

## Characterizing Airborne Particulates in Steel Foundries: Using Scanning Electron Microscopy (SEM) Analysis.

Soraya Merzouki<sup>1</sup>, Mohamed Salah Benlatreche<sup>2</sup>, Amel Benbott<sup>3</sup>, Billel Smaani<sup>4</sup>, Ouafa Boukhemis<sup>5</sup>.

<sup>1</sup>Abdelhafid Boussouf University Center of Mila, Environmental and Structural Molecular Chemistry Research Unit (CHEMS), Department of Chemistry, Mentouri Brothers University, Constantine 1, 25000 Constantine (Algeria).

<sup>2</sup>Abdelhafid Boussouf University Center of Mila (Algeria).

<sup>3</sup> Laboratory of natural substances, biomolecules and biotechnological applications, Larbi Ben M'Hidi University, Oum El Bouaghi, Laboratory of Natural Sciences and Materials, Mila (Algeria).

<sup>4</sup>Abdelhafid Boussouf University Center of Mila (Algeria).

<sup>5</sup>Environmental and Structural Molecular Chemistry Research Unit (CHEMS), Department of Chemistry, Mentouri Brothers University, Constantine 1, 25000 Constantine, Biotechnology Research Center of Constantine (Algeria).

**\*Corresponding author**

**The Author's E-mail:** s.merzouki@centre-univ-mila.dz<sup>1</sup>, msbenlatreche@centre-univ-mila.dz<sup>2</sup>, malika1959@yahoo.fr<sup>3</sup>, billeg.smaani@gmail.com<sup>4</sup>, wafa2535@gmail.com<sup>5</sup>.

Received: 09/2023

Published: 12/2023

### Abstract:

The melting foundry work is challenging, as it is performed in an environment that features high temperatures and the dispersion of nanoparticles (NP) and ultrafine particles (UFP) with multiple chemical compositions, which can significantly impact human health. Hence, it is necessary to study the particle size distribution and chemical composition to assess professional exposure and understand their potential health effects.

This study aims at characterizing the airborne particles at the metallurgical workplace environment. Accordingly, short-term samples were collected from areas and examined using scanning electron microscopy (SEM) coupled with energy dispersive spectroscopy (EDS) to define particle morphology and their chemical composition.

This analysis was conducted to explore the extent of their spread according to height and distance from the source of the furnace. Thus, we collected various-height samples.

Consequently, the spatial mapping revealed high concentrations of particles near the furnaces and plumes of particles rising in the stairwells and moving toward other work areas. The SEM/EDS

results confirmed the high number of nanoparticles measured, indicating that the aerosols were rich in metals, including iron, aluminum, selenium, and zinc.

The survey's findings can be used to deduce appropriate new strategies to mitigate workers' exposure to airborne metals.

**Keywords:** Nanoparticles, Airborne, Ultrafine particles, Scanning Electron Microscope, Workspace healthy, Foundry, Energy-dispersive X-ray spectroscopy (EDS).

*Tob Regul Sci.*™ 2023;9(2):2647 - 2658

DOI: [doi.org/10.18001/TRS.9.2.173](https://doi.org/10.18001/TRS.9.2.173)

## 1. Introduction

The airborne nanoparticles' effects on human health have been a concern for decades. Indeed, epidemiological studies have linked airborne particles to increased mortality and incidence of cardiovascular [1-3], pulmonary [4, 5], and neurological [6].

Numerous workers are exposed to a range of particles present on a nanometric scale. In occupational hygiene, it is common to differentiate manufactured nanoparticles (NP) from ultrafine particles (UFP) from natural, human, or industrial sources. The NP and UFP toxicity has been extensively investigated by several authors [6-10]. Furthermore, studies show that an NPs are generally more toxic than the same chemical size [11, 12].

1-The foundries' dust nature,

2-The dust origin and its development within foundries,

3- The Airborne particles characterization,

4-The importance of the chemical composition of airborne particles,

5-The importance of knowing the size of airborne particles

This study considers a foundry that produces nodular and grey iron castings. Samples were collected throughout the work in various workshops where particles were collected during the metal melting, casting, and the sanding process.

Analytical methods usually include representative sampling protocols to define the worker's exposure [13, 14]. Conversely, this method uses scanning electron microscopy (SEM), and energy-dispersive X-ray spectroscopy (EDS).

## 2.Experimental:

### 2.1. Description of the foundry:

## Characterizing Airborne Particulates in Steel Foundries: Using Scanning Electron Microscopy (SEM) Analysis.

It involves a large workshop of 50 meters long and 90 meters wide, equipped with a furnace comprising fans for the dust removal.

The working area has been divided into three sections, namely:

1. **Zone 1:** Near the furnace and the plumbing machinery. It starts from the location of the furnace and goes up to 30 meters.
2. **Zone 2:** The central area, where the products exit and are treated after heat treatment, ends within 30 meters. The presence of the workers in this area is intense due to the work's requirements.
3. **Zone 3:** The exit zone, where the workers prepare to leave their workplace. This area is the closest to the gate.

Environmental samples were taken following the standard working conditions in the three different workshop areas.

One in the first zone (i.e., near the furnace), the next one near the continuous casting area in the (i.e., the central area) at about 10 m from the treatment zone, and third in the exit zone to investigate the presence of particles in the outdoor environment.

### 2.2.The Elemental Analysis

A Quanta 250 Scanning Electron Microscope was used to examine the samples in powder form. Moreover, to highlight the topography of the corrosion product studied, several high-resolution images were taken in different areas and at different magnifications, ranging from 2000 to 10000 times.

The following parameters have been selected for the analysis;

- Pressure mode: High vacuum ( $> 1.03 \cdot 10^{-4}$  Pascal)
- Primary electron beam: 15Kv
- Electron beam size (spot): 2.5 and 3
- Secondary electron detector: Everhart Thornley Detector ETD ()
- Working distance (WD): 8 mm
- Magnification: from 2000x to 100000x

### Sample Analysis by Energy Dispersion X-Ray (EDX)

The samples' morphology and the chemical composition are illustrated in the images resulting from the Scanning Electron Microscope (SEM) analysis coupled with the EDX.

### 3. Results and discussion

The Airborne PFU and NPs concentration data were collected during normal working hours (i.e., 8 to 4 p.m.) when furnace operation was intensive.

Accordingly, the analysis was based on two variables to study the dispersion of ultrafine particles and NPs resulting from the iron liquefaction and casting processes:

- Distribution according to the size of the dust particles
- Distribution according to the chemical composition of the dust

The different samples and the three workshop areas investigation revealed a high concentration of the metal particles near the first area of the kiln. This concentration is moderate near the second area, and relatively low near the third area. This initial study established a baseline for the distribution of the metal particle concentrations of the UFPs and NPs in the furnace, as shown in Figure 1 (a).

The element distribution is illustrated and will be very important in Figure 1(b) (treatment zone) with high concentrations by introducing processes that require other elements. Furthermore, a noticeable change in the concentration of some metallic elements is confirmed in Figure 1(b).

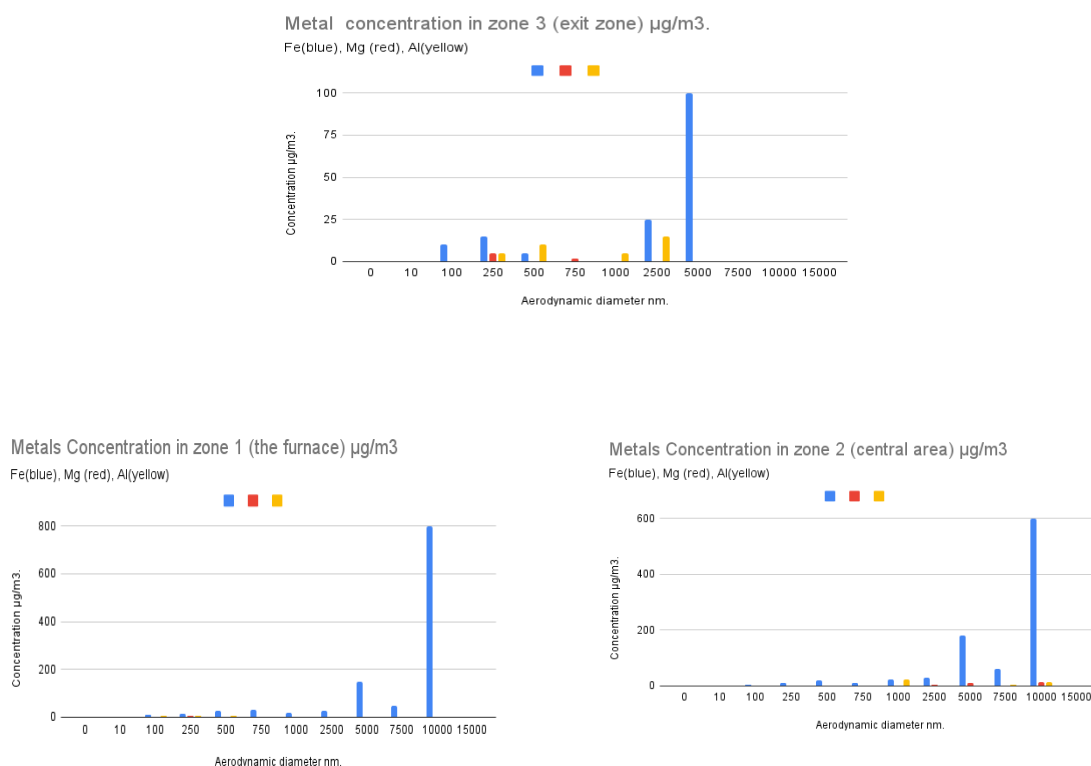


Figure 1: Distribution of the most important metal elements used in the foundry industry by zone.

Regarding zone 3 (i.e., the exit zone) there is a decrease in the airborne density compared to zones 1 and 2, with a change in metal element concentration, as confirmed by Figure 1(c).

### 3.1. SEM examination

The distribution of the airborne particles in the form of glomerules of regular spherical size up to a diameter of 30  $\mu\text{m}$  can be observed from the first view in the SEM with a magnification of (a) x1200 and (b) x2000 is at 5kv. The presence of objects of irregular size is also visible. It is noteworthy to state that this research only considers the spherical objects.

An irregular distribution of PFU and NP from 1 nm to over 2500 nm in diameter is shown in Figure 2.

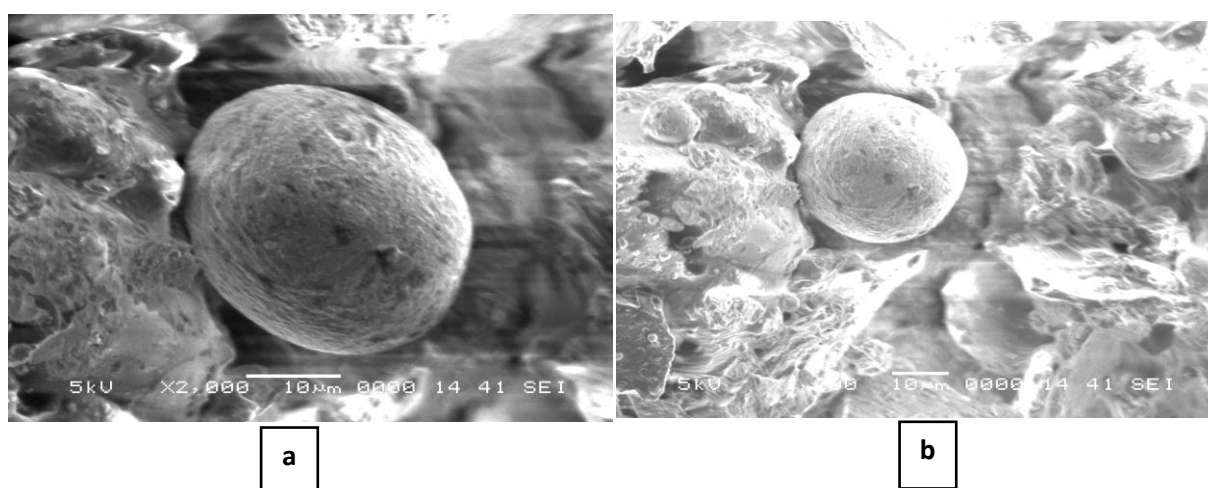


Figure 2: SEM images of the powder at different magnifications (a) x1200 and (b) x2000 at 5kv.

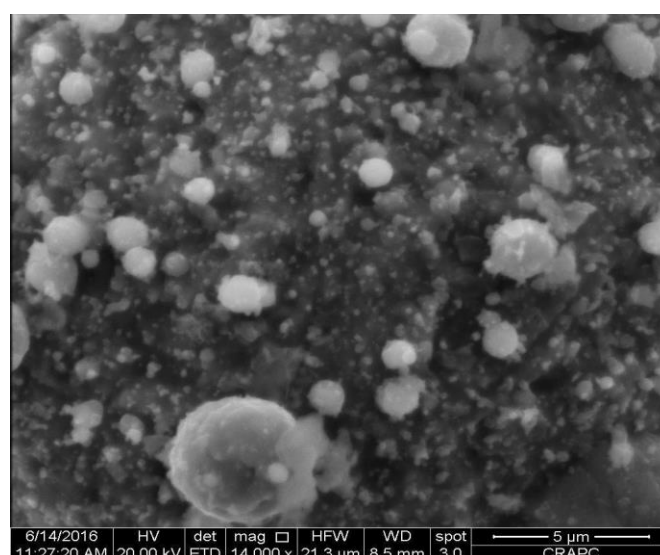
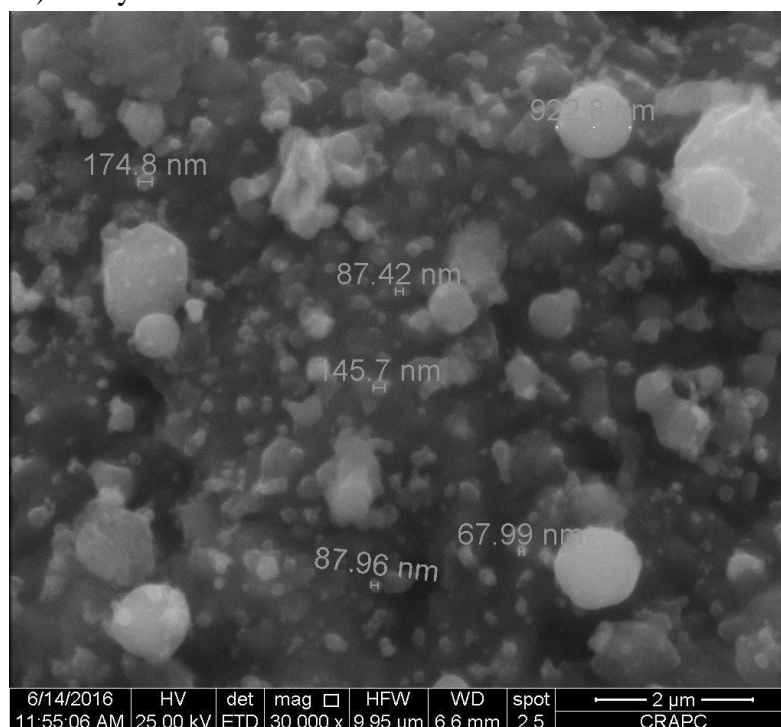


Figure 3: spherical particles of various sizes and diameters.



**Figure 4:** Morphology of metal powders observed by scanning electron microscopy in secondary electron mode are spherical particles and agglomerates of various sizes, ranging from 10 to 589 nm.

The images in Figures 3 and 4 are given at a low magnification, where spherical particles of various sizes and diameters can be seen. It is possible to distinguish show particles of different shapes, including micron-sized particles, which are the finest sand grains. Nevertheless, the most important thing to note, especially in Figure 4, are the large spherical particles with smaller particles deposited on their surfaces, which are sub-assemblies of nanoparticles.

The images demonstrate that in Figure 3 the small nanoparticles are homogeneous in contrast to the large ones that are clearly visible. Therefore, we can see some bodies adhering to their external face (Figure 3), such as the small nanoparticles. It is also evident that the NPs condense on each other to form aggregates of large-volume NPs or relatively large micro-NPs compared to the NPs.

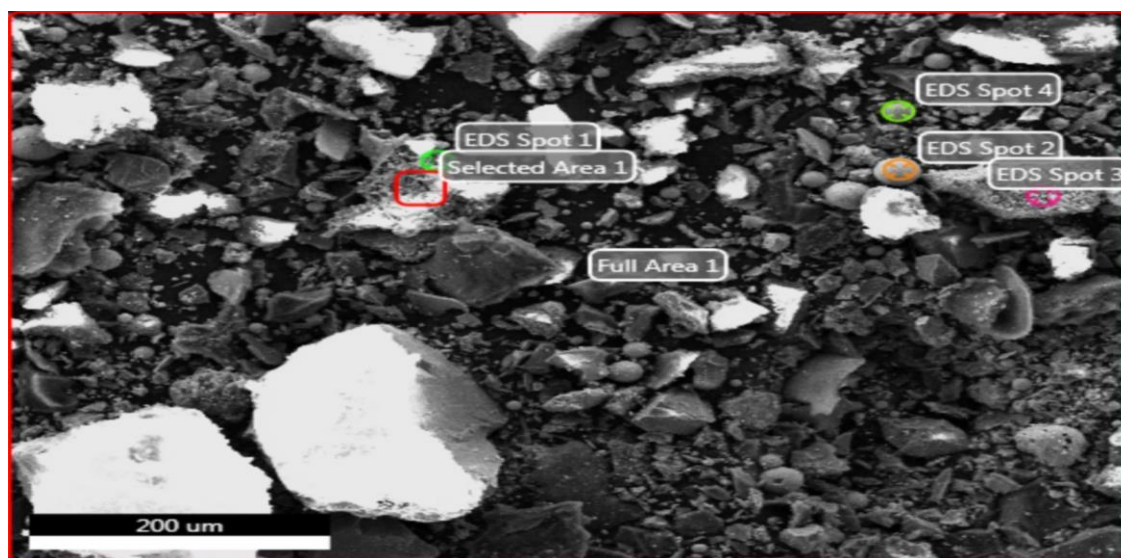


Figure 5: SEM-EDS image to obtain spectra in the selected areas (points)

A micro-analyzer (EDX), is installed in the scanning electron microscope. The latter collects the photons released by the primary electron beam. The x-ray detector can determine the photons' energy. In addition, data describing the chemical type of the atom is obtained by analyzing these beams. Figures 5-7 provide examples of particle collection.

The following observations can be made from the established analysis

Conversely, the EDX data of the nanoparticles below 150 nm (EDS Spot 4) highlights the concentration of chemical components in several micro areas, indicating only oxygen (O) and iron (Fe) atoms. With ratios of 56.15 for the oxygen and 43.85 for the iron. This finding demonstrates that these nanoparticles are NPs of  $\text{Fe}_2\text{O}_3$  iron oxide particles (Figure 5, Table 3).

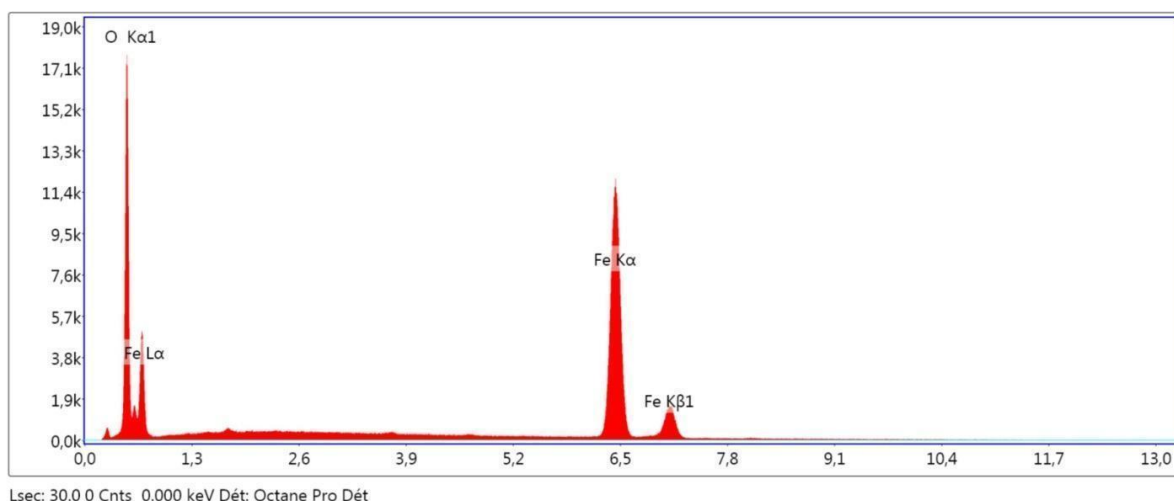


Figure 6: EDX spectrum of the powder on the EDS Spot4 area

Chemical element	mass rate	atomic mass rate
O K	26.84	56.15
Fe K	73.16	43.85

Table 2: Quantitative results for the EDS Spot4 areas

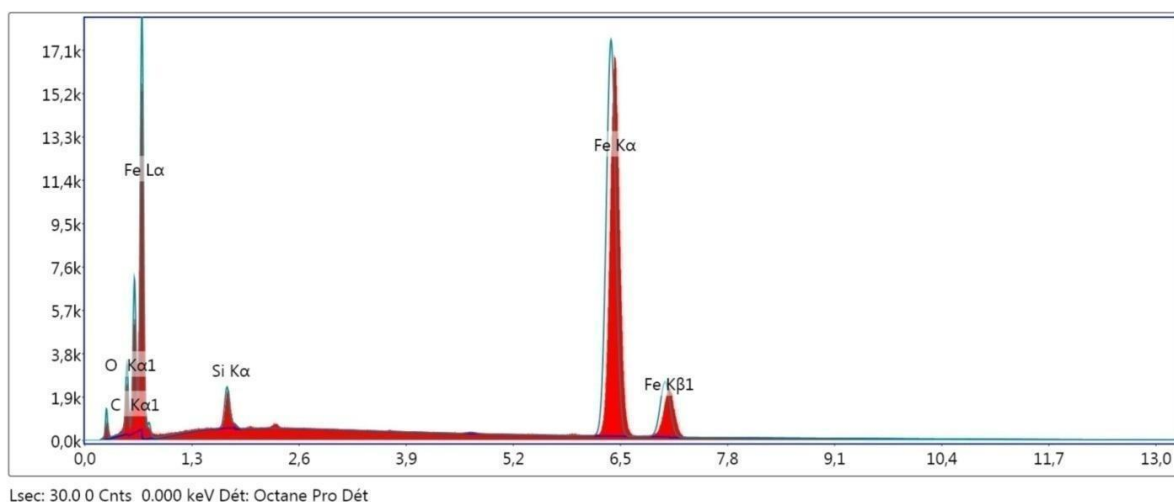


Figure 7: The EDX spectrum of the powder on the EDS Spot2 area.

Chemical element	mass rate	atomic mass rate
C K	5.84	20.26
O K	4.25	11.06
Si K	2.25	3.34
Fe K	87.65	65.34

Table 3: Quantitative results for the EDS Sport2 range of points of interest

The presence of non-metallic elements such as C and Si, with a significant rate of Fe and O in various percentages, is the most important observation that can be made in Table 3 of the Spot 2 illustrated in Figure 7.

### 3.2. Form, distribution, and degree of aggregation of the emitted particles



### Characterizing Airborne Particulates in Steel Foundries: Using Scanning Electron Microscopy (SEM) Analysis.

The size of the particles ranges from 27 to 240 nm (Figure 6 to 10). However, aggregates can be larger depending on the aggregation degree.

In line with our previous analysis, we can gather that when the NPs are small and of constant spherical size, their components are pure, comprising two elements, namely: iron, and oxygen. These element found in the proportions of 43.85% and 56.15%, for iron and oxygen, respectively (Figure 5 and 6 and Table 2). This confirms that they are iron oxide nanoparticles since they are widely distributed in area 4.

As mentioned earlier (Figures 3 and 7, Table 7), the nanoparticles above 147 nm in diameter are irregular and have several compounds attached to their external surface, which is coherent with the description of Natalia et al. [17].

This implies that nanoparticles start forming from a core of iron and oxygen ( $\text{Fe}_2\text{O}_3$ ). This core may be a single molecule combining various chemical components to form large nanoparticles of various chemical elements.

### 5- Conclusions:

The main purpose of the present research is to analyze the morphology and distribution of particulates in a steel workshop using electron microscopy and EDX.

The samples have been collected while melting and during intensive working. Accordingly, measurements considered the number of PFUs, morphology, and composition of metallic elements.

The results improved the knowledge regarding exposure to ultrafine particles and metal element fractions in nano-scale particles, which were in accordance with the previous studies conducted in similar industrial conditions [18-22]. Indeed, The study findings contribute to the characterization of occupational exposure to UFPs and may be useful to identify preventive measures to limit the workers' exposure.

Moreover, our results provide relevant information on the evolution of nanoparticles under UFPs, which confirms that a nanoparticle arises from an initial core consisting of iron oxide, then with the adhesion of several chemical elements to its surface, it begins to grow until reaching a diameter of a few hundred nanometers. This case has been verified by the presence of particles attached to the surface of the nanoparticles.

Similar NP collectors have been observed in factories performing MIG welding [22] and in particles collected in polluted air studies [23], indicating that they could be as common as aggregates of this type. Thus NP should be studied in detail, as they transport accidental nanoparticles on their surface [18, 21].

## Characterizing Airborne Particulates in Steel Foundries: Using Scanning Electron Microscopy (SEM) Analysis.

These results contribute to the characterization of occupational exposure to PFU chemicals and may be useful in identifying preventive measures to limit worker exposure. Furthermore, the study findings provide relevant information to improve knowledge of health effects associated with workplace exposure to UFPs. The description provided by this paper could help to better structure a workplace exposure matrix and plan an early biological response study.

### 6- References:

- [1] Robertson S, and Miller MR: Ambient air pollution and thrombosis. *Particle and Fibre Toxicology*, 15(2018).
- [2] Araujo JA, and Nel AE: Particulate matter and atherosclerosis: role of particle size, composition, and oxidative stress. *Particle and Fibre Toxicology* 6(2009).
- [3] BENLATRECH, Mohamed Salah, et al. Describing the Diffusion and Distribution of Airborne Nanoparticulates (NPs) and Ultrafine Particulates (UFPs)-Investigation of Size, Composition and Concentration in a Metallurgy and Smelting Plant by Scanning Electron Microscopy (SEM). *Tobacco Regulatory Science (TRS)*, 2023, 5017-5025.
- [4] Rao XQ, Zhong JX, Brook RD, and Rajagopalan S: Effect of particulate matter air pollution on cardiovascular oxidative stress pathways. *Antioxidants & Redox Signaling* (2017).
- [5] Vignal C, Pichavant M, Alleman LY, Djouina M, Dingreville F, Perdrix E et al “ Effects of urban coarse particles inhalation on oxidative and inflammatory parameters in the mouse lung and colon”. *Particle and Fibre Toxicology* 14(2017). Claude Ostiguy et al 2013 *J. Phys.:* Conf. Ser. 429 012058
- [6] Stone, V., Hankin, S., Aitken, R., Aschberger, K., Baun, A., Christensen, F., Fernandes, T., Hansen, S.F., Hartmann, N.B., Hutchison, G., Johnston, H., Micheletti, C., Peters, S., Ross, B., Sokull-Kluettgen, B., Stark, D., Tran, L. 2009, ENRHES - Engineered nanomaterials: review of health and environmental safety, EC contract number 218433 448 p.
- [7] Ostiguy C, B, Roberge C Woods, B Soucy, G Lapointe, L Ménard, 2010. Les nanoparticules de synthèse: connaissances actuelles sur les risques et les mesures de prévention en SST, Seconde édition, IRSST, Études et recherches / Rapport R-646, 159 p.
- [8] Benlatreche, Med Salah, Ouafa Boukhmis, and Kahina Slimane. "Investigation of Nanoparticles and Ultra Fine Particles in the Workplace." *Recent Advances in Environmental Science from the Euro-Mediterranean and Surrounding Regions: Proceedings of Euro-Mediterranean Conference for Environmental Integration (EMCEI-1)*, Tunisia 2017. Springer International Publishing, 2018.

- [9] AA Shvedova, An Pietroiusti, B Fadeel, VE Kagan, 2012. Mechanisms of carbon nanotubes-induced toxicity : focus on oxidative stress. Review article. *Tox Appl Pharm* 261:121-133.
- [10] Reijnders L, 2012. Human health hazards of persistent inorganic and carbon nanomaterials.Review. *J Mater Sci* 47:5061-5073.
- [11] Eckhoff R.K., 2011. Are enhanced dust explosion hazards to be foreseen in production, processing, and handling of powders consisting of nano-particles? *Journal of Physics: Conference Series* 304 (2011) 012075
- [12] R E Clinkenbeard, E C England, D L Johnson, N A Esmen, T A Hall, A field comparison of the IOM inhalable aerosol sampler and a modified 37-mm cassette, *Appl Occup Environ Hyg.* 2002 Sep;17(9):622-7. doi: 10.1080/10473220290095943.
- [13] Gabriele Marcias, Jacopo Fostinelli, Simona Catalani, Michele Uras, Andrea Maurizio Sanna, Giuseppe Avataneo, Giuseppe De Palma, Daniele Fabbri, Matteo Paganelli, Luigi Isaia Lecca, Giorgio Buonanno, Marcello Campagna. Composition of Metallic Elements and Size Distribution of Fine and Ultrafine Particles in a Steelmaking Factory, *Int J Environ Res Public Health.* 2018 Jun; 15(6): 1192. doi: 10.3390/ijerph15061192
- [14] Chun-Nan Liu, Amit Awasthi, Yi-Hung Hung, Chuen-Jinn Tsai. Collection efficiency and interstage loss of nanoparticles in micro-orifice-based cascade impactors, *Atmospheric Environment* Volume 69, April 2013, <https://doi.org/10.1016/j.atmosenv.2012.12.003>
- [15] Larissa V Stebounova, Natalia I Gonzalez-Pech, Jae Hong Park, T Renee Anthony, Vicki H Grassian, Thomas M Peters. “Particle Concentrations in Occupational Settings Measured with a Nanoparticle Respiratory Deposition (NRD) Sampler” *Annals of Work Exposures and Health*, Volume 62, Issue 6, July 2018, Pages 699–710, <https://doi.org/10.1093/annweh/wxy033>
- [16] Park JH, Mudunkotuwa IA, Crawford KJ, Anthony TR, Grassian VH, and Peters TM: Rapid analysis of the size distribution of metal-containing aerosol. *Aerosol Science and Technology* 51(1): 108–115 (2017). [PubMed: 28871214].
- [17] Natalia Isabel Gonzalez-Pech, Larissa V. Stebounova, Irem B. Ustunol, Jae Hong Park ‘ Size, composition, morphology and health implications of airborne incidental metal-containing nanoparticles’ *J Occup Environ Hyg.* 2019 June ; 16(6): 387–399. doi:10.1080/15459624.2018.1559925.
- [18] Pr  sum   M, et al. Exposure to metal oxide nanoparticles administrated at occupationally-relevant doses induces pulmonary effects in mice *Nanotoxicology*, Sept 2016. <https://doi.org/10.1080/17435390.2016.1242797>.

- [19] Nielsen, Maria Bille, et al. "European nanomaterial legislation in the past 20 years—Closing the final gaps." *NanoImpact* (2023): 100487.
- [20] Ault AP, Peters TM, Sawvel EJ, Casuccio GS, Willis RD, Norris GA, et al.: Single-particle SEM- EDX analysis of iron-containing coarse particulate matter in an urban environment: Sources and distribution of iron within Cleveland, Ohio. *Environmental Science & Technology* 46(8): 4331–4339 (2012). [PubMed: 22435663].
- [21] Park JH, Mudunkotuwa IA, Crawford KJ, Anthony TR, Grassian VH, and Peters TM: Rapid analysis of the size distribution of metal-containing aerosol. *Aerosol Science and Technology* 51(1): 108–115 (2017). [PubMed: 28871214].
- [22] Gonzalez LT, Rodriguez FEL, Sanchez-Dominguez M, Cavazos A, Leyva-Porras C, Silva-Vidaurre LG, et al.: Determination of trace metals in TSP and PM<sub>2.5</sub> materials collected in the metropolitan area of Monterrey, Mexico: A characterization study by XPS, ICP AES, and SEM-EDS. *Atmospheric Research* 196:8–22 (2017).