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IRON HYDROXIDES OCCURRENCE IN WINTER AIR PARTICULATE MATTERS SUSPENSIONS IN CLUJ – NAPOCA, ROMANIA

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Abstract: Our performed measurement on the air particulate matters level during 2011winter in Cluj – Napoca, on the Dambovitei Street air quality monitoring platform, shows significant values around of 4 g/m^2/month. The PM_{10} (Particulate Matters) fraction feature the average diameter of 9 µm and the $PM_{2.5}$ fraction feature an average diameter of 2µm. The performed mineralogical and crystallographic investigations shows large amount of quartz 50%, kaolinite 25%, lepidocrocite 20% and tridimite 5% in the collected PM. The tridimite and lepidocrocite is quite unusual for naturally formed PM. The enhanced analysis over the possible anthropogenic PM sources reveals the source of lepidocrocite and tridimite in the used nonskid material, via street dust. We found over 30 % ferro hydroxide in nonskid material. The respiratory test reveals that such PM interact with the nose mucosa presenting a potential health risk. We developed some magnetic separation strategy in order to diminish the iron hydroxides occurrence in nonskid material. We achieve a good extraction coefficient of ferro hydroxide particles around 23%. This result proves to be useful for developing of an ecological management of streets maintenances during winter.

Keywords: air PM, lepidocrocite, goethite, magnetic separation.

1. INTRODUCTION

The air quality is one of the most important environmental parameter monitored by the agencies over the whole world (Williams, 2004; Gidhagen et al., 2009; Gerboles et al., 2011). Particulate matters (PM) are one of the most important reference monitored parameter due to the inhalation risk. There appear a major risk of silicosis, lung cancer and even skin allergy at long exposure (Harris, 1999; Hartog et al., 2003; Polichetti et al., 2009).

The usual PM found in the urban areas is composed by various types of material particles ranging from the minerals, chemical compounds and even biologic residues. The most prevalent constituent of air PM are mineral compounds due to their proximity in the normal environment. The most usual PM mineral fractions are formed by quartz, calcite and clay particles (Ferguson & Ryan, 1984; Joshi et al., 2009) resulted from erosion of natural environment. Some other different minerals (e.g. portlandite, hematite, tridimite) along with chemical compounds are related to the industrial and anthropogenic activities (Joshi et al., 2009; Hosu et al., 2010).

The elemental measurements performed over many years prove that the anthropogenic built environment presents high amount of metals such Fe, Al and Pb in air PM (Ferguson et al., 1984; Joshi et al., 2009). Iron is one of the most representative due to its utilization in all built environment aspects.

The iron parts from buildings, metallic structure and car and lorry chasses are subjected to an intense weathering which is able to induce corrosion (Evans & Taylor 1979; Balasubramaniam et al., 1999). There are multi layered ferro – oxides and hydroxides on the rusted surfaces (Balasubramaniam, 2000), which are able to transform in different allotropic forms according to the environment conditions (Schwartzmann & Taylor, 1972; Cuddenec & Lecerf, 2005; Garcia et al., 2008). The most degraded state of the iron is lepidocrocite and goethite (α and β – iron hydroxide. They are often found as red brownish rust scale (Schwartzmann & Taylor, 1972; Garcia et al., 2008). The more intense rusting processes are related to the industrial activities and especially metallurgical industry (industrial plants and dumps) conducts to larger metals amount in PM range (Damian et al., 2010; Manasreh, 2010).

The iron hydroxide occurrence in air PM fraction it is an interesting problem to investigate. There could be traced some anthropogenic pollution sources. The aim of present article is to figure out the composition of the winter PM deposits in Cluj – Napoca in January – February 2011 and to observe the possible occurrence of iron hydroxides.

2. METHODS

The PM samples were collected in winter period over January and February 2011. The collecting procedures are according to the European Union standards, performed on an air monitoring platform managed by Regional Agency of Environment Protection Cluj Napoca. All samples were collected on the Dambovitei monitoring station. The first sample set was collected as weekly representative sample, WPM, (seven days of particle matter deposing) in January 2011 and the second sample set is monthly representative, MPM, (January – February 2011 – thirty days of particle matter deposing). WPM and MPM samples are average representative due to the collecting mode.

The street dust was collected by the Dambovitei Street nearby the monitoring platform from at least 10 different points. Equal quantities of dust from each collecting point was mixed onto an average representative dust sample noted ASD. The nonskid material samples were collected from 5 street tanks nearby the Dambovitei monitoring platform and mixed together into an average representative sample noted ANS.

The X-ray diffraction analyses was performed using a DRON 3 diffractometer equipped with data acquisition module and Matmec VI.0 software. The mineral identification from the resulted X - ray diffraction patterns was done using MATCH 1.0 X – ray diffraction database powered by Crystal Impact Company.

The morphology outline was investigated by dark field optical microscopy on a IOR 8 microscope. The quantitative measurement on the microphotographs was done using the Image J Processing soft.

The cross polarized light microphotographs were done on a mineralogical Karl Zeiss Jena mineralogical microscope Laboval 2. The digital capture used for all microscopy investigation was done with a Samsung camera 8 MPx.

3. RESULTS AND DISCUSSIONS

The eastern city main road (Aurel Vlaicu Ave.) and the eastern industrial pole are situated in the action range of the air parameter monitoring station from Dambovitei Street. The WPM and MPM samples were collected from this point.

The quantitative measurement performed on the WPM collected sample reveals 4.34 g/m^2/month and for the MPM collected sample results 4.55g/m^2/month. We observe that the PM atmospheric emission around Dambovitei Street is relatively constant during winter season. The value around 4g/m^2/month represents an average PM concentration in air according to the accepted limits (Mrkajik et al., 2010; Gerboles et al., 2011). The observed fact is unusual, due to the winter condition observed for many years in Cluj - Napoca featuring significant snow bed or frost soil crust (Tahas et al., 2011). The wet of frost road sides and adjacent surfaces covered with snow are sufficient hypothesis for decreasing of air PM level. Some of the most recent studies supports this hypothesis. (Vardoulakis & Kassomenos, 2008; Gerboles et al., 2011).

The atmospheric condition during sampling was typical for the winter season in Cluj – Napoca with significant snow bed and frost surfaces. The street surface was covered with nonskid material after a precise schedule to prevent traffic difficulties. In such good weather conditions, a question appears: why we have significant atmospheric PM? The answer could be reached out only by an intensive analysis of WPM and MPM collected samples. If we could find some specific mineral traces like iron hydroxides and iron compounds the pollution source could be found precisely.

PM_{10} and $PM_{2.5}$ are the most dangerous components of the particulate matter emissions due to their ability to penetrate onto the lungs in the respiratory process (Polichetti et al. 2009, Carvalho et al., 2011). Some of the recent studies reveals significant amount of PM_1 and even $PM_{0.5}$ in the street dust (Hosu et al., 2010). The particle size and shape of PM_{10} and $PM_{2.5}$ in WPM and MPM sample were investigated by optical dark field microscopy, results are presented in figure 1. The WPM sample morphology is observed in figure 1a. Refined particles appears over the sample surface presenting cohesion tendency. PM_{10} are observed having rounded shape.

The quantitative analysis performed with Image J Processing Soft reveals the average particle size for PM_{10} situated around of 9 µm. $PM_{2.5}$ particles feature small lamellar and tabular shapes distributed around the PM_{10} particles. The quantitative measurement performed with Image J soft procedure reveals the average diameter of $PM_{2.5}$ around 2 µm. The $PM_{10}/PM_{2.5}$ ratio is situated around of 50%.

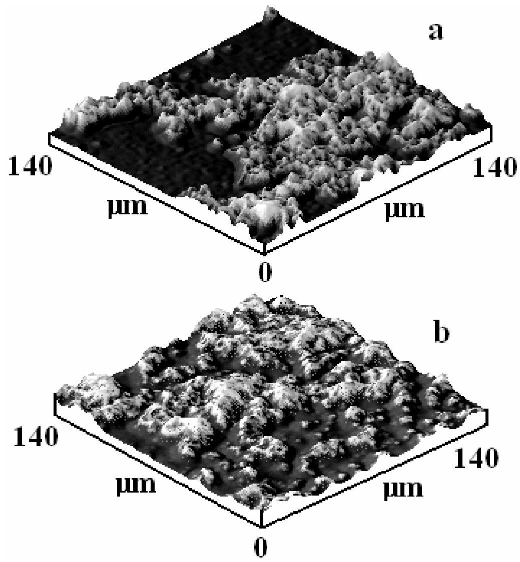

Figure 1. The PM morphology observed by dark field optical microscopy – 3d view: a) WPM and b) MPM.

The MPM sample morphology is presented in figure 1b. We observe a significant difference: the MPM particles are denser in the sample surface presenting a significant increased level of PM_{10} than in WPM. The PM_{10} particle shape is equiaxial – rounded having the average diameter around of 11 µm. The $PM_{2.5}$ particles are similar to the ones observed for WPM sample having the same sharp and the average diameter is situated around of 2 µm. The $PM_{10}/PM_{2.5}$ ratio is situated around of 70%.

All PM_{10} and $PM_{2.5}$ observed in WPM and MPM sample presents the proper shape and size to be subjected to the human inhalation. The fact could be a potential health risk as notice before. The potential risk is significant increased if there appear some chemical and biochemical active minerals instead of inert ones.

In both WPM and MPM samples were found greater microscopic particles having the average diameter around of 50 µm as observed in figure 4 at average and high optical transmitted light microscopy investigation. These particles are less dangerous due to their significant increases size which makes difficult the inhalation process.

The mineral composition of WPM sample was established by X-ray diffraction analysis. The resulted diffractogram is presented in figure 2a. It feature well developed diffraction peaks corresponding to the minerals found in the WPM sample: Quartz 50%, Kaolinite 25%, Lepidocrocite 20% and tridimite 5% (the mineral weight percentage is estimated according to the peaks relative intensities related to the mineral structure factor).

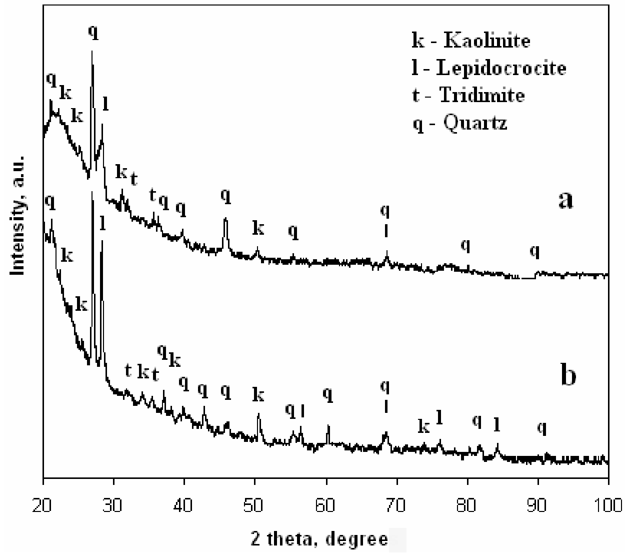

Figure 2. X-ray diffraction patterns for PM samples: a) WPM and b) MPM.

The optical cross polarized light microphotograph at average microscopy, (Fig. 3a), reveals larger quartz particles colored greenish gray having between 30 and 75 µm diameter, trydimite particles pale gray colored have around of 30 µm diameter. There also appear significant amount of lepidocrocite particles (red brown) placed nearby quartz particles. The observed lepidocrocite particles are under 20 µm.

The higher magnification of the cross polarized light microscopy performed on WPM sample, (Fig. 3b), reveals larger quartz and lepidocrocite particles having around 50 µm surrounded by PM_{10} and $PM_{2.5}$. There are observed fine quartz particles as well as lepidocrocite mixed with significant amount of kaolinite (bright white – bright blue color depending on particle orientation to the optical axis of the microscope).

The quartz and kaolinite particles are usual in PM fraction originating from natural sources (e.g. adjacent soil degradation). The lepidocrocite amounts in PM fraction is un usual. The MPM sample was also subjected to the X-ray diffraction, the resulted spectrum is presented in figure 2b. Well developed diffraction peaks are observed. They correspond to the MPM sample mineral composition: quartz 55%, lepidocrocite 25%, kaolinite 17%, and tridimite 3% (the mineral weight percentage is estimated according to the peaks relative intensities related to the mineral structure factor).

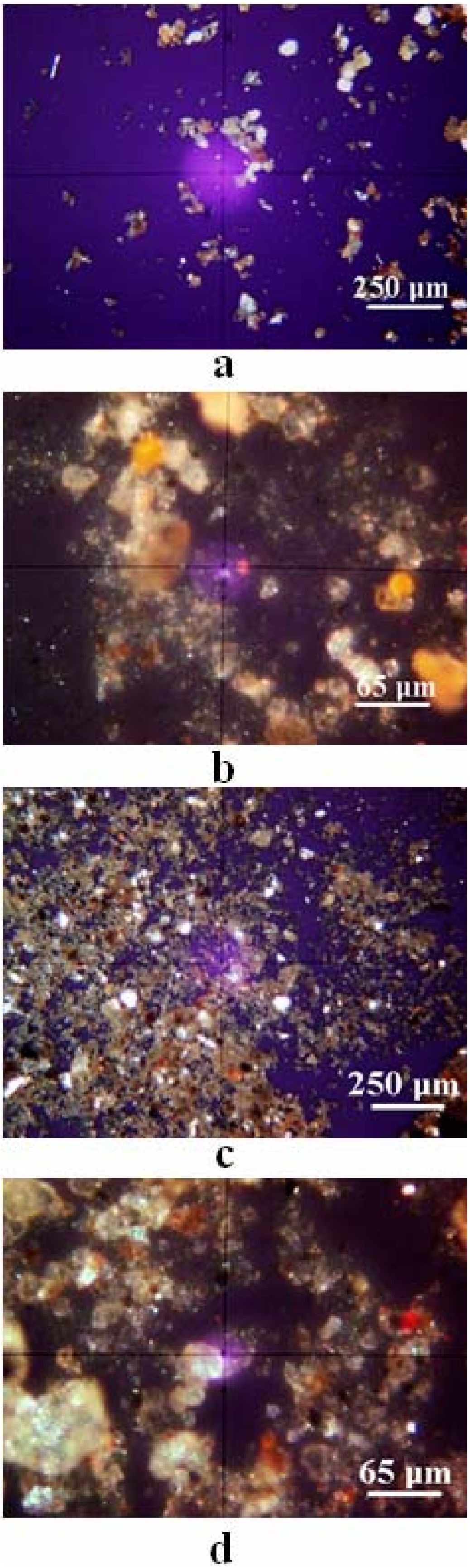

Figure 3. Optical cross polarized light microphotographs for: a) WPM average magnification, b) WPM high magnification, c) MPM average magnification, and b) MPM high magnification.

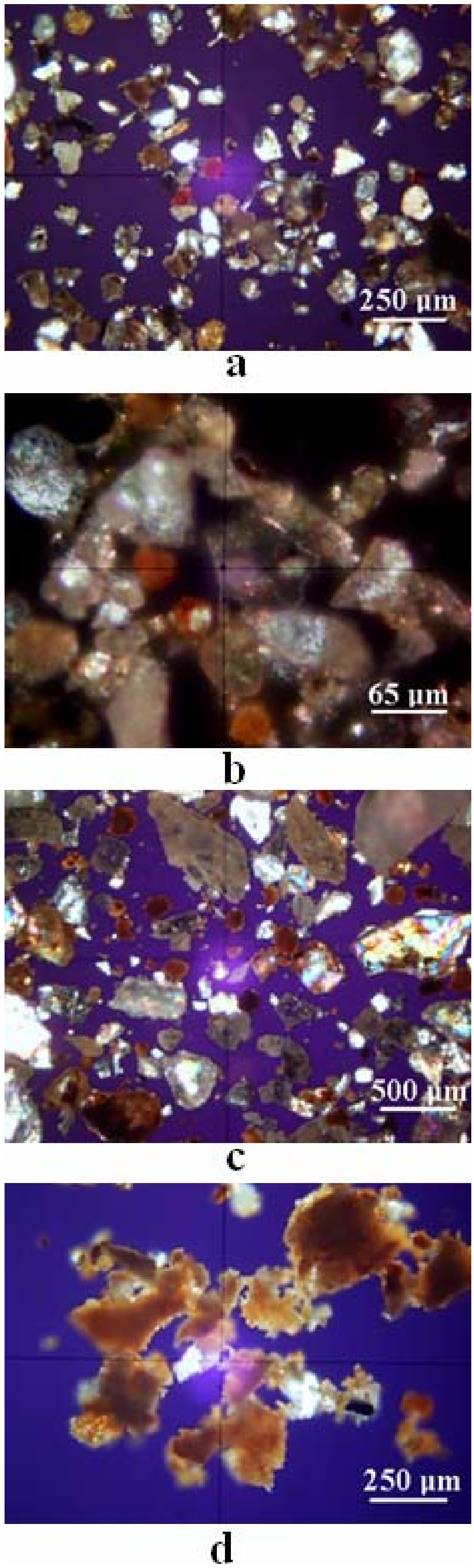

Figure 4. Optical cross polarized light microphotographs for: a) ASD average magnification, b) ASD high magnification, c) ANS low magnification, and b) FHP average magnification.

The MPM mineralogical composition is similar to the WPM sample. The fact prove the constancy of the PM emission on the Dambovitei street during winter season.

Of course, some differences appears at microscopic level of particle distribution. There are observed significant larger amount of greater micro particles between 25 µm and 75 µm, (Fig. 3c). There appear large amount of quartz particles (greenish gray) mixed with significant amount of lepidocrocite particles having under 20 µm (brown red). The explication of the observed distribution is due to the large microscopic accumulation over the long term of sampling (30 days).

The PM_{10} and $PM_{2.5}$ micro distribution in MPM sample is better observed in Figure 3d. The lepidocrocite fine particles are mixed together with small quartz slivers and kaolinite particles surrounding the greater particles (e.g. quartz, and traces of tridimite).

The revealed aspects shows a continuous accumulation of quartz, kaolinite, lepidocrocite and tridimite at almost constant rate of 4 g/m^2/month having large amount of PM_{10} and $PM_{2.5}$. Lepidocrocite is the most degraded state of the iron minerals – chemical formula α FeO(OH) (Schwartzmann & Taylor 1972; Garcia et al., 2008). The iron hydroxides are very dangerous if they are deposited at the respiratory pathways and lung alveoli causing acute inflammation (Harris, 1999; Hartog et al., 2003).

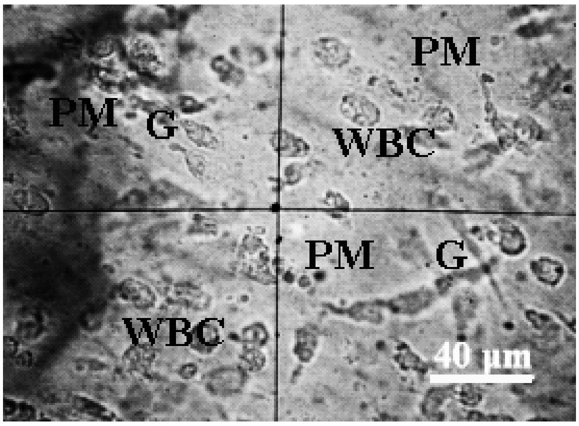

Figure 5. Optical transmitted light microphotograph of nose mucosa after 1 hour in vivo exposure to air PM from area of WPM and MPM sample collecting point.

The invasive ability of PM_{10} and $PM_{2.5}$ resulted from WPM and MPM was tested. One of the article authors, voluntarily was exposed for an hour to the air nearby the monitoring station on the Dambovitei Street walk side in condition of normal road traffic. After exposure a representative sample of in vivo exposed nose mucosa was collected. The micro structural result is presented in figure 5.

PM_{10} and $PM_{2.5}$ particles are observed as black dots surrounded by white blood cells (WBC) and some germs „G". It means that the presence of PM on the nose causes a body reaction. The observed behavior is similar with other reported before (Hartog et al., 2003, Polichetti et al., 2009). Considering this case, the iron hydroxide source in WPM and MPM must to be found in order to establish some diminishing strategies.

The lepidocrocite and tridimite occurrence in observed PM distribution make us to think that there appears an industrial pollution due to the proximity of ceramic and metallurgic factories, very similar with similar cases related to the metallurgical industry (Damian et al., 2010). This is a promising hypothesis.

However, giving a better look on the overview situation: the ceramic factory has improve the particle filters according to the EU regulations and the metallurgical plant has closed the smelters and casting sections (there are no ferro – particles emissions). So, still we have significant PM overflow. In that case we look closer to the air monitoring station in order to chase the iron compound potential source.

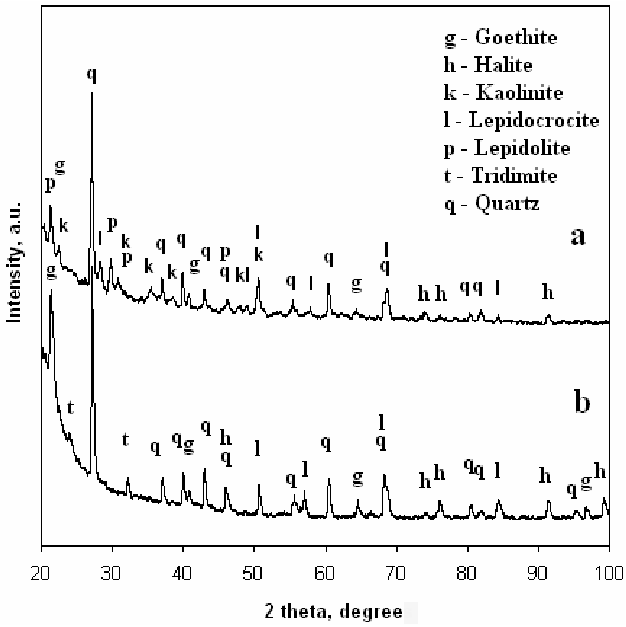

Figure 6. X-ray diffraction patterns for potential PM sources: a) ASD collected from Dambovitei Street, and b) ANS.

The closer PM source in the urban area is the street dust. We collect a representative street dust sample „ASD" from Dambovitei Street, nearby the monitoring station and we tested it as follows.

The X –ray diffraction pattern, (Fig. 6a), feature well developed peaks proving the crystalline state of the dust sample. We found a multi crystal mixture containing large amount of quartz, clay minerals (kaolinite and lepidolite), and iron hydroxides (goethite and lepidocrocite). Quartz particles are the most representative in the ASD sample around of 50%, the other minerals are approximate equally distributed in the sample mass. All these minerals could be explained by natural sources or anthropogenic sources (e.g. industrial PM or traffic made emissions). The „surprise" in figure 6a is the appearance of halite traces (NaCl). The halite peaks intensity is very low, close to the diffractometer sensitivity limit. The halite traces do not affect the mineral distribution but it is very odd since no halite deposit is reported on the Dambovitei Street area.

The optical cross polarized investigation at average magnification of ASD sample, (Fig. 4a), reveal large micro structured quartz particles (greenish gray color) around of 75 μm diameter. There are also featured iron hydroxide particles having around of 50 μm colored brown red. At high magnification (Fig. 4b), these particles could be better observed. The clay particles are very fine (maximum 5 μm) and distributed discretely around the bigger particles. In figure 4b could be observed such clay particles: kaolinite as bright white – blue dots and lepidolite particles feature pink hued dots.

It is obvious that clay particles belongs to the adjacent soil as well as finest quartz particles (under 20 μm diameter), but the iron hydroxide particles are not usual from the soil. These particles, related to the halite traces make us to think that the iron hydroxide contamination of the roadside could belong to the used nonskid material. The halite is very often used in nonskid material to lower the freezing point of the water, respectively to avoid the street frost.

Such material uses macroscopically granulated quartz particles in the range of 5 – 15 mm. The intense road traffic could break the particles in the nonskid material resulting some microscopically fractions. This hypothesis verification was performed on a representative nonskid material sample grinded into an agate mortar „ANS".

The X-ray diffraction pattern resulted for ANS sample is presented in figure 6b. It features very well developed diffraction peaks which corresponds to the high crystalline state of the sample. The minerals found are: Quartz 50%, goethite 15%, lepidocrocite 15%, tridimite 10% and halite 10% (the mineral weight percentage is estimated according to the peaks relative intensities related to the mineral structure factor). The mineralogical composition of ANS sample could be also observed in microphotograph in figure 4c. There are observed quartz and iron hydroxide particles, very similar to those observed in ASD sample. This microstructure prove that the grosser quartz and iron hydroxide particles in ASD derives from the nonskid material.

Goethite and lepidocrocite represents the same chemical substance FeO(OH) crystallized in two different systems due to the different number of water molecules bonded in the crystal lattice (Cudennec & Lecerf 2005). Lepidocrocite is more degraded than goethite due to the larger water amount bonded in crystal structure. Of course it is more brittle and presents the breaking tendency into fine micro particles, similar to such observed in WPM and MPM samples. Goethite is more coherent than lepidocrocite and it is found in intermediary rusted layers (Balasubramaniam, 2000).

The most plausible explanation for coexistence of lepidocrocite and goethite in the same micro structured particles is the dynamic formation in the traffic conditions. The rust over the cars and lorry protection shield is intensive ablated by the nonskid material particles in the winter traffic. The outer most layer of the car shield rust is formed by lepidocrocite meanwhile the inside layer contains more goethite. Such rust layer become particles in the street dust along with quartz and the other mineral particles. The cohesion tendency in wet condition is for the growing of rust particles by sticking each other capping some quartz sliver traces. Recycling the used nonskid material conducts to a contamination with ferro hydroxides particles of the new nonskid material. Such contaminants are brittle. They are easy broken in traffic condition and are easy dispersed in adjacent air as PM emissions.

Some research reveals that the ferro minerals could be separated from the other ones (e.g. clays) via magnetic separation (Schultze & Dixon 1979). In this case, we subject the ANS sample to a classic magnetic separation in order to remove the ferro hydroxide particles from the nonskid material. We assure a uniform mono-granular layer on the magnetic separator belt to assure a better separation. The quantitative gravimetric determinations prove that 23% wt. of ANS sample are ferro hydroxide particles FHP, the rest are the magnetic separation refuse. The removing of FHP is a good treatment for the lepidocrocite amount diminishing in the PM emissions.

The X-ray diffraction pattern presented in figure 7a reveals the mineralogical composition of FHP particles. There appear equal quantities of lepidocrocite and goethite along with significant quantities of quartz. The result confirms the FHP particles formation hypothesis.

The FHP microstructure, (Fig. 4d), presents aggregates having an average diameter of 200 μm. These aggregates contain fine particles of lepidocrocite, goethite and quartz slivers (5 – 20 μm). The aggregates color is light red – brown due to fine dispersion of the quartz slivers. Such particles do not present industrial interest, but they could be further processed via advanced magnetic separation to obtain industrial pure iron hydroxide.

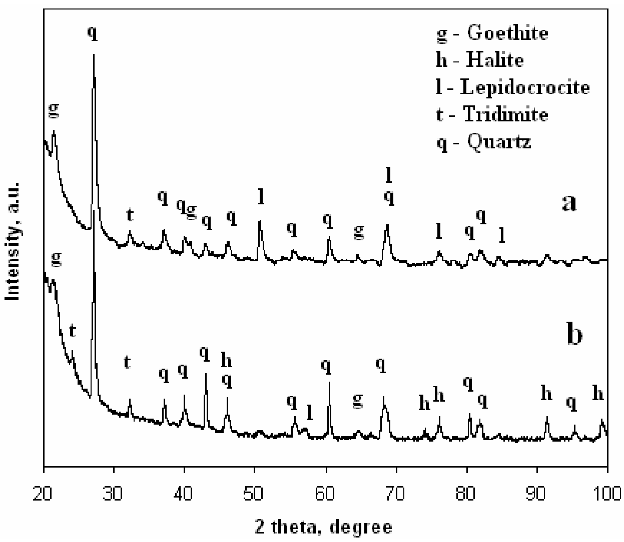

Figure 7. X-ray diffraction patterns for magnetic separated samples: a) the resulted FHP particles and b) the magnetic separation refuse.

On the other hand, magnetic separation refuse feature a very low content of ferro hydroxide (almost traces) as results from the X-ray diffraction analysis, (Fig. 7b). It proves that the ASD decontamination was successfully achieved.

The removal of complex FHP conglomerates reduces significant the risk of PM emission due to the traffic. It could be a fruitful strategy for the considerably reducing the iron hydroxides emissions in air PM along with sensitive diminishing of quartz PM emissions.

4. CONCLUSIONS

The PM monitoring is one of the most important air parameter in European and world wide air regulations. Our performed measurement on the air PM level during 2011winter in Cluj – Napoca shows significant increased values. The air PM feature an almost constant rate of 4 g/m^2/month for both weekly and monthly representative sample. It proves to be a winter season characteristic for Dambovitei Street collecting point. The PM_{10} fraction feature the average diameter of 9 μm and the $PM_{2.5}$ fraction feature an average diameter of 2 μm.

The performed mineralogical and crystallographic investigations shows large amount of quartz 50%, kaolinite 25%, lepidocrocite 20%, and tridimite 5%, in the collected PM. The tridimite and lepidocrocite is quite unusual for naturally formed PM.

The enhanced analysis over the possible anthropogenic PM sources reveals the source of lepidocrocite and tridimite in the used nonskid material via street dust. We found over 15% lepidocrocite and 15% goethite in the used nonskid material representing over 30% ferro hydroxide particles. The nonskid material also contains large amount of quartz 50%, and tridimite 10%, macro particles (5 - 15 mm) along with halite (NaCl) particles 10 %.

The most plausible explanation for coexistence of lepidocrocite and goethite in the same micro structured particles is the dynamic formation in the traffic conditions. The rust over the cars and lorry protection shield is intensive ablated by the nonskid material particles in the winter traffic.

We observe that the cohesion tendency in wet condition is for the growing of rust particles by sticking each other capping some quartz sliver traces. Recycling the used nonskid material conducts to a contamination with ferro hydroxides particles of the new nonskid material. Such contaminants are brittle. They are easy broken in traffic condition and are easy dispersed in adjacent air as PM emissions.

The respiratory test reveals that such PM (e.g. lepidocrocite, fine quartz or tridimite slivers) interact with nose mucosa presenting a potential health risk.

We developed some magnetic separation strategy in order to diminish the iron hydroxides occurrence in nonskid material. We achieve a good extraction coefficient of 23% wt. removal of ferro hydroxide particles from the nonskid material. This result proves to be useful for developing of an ecological management of streets maintenances during winter.

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