DOI:10.5937/IIZS26498I

# EDS-BASED CHARACTERIZATION OF INHALABLE METAL AEROSOLS IN OCCUPATIONAL ENVIRONMENTS

*Research paper*

**Milana Ilić Mićunović[1*], Borut Kosec[2], Zoran Čepić[3], Slađana Jovanović[4], Boris Agarski[5]**

[1,3,4,5]University of Novi Sad, Faculty of Technical sciences, Novi Sad, Serbia
[2]University of Ljubljana, Faculty of Natural Sciences and Engineering, Ljubljana, Slovenia

**Abstract:** Workplace exposure to airborne metallic aerosols during thermal joining processes represents significant occupational health risks. This study presents a comparative microstructural and elemental characterization of inhalable welding aerosols generated during Metal Active Gas (MAG) and Tungsten Inert Gas (TIG) processing of structural steels. Personal aerosol sampling was conducted in the operator's breathing zone using mixed cellulose ester (MCE) filters at a flow rate of 3.5 L/min. Microchemical analysis was performed using Field Emission Scanning Electron Microscopy with Energy-Dispersive X-ray Spectroscopy (FEG-SEM/EDS), utilizing both point-spectrum acquisition and EDS Layered Imaging for spatial mapping. The results revealed distinct chemical signatures between the two processes: TIG-generated particles were dominantly composed of iron oxides (79–83 wt% Fe) with trace amounts of Ni and Cr, whereas MAG aerosols mainly contained manganese (31.37–42.28 wt% Mn) and silicon (0.45–7.29 wt% Si) due to the intense vaporization and condensation of filler wire deoxidizers. EDS elemental mapping confirmed the co-localization of Mn, Si, and O within complex spherical submicron aggregates. These findings highlight the efficacy of SEM-EDS in resolving single-particle chemical heterogeneity and providing process-specific elemental profiles essential for targeted occupational health risk assessments and exposure control strategies.
**Key words:** EDS, welding aerosols, occupational exposure

## INTRODUCTION

Measurement and characterization of airborne particulate matter in the occupational environment are of critical importance for the prevention of occupational respiratory diseases. Particle size directly determines the site of deposition within the respiratory tract, which is why a standardized classification into inhalable and respirable fractions is applied in industrial hygiene (ISO 7708, SRPS EN 481).

Inhalable fraction includes particles with an aerodynamic diameter of up to 100 μm that are inhaled through the nose and mouth. The primary site of deposition is the upper respiratory tract (nasal cavity, pharynx, and trachea). This fraction is predominant in processes such as grinding, stone processing, and bulk material handling (Ilic Micunovic, 2024, SRPS EN 481).

Thoracic fraction consists of particles with an aerodynamic diameter of less than 10μm (with a 50% collection efficiency cut-point at 10μm) that pass through the larynx and penetrate into the conducting airways of the lungs, the tracheobronchial tree. These particles primarily deposit in the trachea, main bronchi, and bronchioles, causing localized inflammatory responses, bronchospasms, and exacerbation of pre-existing airway conditions such as asthma and chronic bronchitis (Brown, 2013).

Respirable fraction consists of particles with an aerodynamic diameter of less than 2.5 μm (with a 50% collection efficiency cut-point at 4 μm) that penetrate deep into the respiratory bronchioles and alveoli. Due to their size, these particles bypass the respiratory tract's natural clearance mechanisms and remain permanently deposited in the lung tissue or enter systemic circulation (Ribalta, 2019, Brown, 2013).

Monitoring fractions enables accurate risk assessment and comparison with occupational exposure limits (OELs), as coarser particles cause local irritation and mucosal damage, whereas respirable particles lead to chronic fibrotic and systemic diseases (Ribalta, 2019).

Transition metals present in industrial aerosols, during welding, casting, thermal cutting and electroplating, exhibit high toxicity, primarily through the generation of reactive oxygen species

[1] Corresponding author, milanai@uns.ac.rs, ORCID: 0000-0002-5673-4690
[2]borut.kosec@ntf.uni-lj.si, ORCID: 0009-0001-8117-9721
[3]zorancepic@uns.ac.rs, ORCID: 0000-0001-5853-4767
[4]sladjana.jovanovic@uns.ac.rs, ORCID: 0009-0001-2226-1645
[5] agarski@uns.ac.rs , ORCID: 0000-0002-7206-5714

through Fenton and Fenton-like reactions, leading to oxidative stress, DNA damage, and cellular inflammation. Chronic inhalation of iron oxides leads to siderosis ("welder's lung"). Although siderosis was traditionally considered a benign pneumoconiosis, contemporary research indicates that the presence of respirable iron causes persistent tissue inflammation and synergistically increases the risk of chronic obstructive pulmonary disease (COPD) (Liu, 2025). Respirable manganese aerosols cross the alveolar-capillary membrane and reach the central nervous system via retrograde axonal transport along the olfactory nerve. Chronic exposure to manganese causes an irreversible neurodegenerative disorder that clinically mimics Parkinson's disease. Hexavalent chromium is a potent mutagen and Group 1 carcinogen (IARC). It readily crosses cell membranes, where it is reduced, generating ROS and DNA adducts/crosslinks, leading to lung cancer, nasal ulcerations, and chronic asthmatic bronchitis. Inhalation of nickel compounds (particularly nickel subsulfide and nickel oxide) is associated with a high risk of developing lung and nasal cavity cancers. Nickel inhibits DNA repair mechanisms and acts as a strong allergen, causing occupational asthma and contact dermatitis (Khoshnaw, 2023). Titanium is predominantly present in the form of titanium dioxide ($TiO_2$). While bulk particles are considered less toxic, ultrafine and nano-sized $TiO_2$ particles possess high surface reactivity, inducing alveolar inflammatory responses and pulmonary fibrosis, and are classified as possibly carcinogenic to humans (IARC Group 2B) (Rahul, 2021). Standard gravimetric and bulk chemical analyses of airborne particulate matter provide essential data on total mass concentration, but they often mask the intrinsic heterogeneity of complex industrial aerosols. Advanced microanalytical techniques such as Scanning Electron Microscopy coupled with Energy-Dispersive X-ray Spectroscopy (SEM-EDS), Transmission Electron Microscopy (TEM), and automated digital image analysis offer single-particle resolution that is indispensable for comprehensive occupational health risk assessment.

SEM-EDS mapping allows for the identification of specific toxic elements (e.g., transition metals like Cr, Ni, Mn, Fe) within individual particles. Microanalysis distinguishes between externally mixed aerosols where distinct chemical species exist as separate particles, and internally mixed aerosols where heterogeneous agglomerates containing multiple elements. This distinction is critical because internal chemical interactions can significantly alter particle solubility, bio accessibility, and toxicity (Stefaniak, 2010).

The main objective of this study is to perform a comparative chemical and microstructural characterization of workplace metallic aerosols generated during MAG and TIG material processing. Utilizing Energy-Dispersive X-ray Spectroscopy (EDS) via point-spectrum analysis and elemental mapping, the study aims to identify the primary elemental composition (wt%) of inhalable particles produced under different process conditions, specifically comparing aerosol characteristics generated during MAG welding with Mn and Si containing filler wire and TIG welding under an argon shielding atmosphere. Furthermore, it seeks to determine the spatial distribution and localization of key alloying and deoxidizing elements such as Fe, Mn, Si, Cr, Ni, and O within individual particles and agglomerates. Ultimately, this approach establishes a clear spectroscopic correlation between processing parameters and process-specific elemental signatures relevant to occupational exposure assessment, thereby providing concise data for workplace risk assessment.

## MATERIAL AND METHODS

### Aerosol Sampling and Preparation

Workplace aerosol particles generated during MAG and TIG metal processing were collected to evaluate the operator's direct exposure to airborne emissions. Sampling was conducted using a ZAMBELLI EGO PLUS TT personal air sampler equipped with a 25 mm mixed cellulose ester (MCE) filter. The device was calibrated prior to each experimental run and operated at a constant flow rate of 3.5 L/min with a conical inlet, specifically chosen for capturing the inhalable aerosol fraction. To capture representative breathing-zone samples, the filter inlet was secured near the operator's clavicle, approximately 30 cm from the breathing area.

The sampled particulate matter originated from the thermal joining of structural and tool steel specimens. Aerosol generation was evaluated across two distinct process configurations: Metal Active Gas (MAG) welding using solid wire filler materials rich in manganese (Mn) and silicon (Si) deoxidizers, and Tungsten Inert Gas (TIG) welding conducted under a protective argon atmosphere. The collected airborne samples thus represent a mix of condensed metallic vapors, oxidized fumes, and micro-particles emitted directly into the breathing zone during the melting and solidification of the base and filler metals.

### Scanning Electron Microscopy (SEM)

Microstructural and morphological evaluations of the collected airborne particles were conducted using a Field Emission Scanning Electron Microscope (FEG-SEM, Thermo Fisher Scientific Quattro S). The instrument is equipped with multiple imaging modes, including Secondary Electron Imaging (SEI) for high-resolution surface topography and Backscattered Electron Imaging (BEI) for compositional contrast. High-magnification micrographs (ranging from 1 to 10 μm scale bars) were acquired to evaluate individual particle geometries, sphericity, and agglomeration phenomena across the MCE filter matrix.
For elemental and microchemical analysis, the FEG-SEM system is integrated with an Energy-Dispersive X-ray Spectroscopy (EDXS) detector (Ultim® Max). Microchemical characterization was performed using both localized point-spectrum acquisition and EDS elemental mapping (EDS Layered Imaging). Accelerating voltages and acquisition parameters were optimized to capture characteristic Kα and Lα emission lines for key metallic species specifically Fe, Mn, Si, Cr, Ni, and Ti as well as light elements (O, C) originating from the substrate and oxidized phases. Spatial element distribution maps were generated to evaluate element co-localization within individual micro-spheres and complex particle aggregates.

## RESULTS AND DISCUSSION

### Elemental Composition of TIG and MAG Aerosols

The EDS point-spectrum analysis revealed distinct chemical profiles for aerosol particles generated by the two investigated processes (Table 1.). Particles captured during TIG processing (without additives) are characterized by a high iron content ranging from 79 to 83 wt% Fe alongside oxygen, indicating the formation of primary iron oxides (Fig. 1.). Minor trace amounts of nickel (up to 3.63 wt% Ni) were also detected, closely reflecting the base and filler metal compositions. Conversely, MAG aerosols generated with additives exhibit a strong enrichment in manganese, reaching 31.37 to 42.28 wt% Mn, and silicon between 0.45 and 7.29 wt% Si, together with moderate amounts of chromium (up to 3.89 wt% Cr). This chemical signature confirms the intense evaporation and condensation of Mn-Si deoxidizing agents and alloying elements present in the MAG wire. Additionally, minor aluminum signals (0.18–0.49 wt% Al) observed across both process conditions likely originate from background scatter or trace constituents in the sampling setup. Furthermore, high carbon and nitrogen contents detected in certain area spectra, such as Spectrum 2, originate from the underlying mixed cellulose ester filter matrix rather than the metallic particles themselves (Fig. 2.).

**Table 1.** Elemental composition of representative aerosol particles derived from EDS point spectra.

| Process Condition | Spectrum Label | Fe (wt%) | Mn (wt%) | Si (wt%) | Ni (wt%) | Cr (wt%) | O (wt%) | Al (wt%) |
|---|---|---|---|---|---|---|---|---|
| **TIG (without additives)** | Spectrum 7 | 79.24 | — | — | — | — | 20.76 | - |
| **TIG (without additives)** | Spectrum 3 | 62.01 | — | — | 3.63 | — | 34.09 | 0.26 |
| **MAG (with additives)** | Spectrum 2 | 16.21 | 31.37 | 6.92 | — | 3.61 | 41.40 | 0.49 |
| **MAG (with additives )** | Spectrum 3 | 14.50 | 42.28 | 7.29 | — | 3.89 | 31.86 | 0.18 |

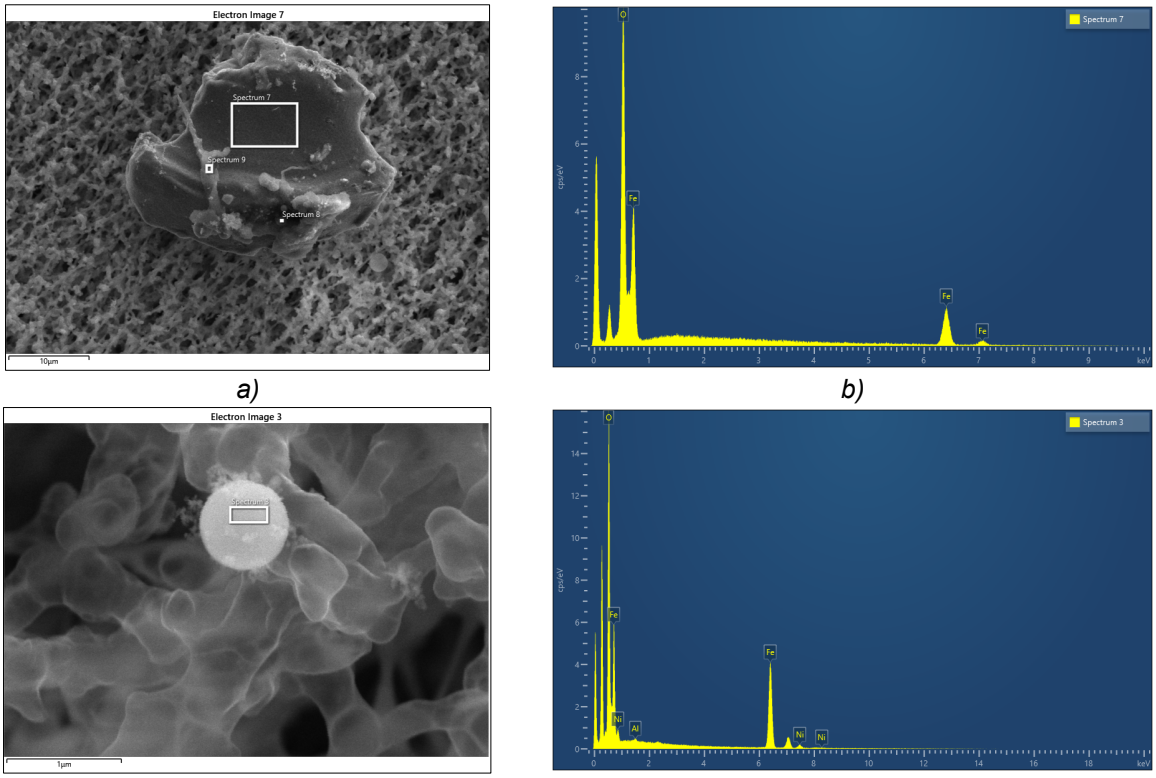



***Fig. 1.*** *Representative SEM micrographs and corresponding EDS point spectra of TIG aerosol particles: (a, c) particle morphology, and (b, d) their elemental composition.*

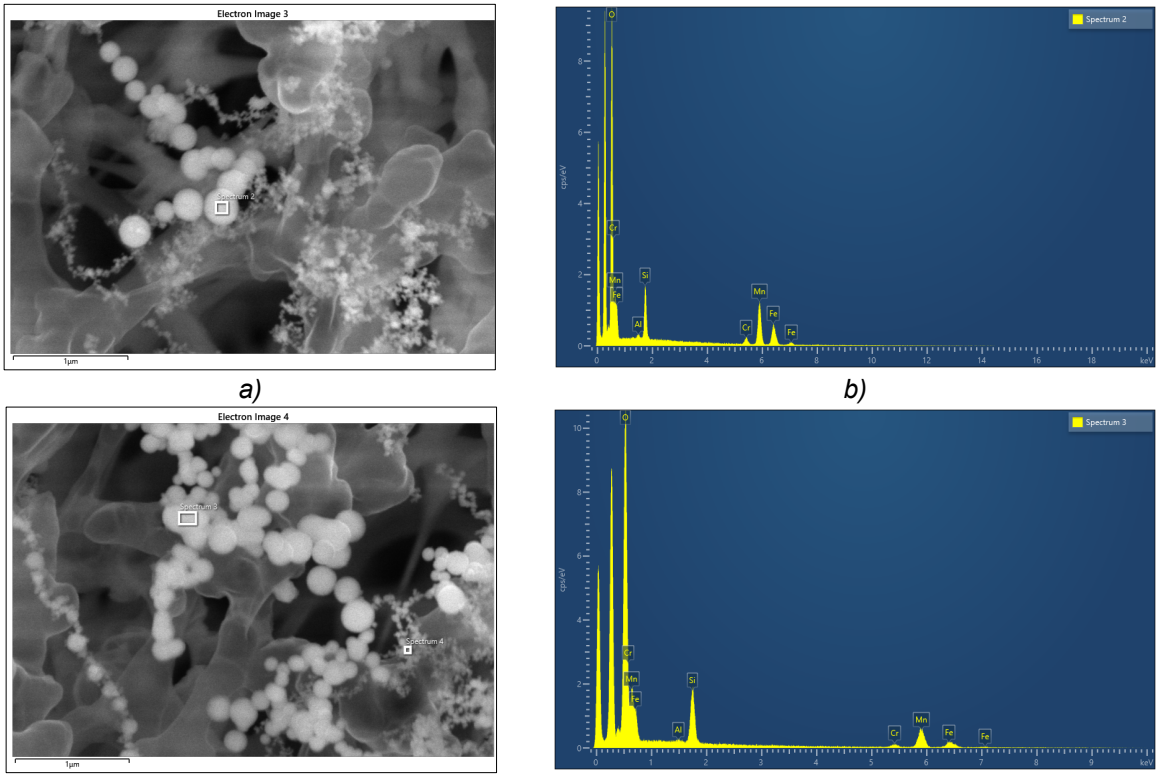
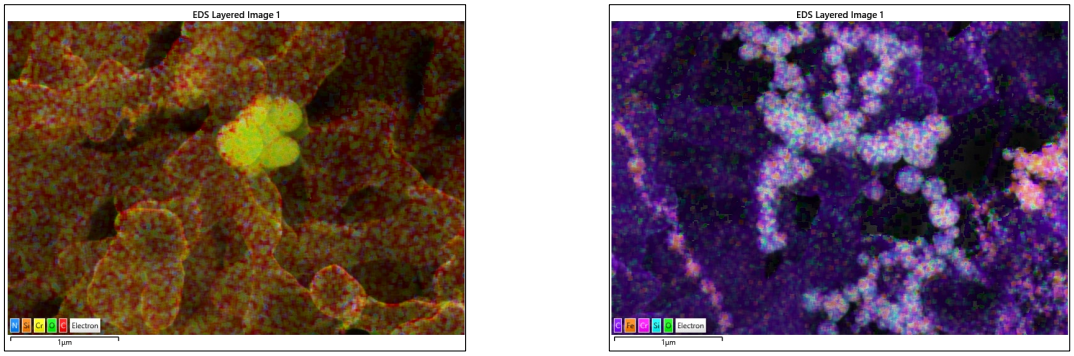



***Fig. 2.*** *Representative SEM micrographs and corresponding EDS point spectra of MAG aerosol particles: (a, c) particle morphology, and (b, d) their elemental composition.*

Elemental mapping via EDS Layered Imaging confirmed the localization of key elements within individual particles across the investigated process conditions. In MAG aerosols, the spatial signals for manganese, silicon, and oxygen overlap directly over the spherical particles, confirming the formation of complex mixed Mn-Si-Fe oxides resulting from vapor condensation (Fig. 3a). Conversely, larger and more irregularly shaped particles exhibit a distinctly heterogeneous element distribution, characterized by iron-rich cores surrounded by localized surface oxidation (Fig. 3b).




*a)* *b)*

***Fig. 3**. Representative EDS elemental mapping (Layered Imaging) showing the spatial distribution and co-localization of key elements (Fe, Mn, Si, O, Cr) in: (a) MAG spherical aerosols, and (b) irregular TIG particles.*

These findings demonstrate the value of combining SEM morphology, EDS point spectra, and elemental mapping for characterizing the chemical heterogeneity of individual airborne particles. From an occupational exposure perspective, the pronounced manganese enrichment observed in MAG particles is particularly relevant due to associated inhalation risks. However, EDS weight percentages describe comparative, process-specific elemental profiles rather than absolute airborne concentrations or inhaled doses. The semi-quantitative nature of EDS, as well as substrate interaction with the MCE filter, should be considered during interpretation.

## CONCLUSION

This study demonstrates that Energy-Dispersive X-ray Spectroscopy EDS provides a rapid and spatially resolved approach for characterizing and differentiating airborne metallic particles generated during welding processes. The elemental analysis clearly distinguished the physical and chemical mechanisms of two distinct operations: MAG processing produces aerosols heavily enriched in manganese (up to 42.28 wt% Mn) and silicon from filler wire vaporization, whereas TIG processing yields predominantly iron rich oxide particles. Recognizing these distinct chemical profiles is vital for assessing process-specific occupational health risks and implementing targeted exposure controls. While EDS provides exceptional speed and spatial precision for elemental identification, future research should incorporate complementary analytical techniques such as X-ray Diffraction (XRD), X-ray Photoelectron Spectroscopy (XPS), or Transmission Electron Microscopy (TEM) to precisely determine the underlying crystal structures, phase transformations, and oxidation states of the collected airborne particulates.

## ACKNOWLEDGEMENT

This research has been supported by the Ministry of Science, Technological Development and Innovation (Contract No. 451-03-34/2026-03/200156) and the Faculty of Technical Sciences, University of Novi Sad through project "Scientific and Artistic Research Work of Researchers in Teaching and Associate Positions at the Faculty of Technical Sciences, University of Novi Sad 2026" (No. 01-3609/1).

## REFERENCES

ISO 7708 (1995): Air quality — Particle size fraction definitions for health-related sampling.

SRPS EN 481 (2010): Vazduh na radnom mestu - Definisanje veličine frakcija čestica za merenje u vazduhu.

Ilic Micunovic, M., Budak, I., Vukelic, D., Djurovic Koprivica, D., Kuzmanovic, M., Agarski, B., Puskar, T (2024). Investigation of the Geometric Characteristics of Inhalable Particles Emitted from the Process of Grinding Dental Restorations. *Applied Sciences*, 14, 5169.

Brown, J.S., Gordon, T., Price, O. (2013). Thoracic and respirable particle definitions for human health risk assessment. *Particle and Fibre Toxicology,* 10, 12.

Ribalta, C., López-Lilao, A., Estupiñá, S., Fonseca, A.S., Tobías, A., García-Cobos, A., Minguillón, M.C., Monfort, E., Viana, M. (2019). Health risk assessment from exposure to particles during packing in working environments, *Science of the Total Environment*, 671, 474-487.

Liu, Y., Yang, R., Chai, Y., Wang, Y., (2025). Welding and burnishing dusts' exposure characteristics and health risk assessment in electromechanical processing workshop: Based on oxidation potential and EPA, *Building and Environment*, 279, 112971.

Khoshnaw, F., (2023). Chapter 1 - An introduction to welding of metallic materials, Editor(s): Fuad Khoshnaw, *Welding of Metallic Materials*, Elsevier, 1-35.

Rahul, M., Sivapirakasam, S.P., Vishnu, B.R., (2021). Health issue owing to exposure with welding fumes and their control strategies at the source–a review, *Materials Today*, Proceed., 46, 9239-9245

Stefaniak, A. B., Virji, M. A., & Day, G. A. (2010). Characterization of chemical and physical properties of airborne particles to inform exposure and risk assessment. *Inhalation Toxicology*, 22(S2), 19–38.