



Unintended emission of nanoparticle aerosols during common laboratory handling operations



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HIGHLIGHTS

- Aerosol nanoparticles generation during common laboratory operations was studied.
- Dust concentration and NEFs were similar for common laboratory operations.
- NEF for the handling processes were in the range of $10^8 \# \text{h}^{-1}$.
- Ce/TiO₂ showed rapid interaction between emitted and ambient nanoparticles.

ARTICLE INFO

Article history:

Received 6 February 2014

Received in revised form 24 June 2014

Accepted 28 June 2014

Available online 5 July 2014

Keywords:

Nanoparticle
Handling
Aerosol emission
Laboratory operations

ABSTRACT

Common laboratory operations such as pouring, mashing in an agate mortar, transferring with a spatula, have been assessed as potential sources for emission of engineered nanoparticles in simulated occupational environments. Also, the accidental spilling from an elevated location has been considered. For workplace operations, masses of 1500 or 500 mg of three dry-state engineered nanoparticles (SiO₂, TiO₂ and Ce-TiO₂) with all dimensions under 30 nm, and one fibrous nanomaterial (MWCNT) with diameter under 10 nm and length about 1.5 μm were used. The measured number emission factors (NEF) for every operation and material in this work were in the range of $10^5 \# \text{s}^{-1}$. The traceability of emitted nanoparticles has been improved using Ce-doping on TiO₂ nanoparticles. With this traceable material it was possible to show that generated aerosol nanoparticles are rapidly associated with background particles to form large-sized aerosol agglomerates.

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1. Introduction

Although the presence of the nanotechnology in the marketplace is rapidly growing, in many cases consumers remain unaware of the nature and characteristics of nanomaterial-containing products [1]. Products and utensils containing engineered nanomaterials (ENMs) such as nanoparticles or carbon nanotubes may become sources of unintended human exposure, mainly through

dermal and respiratory routes. Nanoparticles are also found widely in nature, and natural sources include ash, desert dusts, aerosols and metal oxide particles. Although evolved to deal with natural nanomaterials and their fluctuations over millennia, it is not known how organisms will cope with high discharges of anthropogenic nanomaterials into the environment [2]. Once released, the behavior of ENMs in the environment depends on their surface area and size, among other material parameters [3]. Due to their small size (under 100 nm), the exposure to ENMs could imply hazards beyond the capabilities of conventional industrial safety and hygiene procedures. According to O'Shaughnessy, a worst-case scenario regarding exposure to ENMs concerns the manufacture of nanoparticles, especially in the dry state [4]; even though accidental spills during manufacturing would be even a worst situation. Many common tasks in occupational settings involved in

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nanomaterials production and use require the handling of ENMs in a dusty form, and this could lead to a significant increase in the risks of exposure to nanosized aerosols [5–8].

There is no protocol of general validity to assess the exposure to ENMs during common handling operations. Several authors have developed different standardized dustiness tests to assess the materials risks [9]. One common test uses a rotating drum process, normalized under the European standard EN 15051, and involves the application of mechanical energy to a bulk sample while a steady stream of air is supplied through the drum. Emitted particles are alternatively collected on a filter or measured with direct-reading instrumentation [6,8,10,11]. Similarly, the single-drop method consists of a vertical tube that generates dust by allowing the sample to drop through the tube [12]. A third dustiness test uses a vortex shaker to agitate the powder placed in a test tube [13]. All the mentioned methods use large amounts of materials (about 500 g), which entail obvious difficulties when applied to nanotechnology laboratory operations, where sub-gram quantities are often handled. Therefore adaptations have been developed to reduce the sample amount down to several grams [11] or even to a milligram scale [9]. Evans et al. [14] dispersed 10 mg of powder utilizing an energetic Venturi aerosolization, as a more energetic alternative to conventional methods such as rotating drum testers, and determined the total and respirable dustiness of different materials. The aerosolization of the powder in their work takes place under turbulent conditions that according to the authors may be representative of energetic cleaning operations such as cleaning dry work surfaces with compressed air. However, their results cannot be extrapolated to manual handling of nanomaterials in laboratory operations (e.g. transferring with a spatula to a weighting dish), where the energy involved is orders of magnitude lower.

In this work, we have calculated the Number Emission Factors (NEF), rather than dustiness. In fact, both methods (dustiness tests vs. NEF calculation) are measuring a similar property, (the propensity of powdered materials to become airborne upon different operations), but provide complementary information, since there are differences regarding the amount of matter handled in every case and in the fundamental parameters measure, which for dustiness are based in the mass of particles whereas NEFs are calculated in a number basis.

One of the first attempts for analyzing the emission of airborne nanosized matter when handling ENMs was performed by Tsai et al. [15] showing that handling dry ENMs inside a fume hood results in a significant release of nanoparticles into the laboratory environment and the researcher's breathing zone. Moreover, several laboratory and field studies have reported that the sonication of both hydrophobic and hydrophilic carbon-based nanomaterials (CNM) in water generates airborne nanoparticle concentrations similar to those found during dry handling [16–18]. Other laboratory operations have been identified as possible exposure risk sources, namely synthesis [19,20], accidental spilling [21] or even common activities such as weighting or pouring [16,17]. Ham et al. [17] studied nanoparticle manufacturing workplaces producing titanium dioxide, silver, aluminum and copper. They found that the nanoparticle concentration varied depending on the task performed, metric adopted and working or off duty. On the other hand, Methner et al. [18] studied the release of carbon nanofibers (CNF) on dry material handling, wet cutting, grinding and sanding of plastic composites containing CNF. They point out that surface grinding of composites and manually transferring dry CNFs produce substantial increases in particle number concentration.

The complex nature of indoor environments in a research laboratory where different nanomaterials are manually handled plays a key role in the impact of ENMs. Workplace atmospheres generally contain numerous nanoparticles of a varied nature that may be of natural and/or anthropogenic origin. Airborne nanoparticles in

research laboratory environments undergo continuous changes in concentration, size distribution and particle structure and nature. Even working inside hoods nanoparticles are released to the laboratory air, concentration as high as $1.3 \times 10^5 \text{ cm}^{-3}$ can be reached in the breathing zone [15]. The use of products and utensils containing engineered nanomaterials could lead to the release of these ENMs to any indoor environments, significant sources that can generate episodic emissions include every day actions such as cooking or cleaning [22]. These environmental nanoparticles represent a strong interference in the detection of those emitted during synthesis and handling operations, which are often present in much smaller concentrations. Methner et al. [23] have stressed the influence of background particles when measuring the concentration of ENMs during workplace monitoring. In an exhaustive work, Seipenbusch et al. [24] studied the evolution of aerosols with concentrations of 7×10^4 – $5 \times 10^6 \text{ cm}^{-3}$ in the presence of background particles, showing changes in the size and number concentration of nanoparticle aerosols due to heterogeneous coagulation processes with background airborne matter. The magnitude of these processes was found to depend on the concentration of background aerosol; representative indoor aerosols have typically concentrations in the range 10^3 – 10^4 cm^{-3} . Furthermore, binary hybrid particles consisting of background particles decorated with emitted nanoparticles evolve, and therefore the chemical composition of the ENM aerosols changes. Ono-Ogasawara et al. [25] reviewed some examples where measurements of ambient nanoparticles did not correlate with workplace operation conditions, due to the presence of background particles. Thus for instance, in the monitoring of nanoparticles at a lithium titanate production facility the number concentrations did not correlate with workplace operation. Possible contaminants included welding fumes, particles from grinding and particles from outside sources. In a different example, the total number concentration in a MWCNT laboratory responded to oil pump operation more strongly than to CNT release. Sources of ENM are difficult to detect in laboratory indoor environments because the generated aerosol can be diluted or hidden under natural or man-made background particles. At large, the extent of this interaction depends on the concentration and particle size of the background nanoparticles and the concentration of the studied aerosol. However, the presence of these background nanoparticles is unavoidable in real life exposure and therefore its influence must be taken into account.

In this work, we have attempted to shed light on the generation of nanoparticle aerosols in several common laboratory operations and events. To this end, we have restricted ourselves to handling small amounts of powders (always below 2 g of material). This presents some additional complications for measurement, but is deemed more realistic in the scenarios considered. We have used commonly encountered ENMs of a different nature: TiO_2 , SiO_2 and MWCNT. A novel experimental strategy was designed to investigate the influence of background nanoparticles: First, using a clean-room approach (a clean glove chamber with HEPA-filtered air) handling operations were performed, which allowed monitoring aerosol generation without the influence of environmental particulate matter. Handling experiments were then performed after filling the chamber with ambient air, containing the background particles. The generated aerosols were studied in both cases using online methods (airborne nanoparticle counters) and offline methods (electron microscopy analysis of the collected particles). Finally, to address the problem of discriminating the released ENMs from background material, which is relatively straightforward by transmission electron microscopy for CNTs but highly challenging for TiO_2 or SiO_2 , cerium-doped TiO_2 nanoparticles were synthesized and employed in pouring operations. This facilitated the identification of ENMs by offline analysis since Ce is absent from background material.

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