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RESEARCH

The Structural Influence of Airborne Particles on their Toxicity

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ABSTRACT

This paper studies human exposure to microplastics in real-life situations. The concentration of the polyamide nano/microplastics in the proximity of textile workplaces was made by the device Laser Aerosol Spectrometer. The descriptive statistical analysis was elaborated for: variables TSC (total concentration µg/m3), PM10 (µg/m3), PM2.5 (µg/m3), PM1 (µg/m3) and TC (total number of particles, 1/l). The histograms of each variable highlight the asymmetry of the distribution, with the predominance of frequencies or variables; representation of level indicators (average, median) and dispersion-box-plot graphs were obtained. Collection of the particle made with TECORA-SKYPORT PM-HV and GilAirPlus pumps, by using Quartz and Nucleopore filters with different diameters highlighted that the highest mass (683µg) was collected by using GilAir Plus pump and Quartz filter 37mm. A strong correlation was obtained between the variable: "Collected mass/ Air concentration": r = 0997775 ÷ 0,999477 and "Collected mass/ Air volume", r = -0,97473 ÷ 0.80064. The prediction of the value of the collected mass as a function of the diameter of the filters, particle concentration, airflow, and total air volume was made by obtaining the regression equations. Optic microscope analyses highlighted the dimension of microfibres (50% are <10µm) and SEM the dimension: 25% < 68.47 nm and the spherical shape of particles. FT-IR, µRaman and TG-DSC analyses showed the presence of PA particles in the collected particles.

KEYWORDS

Particles, heath, filter, plastic, polyamide.

INTRODUCTION

Plastics can be manufactured at low cost, and their low weight and adaptability are recommended for use in a variety of everyday applications, including food packaging, consumer products, medical devices, and construction. However, by 2050, an additional 33 billion tonnes of plastic is expected to be added to nature. Given that most plastic polymers currently used are highly resistant to degradation, this influx of persistent and complex materials poses a risk to human health and the environment. Numerous studies demonstrate the omnipresence of MP and NP in various areas of the environment, including water, sediments, soil and biota, with a major impact on life, being difficult to control and manage [**1**]. Airborne particulate matter is one of the main air pollutants.

Their impact on mortality, and occurrence of pulmonary and cardiovascular complications, have been the subject of other numerous studies.

It has also been shown that long-term effects are more serious than short-term effects. Among the many pollutants present in the air, of natural or anthropogenic origin (due to human activity), suspended particles have been the subject of a great amount of work. Airborne particles are complex mixtures of organic and inorganic substances from different sources of particulate emissions. It is still impossible to make a routine and precise distinction between the different constituents of particles (see Air Pollution Particles: What are they?); it is, therefore, impossible to attribute this or that

effect to this or that component of particles. Except in the laboratory and under strict experimental conditions. The effects attributed to particulate matter at the population level are therefore the effects observed overall without being more precise as to the component(s) responsible for these effects. Finally, the socio-economic cost of this pollution is considerable and preventive measures are still insufficient.

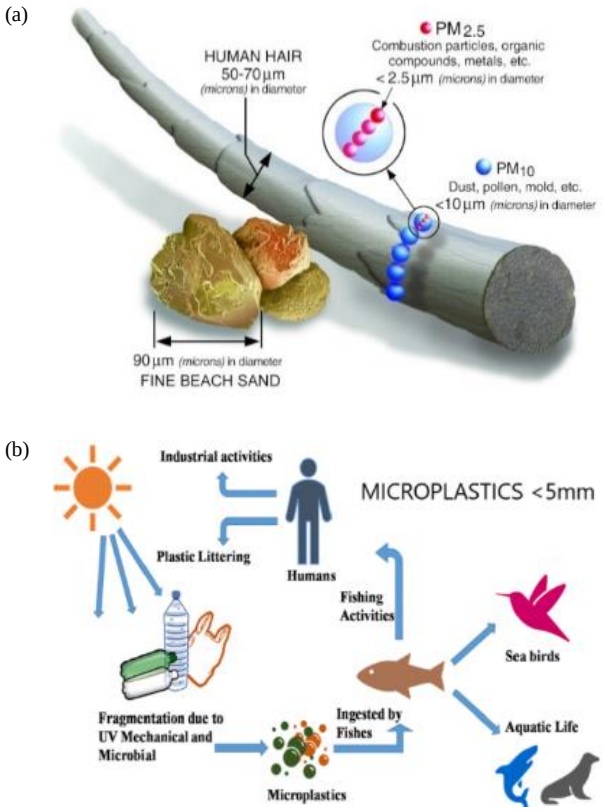

Fig. 1. Comparison of hair size with PM10 and PM2.5 [2].

Particulate Matter (PM) particles are classified according to their aerodynamic diameter expressed in µm [**2**]. The particles that are routinely analysed by air quality monitoring devices are 10 µm in diameter; they are noted as PM10; they are referred to as "coarse" particles. Fine particles are defined as those with a diameter of less than 2.5 µm (PM2.5) (**Fig. 1**). These diameter considerations are fundamental because they condition the penetration of particles into the bronchopulmonary system and into the body. And in recent years, there has been an interest in so-called "ultra-fine" particles, with a diameter of 0.1 µm (or 100 nm), or PM0.1. They are nanoparticles.

This broad composition and size distribution ranges limit the efficiency of detection methods, often inherently focused on a single and narrow class of NP/MPSs sizes. In addition to their demonstrated native toxicity, NP/MPSs may act as efficient carriers of pollutants and pathogens onto their surface, facilitating the transfer and penetration of other classes of hazardous materials [**3**]. Their high persistence nature and release of chemicals/additives used in the synthesis of plastics materials may pose cascading impacts on living organisms across the globe. Natural connectivity of all the environmental compartments (terrestrial, aquatic, and atmospheric) leads to the migration/ dispersion of MNPs from one compartment to another (**Fig. 1b**) [**4**]. In 2016, approximately 27.1 mt of plastic waste was collected in the European Union (EU), of which 31.1%, was recycled, 41.6% reused (for energy production) and 27.3% landfilled [**5**].

To be eligible to be introduced as a PLC, a polymer must: have a number average molecular weight ≥ 1,000 g/mol as well as meet the low molecular weight species and reactive functional group requirements or be a polyester manufactured solely from prescribed reactants. To be introduced as a PLC, a polymer must also meet the criteria for low cationic density, approved elements, difluoro methylene or trifluoromethyl groups, stability, water absorption and known hazard classification. Consider a polyamide with a NAMW 7,000 g/mol manufactured from equimolar amounts of ethylenediamine and isophthalic acid. On average, the polymer will have 1 unreacted amino group at 1 end of the polymer chain and an unreacted carboxylic acid group at the other end. As the amino group is potentially cationic, it needs to be included in the calculation of the FGEW of cationic groups in this polymer. The FGEW for the amino group can be calculated by end-group analysis, that is 7,000/1 g/mol. Therefore, the polymer meets the criteria for low cationic density as the FGEW is above 5,000 g/mol. If the NAMW had been less than 5,000 g/mol, or if the polymer had 2 free amine groups, then the polymer would not be eligible to be introduced as a PLC [**6**].

Total suspended particles (TSPs), $PM_{2.5}$ and size-fractioned inhalable particles ($PM_{10\text{-}5.8}$, $PM_{5.8\text{-}3.3}$, $PM_{3.3\text{-}1.1}$ and $PM_{1.1\text{-}0.43}$) were collected in a number of sites in Guangzhou, a megacity of Southern China. The main objectives of the study were the following: (a) determining the levels and composition of TSPs and $PM_{2.5}$ from various industrial and commercial activities (**Fig. 2**) (b) examining the cytotoxicity of size-fractioned breathable particles from different industrial/commercial activities to A549 cell lines, and (c) exploring the components that could influence the cytotoxicity of particles to A549 cell lines [**7**].

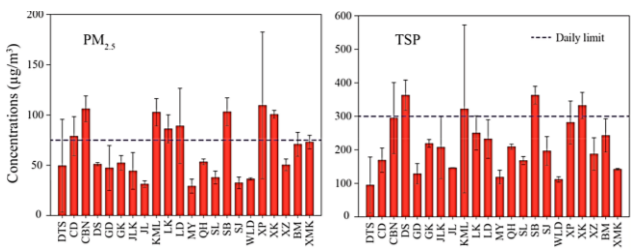

Fig. 2. Levels of TSPs and $PM_{2.5}$ at different industrial/commercial sites of Guangzhou.

The Textile and Clothing sector (T&C) is an important part of the European manufacturing industry, with over 1.5 million employees. Sources of particulate pollution are emissions from the spinning preparation (blow room, carding), combing and woven fabrics, knitted fabrics and non-woven fabrics manufacturing that form a suspension, which over time settles on each surface [**8**].

An important component of the solid particles that generate air pollution in the textile industry are microplastics (MP) and nano plastics (NP), which also include microfibers (<5mm) and nanofibers (<100 nm), respectively. The quantity of the particles released into the air during fibre and yarn processing ranges from 1 µg/m^3 to 50 µg/m^3 [**9**].

The study highlights the concentrations of plastic particles (polyamide), the collection methods and the main characteristics of NMP: shape, size, dimensions, type, etc.

EXPERIMENTAL

Toxicology makes it possible to study the effects of pollutants on health using mathematical or experimental models, at the cellular, tissue or sometimes animal level. Under rigorous conditions, the effect of a specific pollutant on a particular organ can be studied and a so-called risk assessment conducted. The ultimate objective of risk assessment is to compare the level of risk measured in the laboratory with the level of risk to which is exposed in reality, by studying and quantifying the exposures to which we are exposed (exposure scenarios). The level of particle concentration [TSP, PM10, PM2.5 and PM (µg/m^3), TC-l/l] in different work areas in the weaving and knitting mill are determined by using an online recording system with the Laser Aerosol Spectrometer MINI LAS model 11-EL-Laser). The database created as a result of the records made for each fraction was populated with at least 50 values for each variable. Descriptive statistics used in the analysis allowed:

(i) determining the parameters that highlight the extent to which the data are homogeneous or not, measuring their spread and implicitly the differences that exist within the data series, respectively: amplitude, standard deviation and dispersion;
(ii) calculating the coefficients of variability that demonstrate to what extent the data groups are homogeneous or heterogeneous;
(iii) tracing the histograms of each variable that highlights the asymmetry of the distribution, with the predominance of frequencies or variables;
(iv) representation of level indicators (average, median) and dispersion – box-plot graphs.

The collection of particles was performed by using 2 methods: Method 1 - according to SR EN 12341: 2014 standard which is based on sampling the particles on the filters and weighing them using a balance. The TECORA-SKYPORT PM-HV type device was used for sampling, which ensures the volumetric measurement with dry gas ether with an accuracy of ±2%. Airflow 38 l/min, 6 m^3/h rotary vane pump type. Quartz filters with a diameter of 47 mm were used. The volume of air was recorded during the collection period. Particles were taken from each type of 3 filters (A1, A2, A3). Method II - is based on sampling the particles on the filters using a GilAirPlus-type device equipped with cyclone Higgins-Dewell (HD) and a 2 l/min air flow pump. 37 mm diameter quartz filters (B1, B2, B3) and 25 mm diameter nucleopore filters coated with the gold membrane (C1, C2, C3) were used for sampling. For each type of particle and filter, 3 filters were taken. The amount of dust collected was determined by the difference in mass between the white filters and the filters after collection.

Characterization of polyamide particles collected was performed by analysis: Optic microscope, Scanning Electron Microscopy (SEM), FT-IR, µRaman spectroscopy analyses, and TG-DSC allowed the identification of shape, size and structural footprint of particles.

RESULTS AND DISCUSSION

The concentration of airborne particles

The descriptive statistical analysis was elaborated for: variables TSC (total concentration µg/m^3), PM10 (µg/m^3), PM2.5 (µg/m^3), PM1 (µg/m^3) and TC (total number of particles, 1/l).

The histograms and the boxplot graphs for the analysed variables are presented in **Fig. 3(a)** and **Fig. 3(b)**.

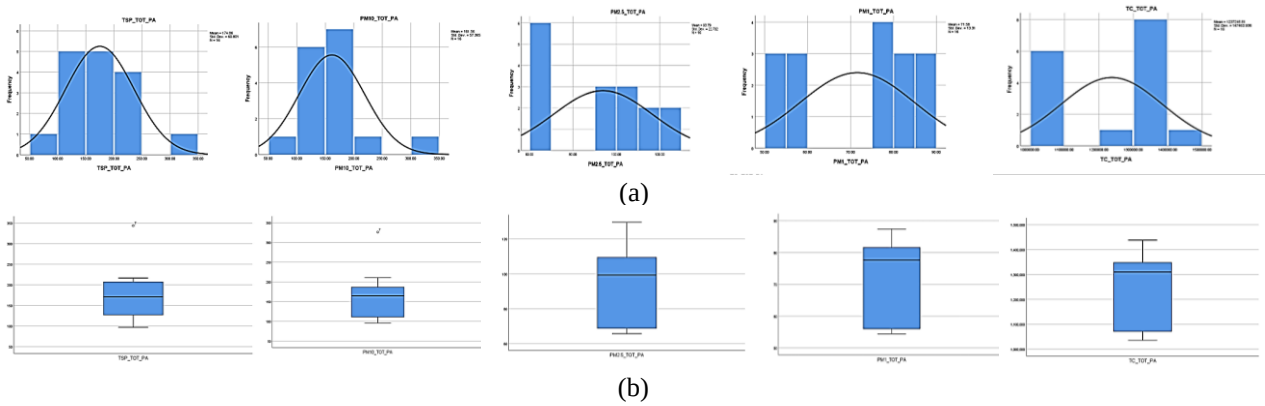

Fig. 3. (a) histograms; (b) box-plot of TSP, PM10, PM2.5, PM1, TC

Data analysis highlighted

Variability

The variable TSP has a value with an indicative: 7 with a value of 343.8 l/l (weaving mill) located in the range of 1.5-3 box lengths. The variable shows the distribution of 50% of the values directed to the right, the median being directed towards the top of the box-plot box, so the large values are predominant.

Distribution form indicators TSP (µg/cm³): 25% of the values obtained for the total concentration of particles are below 126,8 (µg/cm³), 50% are between 126,8-170,9 (µg/cm³) and 25% over 170,9 (µg/cm³).

The variable PM 10 has a value with an indicative: 7 with a value of 326,3 µg/cm³ - located in the range of 1.5-3 box lengths. The variable shows the distribution of 50% of the values directed to the right, the median being directed towards the top of the box (box plot), so the large values are predominant.

Indicators of the distribution shape of PM 10 (µg/cm³) - 25% of the values obtained for the concentration of PM 10 (µg/cm³) are below 78,5(µg/cm³), 50% are between 78,5 -93,2(µg/cm³), and 25 % over 93,2(µg/cm³).

The variable PM 2.5 does not show values with indicative. The variable shows the distribution of 50% of the values directed to the right, the median being directed towards the top of the box (box plot), so the large values are predominant.

Indicators of the distribution shape of PM 2.5 (µg/cm³) - 25% of the values obtained for the concentration of PM 2.5 (µg/cm³) are below 68,7(µg/cm³), 50% are between 68,7-99,3(µg/cm³), and 25 % over 99,3(µg/cm³).

The variable PM1 has no indicative values. The variable shows the distribution of 50% of the values directed to the right, the median being directed towards the top of the box (box plot), so the large values are predominant.

Indicators of the distribution shape PM 1 (µg/cm³), 25% of the values obtained for the concentration of PM1 (µg/cm³) are placed below 56,0 (µg/cm³), 50 % between 56,0-77,6(µg/cm³) and 25 % over 77,6(µg/cm³).

The TC variable does not show indicative values. The variable shows the distribution of 50% of the values directed to the right, the median being directed towards the top of the box (box-plot) so the high values are predominant.

Indicators of the distribution shape TC (1/l) - 25% of the values of the number of registered particles are below 1071226,0(1/l), 50% between 1071226.0 -1309556,0(1/l) and 25% over 1309556.0(1/l).

Skewness indicators have positive values for TSP (µg/cm³): 1,297, and PM10 (µg/cm³): 1,511 the normal distribution curve moves away from the middle, moving to the right. Negative values are registered for PM2,5 (µg/cm³): -0,026, PM1 (µg/cm³): - 0,376, TC(1/l): -0,385, the normal distribution curve moves away from the middle, moving to the left.

The vault indicators (kurtosis) have negative values for: PM2.5: -1,450, PM1: -1,799 and TC: -1, the curves being platykurtic and positive TSC: 2,979 and PM10: 3,610 curves being of leptokurtic type.

The analysis of the average values of the variables PM 10 (µg/m³), PM2.5 (µg/m³) calculated for the polyamide airborne particles registered in the vicinity of the workplaces shows that the level of PM10 concentration of 161,56 (µg/m³) falls within the pollution threshold, orange (15-300 µ/m³) and PM2.5 of 46,0 µg/m3 within the green pollution threshold (0-75µg/m³) - **Table 2** [**5**].

Table 2. The pollution levels.

Color	green	yellow	orange	red	purple	dark purple
Concentration PM 2.5	0-35 µg/m³	35-75 µg/m³	75-150 µg/m³	150-200 µg/m³	200-250 µg/m³	>250 µg/m³
Concentration PM 10	0-75 µg/m³	75-150 µg/m³	150-300 µg/m³	300-400 µg/m³	400-500 µg/m³	>500 µg/m³
Air quality	Good	Normal	Lightly polluted	Medium polluted	Strong polluted	Seriously polluted

Particle collection

Quartz filters with 47mm, 35mm diameter and 25mm Nucleopore filters were weighed before and after sampling with an A&D BM20-type analytical balance, after conditioning for 24 hours. The results obtained after sampling the airborne particles in the vicinity of the workplaces are presented in **Table 3**.

Table 3. Collected mass particle.

Variants		Collected parameters				
		Initial mass, mg	Mass after collecting, mg	Collected mass particles, µg	Air concentration, µg/m³	Air volume, l
Quarz filter, 47mm	A1	141.92	142.27	35	0.124	4527.0
	A2	138.85	139.7	87	0.358	2430.0
	A3	143.3	143.94	71	0.272	2610.0
Quartz filter 37mm	B1	87.699	88.382	683	1.208	247.42
	B2	87.703	88.304	601	0.896	242.11
	B3	92.335	92.797	462	0.298	261.6
Nucleopore filters, 25mm	C1	9.014	9.030	16	117.41	136.3
	C2	9.569	9.579	10	68.51	146.0
	C3	9.077	9.094	17	123.43	137.7

Correlation coefficients and regression coefficients

Correlation - is a general term used to define the interdependence or link between the variables observed in statistical populations. In a narrow sense, it is a measure of the degree of a statistical link between quantitative variables, under the name of "correlation coefficient", we note **r = CORREL (X, Y)**.

The equation for the correlation coefficient is:

$$Correl(X,Y) = \frac{\sum (x-\bar{x})(y-\bar{y})}{\sqrt{\sum (x-\bar{x})^2 \sum (y-\bar{y})^2}} \quad [1]$$

The correlation coefficient r has values between -1 and 1.

The correlation coefficients calculated between the collected mass particles[µg] and air concentration (µg/m^3), air volume (l) and diameter (mm) of the filters were calculated.

There is a strong correlation between the variable: "Collected mass/ Air concentration": r = 0997775 ÷ 0,999477 and "Collected mass/ Air volume", r = -0,97473 ÷ 0.80064 for all three diameters studied. A weak correlation is between collected mass (µg) and diameter of the filters (mm): r = 0,131679519.

In **Fig. 4(a)** the real distribution of the data series (scatter) from the previously analysed correlations is presented.

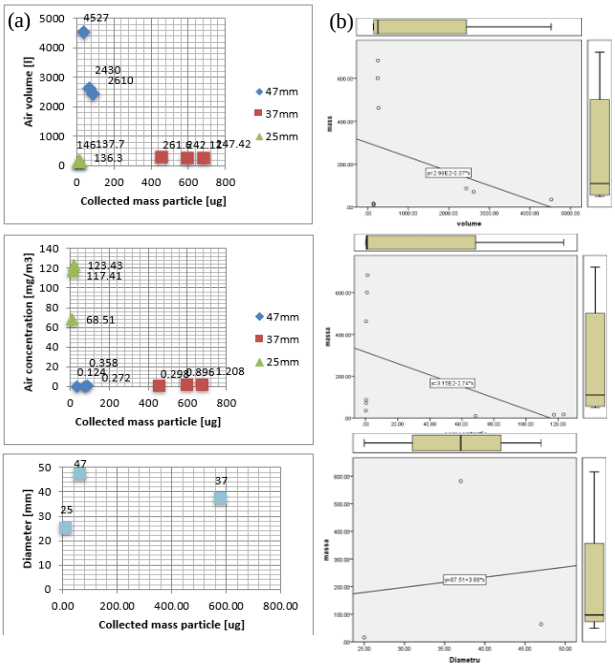

Fig. 4. (a) The real distribution of the data series (scatter); (b) the regression curve.

The prediction of the value of the mass of the collected particle as a function of the diameter of the filters, particle concentration in the air, air flow of the pumps, and total air volume was made by plotting the regression curves and box-plot graphs and obtaining the regression equations (1,2,3), **Fig. 3(b)**.

The regression equations obtained:

Y=87.51+3.66*x collected mass (µg) as a function of filter diameter (mm). [1]

Y=3.15E2-2,7*x - collected mass (µg) as a function of particle concentration in the air (µg/m3). [2]

Y=2.99E2-0,07*x - collected mass (µg) as function of the air volume (l/min). [3]

Characterization of polyamide particles

Optic microscope analyses

Optical microscopy analysis with phase contrast (100x) filter MCE 201 (PA) - was performed with the following parameters: A total number of microscopic fields viewed: 122, the total number of identified fibres: 34, the diameter of the fibre between 1-3 µm; fibre length: 14 fibres <10 µm, 10 fibres. > 10 µm and 10 fibres >100 µm. Optical microscope images are presented in **Fig. 5(a)**.

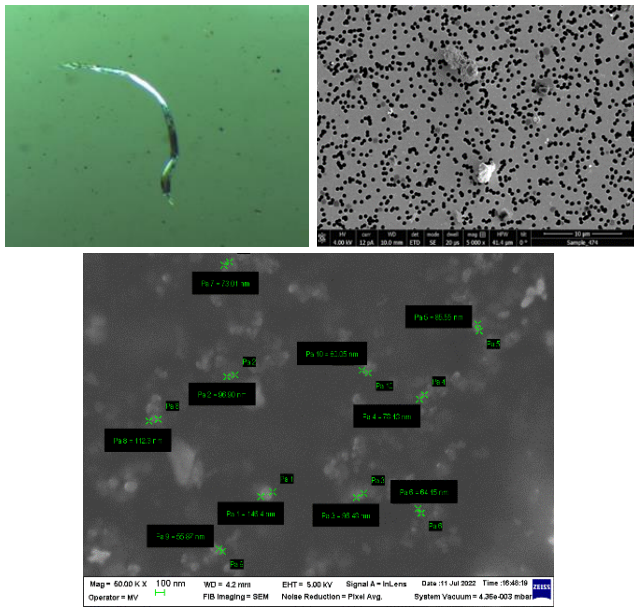

Fig. 5. (a) Optical microscope; (b) and (c) SEM imagine nucleopore filters.

Scanning electron microscope

Versa 3D electron microscope from TermoScientific (formerly FEI) was used for the analysis of the microparticles. In order to make samples accessible for further characterization methods, the sample was not covered with metal or carbon. The acceleration voltage used was 4 kV with a spot size between 2 and 4 (sample current between 0.5 pA and 12 pA). The WD distance used is 10 mm. In the case of quartz filters, most particles would likely be deposited further inside the filter instead of on the surface, so any microscopical method will not work to determine concentrations (**Fig. 5(b)**). The nucleopore sample has a circular trace with an area of about 336 mm^2. This area was taken into account to be the active area of the filter. For the SEM analysis of the sample, five different regions with a surface of 0.46 µm^2 were selected on different parts of the sample. The characterized surface is about 0,6% of the entire active area of the filter.

High-resolution images highlighted a low concentration of particles present on the nucleopore filter (**Fig. 5(c)**).

The shape and size of the particles were obtained. The statistical analysis of the particle sizes showed that 25% have dimensions smaller than 68.47 nm, 25-50 % have dimensions between 68.47nm - 80.22 nm, 25 % between 80 nm - 94.99 nm and 25 % above 94.99 nm (**Fig. 5(d)**). In **Fig. 6(a)** and **Fig. 6(b)** the Normal P-P plot and Detrended Normal P-P plot of the particle dimensions are presented. Most of the particles have a spherical shape.

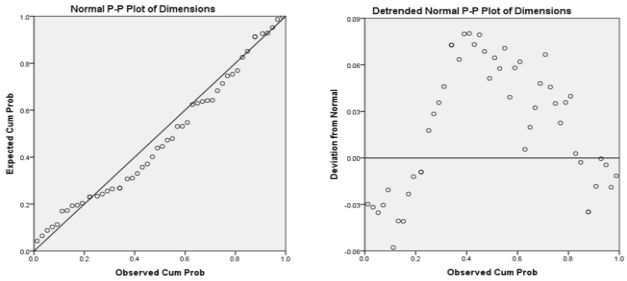

Fig. 6. (a) Normal P-P plots; (b) detrended P-P plot of dimensions.

Fourier Transform Infrared Spectroscopy (FTIR)

The ATR - FTIR spectra of the PA samples were recorded at room temperature using a Perkin–Elmer Spectrum Two IR spectrometer. Attenuated total internal reflection FTIR measurements were performed by averaging 20 scans, with a resolution of 2 cm^{-1}, over the 4000-500 cm^{-1} wavenumber range. The peaks in the FTIR spectra are assigned to polyamide and to the membrane where the samples were collected. The peaks assigned to polyamide are (**Fig. 7(a), Fig. 7(b)**: 1925: asymmetrical stretching CH_2, 2853: symmetrical stretching CH_2.

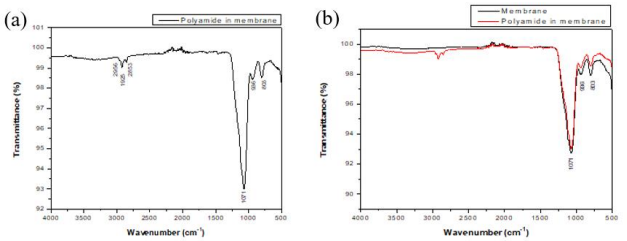

Fig. 7. (a) FTIR PA spectra; (b) FTIR PA and membrane spectra µRaman spectroscopy.

µ-Raman spectroscopy

Unpolarized µ-Raman measurements were performed with a triple 557 TriVista spectrometer (S&I Imaging GmbH) in reflection geometry. The excitation was carried out with the 514.5 nm line of the DSS laser having an output power level of approximately 100 mW. The laser was focused on the sample with a 100x/0.9 microscope objective to a spot size of less than 2 µm. The reflected laser light was rejected edge filter. Raman spectra were collected with a monochromator having a 1500 g/mm grating and a focal length of 750 mm. The spectra acquisition was done with a nitrogen-cooled 1024 x 256 Si-CCD camera. The silica filters with a 9mm diameter for the collection were used for these analyses.

PA particles and fibres were identified using µ-Raman spectroscopy. In **Fig. 8(a)** are presented: optical microscope image of microparticles and fibres, PA Raman spectra and Raman Mapping centred at 1100 cm-1 line of the PA fingerprint and 1600 cm^{-1} (SWNT) and **Fig. 8(b)** standard spectra for the comparison with other particles. In the PA spectrum, the difference between the reference and the measurement on the sample is due to the type of PA (PA 6 or PA6.6). In the case of the sample, the double peak from 1075-1200 appears with a different intensity compared to the comparative sample.

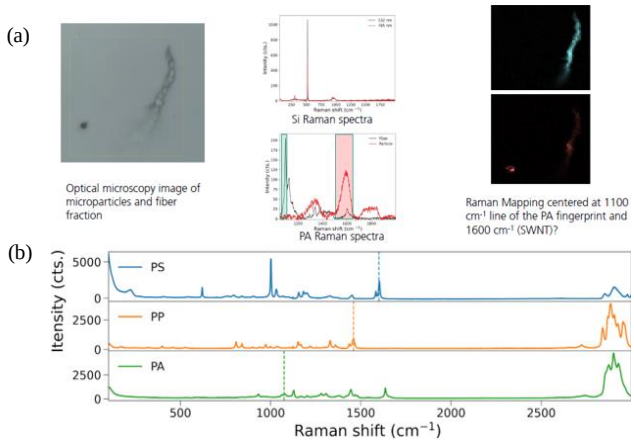

Fig. 8. (a) µRaman spectra; (b) standard spectra.

Thermogravimetry and differential scanning calorimetry - TG-DSC

GA/DTA and DSC measure temperature-related material properties such as mass loss, melting points and other phase transitions. The device used is a thermogravimetric balance model STA-449 produced by Nietzsche Germany. The sample mass was about 5 mg placed in layers in a high sintered alumina crucible of 85 microliters using a temperature program from 30^0C - 900^0C with a heating rate of 10^0 C/minute in an inert atmosphere of Nitrogen purged in the sample room as well as at the scale. The analysis was carried out for the particles collected on the 25 mm quart filters.

The TG (green) curve is stable, without weight loss but with drift, the mass increased by 105% ~~is~~ due to the thermal balance with the vertical system. The DSC (BLUE) curve shows some very small peaks that represent two processes of melting and then decomposition of the PA polymer. (**Fig. 9**) The silica fibres that formed the filter did not undergo any changes and were stable up to 900 ^{0}C.

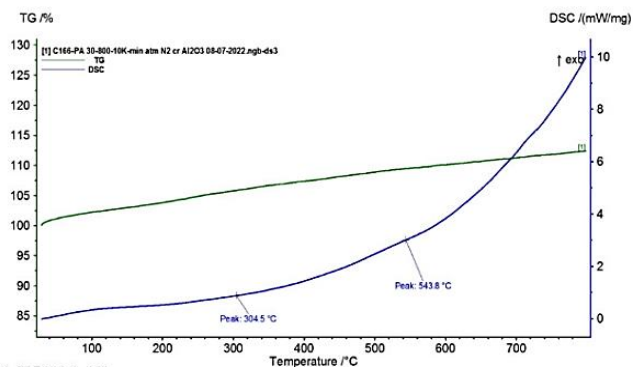

Fig. 9. TG-DSC curves.

CONCLUSION

Studies have been conducted on the assessment of the concentration of particles in the vicinity of workplaces and the environment outside textile companies.

Characterization of statistical populations was performed for the variables: total concentration (TSP) µg/m^3, fractions PM10, PM2.5 and PM1 (µg/m^3) and the total number of particles (TC) 1/l, polyamide particles registered in the vicinity of workplaces. Among the companies that process polyamide yarns, the highest value of TSC (µg/m^3) was registered at the company (590.1 µg/m^3) which processes 334 x 1/ 300,6 dtex/den polyamide yarns in order to make belts/strips with various destinations by applying weaving technology on narrow width fabrics.

For the evaluation of the characteristics, polyamide particles were collected by using pumps with 2 l/min and 38 l/min flow and quartz and polycarbonate filters coated with a membrane covered with gold.

The calculation of correlation coefficients highlighted a strong correlation between the variables: "Collected mass/ Air concentration" and "Collected mass/ Air volume" for all diameters of the filters.

SEM analyses of the polyamide particle highlighted the size and shape of the particles. The statistical analysis of the particle sizes showed that 25% have dimensions smaller than 68.47 nm, 25-50 % have dimensions between 68.47nm - 80.22 nm, 25 % between 80 nm-94.99 nm and 25 % above 94.99 nm. Raman, FTIR and TG-DSC highlighted the presence of PA particles and fibres.

In the next stage of the project:

- CSP-A will be used to identify PA particles;
- The skin tolerance/irritability test of PA particles will be performed in vitro according to ISO 10993;
- After biological samples collection: the transfer values of MNP across lung and intestinal epithelial linings, assessment and characterization concentration-relationships of toxic effects of MNP on epithelial cells of lung, intestine and skin, assessment and characterization concentration-relationships of toxic effects of MNP on specific immune cells, will be developed.

ACKNOWLEDGEMENTS

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CONFLICTS OF INTEREST

There are no conflicts to declare.

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GRAPHICAL ABSTRACT

Polyamide polymer is processed into fibres or into multifilament yarns. Spinning and multifilament yarns are processed into textile structures by weaving, knitting or non-woven processes. During the processing as a result of the intense friction phenomena, NMP releases occur in the surrounding air. The concentration of the particle in the air and their collection was done in order to characterize them by μ-Raman, SEM, FT-IR and DSC analyses.