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Environmental release of engineered nanomaterials from commercial tiles under standardized abrasion conditions

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HIGHLIGHTS

- Nanotreated commercial tiles under abrasion release 175 times more fine and nanosized particles.
- The generated aerosol is dominated by the presence of composite nanoobjects, agglomerates and aggregates (NOAAs).
- The big majority of the generated particles is inhalable.
- An experimental release scenario can generate real life particles and provide them to ecotoxicological studies.

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ABSTRACT

The study presented here focuses on commercial antibacterial tiles whose emissivity of (nano) particles due to abrasion has yet barely been investigated. The tiles have been characterized regarding their surface properties and composition throughout their chain-of-use, *i.e.* from their state of commercialization until the experimental end-of-service life. In contrast to plane standard tiles, their surfaces form hilly surfaces. In the depressions, titanium dioxide is found at the surface, thus theoretically protected by the hilly areas against abrasion on the tile's surface.

Furthermore, a deposition technique has been put in place by producers allowing for coating the before mentioned commercial tiles with titanium dioxide, thus being similar to those commercially available. It consists in depositing titanium dioxide on the surface, latter one allowing fixing the first. This development allows for better understanding the future options for product formulation and thus improvement with respect to particle release.

The tests reveal the aerosolization from commercial antibacterial tiles of micronic and submicronic particles in the inhalable region or particles that can be subjected to be released in the environment (<10 μm). The aerosolization of the particles from the coated tiles was found to be significantly higher compared to the non coated tiles.

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1. Introduction

Nanoparticles are nowadays often found embedded in commercial consumer products, endowing the latter with new and innovative properties [1,2]. So called self-cleaning or antibacte-

rial coatings for instance, selectively bind and remove bacteria, *e.g.* when using silver or titanium dioxide nanoparticles [3].

Sole the soaring presence of nanoparticles in consumer goods increases the risks along their life cycle, *i.e.* to workers, consumers and the environment. Nanoparticles incorporated into products can indeed be released over the whole product life cycle, particularly during use and disposal, both into the gas and/or the aqueous phase (*i.e.* via mechanical solicitation and/or leaching processes). Looking at the possible routes for nanoparticles or nanomaterials to enter the environment, both the aquatic and terrestrial compartment can be substantially exposed [4]. As an example, many studies have

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confirmed the potential release of nanoparticles from antibacterial textiles or antibacterial wound dressing as well as from coatings [5]. There is also evidence that nanoparticles can affect the terrestrial compartment, e.g. through the use of sewage sludge as fertilizer in agricultural process [6]. From a toxicological point of view, out of the dermal, ingestion or inhalation penetration routes to the human body, the inhalation route is considered to be critical [7].

The assessment of the ecotoxicological and toxicological effects of nanoparticles and nanomaterials has, consequently, become a major issue worldwide, and in the last five to ten years an increasing number of studies concerning the nanoparticles or nanomaterials hazard assessment were published [8–12]. For example, the “pristine” forms of titanium dioxide nanoparticles are reported to cause acute and sub-lethal effects to several freshwater or marine species including bacteria, micro-algae, micro-invertebrates, mollusk, insect larvae and fishes [10,13,14]. Nano titanium dioxide is also identified for its toxicological damage [15], such as reproduction troubles [16,17] or splenic [18], kidney [19] injury or hippocampal apoptosis [20], which is supposed to be a consequence of its large surface area [21]. For given nanometric particle sizes, titanium dioxide was however shown to have lesser effects on the respiratory ducts than zinc oxide or silver [22].

However, most of the works published on nanoparticles or nanomaterials is considering their so-called “pristine” forms, as produced, whatsoever regardless of ecotoxicity studies, toxicity studies or environmental fate studies. Although providing useful (eco)toxicological informations, the quantity of available data for the “pristine” form contrasts with the very few studies that have investigated the environmental exposure and the ecotoxicity or toxicity of nanoparticles released from consumer products according to relevant scenarios. In addition, some authors have shown that modifications of the nanoparticles can happen when they are included in consumer products and/or released during their life cycle [23,24]. Therefore the physico-chemical properties of the nanoparticles released from consumer product could markedly differ from the pristine form and, as a consequence, the potential ecotoxicological or toxicological effect could change in equal measure.

Currently, both with nanoaerosol formation as well as with nanoleachate formation, two fundamental paths of nanoparticle transport away from the source are investigated, i.e. either in gas or liquid phase [25].

The formation of aerosols and suspensions prior to this transport is the result of mechanical solicitation, i.e. the exertion of mechanical stresses going along with deformations and cracking fissuration of asperities on the surface [26]. Here, abrasion is one of the most prominent processes allowing for experimentally simulating these stresses [27–32].

Further to the (eco)toxicological studies on pristine nanoparticles, a realistic risk assessment with respect to the exposure to engineered nanomaterials should thus lead to a holistic approach. Processes that lead to the particle release must be taken into account, since the environmental behavior supposedly strongly depends on the respective *release scenario*. Yet, very few is known about this and further research is needed here [4].

In this context, the agglomeration behavior of nanoparticles should also be brought into focus, which is – in the wet phase-known to happen rapidly for TiO₂ [33]. For the dry phase, some researchers argue that agglomeration might reduce the risk of inhalation [34]. On the contrary, no exposure alleviation has up to now been demonstrated for the so called nanoobjects, agglomerates and aggregates (NOAAs), endorsing the imperative of experimental strategies based on holistic release scenarios [35,36].

Conscious of the concerns related to the nanorisks, tile producers might try to modify their production process in order to avoid any possible nanoparticle release. As a result a new generation

of photocatalytic tiles were for example prepared by the manufacturer GranitiFiandre deploying a commercial micron-sized TiO₂ suspension [37].

A second prominent *release scenario* is the washing out of nanoparticles from their matrix. It is known that this happens easily to nanosilver [5] or to nanometric titanium dioxide, which has been traced from exterior façade paints via its detachment from new and aged façade paints by natural weather conditions to its release into surface waters [38]. Titanium dioxide can deteriorate its surrounding paint matrix by its in principle desired photocatalytic action, whereby the question raises whether there is a point in embedding nanoparticles into paints [39].

Further to the basic functionalities of tiles, such as excellent mechanical and chemical resistance which make them ideal for professional and hygienic environments, targeted functionalities are reached today, owing to the embedding of nanoparticles. The temperature resistance of tiles, unlike plastics, makes them an ideal substrate for directly attaching photocatalytic layers by sintering [40]. By reason of their self-cleaning properties, the so produced tiles are becoming widely spread used, which still limited to a worthwhile outdoor use [41]. Thousands of tall buildings are reported to have been covered with self-cleaning tiles in Japan [42]. Tiles, together with other products, have already been the topic of release studies about a decade ago [43], which, due to the absence of references in sample preparations, exposure scenarios and measurement equipment unfortunately make clear conclusions impossible [44]. Moreover, the manufacturing processes of these nanocoated tiles still requires much research progress [45], which also leads to the different scenarios which need to be investigated.

Consequently, the investigation of the release of engineered nanomaterials from commercial tiles under mechanical solicitation is of vital importance for this growing, at this stage established market of consumer goods. The connection of both manufacturing considerations as well as release scenarios, to the worker, to the consumer or to the environment, to the experimental work contributes to a realistic study of the hazardousness of the tiles.

2. Materials and methods

2.1. Antibacterial tiles

2.1.1. Specificities of the ceramic materials and characterization of the commercial products

The majority of commercial tiles are obtained by molding and pressing a suspension containing the ceramic material. The obtained raw tile is then heated in a roller kiln, allowing for stoving the tiles during short cycles, to temperatures ranging from 1200 °C to 1250 °C [46,47]. At this temperature, its future mechanical resistance is conferred to the tile. The so-produced tile which did not yet receive any antibacterial treatment, is denominated ‘TNT’ in this paper.

The antibacterial properties are obtained by adding titanium dioxide in a further process step. This mineral can exhibit different crystal structures varieties: The anatase phase forms at temperatures below 800 °C, while as the rutile phase formed above 800 °C does not exhibit the targeted antibacterial properties and is for this used as pigment only. Further to the crystal structure, their presence with or without a matrix and the granulometry govern their activity [48]. The annealing of the TiO₂ to the tile must thus take place below 800 °C, i.e. much below the stoving temperature, and is often done at about 500 °C, whilst an optimum temperature has been reported for a lower temperature of 300 °C [40]. The so-treated tile is denominated ‘TT’ in this paper.

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