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МЕЃУНАРОДНО УЧЕСТВО**

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**ТЕХНОЛОГИЈА НА ПОДЗЕМНА И ПОВРШИНСКА
ЕКСПЛОАТАЦИЈА НА МИНЕРАЛНИ СУРОВИНИ**

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ЗРГИМ

XII СТРУЧНО СОВЕТУВАЊЕ НА ТЕМА:

“Технологија на подземна и површинска експлоатација на минерални сировини”

ПОДЕКС – ПОВЕКС '19

**Струмица
01 ÷ 03. 11. 2019 год.**

ПРЕДГОВОР

Меѓународното стручно советување за подземната експлоатација на минералните сировини (ПОДЕКС), за првпат се одржа на 06.12.2007 год. во Пробиштип во организација на Сојузот на Рударските и Геолошките Инженери на Македонија (СРГИМ).

Од 2012 година советувањето е проширено со трудови од површинската експлоатација на минерални сировини и е именувано како ПОДЕКС-ПОВЕКС.

Стручното советување, на тема: технологија на подземна и површинска експлоатација на минерални сировини, традиционално се одржува секоја година во месец ноември. На ова советување земаат учество голем број на стручни лица од: рударската индустрија, универзитетите, научно-истражувачките и проектантските организации, производителите на опрема и др.

На досегашните единаесет советувања (2007, 2008, 2009, 2010, 2011, 2012, 2014, 2015, 2016, 2017 и 2018 год.) учествуваа повеќе автори од 12 држави, кои презентираа 312 стручни трудови.

За ова дванаесетто советување (ПОДЕКС - ПОВЕКС '19) пријавени се 25 труда, на автори од 2 држави.

Големиот број на трудови од домашните автори произлезе како резултат на научно-истражувачката работа реализирана на високообразовните институции во Р. Македонија. Меѓутоа, посебно не радува учеството на автори од непосредното рударско производство, кои што презентираат постигнати резултати во рударската пракса.

Се надеваме дека традицијата за собирање на сите специјалисти од областа на подземната и површинската експлоатација на минералните сировини, ќе продолжи и дека во идниот период ова советување ќе прерасне во меѓународен симпозиум.

Уредници



AMGEM

XII EXPERT CONFERENCE THEMED:

“Technology of underground and surface mining of mineral raw materials”

PODEKS - POVEKS '19

Strumica

01 ÷ 03. 11. 2019.

FOREWORD

The International expert conference on underground mining of mineral raw materials (PODEKS), organized by the Association of Mining and Geology Engineers of Macedonia (AMGEM), was first held on 06.12.2007 in Probishtip.

Since 2012, in this counseling, surface exploitation of mineral resources is included too, and it is called PODEKS-POVEKS.

This expert conference called: Technology of underground and surface mining of mineral raw materials, traditionally, has been organized annually during November. A number of experts from the mining industry, universities, research institutions, planning companies, and equipment manufacturing companies participate in this conference.

Many authors from 12 countries participated in the previous eleven conferences (2007, 2008, 2009, 2010, 2011, 2012, 2014, 2015, 2016, 2017 and 2018) presenting 312 expert papers.

Twenty-five authors from 2 countries have registered their expert papers for the XIIth conference (PODEKS - POVEKS '19).

The large number of expert papers from the domestic authors has emerged as a result of the research work carried out at the higher education institutions in the Republic of Macedonia. We are particularly delighted by the participation of the authors involved in the immediate mining production who will be presenting the achieved results in the mining practice.

We hope that the tradition of gathering of all specialists from the field of underground and surface mining of mineral raw materials will continue and that this conference will grow up to an international conference in the future.

The Editors



ЗРГИМ
Здружение на
рударски и
геолошки инженери
на Македонија

XII СТРУЧНО СОВЕТУВАЊЕ НА ТЕМА:

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на минерални сировини**

ПОДЕКС – ПОВЕКС '19

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ЗРГИМ
Здружение на
рударски и
геолошки инженери
на Р. Македонија

XII^{TO} СТРУЧНО СОВЕТУВАЊЕ НА ТЕМА:
Технологија на подземна и површинска експлоатација на
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**ЗАГАДУВАЊЕ НА ВОЗДУХОТ СО ПРАШИНА ВО ГРАДОТ КАВАДАРЦИ.
ОДРЕДУВАЊЕ НА ФАЗНАТА ЗАСТАПЕНОСТ СО ПРИМЕНА НА СКАНИНГ
ЕЛЕКТРОНСКА МИКРОСКОПИЈА (СЕМ) И ЕНЕРГЕТСКА ДИСПЕРЗИВНА
СПЕКТРОСКОПИЈА (ЕДС)**

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Абстракт: На основа на добиените резултати може да се заклучи дека примероците од прашина (ПМ-10) содржат неколку минерални фази и тоа: алумосиликатна фаза која ги вклучува (илит, плагиоклас, кварц, амфиболи, пироксени и хлорит). Други присутни минерални фази се: калцит, гипс, железни оксиди/хидроксици, хромит, минерали на среброто и метални фази. Не е утврдено присуство на фиброзни минерални фази.

Клучни зборови: ПМ-10, минерални фази, Сканинг Електронска Микроскопија.

**AIR POLLUTION OF DUST IN TOWN OF KAVADARCI.
DETERMINATION OF PHASE CONTENT BY SCANNING ELECTRON
MICROSCOPY (SEM), ENERGY DISPERSIVE SPECTROSCOPY (EDS)**

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Abstract: Based on obtained results it can be concluded that PM10 samples contained several aluminosilicate phases, including illite, plagioclase, quartz, and possibly amphibole/pyroxene and chlorite. Other phases observed were calcite, gypsum, iron oxides/hydroxides, chromite, silver minerals, and metallic phases. Minor nickel was found associated with metal oxides and stainless steel. No indication of fibrous materials. Particles such as illite, plagioclase, quartz, amphibole/pyroxene, chlorite, calcite and gypsum are mainly from natural origin, while iron oxides/hydroxides, chromite, silver minerals and nickel come from human activities such as industrial processes, especially the ferronickel smelter plant which is situated in this area.

Key words: PM-10, mineral phase, Scanning Electron Microscopy (SEM-EDS).

1. INTRODUCTION

The quality of the environment in the city of Kavadarci is affected by several most important factors: industrialization of the area, using of fertilizers in the agriculture and

local infrastructure. The highest impact to the environment in the area was registered after 1982 when the ferronickel smelter plant was started with the production. This impact is manifested differently in various environmental media (Fig 1).

The term dust means all fine grain solid particles which under certain conditions can fly in the air environment and gradually deposited by the action of gravity (Glossary of Atmospheric Chemistry Terms, IUPAC, 1990).

Solid particles are retained longer in the air forming *dispersion system* (dispersoid), in which air is a dispersion medium and the particles are dispersion phase. Dispersion system in which dispersion environment (air) prevail is called aerosol. When in the system prevail solid particles, it is called airgel. Such a system represents the dust deposited on the floor or on the walls. These dispersion systems are characterized by the following basic parameters: concentration of dust in the unit volume of air, the degree dispersion, dispersion (granulometric) and chemical content. Significant impact on the properties of aerosols shows the form of solid particles (Harrison, R. M. 1999).

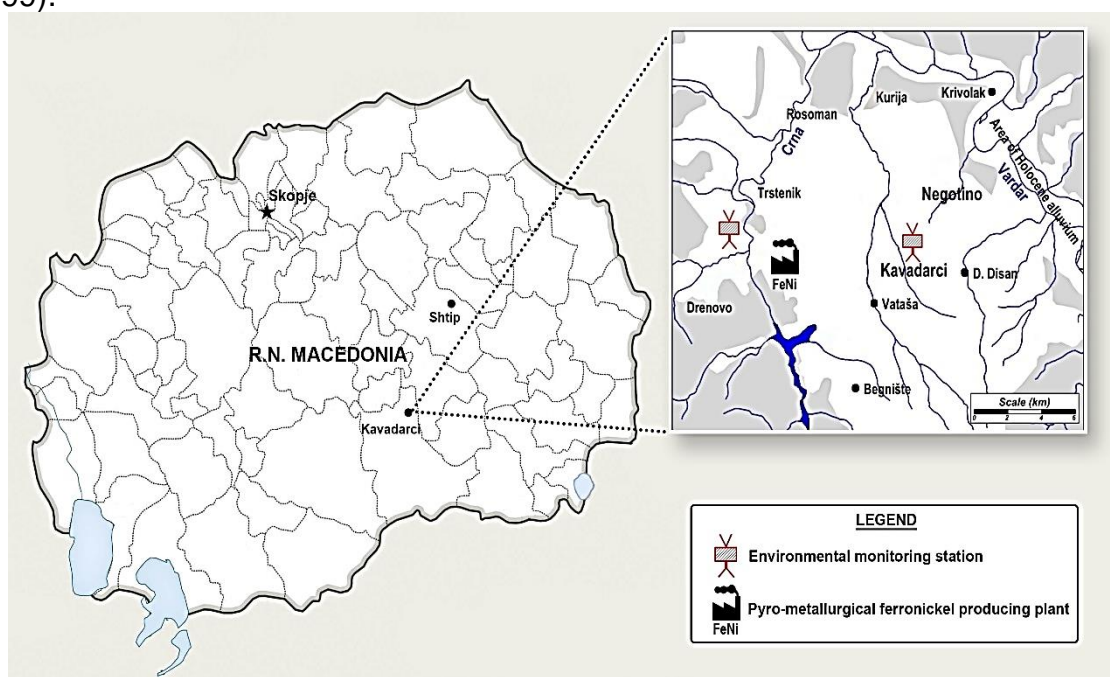


Figure 1. Geographical position of investigation region

The concentration of dust can be expressed as *weight* (gravimetric) in mg/m^3 , and numerical (coniometric) in number of particles/ m^3 . According to the size of the particles, a degree of dispersion, aerosols may be: rough dispersal, fine dispersal, colloidal and molecular. Mineral dust is polydispersal system in which the size of the particles varies from 0.01 to 100 μm . Larger particles due to their weight precipitate relatively fast and do not remain longer in the air environment.

The content of specific factions (groups) with different particle sizes is called dispersion (granulometric) composition of aerosols. The degree of dispersion and dispersed content depend on several factors, primarily the petrographic composition of the rocks, their physical and mechanical characteristics, the nature of the creation of dust, the characteristics of the airflow etc (Ebert, M, et al, 2000).

The shape of the particles from the mining aerosols is very arbitrary and is directly determined by the physical-mechanical and petrographic characteristics of the environment that their are formed. The most common are plated forms and rarely cubic or prismatic.

For the determination of the phenomena associated with the appearance and existence of aerosols in the atmosphere, besides the basic physical characteristics, the following properties are also important: physical and chemical activity of particles, electric and magnetic properties, aggregation, aerodynamic characteristics, wetting ability and condensation of moisture on the particles and others.

By increasing the degree of dispersion the chemical and physical activities of particles are also increase. Namely, as a result of substantial increase of particle surface with respect to their weight, the speed of oxidation significantly increases, the dissolution and absorption of gases is facilitated, etc. For example, heavily soluble finely dispersed silica (SiO_2) is easier to dissolve in a basic solution, which means that the biological activity of fine (respirable) silica dust is significantly higher.

Flying particles in the aerosol very easily absorb the gas molecules on the free surface, forming around them a gas layer. This layer depending of the properties of the absorbed gas, significantly change the properties of aerosol particles, making them primarily hydrophobic, and if the gases are toxic (CO , N_2O_5 ...) or radioactive (radon) they are hazardous to living organisms.

The retention time of particles in the air environment is conditioned by their aerodynamic properties. They basically depend on the form, dimensions and density of the particles, and the nature of the air flows.

In turbulent flow, the aerosol particles and the air participate in cross (whirlwind) movements. Under such movements they collide with increased velocity and form *aggregates* (compounds of two or more particles). The causes of the appearance of the aggregation of particles are not fully studied, although it is known that the highest impact show the form and dimensions, the chemical composition of the particles, as well as the sign of the absorbed gas ions.

Particles of aerosols are electrically charged. This charge is a result of the processes of absorption of gaseous ions, collision with the dry hard surfaces (walls of the rooms or pipelines), as well as the collision between the particles themselves. It must be noted that even in the moment of separation of dust or crumbling of the mass, the particles are charged. This is explained by the appearance of free energy of the grinding points due to the destruction of the crystal lattice of minerals.

The tendency of the dust particles to moistening is very important to define the appropriate techniques for dust collection. It depends on several factors such as: mineralogical and petrographic composition of the working environment, the degree of dispersion of particles, the degree of oxidation of their surfaces, absorption of gases and so on. In certain cases, fine dispersed particles can become condensed nuclei in the air environment and remain in droplets with a size up to 5 μm .

2. METHODOLOGY

2.1. Sampling

The dust samples (particulate matters, PM10) were collected by the standard procedures by setting up two mobile stations, one in the area of the village of Vozarci (near the iron ferronickel smelter plant) and the other in the urban part of the town of Kavadarci. Samples have been collected in the area of the village of Vazarci, and 13 from the urban part of Kavadarci.

The sampling device consists of three integral, conductive plastic cassettes sampling head, where the design does not allow a significant spilling around the filter. The sampling head consists of a cylindrical protective casing and filter holder with an

auxiliary filter. The protective layer of the filter holder is made of stainless material. The filter should be tight so that no significant leakage around the filter at various pressures up to approximately 50 kPa. The length of the shell should be from 0.5 to 2.5 times of the diameter of the filter. If the wind speed measurement shows higher velocity than 5 m/s, the longer length of casing diameter of 2.5 should be used. Flow control should also be achieved by using flow controller. The sampling pump should be capable of maintaining a pressure through the filter at least 50 kPa, flow between 8 l/min and 30 l/min, depending on the diameter of the filter used.

In order to obtain better analytical sensitivity, the flow rate of 8 m/l is required if a filter with a diameter of 25 mm is used. The flow is equivalent to the main side of the filter with a speed of approximately 35 cm/s, resulting the pressure of approximately 50 kPa. Sampling pump should be able to maintain the overall flow with a $\pm 10\%$ throughout the sampling period.

To start a measurement, the sampling head should be placed at a height of approximately 1.5 meters from the ground. If the sampling head used previously is mounted, a 5 μm celulous auxiliary filter should be put as a base for the sampling head, to set up a filter previously treated with a gold vapors. Then, the filters are tighten in the sampling head so that the filter lies on the auxiliary filter.

The membrane filter should be coated with the thin golden layer before sampling. Gold will protect the filter in the process of plasma ashing and will improve the contrast between the fibers of SEM image. This gold layer should be applied on the side for dust collection, i.e. the smooth and brighter side with a layer of approximately 30 nm thickness. Within 2 minutes from the start of sampling, the flow should be adjusted to 2 l/min per square centimeter (this value should not vary more than $\pm 10\%$ during the sampling). This corresponds to a volume of 1000 liters per square centimeters of effective filter area in a sampling period of approximately 8 hours.

2.2. Determination of mineral phases

For the determination of the phase and mineralogical content of PM-10 collected from Kavadarci and Vozarci areas the filters were cut and mounted on 25 millimeter Cambridge-style SEM stubs using double sided carbon tape, and graphite coated to prevent charging. The coated samples were analyzed by Quanta 650F SEM, fitted with a back-scattered electron detector (BSED) and a Bruker 5030 X-ray detector. The Esprit Quantax 1.9 EDS Analysis System was used to determine the elemental composition of particulate matter. Point Analysis was used to characterize the samples in high-vacuum mode, using an accelerating voltage of 15 kV and a spot size of 6. BSE images of selected fields of view were taken to examine SEM-based characteristics.

2.3. Obtained results and discussion

It was found by EDS analyses that the filters contained several aluminosilicate phases, including quartz, illite, plagioclase, and possibly amphibole/pyroxene and chlorite. Silica (SiO_2) particles are characterized by high content of Si and O. The pure silica particles have natural and anthropogenic origins (Li et al., 2010). These particles have tubular structure. It is the most abundant chemical constituent of Earth's crust and a major component of sandstone and granite. Thus, the most abundant source for this particle type is soil related.

Alumosilicate particles are composed primarily of feldspar (Si, Al, Ca or Si, Al, Na) and clay (Si, Al or Si, Al, Fe). Their origin is mainly crustal, but they can also come from erosion of buildings constructions and from road dust. Other elements are present in minor content in the aluminosilicate particles. These particles mainly present an angular shape, ranging from polyhedral to sharp one. Illite and plagioclase were identified in sample 4 (Figure 2). Chemical composition of these minerals is shown in Figure 2. In the image given in Figure 2 it can be seen the particles of illite, plagioclase and spores or pollen while the EDS spectra of illite and plagioclase are given on Figure 2 (A and B).

Particles with a high content of Ca (mass fraction <50%) fall into the calcium rich particles. Carbonate minerals include calcite (CaCO_3) with the traces of other dust related elements which are the common constituent of soil and often observed in the individual aerosol particle analysis (Lu et al., 2006). These particles are irregular fragments with distinct rough surfaces on all faces originated from building construction and demolition and commonly found in Earth's crust. The morphology of this small particle suggests its mineral origin. Calcium carbonate was observed on and around clay minerals in sample 4 (Figure 3). The content of major elements is given also Figure 3.

The particles of manganochromite and stainless steel were also observed in the sample No. 4. Figure 4A shows BSE images of manganochromite while 3B shows EDS spectra of manganochromite. BSE images and EDS spectra of stainless steel is shown in Figs. 4 and 4D, respectively. Metals, metal oxides, and metal oxyhydroxides were also found with clay minerals. BSE images and EDS spectra are given in Figures 5E and 5F, respectively. Minor nickel was found associated with metal oxides and stainless steel (Figure 5D). Also, metals, metal oxides, and metal oxyhydroxides were found with clay minerals (Figure 5). Hydrated phases, observed to be volatile under the electron beam, presumably produced water vapor or carbon dioxide as an effect of heating (Figure 5E, 5F). Minor nickel was found associated with metal oxides (Figure 5B).

Several aluminosilicates with varying elemental compositions could not be conclusively identified (Figure 6). The content of major elements of iron magnesium silicate (Figure 6 B) and iron magnesium aluminosilicate (Figure 6D) are also determined. Gypsum was observed in sample 35 (Fig. 7A,B). Metals, metal oxides, and metal oxyhydroxides were found with clay minerals (Fig. 7C,D). Hydrated phases, observed to be volatile under the electron beam, presumably produced water vapour or carbon dioxide as an effect of heating (Fig. 7 C,D). Silver and associated minerals were observed in sample 11 (Fig. 8). Chlorargyrite (Fig. 8 A,B), with minor acanthite. Chlorargyrite and silver (bright phase indicated in C) (Fig. 8 C,D). Silver mineral (Fig. 8 E,F), can be of crustal origin, but may also come from human activities such as industrial processes.

In the dusts from this region metal oxides and sulfides; magnesium silicate with iron oxide (Fig. 9 A,B); magnesium silicate with zinc, iron oxide (Fig. 10 C,D); and aluminosilicate coated by iron, chromium, titanium and nickel oxides and/or sulfide (Fig. 9 E,F) have been also identified. This group of particles mainly corresponds to Fe oxides with irregular morphology which are assumed to be soil related. Minor nickel was found associated with metal oxides (Fig.9 E). Metals, metal oxides, and metal oxyhydroxides were found with clay minerals. Because these minerals are present in the ore processed in the ferronickel smelter plant which is situated in this area, we can conclude that the content of these particles in the analyzed samples come from human activities in the metallurgical plant.

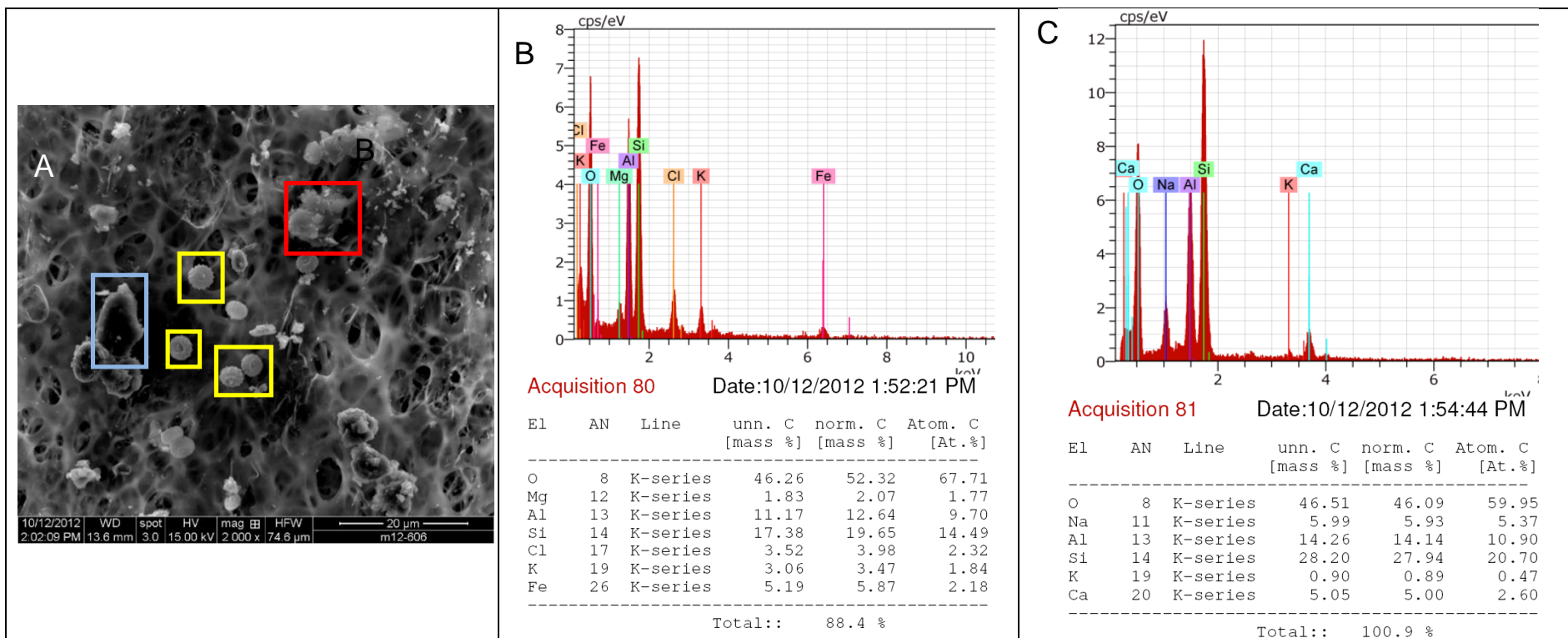


Figure 2. BSE photos of sample No. 4 showing illite (red), plagioclase (blue), pollen spores (yellow). B) EDS spectrum of illite, C) EDS spectrum of plagioclase

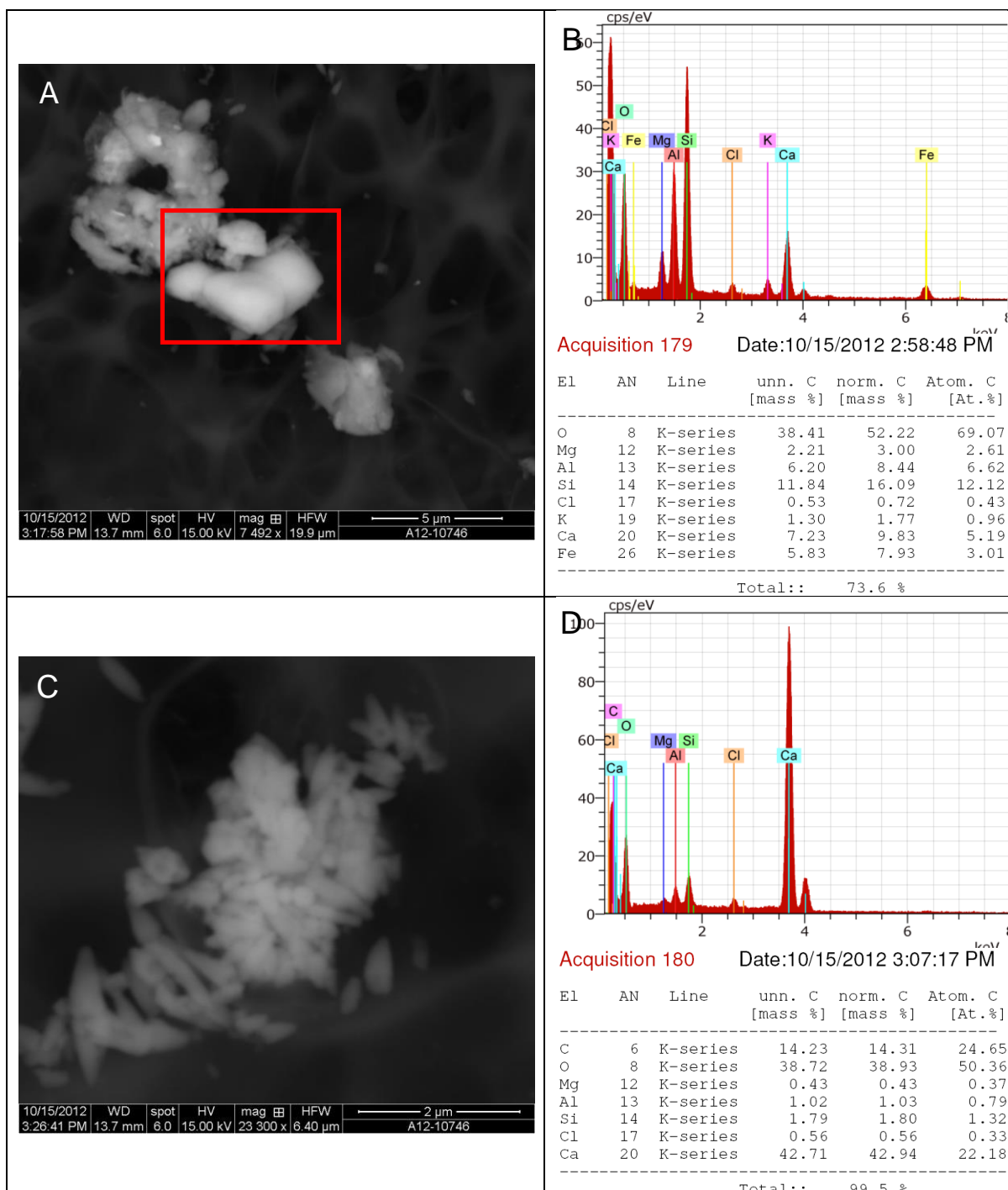


Figure 3. BSE image and EDS spectra of sample No. 4. A, B) calcium carbonate (A) around the aluminosilicates. C, D) Calcium carbonate and aluminosilicates

Based on obtained results it can be concluded that dust on the filter samples contained several aluminosilicate phases, including illite, plagioclase, quartz, and possibly amphibole/pyroxene and chlorite. Other phases observed were calcite, gypsum, iron oxides/hydroxides, chromite, silver minerals, and metallic phases. Minor nickel was found associated with metal oxides and stainless steel. No indication of fibrous materials. Particles such as illite, plagioclase, quartz, amphibole/pyroxene, chlorite, calcite and gypsum are mainly from natural origin, while iron oxides/hydroxides, chromite, silver minerals and nickel come from human activities such as industrial processes, especially the ferronickel smelter plant which is situated in this area.

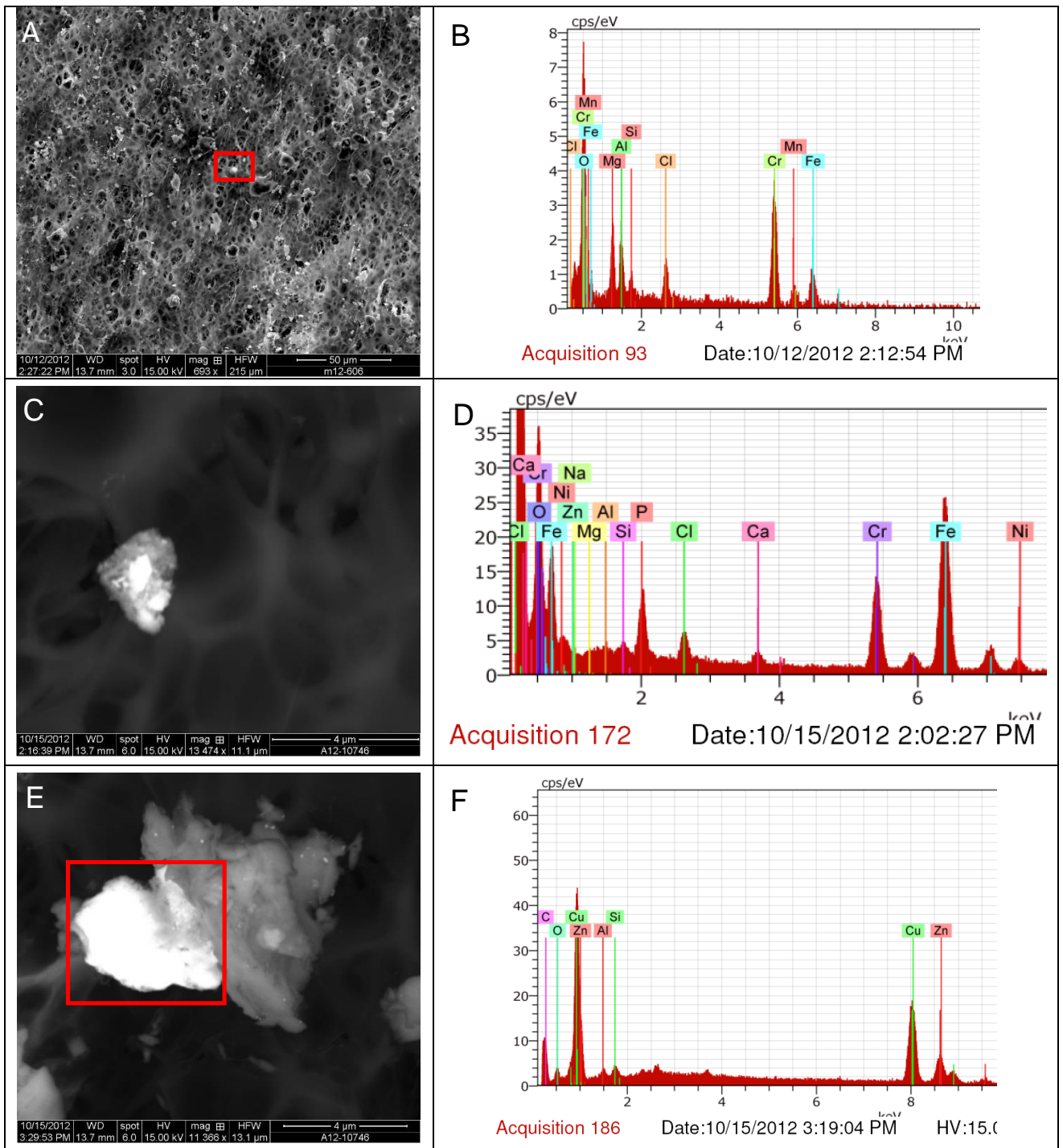


Figure 4. BSE image and EDS spectra of metal phase in sample No. 4. A, B) manganochromate (A). C, D) Particles of stainless steel. E, F) metallic forms of copper and zinc (E) on a sodium magnesium aluminosilicate

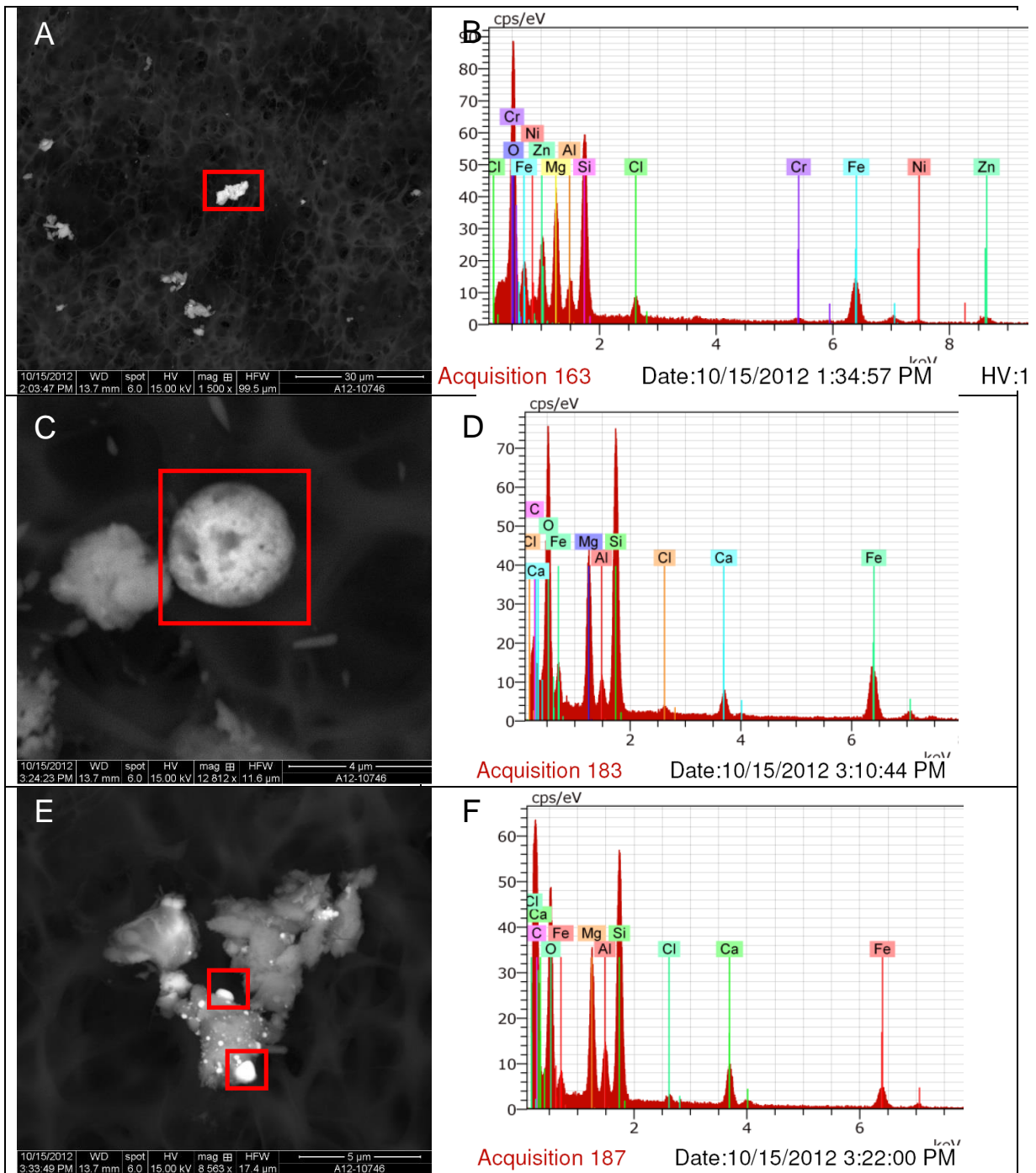


Figure 5. BSE image and EDS spectra of oxides in the sample No. 4. A, B) Magnesium silicate with metal oxide (A). C, D) Iron oxide and magnesium silicate (most probably pyroxene/amphibole) (C). E, F) iron oxyhydroxide (E) and hydrated magnesium clay minerals

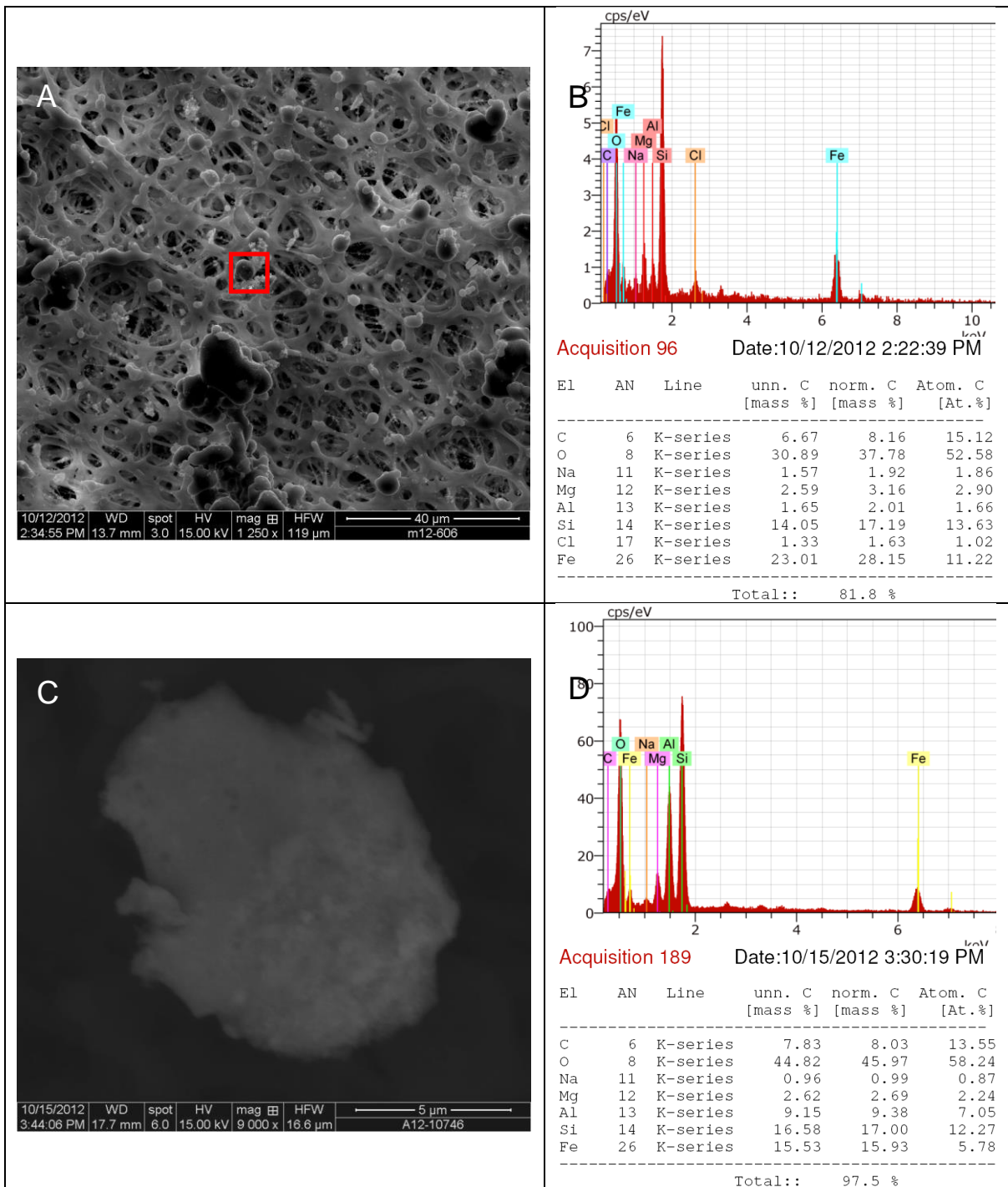


Figure 6. BSE image and EDS spectra of sample No. 11. A, B) Iron magnesium silicate (A). C, D) iron magnesium aluminosilicate (probably chlorite)

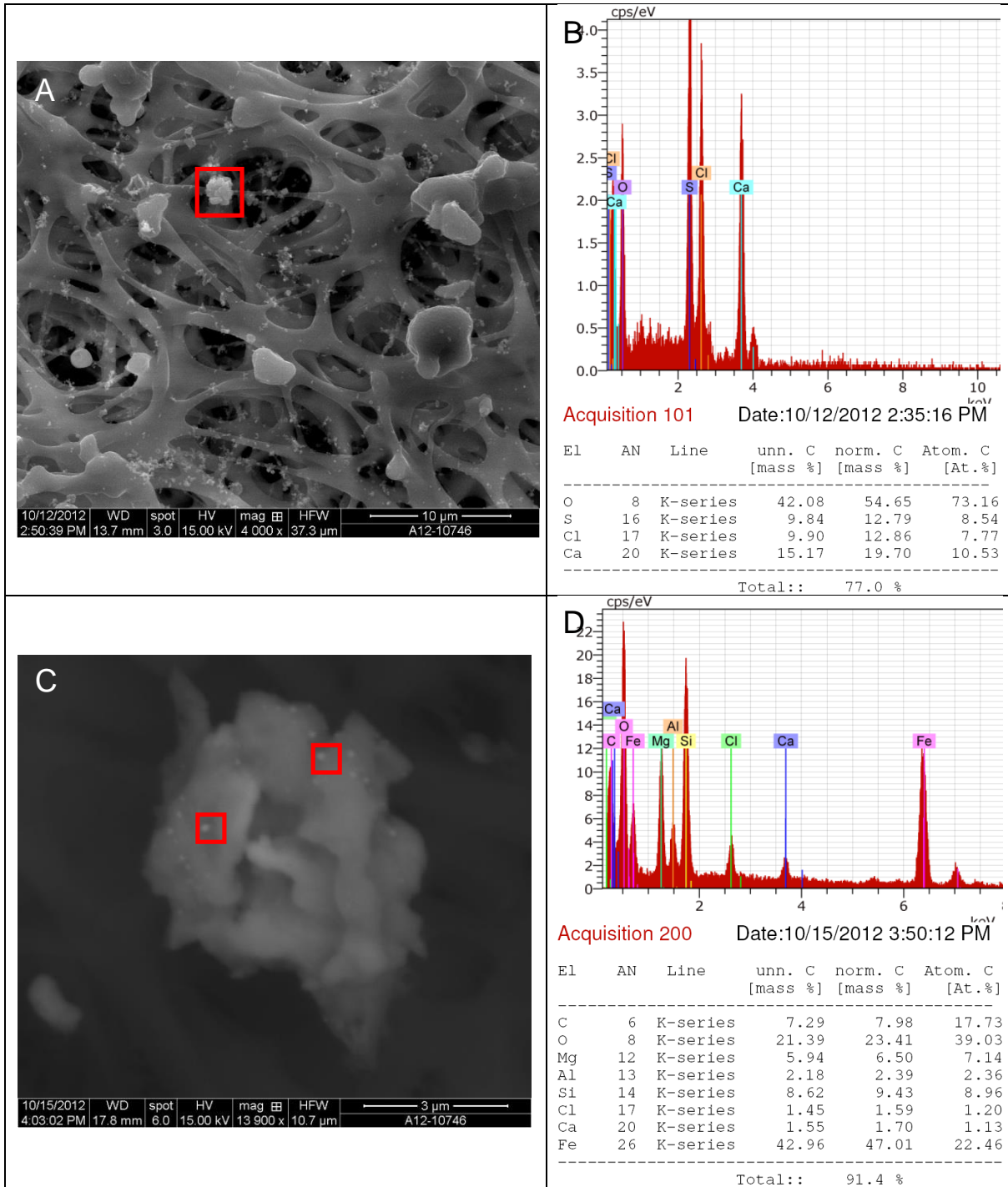


Figure 7. BSE images and EDS spectra of sample No. 11. A,B) Gypsum (A). C,D) iron oxide/oxyhydroxide (C) and magnesium silicate minerals

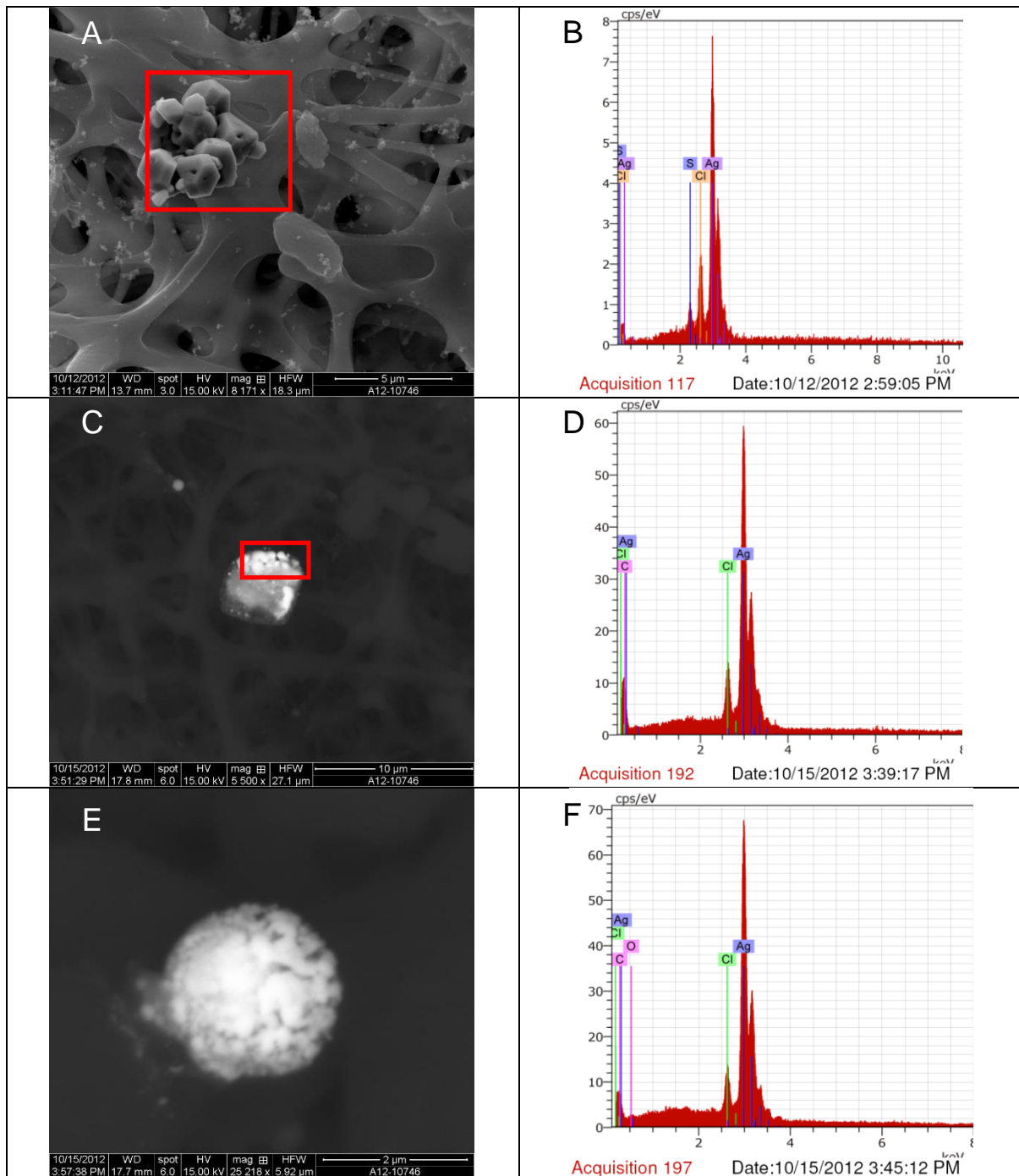


Figure 8. BSE images and EDS spectra of silver mineral in sample No. 11. A,B) chlorargarite (A) with small content of acantite (B). C, D) Chlorargarite and silver (gray phase in C). E,F) silver mineral

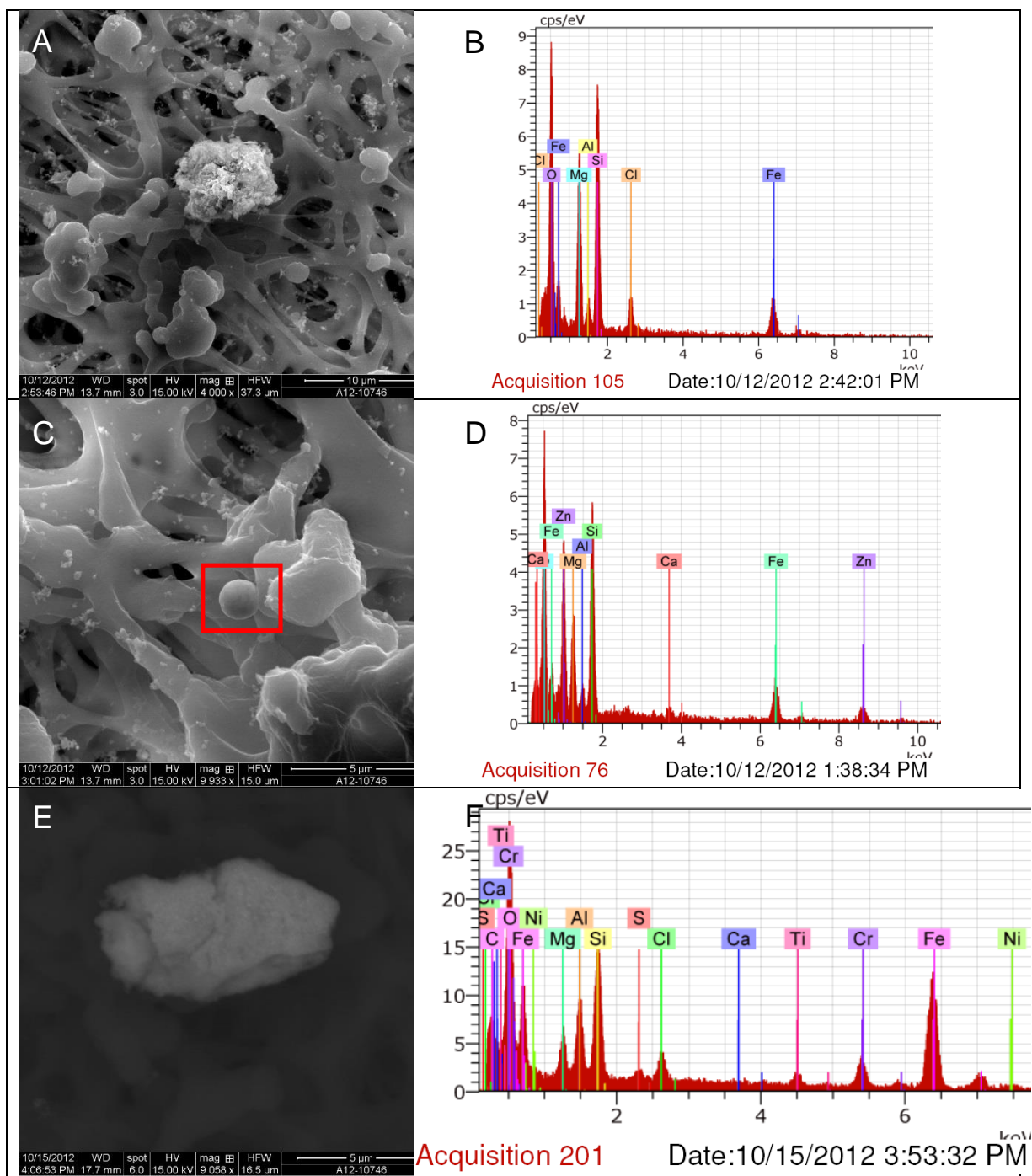


Figure 9. BSE images and EDS spectra of metal oxides and sulfides in sample No. 11. (A,B) magnesium silicate with iron oxide, (C,D) magnesium silicate with zinc, iron oxide (C). (E,F) aluminosilicates around iron, chromium, titanium and nickel oxides and/or sulfides

The investigations performed by applying electron microscopy (SEM-EDS technique) unequivocally confirmed the results made by using XRF, the X-ray diffraction and the results from the determination of chemical composition of particles PM-10 with the application of the ICP-AES and ICP-MS (Boev et al, 2013, Stafilov et al, 2010, Stafilov et al, 2012). From the results performed with all of the applied techniques it can be concluded that the presence of specified mineral phases that have typically anthropogenic origin are registered as well as the mineral phases that have lithogenic

origin or the origin of the present geological structure. Therefore, it can be concluded that the urban dust with a sizes below 10 μm (PM-10) in the Tikveš area originated from lithogenic and anthropogenic processes. It can be concluded that the phase composition of the particles PM10 from Tikveš area consisting of mineral phases which have anthropogenic origin confirmed by the high content of Fe, Ni, Cu, Zn, Ag, Cr (present in higher content in the ore processed in the ferronickel smelter), and by the presence of the minerals like: chlorite, amphibole, pyroxene, magnetite, chromites, Ag-minerals, metallic forms of Mn-Cr, Cu-Zn (also present in the ore processed in the smelter plant). The lithogeneic origin of the part of PM10 is confirmed by the presence of minerals quartz, calcite or plagioklas clay minerals.

3. CONCLUSION

By the application of Scanning Electron Microscopy (SEM) - Energy Dispersive Spectroscopy (EDS) mineralogical phases in the PM10 samples were determined. It was found that the particles contained several aluminosilicate phases, including quartz, illite, plagioclase, and possibly amphibole/pyroxene and chlorite. Silica (SiO_2) particles are characterized by high content of Si and O. The pure silica particles have natural and anthropogenic origins. These particles have tubular structure. It is the most abundant chemical constituent of Earth's crust and a major component of sandstone and granite. Thus, the most abundant source for this particle type is soil related. Aluminosilicate particles are composed primarily of feldspar (Si, Al, Ca or Si, Al, Na) and clay (Si, Al or Si, Al, Fe). Their origin is mainly crustal, but they can also come from erosion of buildings constructions and from road dust. Other elements are present in minor content in the aluminosilicate particles. These particles mainly present an angular shape, ranging from polyhedral to sharp one. Illite and plagioclase were identified in some samples. From the obtained image it can be seen the presence of particles of illite, plagioclase and spores or pollen.

Particles with a high content of Ca (mass fraction $<50\%$) fall into the calcium rich particles. Carbonate minerals include calcite (CaCO_3) with the traces of other dust related elements which are the common constituent of soil and often observed in the individual aerosol particle analysis. These particles are irregular fragments with distinct rough surfaces on all faces originated from building construction and demolition and commonly found in Earth's crust. The morphology of this small particle suggests its mineral origin. Calcium carbonate was observed on and around clay minerals. The particles of manganochromite and stainless steel were also observed in this sample as well. From the BSE images and EDS spectra the following minerals fractions were determined: manganochromite, stainless steel, metals, metal oxides, and metal oxyhydroxides. Minor particles of nickel were found associated with metal oxides and stainless steel. Also, metals, metal oxides, and metal oxyhydroxides were found with clay minerals. Hydrated phases, observed to be volatile under the electron beam, presumably produced water vapor or carbon dioxide as an effect of heating. Minor nickel was found associated with metal oxides.

Several aluminosilicates with varying elemental compositions could not be conclusively identified. The contents of major elements of iron magnesium silicate and iron magnesium aluminosilicate are also determined. Gypsum was observed in sample. Metals, metal oxides, and metal oxyhydroxides were found with clay minerals. Hydrated phases, observed to be volatile under the electron beam, presumably produced water vapor or carbon dioxide as an effect of heating. Silver and associated minerals were observed in sample No. 11, chlorargyrite, with minor acanthite,

chlorargyrite and silver. Silver mineral can be of crustal origin, but may also come from human activities such as industrial processes.

In the dusts from this region metal oxides and sulfides; magnesium silicate with iron oxide; magnesium silicate with zinc, iron oxide; and aluminosilicate coated by iron, chromium, titanium and nickel oxides and/or sulfide have been also identified. This group of particles mainly corresponds to Fe oxides with irregular morphology which are assumed to be soil related. Minor nickel was found associated with metal oxides. Metals, metaloxides, and metal oxyhydroxides were found with clay minerals. Because these minerals are present in the ore processed in the ferronickel smelter plant which is situated in this area, we can conclude that the content of these particles in the analyzed samples come from human activities in the metallurgical plant.

Based on obtained results it can be concluded that PM10 samples contained several aluminosilicate phases, including illite, plagioclase, quartz, and possibly amphibole/pyroxene and chlorite. Other phases observed were calcite, gypsum, iron oxides/hydroxides, chromite, silver minerals, and metallic phases. Minor nickel was found associated with metal oxides and stainless steel. No indication of fibrous materials. Particles such as illite, plagioclase, quartz, amphibole/pyroxene, chlorite, calcite and gypsum are mainly from natural origin, while iron oxides/hydroxides, chromite, silver minerals and nickel come from human activities such as industrial processes, especially the ferronickel smelter plant which is situated in this area.

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