Determination of the chemical composition of waste dust from process operations – case study

Niculina Sonia Șuvar[1*], *Irina* Nălboc[1], *Maria* Prodan[1], and *Andrei* Szollosi-Moța[1]

[1] National Institute for Research and Development in Mine Safety and Protection to Explosion – INSEMEX, Department for Safety of Mineral Resources, 32-34 Gen. Vasile Milea Street, Petroșani, Romania

Abstract. The main environmental wastes generated by the automotive industry include lubricants and coolants for cars; water and solvent cleaning systems; paint; scrap metal and plastics. It is important to establish the source of the waste release to establish appropriate organizational measures to ensure the correct environmental approach and safety issues. In the present work, several tests have been carried out to identify the source of release for a specific dust waste that appears in the process of car's interior finishing. XRF, ICP-OES, and FTIR analyses were performed for this purpose.

1 Introduction

Waste management is one of the most important issues in the world. This is due to the large amount of waste that is generated daily, its impact being dangerous for the environment and living beings.

With the rapid growth of vehicle volume, there is a need for waste management by governments around the world for the reuse and recycling of waste materials such as metal, solvents, batteries, plastics, etc. [1]. The automotive industry continues to grow, consuming larger amounts of resources for production, but also generating a lot of waste during the end-of-life stage of a product's life cycle [2].

The issue of industrial waste management is one of the biggest challenges for all societies, especially in developing countries where the waste management process includes various stages: production of different wastes, collection, storage, transportation, and processing [3]

In order to reduce the negative effects on the environment and to increase the quality of life, economic agents in the automotive industry have the obligation to ensure the proper management of hazardous waste. In addition, this aims to address environmental concerns and solve the problem of depletion of natural resources [4].

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* Corresponding author: sonia.suvar@insemex.ro

The amount of waste we generate is closely linked to our consumption and production patterns. The very large number of products entering the market is another challenge. Demographic changes, such as an increase in the number of single-person households, also affect the amount of waste we generate (for example, the packaging of products in smaller packages).

The broad spectrum of waste types and complex waste treatment routes (including illegal waste) makes it difficult to get a complete picture of what waste is generated and where it is located. Data exists, albeit of variable quality, for all waste types [5].

From a health point of view, industrial dust is a dispersed solid aerosol, created by human activity, during the mechanical processing of solid materials [6]. The size of the dust particle is between 1 and 100 μm. Particles larger than 30 μm are referred to as coarse dust and they sediment quickly. The particle size is determined by their actual or aerodynamic diameter. Particles smaller than 10 μm predominate in contaminated air, while particles with a size under 1 μm settle very slowly or almost do not settle at all, as their size converges to 0.1 μm. In general, all types of industrial powders are harmful to humans and can cause serious health problems.

The paper aims to determine the chemical composition of the residual powders originating from different specific processes in the automotive industry, aiming at the identification of their release source. The dust samples, collected from different locations inside the production hall, were analyzed using modern analytical methods (Fourier Transform Infrared Spectrometry – FTIR, the inductively coupled plasma – optical emission spectrometry – ICP-OES, and the X-Ray spectrometry – XRF). The IR spectra for each of the five powder sample was compared with the software spectral database, and a probability (match) was obtained. In case of identification of a silicon dioxide peak, the samples will be analyzed by x-ray spectrometry (to determine the approximate silicon dioxide content) and ICP-OES (to determine the metal content) respectively.

2 Materials and methods

The determination of the chemical composition of the residual powders to identify the source of release was carried out for 5 dust samples (Kiefel powder - sample #1, "Bridge crane powder" - sample #2, "Laser drill filter powder" - sample #3, "Ultimod powder" - sample #4, and "Dryer granules powder" - sample #5).

Nicolet IS 50 FTIR Spectrometer (fig. 1) was used to identify the chemicals in the samples. Analysis for the identification of metals and metal oxides was possible using Perkin Elmer ICP-OES (fig. 2), HITACHI XMET 8000 Expert GEO (fig. 3).

2.1 Instrumentation

Infrared absorption spectra were recorded at a resolution of 4 cm^{-1} using the Attenuated Total Reflectance (ATR) analysis technique, thus:

- ATR analysis was performed directly on the samples.
- For each spectrum, 32 scans were performed.
- The experimental data obtained were evaluated using OMNIC software (Thermo Nicolet Corporation).

Fig. 1. Nicolet iS 50 FT-IR Spectrometer, with Built-in mid- and far-IR capable diamond ATR

The Inductively Coupled Plasma - Optical Emission Spectroscopy (ICP-OES) equipment is used to determine the concentrations of Cadmium, Cobalt, Chromium, Copper, Iron, Manganese, Molybdenum, Nickel, Lead, Zinc, Beryllium, Bismuth, Arsenic, Calcium, Lithium, Magnesium, Sodium, Strontium, Stybium, Titanium, Vanadium, Selenium, Thallium, in liquid and solid samples, using the digester. The principle of the method is the measurement of atomic emission by optical spectrometry. Inductively coupled plasma mass spectrometry uses liquid samples which, after the preparation process, are transformed into aerosols and introduced into the plasma. The substance of interest in (or passed in) the liquid state is nebulized, and the resulting aerosol is carried by a gas stream (Argon) to the plasma torch [7, 8].

a   b

Fig. 2. a. Inductively coupled plasma optical emission spectrometer ICP-OES Perkin Elmer: b.Microwave oven for solid sample digestion

The X-ray Spectrometry method is widely used for the qualitative and quantitative determination of the elemental chemical composition of a sample, especially for inorganic substances (minerals, ceramics, metals, soils, etc.) - mainly in industry. This is based on the fact that the wavelength of X-radiation is of the same order of magnitude as the distances between the nodes of crystal lattices (atomic, metallic, ionic, or molecular) and, as a

consequence of diffraction and interference, peaks in the intensity of emerging rays occur at certain angles.

In X-ray Fluorescence (XRF) the following processes take place successively: the inner electrons of the sample atoms are expelled due to collisions with the X photons of the primary X-ray source; the electrons in the outer layers occupy the vacancies on the lower layers (K, L, M); as a result of the transitions that take place, energy quanta in the X-ray domain are released and leave the sample in all directions. The energy of the X-ray source is between 5 and 100keV. Primary X-rays, as the radiation from the source (often an X-ray tube) is called, are directed towards the sample under analysis. Characteristic X-rays - specific to the elements of the sample and re-emitted by the sample - leave the sample, containing information that allows the chemical composition of the material (solid or solution) to be determined. The X-ray tube operates by accelerating the electrons emitted by the cathode in a high-voltage field (and also in vacuum). These electrons strike the anticathode (often the anode of the tube) and generate anticathode-specific X-rays inside the tube. Depending on the nature of the sample, a suitable anticathode is used - made of a metal not contained in the sample (Cu, Fe, Co, Ni, Cr, Mo, Ag, Rh, etc.). Primary X-rays are emitted in all directions but only those coming towards the window leave the tube, made of beryllium - an element with a metallic character, sufficiently rigid, with a low order number, and therefore, at low thickness, it is practically transparent to X-rays. The primary X-rays leaving the X-ray tube are then directed onto the sample to be analyzed. This will emit the so-called *fluorescence radiation* - characteristic of the elements in the sample - and is accompanied by a *continuous radiation* which has a broad spectrum due to the interaction of electrons with the electric field of the nucleus.

Fig. 3. HITACHI XMET 8000 Expert GEO Spectrometer

3   Results and discussions

3.1 Results obtained by infrared spectrometry in the range 4000 - 400 cm^{-1}.

Similar IR spectra were obtained when analyzing residual powder sample #1, sample #2, and sample #3, suggesting that the three powders originate from the same source (Fig. 4). Characteristic peaks for silicon dioxide at 1100 cm^{-1} were identified (Fig. 5). By comparing

the IR spectra with the database, a probability (match) of the spectra with that of the cement was obtained, probably due to the high concentration of silicon dioxide.
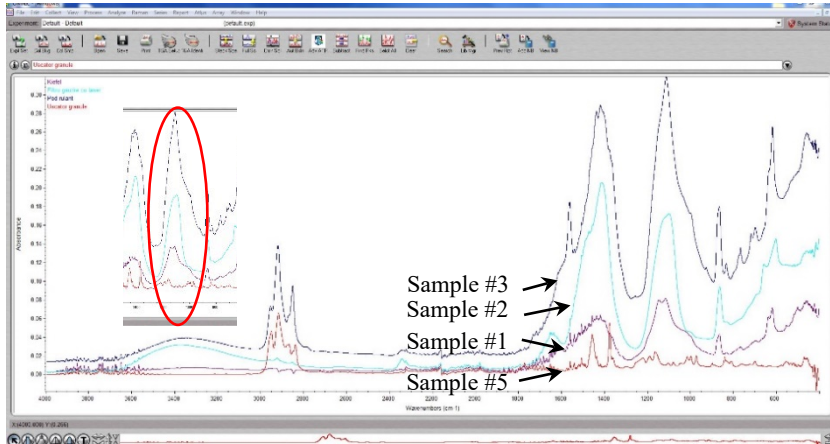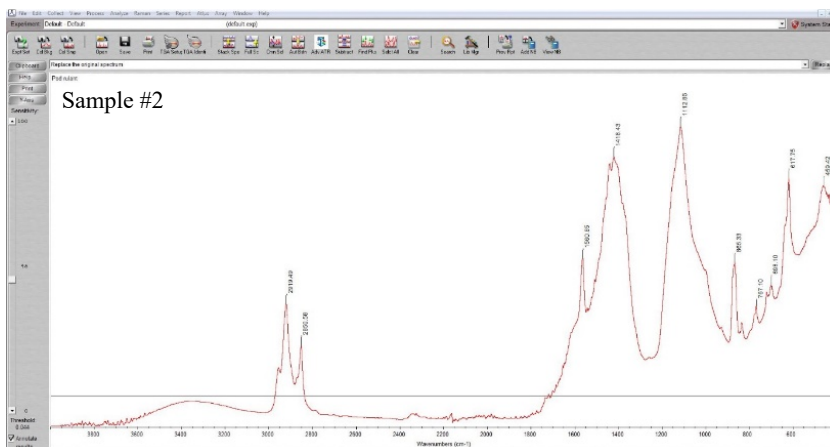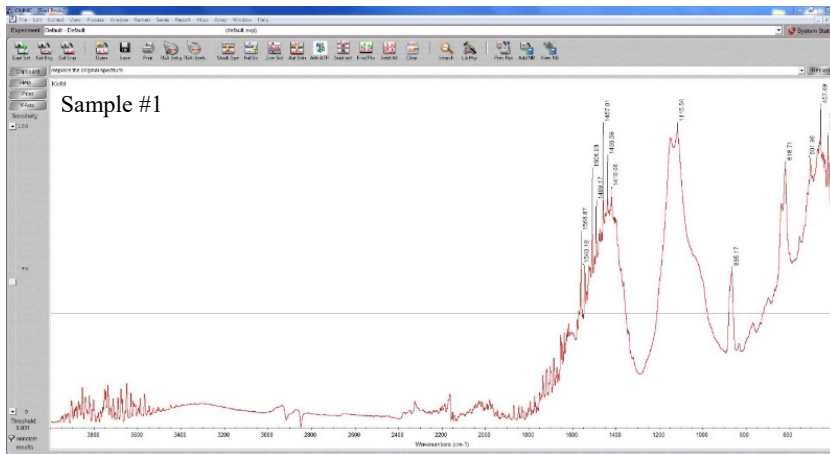

Fig. 4. IR spectra for powders from Sample #1, Sample#2, Sample #3, and Sample #5, stacked in the same chart
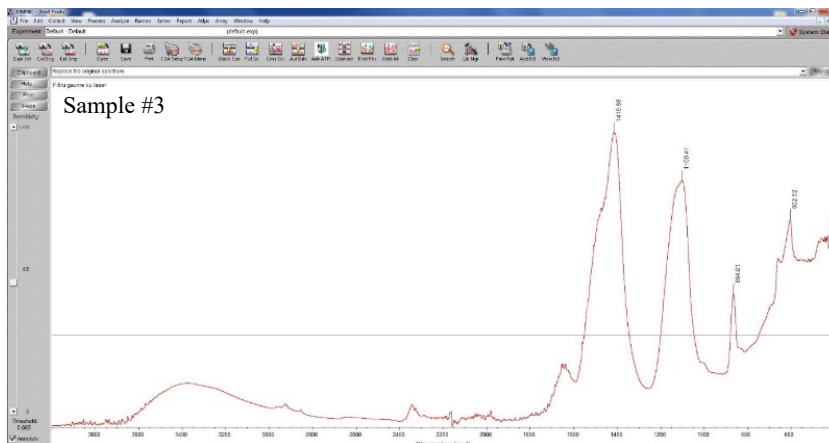
Fig. 5. Characteristic SiO_2 peaks, for the first three powder samples

No characteristic silicon dioxide peak at 1100 cm^{-1} was identified in the analysis of sample #5 (Fig. 6). By comparing its IR spectrum with the database, a spectral match of approx. 90% with that of polypropylene - PP) was obtained (Fig. 7).
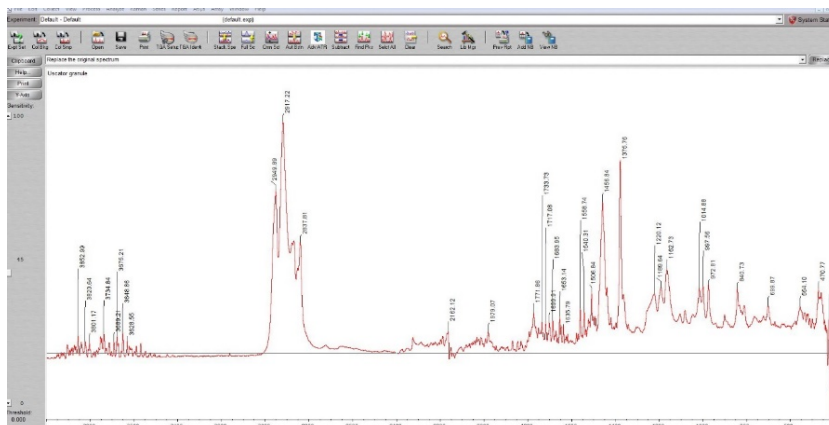

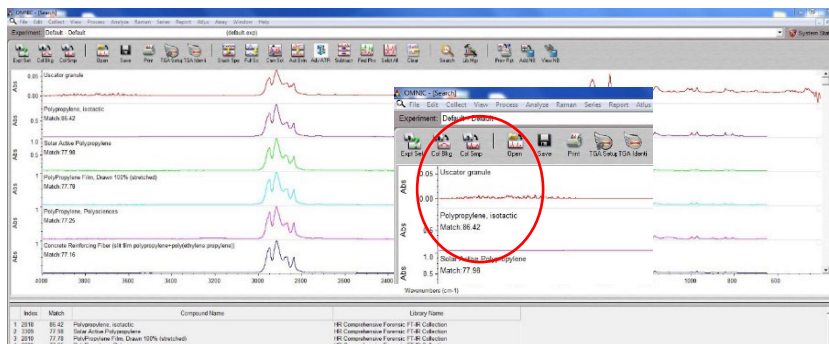**Fig. 6.** Characteristic peaks for Sample #5, "Dryer Granules powder"

Fig.7. Sample #5 identification

Analysis of Sample #4, identified a peak characteristic of silicon dioxide at 1100 cm^{-1} (Fig. 8). By comparing its IR spectrum with the database, a spectral match of approx. 90% with that of silicon dioxide was obtained. (Fig. 9).
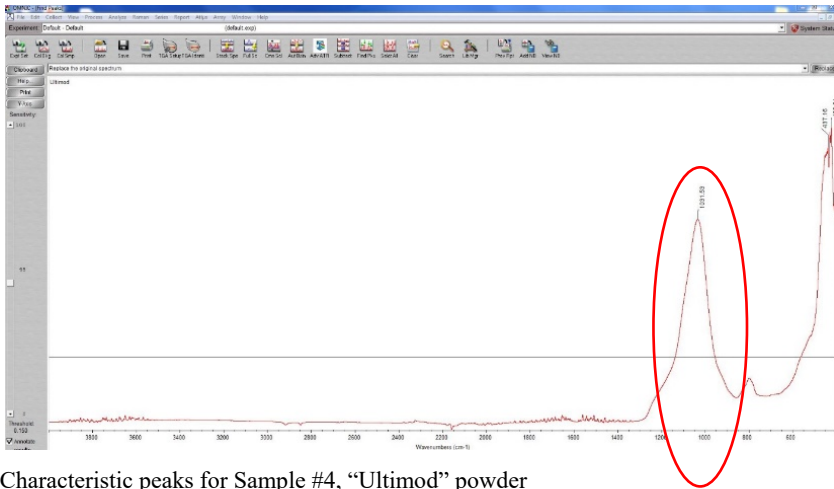

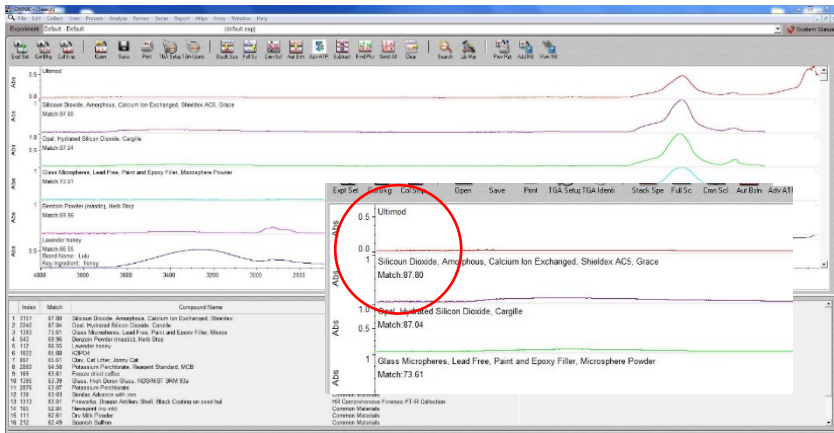**Fig. 8.** Characteristic peaks for Sample #4, "Ultimod" powder

Fig. 9. Sample #4 identification

Given the identification of the peak for silicon dioxide, as can be seen in table 1, all 5 samples were further analyzed for metal content by two other methods, namely X-ray spectrometry for the determination of the approximate silicon dioxide content, and inductively coupled plasma-optical emission spectrometry for the determination of metal content.

Table 1. Results obtained with FTIR method

Identification	Match results
Sample 1	Cement (silicon dioxide peak)
Sample 2	Cement (silicon dioxide peak)
Sample 3	Cement (silicon dioxide peak)
Sample 4	Silicon dioxide
Sample 5	Polypropylene

3.2 Results obtained by the inductively coupled plasma - optical emission spectrometry method (Table 2).

Table 2. Concentrations of metals identified in the five samples.

Analyzed Item	Sample #1	Sample #2	Sample #3	Sample #4	Sample #5
Calcium	4.73 %	4.96 %	8.19 %	0.38 %	989 ppm
Iron	2.83 %	7.23 %	5.63 %	2.91 %	849 ppm
Magnesium	0.5 %	0.5 %	1.36 %	3.39 %	294 ppm
Titanium	0.14 %	0.01 %	9.9 ppm	0.14 %	7.5 ppm
Aluminum	0.74 %	0.73 %	0.13 %	2.91 %	557 ppm

3.3 Results obtained by the X-Ray spectrometry method (Table 3).

Table 3. Concentrations of oxides identified in the five samples.

Analyzed Item	Sample #1	Sample #2	Sample #3	Sample #4	Sample #5
Silicon Dioxide	4.44 %	7.62 %	2.97 %	69.2 %	20.62 %
Calcium Oxide	11.03 %	15.36 %	29.37 %	1.48 %	-
Iron Oxide	8.49 %	23.22 %	15.8 %	10.2 %	-
Magnesium Oxide	0.86 %	1.47 %	2.37 %	1.38 %	-
Titanium Oxide	0.16 %	0.32 %	-	1.55 %	-
Aluminum Oxide	0.70 %	1.57 %	0.31 %	9.75 %	-

The obtained results suggest that the resulting powders come from the use of the substance "ULTimod 01 - Precoating powder" for the first three samples, namely Kiefel, Laser drill filter, and Bridge crane. In the Dryer Granules test (sample #5), the major component is polypropylene. The chemical analysis shows that it also contains silicon dioxide, the major component of ULTimod 01, so it can be concluded that sample #5 can come from both the use of ULTimod 01 and another substance containing polypropylene, possibly the substance "PP-Compound + 20% Mineral, ME266U".

4. Conclusion

5 samples were analyzed namely Kiefel powder, Laser drill filter powder, Bridge crane powder, Ultimod powder, and Dryer Granules powder.

- Evaluating the results obtained from the analyzes and the technical data sheets of the products used, the analyzed powders from Samples 1 to 3 represent micronic powders (with time deposition) resulting from the use of the compound ULTimod 01 - Precoating powder.
- Sample #5 (Dryer Granules powder) may come from the use of the substance ULTimod 01 due to the content of silicon dioxide but most likely comes from a substance containing polypropylene, respectively possibly the substance PP-Compound + 20% Mineral, ME266U.
- For Sample #4, the characteristic peak of silicon dioxide at 1100 cm^{-1} was identified. By comparing its IR spectrum with the database, a spectrum match of approx. 90% with that of silicon dioxide was obtained. The identified

concentrations also fall within the concentration range indicated in the safety data sheet of ULTimod 01.

- The use of the three analytical techniques (Fourier transform infrared spectrometry technique, X-ray spectrometry method, and inductively coupled plasma optical emission spectrometry method), provided a versatile tool for identifying and quantifying the metals and oxides in the complex samples analyzed.

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