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MICROSCOPIC INVESTIGATION ON PM_{10} AND AIR-CLEANING COATINGS BASED ON NANO-TIO_2

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ABSTRACT: Fine atmospheric particles have physiological toxicity affecting human health. This study investigates the compositional and morphological properties of the particulate matter smaller than 10 μm (PM_{10}) collected from five different monitoring stations in Chelyabinsk, Russia. We used scanning electron microscopy-energy dispersive X-ray spectroscopy (SEM-EDS) for the investigation of PM_{10}. In order to determine the possible sources of emissions of atmospheric particulate matter, a principal component analysis (PCA) was performed. The results revealed the presence of atmospheric $PM_{10,}$ such as aluminosilicates, calcium particles, sulfates, and metallic particles differentiated on the basis of the chemical composition and morphological parameters. Sulfate PM_{10} with metal inclusions contained Mn, Ti, Zn, Cu, and Cr. It obtained a higher content of potentially toxic elements in traffic-related S-rich particles compared with industrial-related PM_{10}. It was the result of the solubilization of metals in airborne particles and increased toxicity. We obtained new coatings based on titanium nanooxide synthesized by the peroxy method for air purification from $PM_{2.5}$ and PM_{10}. They were macroporous and very effective means against industrial dust and PM air pollution.

Keywords: PM_{10}, Air-cleaning coatings, Nano-$TiO_{2,}$ Scanning electron microscopy, Anthropogenic sources

1. INTRODUCTION

Control over air pollution in Russian cities is increasing every year, as residents are worried about the increasing smog driven by elevations in atmospheric particulate matter (PM), especially particles with aerodynamic diameters <2.5 μm ($PM_{2.5}$) and 10 μm (PM_{10}). By entering the body and penetrating deep into the respiratory tract, PM causes significant harm to health [1,2].

In 2010, Russia set maximum permissible concentrations of $PM_{2.5}$ and PM_{10} (35 and 60 μg/m^3 (24-hour mean concentrations), 25 and 40 μg/m^3 (annual mean concentrations), respectively) [3]. Since then, the number of automatic air monitoring posts has grown in Russian cities. However, these concentrations are higher than $PM_{2.5}$ and PM_{10} concentrations recommended by WHO (15 and 45 μg/m^3 (24-hour mean concentrations), 5 and 15 μg/m^3 (annual mean concentrations), respectively [4]. The industry is one of the main sources of PM pollution in Russian industrial cities. The presence of fine particles in the emissions of various industries has been confirmed: in mechanical engineering, up to 13% $PM_{2.5}$ and up to 40% PM_{10}; in ferrous metallurgy-up to 79% $PM_{2.5}$ and up to 84% PM_{10} [5]. Another major source of air pollution in the urban environment is vehicular transport and it is important to identify the sources of PM air pollution [6,7].

Various factors contribute to the formation of airborne pollutants, and the spatiotemporal variability of such pollutants is expected to be significant depending on the processes of pollutant formation and local meteorological conditions. The road dust and PM collected from the industrial city area are enriched with trace elements [8-10]. It seems reasonable, therefore, to assess potential health risks associated with the exposure of inhabitants of large cities.

Scanning electron microscopy analysis is a useful tool for the characterization of aerosol samples at the level of individual particles [11,12]. Characterization of aerosol samples at the level of individual particles permits to obtain of detailed information on the sources of pollution and their processes. Scanning electron microscopy-energy dispersive X-ray spectroscopy (SEM-EDS) examines particles that are too small to be studied with conventional optical microscopes and gives information on the nature and origin of the particles.

Porous material has been recently realized and proposed as the first viable technical solution for the reduction of aero-dispersed PM [13]. The idea of the present study was to obtain coatings that can be applied to a nonwoven material and used as a filter for air purification from the industrial and atmospheric PM.

The aims of this work were: (1) the identification and characterization of particles present in samples of PM_{10} as part of an air quality study by SEM-EDS; (2) to obtain new coatings based on nano-titanium oxide for air purification from $PM_{2.5}$ and PM_{10}.

2. RESEARCH SIGNIFICANCE

Using SEM-EDS, this work identifies and characterizes the morphology and chemical composition of particles present in samples of PM_{10} collected from the Chelyabinsk urban area. With the data obtained from the characterization, a principal component analysis (PCA) was carried out, from which it was possible to identify some of the potential emitting sources of PM_{10} in the region. We proposed material based on nano-TiO_2 to reduce PM_{10} and $PM_{2.5}$ air pollution. The coating of the proposed material contains macropores that can absorb PM_{10} and $PM_{2.5}$.

3. MATERIALS AND METHODS

3.1 Study Area, Data, and Sample Collection

The sampling stations were located in Chelyabinsk (55°09′14″°N, 61°25′44″ E, Elevation: 219 meters). Chelyabinsk is located on the eastern slope of the Southern Urals. The city has a humid continental climate. The average temperature in January is -14°C/6.6°F. July has a relatively cool average of 19°C/66.7°F, while the annual average is 3°C/37.8 °F.

The results of long-term observations show that the prevailing winds are from the south, southwest, west, and northwest. In Chelyabinsk, the lowest frequency is recorded for east, southeast, and northeast winds. Wind directions vary slightly throughout the year. The population of Chelyabinsk was 1,130,132 in 2010. The city has a land area of roughly 530 km^2.

Chelyabinsk is one of the most air-polluted cities in Russia. According to a report by the Ministry of Ecology of the Chelyabinsk Region, Chelyabinsk showed heavy air pollution, about 110 days per year when pollution is above the national standards [14].

The five sampling sites were located in urban residential areas. Table 1 shows the main characteristics of each site.

PM_{10} sample collection was performed using cascade impactor samplers (Fig. 1). Impactors were operated at a flow rate of 16 L min^{-1} for 24 hours.

3.2 Scanning Electron Microscopy

The samples were characterized by SEM-EDS. SEM analysis was performed on a Jeol JSM-7001F Scanning Electron Microscopy Complex. SEM-EDS is a non-destructive analytical method for surface elemental analysis, with a potential detection limit of 0.1–0.5 wt.% for most elements. For SEM-EDS analyses, PM_{10} samples were mounted with double-sided tape on an aluminum SEM stub for analysis. With the help of a vacuum coating unit (Gold Sputter Coater, SPI-MODULE, US), a very thin layer of gold was deposited on the surface of each sample in order to achieve better conductivity and less electron charge.

Table 1 Description of sampling sites

Code of site	Description
S1	The residential area is located near small industrial enterprises, for example, rock wool production plants. High traffic volume, intercity buses and heavy vehicles.
S2	Residential area. This is a lower-relief area. High traffic volume, intercity buses and heavy vehicles. Distance to the metallurgical plants and their slag dumps is 2.5 km.
S3	Residential area. Distance to the average traffic avenue is 100 m. Average traffic volume. The high volume of heavy vehicles.
S4	The average volume of intercity buses. The distance to the coal-fired power plant is 2 km.
S5	Low traffic volume. It is located near (100 m) an urban park. Distance to the metallurgical plant and its slag dumps is 1.5 km.

Fig. 1 The aerosol cascade impactor sampler

300 particles were manually analyzed 60 particles selected randomly from each of the 5 samples.

3.3 Porous Material Synthesis

3.3.1 Chemicals and characterization

Titanium oxysulphate containing 33% of TiO_2 provided by Alfa Aesar, propanol-1, aqueous ammonia, hydrogen peroxide, methylene blue dye, hydrochloric acid supplied by Reachim, polyvinylalcohol 4-98 (PVA) was provided by Fluka, LUDOX® HS-40 colloidal silica was used, all chemicals were of analytical grade.

Morphological investigation of the prepared composite silica-titania spheres was performed by a field emission scanning electron microscope (SEM) JEOL JSM 7001F. SSA, volume and size distribution of micro and mesopores were probed by N_2 adsorption at 77K using an ASAP 2020 Micromeritics analyzer. UV-vis spectrum was registered with the aid of Shimadzu UV2700 UV-vis spectrometer with $BaSO_4$ as the inert layer with high reflectance.

3.3.2 Preparation of titanium nanooxide

Preparation of titanium nanooxide (nano-TiO_2 or TiO_2 microspheres) was performed via the peroxo-route reported in [15]. For the synthesis, 2.4 g of titanium oxysulfate (containing 10 mmol of TiO_2) was dissolved in 20-30 ml of deionized water. Subsequently, 10 ml of ammonia solution was added with the immediate formation of the colloid precipitate. After this, the precipitate was washed 8 times with deionized water and dissolved in 10 ml of H_2O_2 in the ice bath under vigorous stirring. Dissolution caused the formation of transparent orange colour solution, which was adjusted to 100 ml and pH value to 9.5 by diluted ammonia.

Separately 100 ml of n-propanol was taken and poured into the aqueous titanium peroxo complex solution. The mixture turned turbid instantaneously and was left under agitation for 24 h. Subsequently, the colloidal precipitate was centrifuged, washed 6 times with deionized water and dried under vacuum at 50 ºC for 6 h.

The TiO_2 spherical particles were non-porous after the preparation. For the increase of the specific surface area of TiO_2 microspheres, a reflux procedure was used. The as-prepares titania particles were put into 50 ml of ethanol: water solution (1:1 volume ratio), pH value was adjusted to 5 by the diluted hydrochloric acid. The solution was put to boiling with the reflux condenser for 21 hours. The resulting colloidal precipitate was washed several times with deionized water and dried at 50 ºC for 24 h. After drying, TiO_2 material was calcined at 450°C for 1 hour.

3.3.3 Obtaining nonwoven material with coating based on nano-TiO_2

A suspension of 100 mg of titanium nanooxide in 10 ml of distilled water was mixed with 90 ml of a binder based on PVA and colloidal silica. Suspension was slowly stirred for at least an hour in ultrasonic homogenizer. Non-woven polymer material Spunbond with a density of 45 g/m^2 was placed in a resulting homogeneous solution for 24 hours. We tried using teflon, polyamide fiber, but Spunbond was the best non-woven polymer material for forming porous coatings. After 24 hours, the resulting filter material was washed with distilled water and dried in air.

3.3.4 Study of the air purification efficiency from PM on the obtained filter materials

The possibility of using the resulting filter material with coatings based on nano-TiO_2 for purification from industrial and atmospheric PM_{10} and $PM_{2.5}$ was studied. We used the special experimental installation designed to study the efficiency of cleaning materials from PM (Fig. 2).

PM_{10} and $PM_{2.5}$ concentrations were determined using dust analyzer Atmas («NTM-Protection», Moscow, Russia) [16]. The elemental composition of PM was determined using Perkin Elmer ELAN 9000 Inductively Coupled Plasma Mass Spectrometry (ICP-MS), as described in [9]. The instrument was calibrated using Inorganic Ventures standards.

Fig. 2 Experimental installation for testing air cleaning efficiency

4. RESULTS AND DISCUSSION

4.1 Overall Chemical Composition of PM_{10}

According to the characteristic SEM-EDX analysis, atmospheric particles collected in winter from Chelyabinsk urban area contained 20 elements (Al, B, C, Ca, Cl, Cr, Cu, Fe, K, Mg, Mn, N, Na, Ni, O, Pb, S, Si, Ti and Zn). The single particles were grouped and labeled as: Fe-containing Fe-Mg-containing, aluminosilicates, particles containing and rich in S, particles containing C and N, metal particles including Fe and Zn, Pb, Cu, Mn, Ni and Cr. The most abundant elements detected in PM_{10} were Fe and Mg which were present in 80–100% of the particles. It could be assumed that Fe-Mg-complexes were included in the composition of natural aluminosilicates. Chelyabinsk stands on sedimentary rocks and granite, typical for the Urals, consisting of oxides Al_2O_3 (14–15%), SiO_2 (70–

72%), Fe_2O_3 (0.7–1.1%), MgO (0.6–1.1%) [17]. Such particles were found at all sites.

4.2 Characteristic PM_{10} Collected from Site 1

Figure 3 shows the predominant morphologies of C-rich particles collected at Site 1. They were regular spherical and spheroidal shapes, with some presenting surface defects such as porosity. The spheres contained sulfur and nitrogen oxides. The particle size of this type varied in the range of 1–5 µm. These particles are observed in cities with large vehicular traffic and are associated with exhaust emissions from automobiles using gasoline or diesel combustibles [18,19].

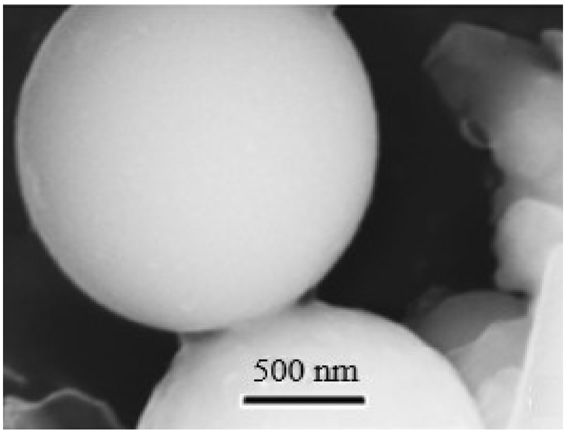

Fig. 3 SEM micrograph (magnification 10,000X) C-rich particles collected in Site 1

Particles in the form of filaments containing aluminosilicate particles enriched with oxides of titanium and other metals were also found (Fig. 4). Such particles can be characteristic of the rock wool production plant located near Site 1 at a distance of 1 km.

4.3 Characteristic PM_{10} Collected from Site 2

Site 2 had the largest number of S-rich particles, with more than half of them containing more than 3 % sulfur. Sulfate PM_{10} is commonly identified as a marker of secondary aerosols related to long-distance transport [20,21].

The majority of the sulfate particles had one or more potentially toxic metal inclusions. Figure 5 shows typical rod-shaped, crystalline, and spherical particles. The size of metal-containing S-rich particles was about 1 µm. Most metal particles were classified as Fe-rich (e.g., hematite), Zn-rich (e.g., zinc sulfate and zinc oxide), Pb-rich (e.g., anglesite), Mn-, Ti-, Cu-, Cr- or W-rich. They were likely emitted from road traffic (exhaust and tire, brake, car body or road surface abrasions) [22]. Site 2 had the highest traffic volume. Zinc, Cu and Ti are often related to brake-pad erosion; Fe, Cu, Pb and Zn are from brake-disc wear. To identify PM from tire abrasion, Cd, Cu, Pb and Zn can be monitored. Metals such as Fe and Zn are also linked to the corrosion of metal car-body parts such as radiators, brakes, and tires. Road dust and roadside soil often contain metals, including Pb, Cu, Cd and Zn, indicative of contamination by road traffic emissions and the abrasion of road surfaces.

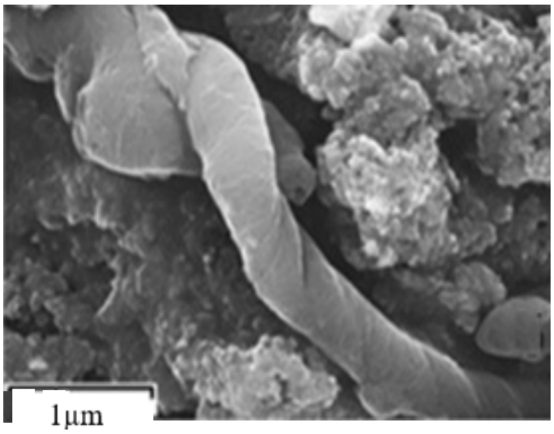

Fig. 4 SEM micrgraph (magnification 5,000X) Ti-rich aluminosilicate particles collected in Site 1

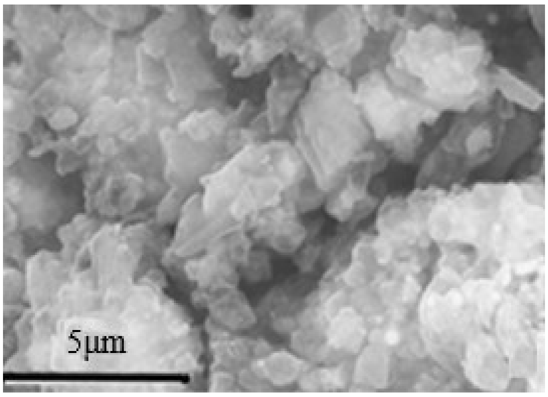

Fig. 5 SEM micrograph (magnification 1,000X) S-rich particles collected in Site 2

It has been shown that sulfates from aqueous SO_2 oxidation catalyzed by transition metals are an important atmospheric process during winter, an alternative to the photochemical pathway that is highly unlikely because of the ultralow O_3 concentrations [23]. Metal catalysis can promote the conversion of SO_2 to sulfates in fog droplets [24]. The internal mixing of metals and acidic constituents solubilizes metals and modifies metal inclusion shapes. The solubilization of metals in airborne particles can increase their toxicity in the particles [25].

4.4 Characteristic PM_{10} Collected from Sites 3-5

At Sites 3 and 4, the morphological characterization results allowed us to classify most of the observed particles as soot due to the presence of large amounts of carbon in the collected sample; the elemental composition spectra were only qualitative and semiquantitative. The soot particles had a spongy aspect and formed spheres of size in the order of nanometers; they are united in chains, forming a mass of amorphous agglomerates, which give rise to larger particles. They had the structural complexity of these particles, and their composition was dominated by carbon (C) and oxygen (O). The soot particles appeared as large clusters of small spheres united by chains and were found to be similar to those in studies of urban regions in Brazil [25]. Such particles are known to be produced by anthropogenic sources such as vehicular emissions and coal burning (Fig. 6).

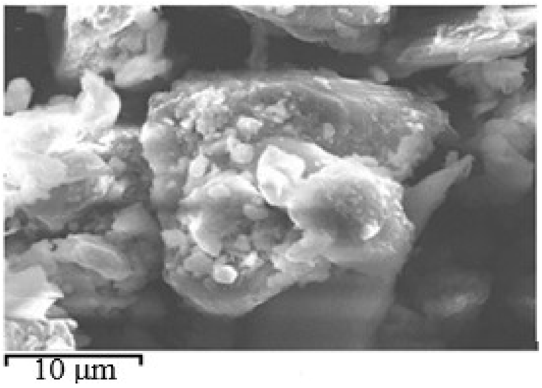

Fig. 6 SEM micrograph (magnification 2,500X) Fe-rich aluminosilicate particles collected in Site 4

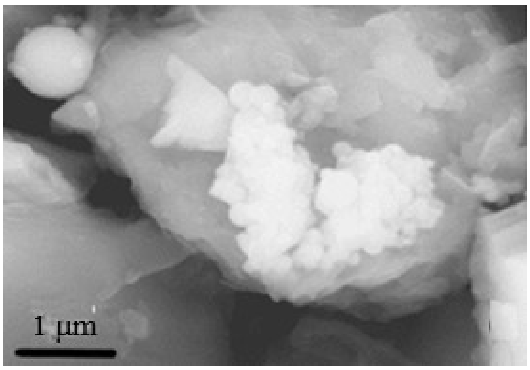

Fig. 7 SEM micrograph (magnification 5,000X) Fe-rich aluminosilicate particles collected in Site 5

At Site 3 the source of the soot is the heavy traffic of trucks that run on a highway through the city. At Site 4 the source of soot is the burning of coal at a thermal power plant. Figure 7 shows spherical Fe-rich PM_{10} found at Site 5. These particles consisted of iron oxide. They could be steel dust emitted by the metallurgical enterprises and placed in numerous slag dumps. Their size ranges between 0.5 and 2 μm. They present a peculiar morphology. They are characterized by perfect sphericity indicating their smelting iron origin or metallurgical activities in general.

4.5 Sources Implied by PCA

To increase the plausibility of the found sources of aerosol particles, a PCA was performed by the Varimax Rotated Factor Matrix method. Total 12 principle components (PCs) were extracted (eigenvalue>1) as which explained 72% of the variance of the data (Table 2).

A large number of PCs is explained by a large number of different sources because various industrial enterprises are located in Chelyabinsk. Atmospheric particles can interact with each other. PC1 explained 11 % variance and was loaded with C, O, Na, Si, S, K, Cl, Fe. It indicated that PM_{10} contained carbonates, silicates and chlorides of sodium, potassium and iron which were mainly from natural sources.

The same can be told about PC6, PC8 and PC9, explaining 6, 4 and 4% of the variance, respectively. They were loaded with aluminosilicates, chlorides and carbonates also indicating natural sources. PC2 titled industrial emission (9% of the total variance) exhibits high loadings of Fe and may be associated with steel production activities. PC4, PC5 and PC10 explain 7, 7 and 4 % of the total variance, respectively, and have high loadings of B, C, O and Fe. They mainly arose from anthropogenic sources such as the burning of coal (B, C and O contained soot PM corresponding to PC4 and PC5) or various transportation fuels (PC10 which has additional Fe loading).

PC7 was responsible for 5% of the total variance, including C, O, Na, Mg, Si, S, Fe and Zn and may be linked to slag dumps dust. PC11 was responsible for 4% of the total variance and high loadings of C, O, Si, S, Fe and Mn. This source was characterized as ferromanganese production. PC3 and PC12 were responsible for 8 and 4 % of the total variance, respectively. They included S and O, besides PC12 was also represented by Na, Si, C, Cr, Ti, Fe, Cu, Ni, Pb and Zn. They were traffic-related S-rich particles.

4.6. Nanocoatings for air purification

Figure 8 represents SEM photographs of coating formed on non-woven polymer material Spunbond with a density of 45 g/m^2 and PM caught in pores after the air purification experiment.

The coating has a porous structure with an average pore size 1 micrometer. Such coatings are of the macroporous type.

Table 2 Results of principal component analysis (Varimax rotation with Kaiser normalization) at the five study sites, only factor loadings ≥0.25 are shown. The highest loadings are in bold

El	PC1	PC2	PC3	PC4	PC5	PC6	PC7	PC8	PC9	PC10	PC11	PC12
Al						**0.91**			0.30			
B				**0.91**	0.33							
C	-0.50			-0.28	**-0.70**		-0.53		0.30	-0.47	-0.43	-0.55
Ca								**0.94**				
Cl	**0.56**					**-0.32**						
Cr												0.27
Cu											**0.35**	0.29
Fe	-0.25	**0.96**					-0.47			-0.26		-0.37
K	**0.25**											
Mg							**0.60**					
Mn											**0.80**	
N										**0.89**		
Na	0.27						-0.30					**-0.31**
Ni												**0.28**
O	-0.28		-0.33		**0.67**	-0.34	-0.53	-0.31	0.35	-0.41	-0.36	-0.45
Pb												**0.29**
S	-0.42		**0.89**				-0.26	-0.27			-0.31	-0.45
Si	-0.26					-0.34	-0.26		**-0.98**		-0.25	-0.33
Ti												**0.8**2
Zn							**0.37**					**0.31**
E	3.1	2.5	2.1	2.0	1.9	1.6	1.4	1.2	1.2	1.1	1.0	1.0
VCR	11	9	8	7	7	6	5	4	4	4	4	4
VCR_c	11	20	28	35	42	47	52	56	61	64	68	72

Note: El-element; E – Eigenvalues; VCR – Variance contribution rate %; VCR_c – Cumulative Variance contribution rate, %.

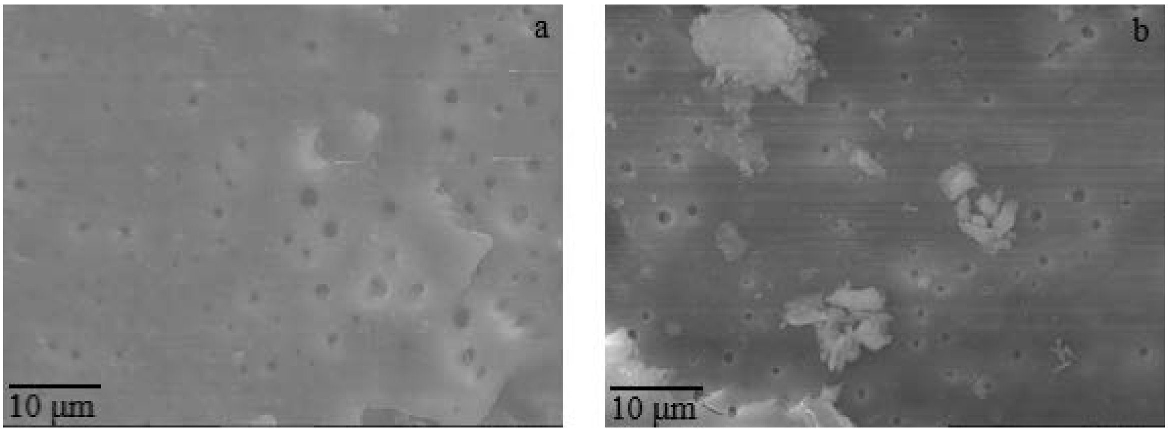

Fig. 8 SEM photographs of (a) TiO_2-based coating and (b) $PM_{2.5}$ in pores of coating

The study of particles using a scanning electron microscope showed that PM is retained by macropores. The cleaning efficiency against PM_{10} and $PM_{2.5}$ for a nonwoven filter coated with titanium nanooxide was 80 and 70%, respectively. Forty-eight elements in 4 different samples were analyzed in collected industrial dust (Table 3). Analysis showed high concentrations of Ti, Cr, Zn, Mn and Cd, among other elements.

5. CONCLUSION

This work studied the morphological and chemical characteristics of metal-containing atmospheric particles collected from 5 sites in the Chelyabinsk urban area. Different magnifications have been selected for SEM-EDS analysis to classify the size range of the particles. SEM-EDS allowed us to provide information about the sources of PM and to highlight differences and similitudes among the sites. Single particle studies gave information for understanding the formation of PM and their potential toxic characteristics.

The five sites are mainly characterized by traffic-related pollution and metallurgical activity. Metal particles were mainly characterized by iron. Sulfur-enriched particles included potentially toxic elements from traffic-related sources.

Table 3 Mean concentrations of elements in different industrial dust

Element	Sample			
	1	2	3	4
Cu	34	18	8888	1588
Zn	510	114	488394	1098
Pb	34.5	5.00	98260	224
Be	2.77	0.47	0.156	0.71
Sc	62.7	8.44	0.57	2.21
Ti	11752	1005	72.3	943
V	196	18.8	12.6	703
Cr	210	10941	49.9	3780
Mn	3756	755	994	14194
Co	1.87	25.0	23.6	943
Ni	13.8	321	42.9	452
Ga	1.26	2.84	40.1	7.32
As	17.9	24.9	4803	25.9
Se	5.97	<2.00	116	<2.00
Rb	26.9	0.95	25.3	15.5
Sr	465	94,7	216	134
Y	30.9	4.81	83.6	4.75
Zr	345	38.6	5.51	19.9
Nb	11.9	0.37	<0.050	2.14
Mo	1.51	0.66	26	1610
Cd	2.02	1.03	7937	4.04
Sn	2.8	1.69	500	36
Sb	2.18	0.88	412	4.81
Te	0.152	<0.30	719	<0.30
Cs	1.06	0.060	2.83	1.31
Ba	698	127	1099	426
La	36.5	4.99	2.28	7.13
Ce	67.5	10.3	2.26	16.6
Pr	7.46	1.16	0.26	1.76
Nd	27.8	4.35	0.98	6.49
Sm	5.66	0.92	0.23	1.47
Eu	1.47	0.22	5.60	0.35
Gd	6.35	1.01	0.24	1.40
Tb	0.87	0.148	0.030	0.191
Dy	5.16	0.77	0.182	0.96
Ho	1.08	0.170	0.051	0.199
Er	3.10	0.40	0.044	0.46
Tm	0.45	0.070	0.014	0.072
Yb	2.91	0.44	0.114	0.51
Lu	0.40	0.074	0.017	0.067
Hf	9.21	0.92	0.133	0.46
Ta	3.55	0.010	<0.010	0.41
W	2.58	2.66	0.36	1563
Tl	0.018	<0.010	32.3	0.52
Bi	0.32	0.088	311	2.65
Th	7.84	1.31	0.21	1.19
U	5.74	0.54	0.49	0.60

Note: sample 1 is blast furnace slag; sample 2 is ferrochromic slag; sample 3 is waelz-oxide; sample 4 is dust from filters

A method of air purification from PM was presented, it shows high PM removal efficacy.

6. ACKNOWLEDGMENTS

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