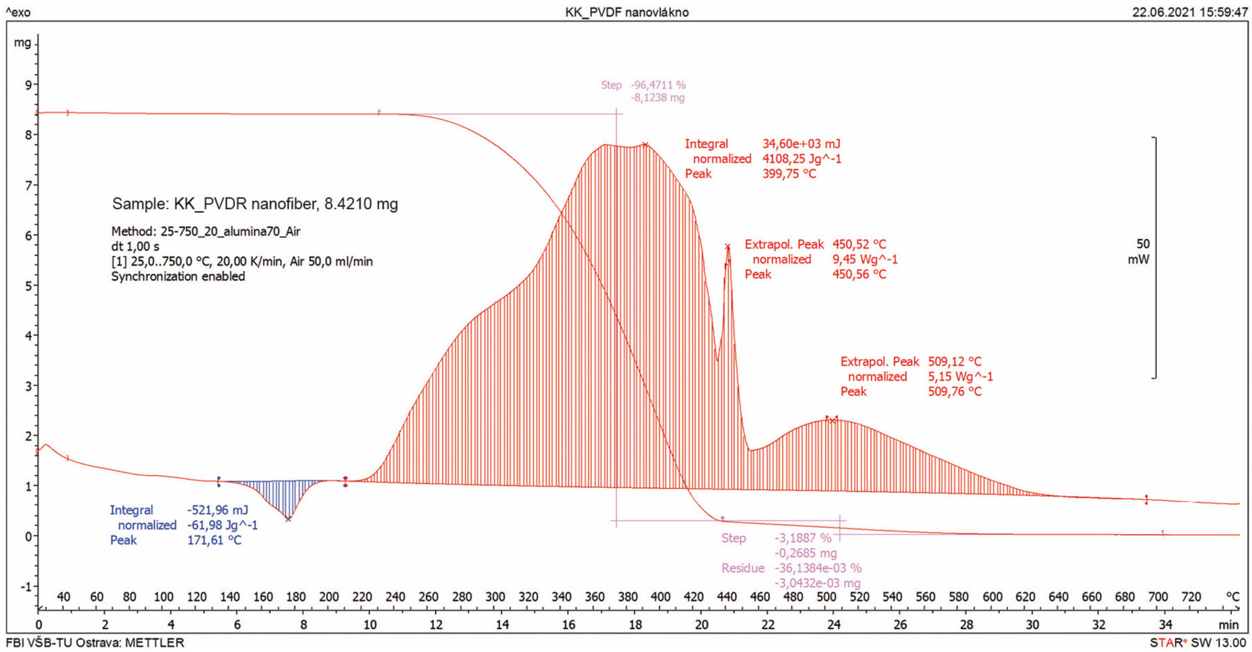


Fig. 9. Thermogravimetric analysis of nanofiber layers formed by PVDF nanofibers.

was recorded at 171 °C without weight loss. From the temperature of 215 °C to 425 °C, decomposition with exo effect with a maximum at 412 °C, essentially with complete weight loss, was observed. The weight loss (remaining from %) was terminated at 550 °C. In the analysis of the separate PVDF nanofiber layer (Fig. 9), endo effect was also recorded at 171.6 °C without weight loss. From 210 °C to 440 °C, exo effect was recorded with weight loss of 96 %. The residue shows exo effect with two peaks, at 450 °C and 509.7 °C. The decomposition of the PVDF nanotextile was completed at 640 °C.

From a comparison of the spectra in Figs. 8. and 9. it is evident that decomposition of the laminated product occurred at a temperature lower by 100 °C than of the separately analyzed nanofiber PVDF layer. This result can be explained by the very close direct contact of PP and PVDF polymers (lamination takes place at elevated temperature and pressure), where they interact at high temperatures which results in faster decomposition.

Static tensile test

Tensile strength test of textiles was performed on a SHIMADZU EZ-LX testing machine (Fig. 10); to achieve adequate measurement results, calibration of the instrument itself was performed one week before the actual measurement. This test was performed only with the sample of laminated material. All samples tested were 15 mm wide and 150 mm long and were prepared differently based on the source material, the weight of nanotextiles was 93.6 g/m^2. LAM 1 samples were cut with the longer side across the winding direction of the fabric during its production, LAM 2 samples were cut with the longer side in the winding direction.

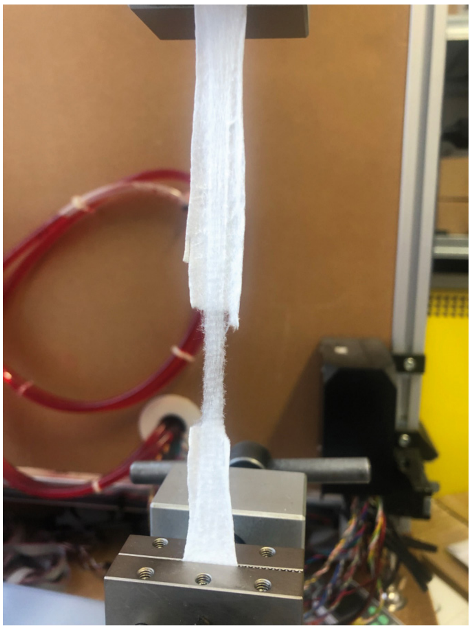
Results of the strength test are summarized in the tensile diagram (Fig. 11). The curves obtained by testing individual samples differ not only in their course, but especially in the value of the maximum force [N] to reach the ultimate strength. For LAM 2 sample, the value of the maximum force was doubled (Fig. 11), but the relative elongation of the sample was greater in LAM 1 (Fig. 10a).

Semichronic toxicity test on seeds

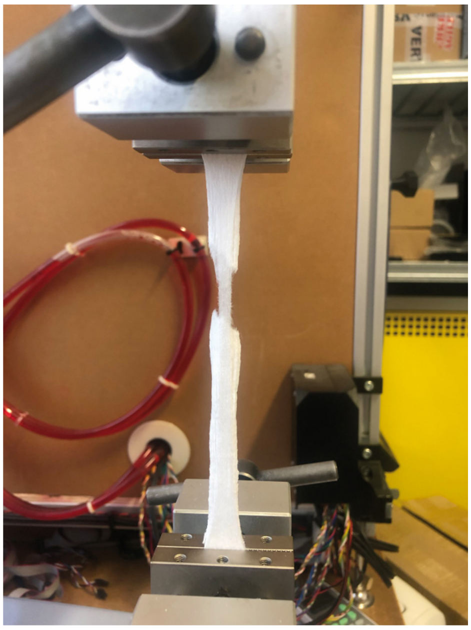
According to the OECD methodology (OECD, 2006), white mustard (*Sinapis alba L.),* rapeseed (*Brassica napus*), fescue (*Festuca pratensis*) and lettuce (*Lactuca sativa*) seeds were used on non-laminated PVDF samples (seeds were placed on the exposed PVDF layer), laminated PP-PVDF, unworn Nanovia Mask 99,97, and worn Nanovia Mask. Germination and inhibition of root growth were determined in seeds placed on and off the test fabric (Figs. 12 and 13). Calculation of root growth inhibition when applying seeds to/off the tested nanotextile is based on the measurement of the root length (root elongation) after the end of the test and follows from the formula:

$$I = \frac{L_C - L_V}{L_C} \cdot 100 \qquad (1)$$

where I is the inhibition or stimulation of root growth (%), L_c is the average root length in the control sample (mm), and L_v is the average root length in the test sample (mm). The resulting



a)



b)

Fig. 10. Tensile test a) LAM 1 sample across the wound fabric; b) LAM 2 sample in the winding direction.

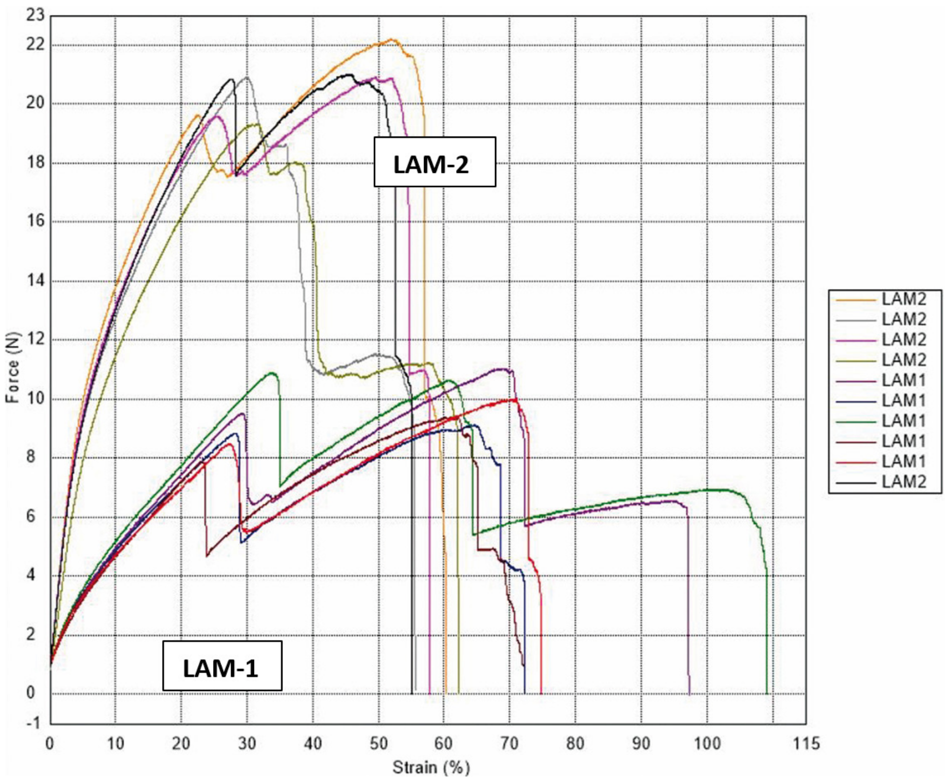


Fig. 11. Tensile diagram of laminated material (LAM 1 and LAM 2 sample).

value of $I > 0$ represents inhibition of root growth, while $I < 0$ shows stimulation of its growth.

Figs. 14 and 15 show charts comparing the inhibition on mustard and rapeseed in case of direct contact with the sample and off of it.

From Fig. 14 it can be seen that a significantly higher inhibition of root growth occurred in mustard and rape seeds in direct contact with the nanotextile. The average inhibition reaches values of more than 50 %. For lettuce, the highest inhibition was found for seeds placed on the unworn Nanovia Mask sample (85.5 %), for seeds placed off the fabric, the highest inhibition was found for the non-laminated PVDF sample (55.42 %). In meadow fescue, on the

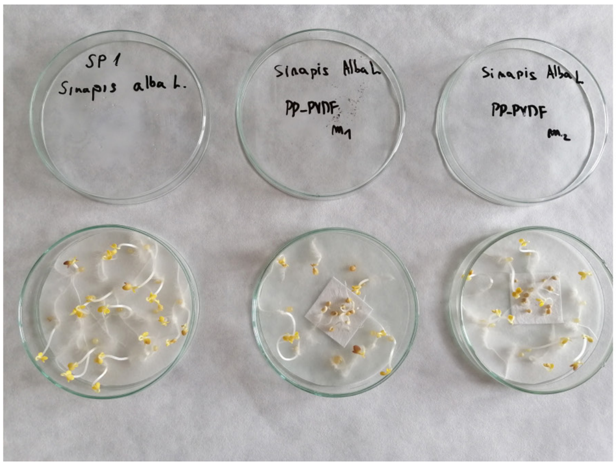


Fig. 12. Semichronic toxicity test on *Sinapis alba* L. (white mustard) after testing (72 hours), experimental temperature: 21 ° C. Left: control determination (control), 2× parallel samples: PP-PVDF lam. m_1 and m_2.

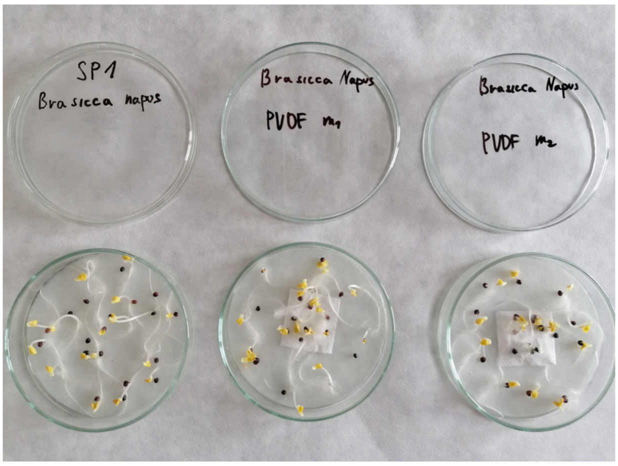


Fig. 13. Semichronic toxicity test on *Brassica napus* (rapeseed) after testing (72 hours), experiment temperature: 21 °C. Left: control set (control group), 2× parallel samples: Nonlam. PVDF m_1 and m_2.

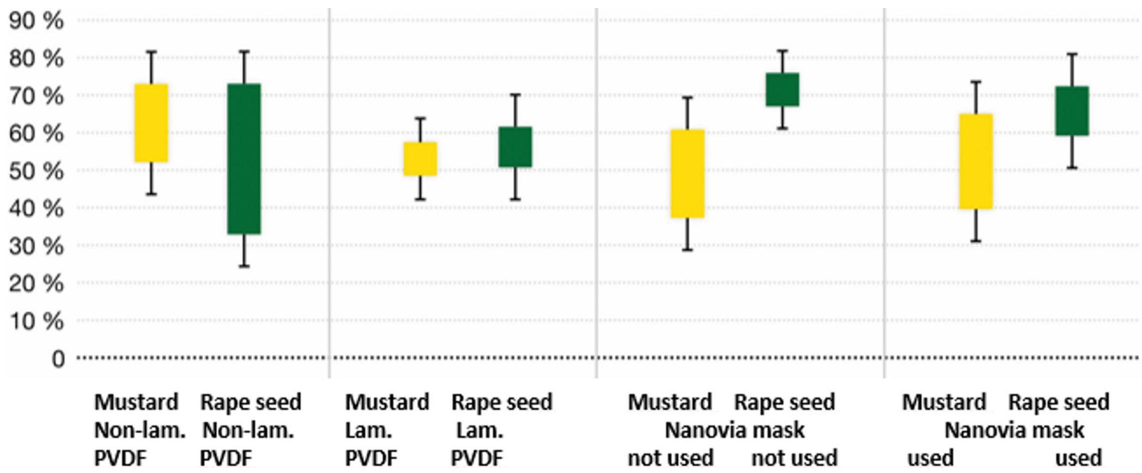


Fig. 14. Chart comparison of inhibition of mustard and rapeseed root growth in % (seeds on sample).

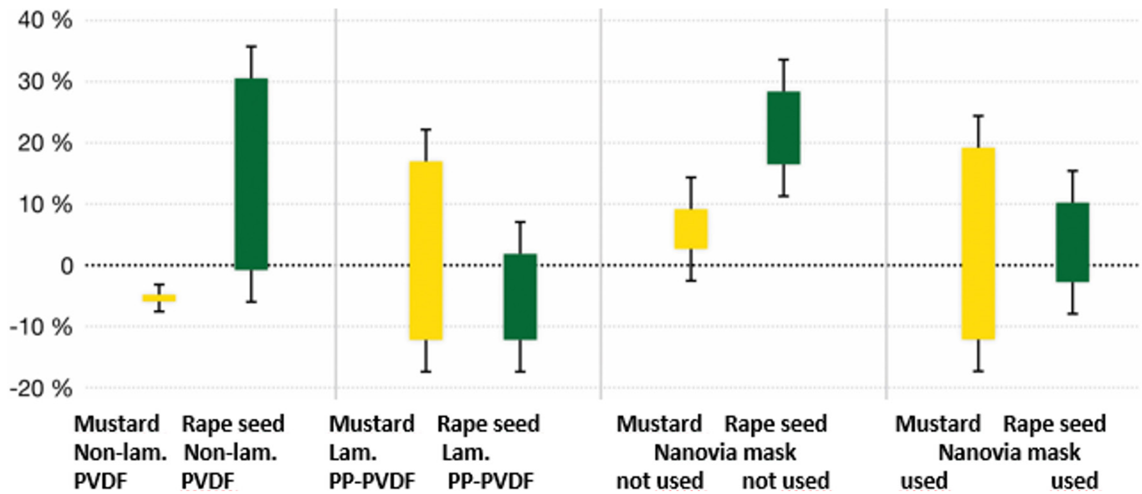


Fig. 15. Chart comparison of inhibition of mustard and rapeseed root growth in % (seeds off sample).

other hand, root growth was stimulated both in seeds in direct contact with the fabric and in seeds placed outside the fabric, e.g., in the non-worn Nanovia Mask veil, the *I* value was set at –131 %.

Obtained results of root growth inhibition correspond with studies focused on the effect of nano- and microplastics performed with terrestrial plants. An increase in total root length and a decrease in root average diameter have been reported in spring onion (*Allium fistulosum*) after exposure to microplasts (de Souza Machado et al., 2019). Bosker et al. state that the change in cress (*Lepidium sativum*) root growth depends on the exposure time and the size of the plastic particles. After 24 h exposure, a decrease in root growth when exposed to 50 nm particles and an increase when exposed 500 nm particles was observed. However, no significant difference in root growth was observed after 48 or 72 h of exposure (Bosker et al., 2019). In addition to root elongation, the presence of nano- and microplastics also affects the germination process itself. Delayed germination of *L. sativum* was observed upon exposure to various doses of plastic particles (Bosker et al., 2019; Pflugmacher et al., 2020). Blocking of pores 'in seed capsules' by particles is considered to be one of the possible mechanisms causing this delay (Bosker et al., 2019).

Conclusion

One of the consequences of the COVID-19 pandemic is an unprecedented increase in the production of respiratory protective devices which include nanotextiles. Because these protective devices are made of fibers and nanofibers of various plastics, they contribute to the increased occurrence of nano- and microplastics in the environment (Sullivan et al., 2021). Based on measurements performed both in the production of material for nanofiber masks and nanofiber respirators and in the production of our own protective equipment, we have shown that if the production line (or its corresponding part) is separated from the rest of the working environment and the air discharged from this separate space is HEPA filtered, the filters capture the nano- and microplastic particles formed. Thus, a greater danger to the environment actually arises from the use of protective equipment, and especially its mechanical stress (abrasion). A tensile test was used to demonstrate that the resistance of the input material for protective equipment production depends on the arrangement of the polymer chains in the production of nonwovens. This finding leads to the assumption that durability as well as effectiveness of protective equipment also depends on the method of processing the sandwich nanotextile used in their production. Based on the performed tests, it can be assumed that the filter layer in extruded nanofiber respirators is not homogeneous.

Due to the COVID-19 pandemic, the use of nanofiber respirators and nanofiber masks has become common not only in workplaces with high risk of infection transmission, but also in general public. While in hazardous workplaces (e.g., medical facilities), adequate disposal of discarded protective equipment is ensured, the method of discarding and liquidation of protective equipment used by the public depends on the information and discipline of its members. The fate of discarded protective equipment in the environment depends on many variables (temperature, humidity, pH, etc.) as well as on the intensity of their mechanical stress. The performed tests of semichronic toxicity on the seeds of selected plants confirmed the influence of the material from the protective agents on the root growth. Direct contact of the fabric with mustard and rapeseed seeds showed more than 50 % inhibition of root growth. In case of fescue seeds, on the other hand, its growth was stimulated.

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