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NANOPARTICLES IN AQUATIC ENVIRONMENTS – FROM PRODUCTION TO WATER TREATMENT: A REVIEW

NANOPARTÍCULAS NO AMBIENTE AQUÁTICO – DA PRODUÇÃO AO TRATAMENTO DA ÁGUA: UMA REVISÃO

Aline Belem Machado

Master's degree and Ph.D. student in Environmental Quality from Universidade Feevale – Novo Hamburgo (RS), Brazil.

Luciane Rosa Feksa

Master's degree and Ph.D. in Biological Sciences from Universidade Federal do Rio Grande do Sul (UFRGS) – Porto Alegre (RS), Brazil.

Daniela Montanari Migliavacca Osorio

Ph.D. in Ecology from UFRGS – Porto Alegre (RS), Brazil.

Daiane Bolzan Berlese

Ph.D. in Biochemical Toxicology from Universidade Federal de Santa Maria – Santa Maria (RS), Brazil.

Endereço para correspondência: Aline Belem Machado – ERS-239, 2755 – Vila Nova – CEP: 93525-075 – Novo Hamburgo (RS), Brazil – E-mail: linebmachado@hotmail.com

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ABSTRACT

Investments in nanotechnology are increasing together with its application in daily products. The use of nanomaterials leads to their release in the environment and the contamination of rivers, which can cause toxicity to the aquatic biota and human beings. Nanomaterials are present in rivers of several countries. However, the detection of nanomaterials in river samples is difficult, so probabilistic methods are being developed to determine their concentration in aquatic environments. Fortunately, water treatments have proven to be effective in removing these nanomaterials. Therefore, the present study aimed to describe the many pathways that nanoparticles can follow from their production to their final destination, along with their possible detection and toxicity, based on the search of manuscripts from ScienceDirect, Wiley Online Library, and *Periódicos Capes* databases.

Keywords: analytical methods; aquatic systems; nanomaterials; toxicity; water treatment.

RESUMO

Os investimentos em nanotecnologia estão crescendo e, juntamente com eles, sua aplicação em produtos de uso diário. O uso de nanomateriais implica em sua liberação no meio ambiente e na contaminação do rio, o que pode causar toxicidade para a biota aquática e para os seres humanos. A presença de nanomateriais em rios ocorre em diferentes países. Entretanto, a detecção de nanomateriais em amostras de rios é difícil, portanto métodos probabilísticos estão sendo desenvolvidos para determinar a concentração de nanomateriais em ambientes aquáticos. Felizmente, os tratamentos de água estão demonstrando eficácia na remoção desses nanomateriais. Portanto, o objetivo do presente estudo foi descrever os diversos caminhos que as nanopartículas podem ter desde sua produção até seu destino final, juntamente com sua possível detecção e toxicidade, baseado na pesquisa de manuscritos nas bases de dados da Science Direct, Wiley Online Library e Periódicos Capes.

Palavras-chave: métodos analíticos; sistemas aquáticos; nanomateriais; toxicidade; tratamento água.

INTRODUCTION

Nanotechnology involves the manipulation of materials within the nanometer size scale between 1 and 100 nm (HANNAH; THOMPSON, 2008; LU; ASTRUC, 2018); however, according to Maurice and Hochella (2008), nanoparticles are those that present at least one nanoparticle with a dimension lower than 100 nm, including spherical, tubular, or irregularly-shaped particles. Nanoparticles have a high surface area to volume ratio and unique physical and chemical properties (GRACA *et al*., 2018).

Due to its great potential, the investments in nanotechnology have been increasing together with the worldwide development in scientific and industrial scale (ASZTEMBORSKA *et al*., 2018). The study of this technology started in 1959 with Richard Feynman's lecture entitled "There's plenty of room at the bottom" given at the Annual American Physical Society meeting (SAVOLAINEN *et al*., 2010).

Engineered nanomaterials are applicable to different kinds of products and fields, such as cosmetics, medicine, engineering, electronics, and environmental protection. However, all these applications result in the release of nanomaterials into the environment and, consequently, in the exposure of organisms to them (QUIK *et al*., 2010). Moreover, sewage and industrial discharge are the main release pathways of engineered nanoparticles. Thus, wastewater treatment plants are essential for controlling the release of these nanoparticles into the environment, such as surface waters through effluent discharge and land through sewage sludge disposal (HOU *et al*., 2012).

Therefore, this review aimed to discuss nanotechnology from different points of view, including its application, release, and the consequent impact on the environment and aquatic biota, as well as the different methodologies that can detect it and possibly remove it from water.

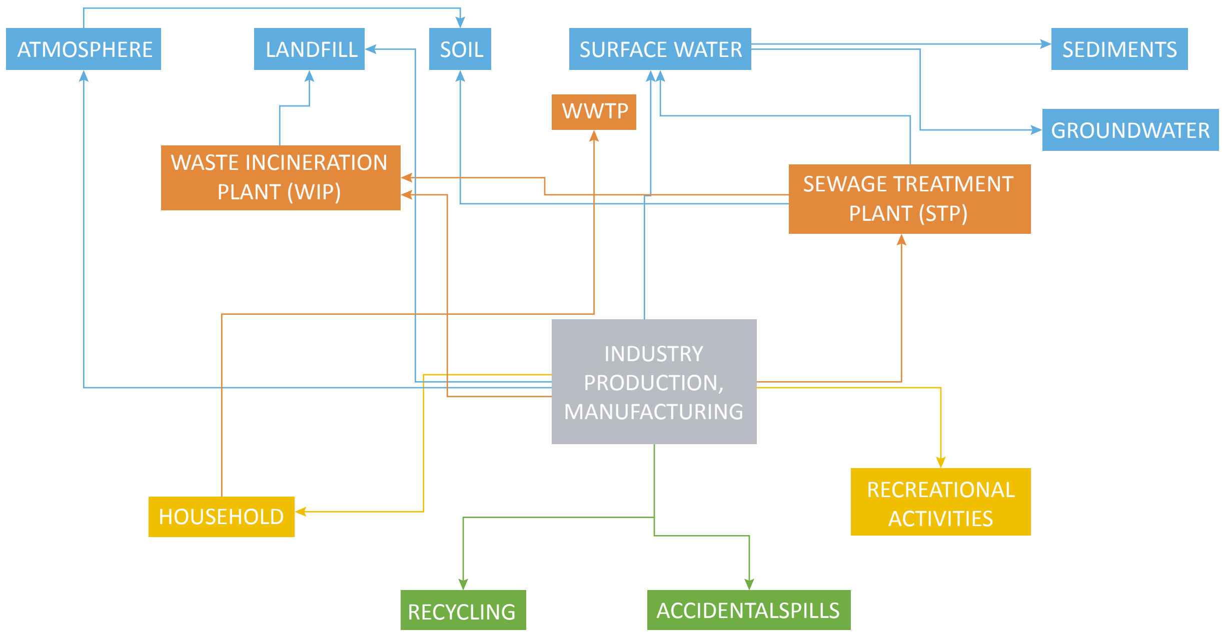
The present review was based on the investigation of manuscripts about the application, detection, water contamination, toxicity, and water treatment related to the production and use of nanoparticles. The review was performed by searching articles from ScienceDirect, Wiley Online Library, and *Periódicos Capes* databases, using the following keywords: analytical methods, nanomaterials, river basin, toxicity, and water treatment.

NANOTECHNOLOGY APPLICATIONS AND CONSEQUENT RELEASE

Nanotechnology can be applied to several kinds of products. Some of them — such as fabrics, personal care items, and food, which contain engineered nanoparticles, including silver (Ag), titanium dioxide (TiO_2), and silica (Si) — can have an easier path to enter the environment, since they can be washed down drains because of their household use (PETERS *et al*., 2018). In addition to the variety of products that contain engineered nanoparticles, such as those mentioned above and also sunscreens, detergents, paints, printer inks, and tires, accidental spills during the manufacturing and transportation, wear and tear, and their final disposal increase the release of these substances into the environment (NAVARRO *et al*., 2008). Figure 1 shows the different pathways that nanoparticles can follow from their production to their final destination.

Many different types of nanoparticles are widely used in cosmetics and sunscreen products, and their consequent disposal into the environment makes rivers and wastewater treatment plants to act as reservoirs of these substances, which can subsequently affect human health through tap water consumption (CHANG *et al*., 2017).

Research performed in 2013 revealed that the production of different kinds of engineered nanoparticles would reach around 350,000 tons by 2016 (GOSWAMI *et al*., 2017). This finding can be attested by the increase in products that contain nanoparticles in their composition. In 2005, a website project named Nanotechnology Consumer Products Inventory (CPI) was created to register products that contain nanotechnology. At first, they listed a total of 54 products, and, by 2014, they had 1,814 products registered (VANCE *et al*., 2015). In 2019, by the time this article was written, the website reported 1946 products with nanotechnology divided into eight categories and 37 subcategories (CPI, 2019). Figure 2 presents the number of products available in 2019, according to the main categories.



Green lines: different destinations; yellow lines: consumer consumption and release fate; orange lines: water and sewage treatment and waste incineration; light blue lines: final destination; dark blue lines: final destination from surface water; WWTP: wastewater treatment plant. Source: adapted from Gottschalk *et al*. (2009) and Peters *et al*. (2018).

Figure 1 – Possible pathways of nanoparticles since their production.

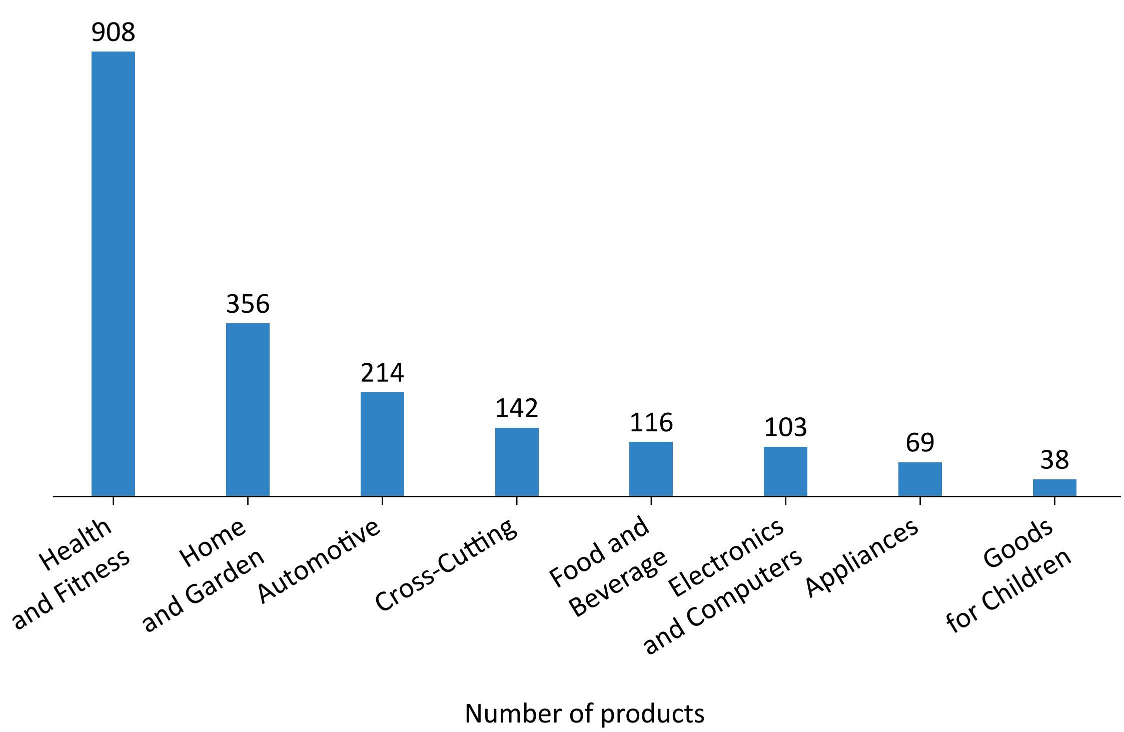


Figure 2 – Number of products available in 2019 divided into categories, according to the Consumer Products Inventory.

MEANS OF DETECTION/MODELING

Evaluating the potential risks of nanomaterials — derived from their production, application, and disposal — to the environment and human health requires suitable analytical procedures with reliable results about the fate and pathways of nanomaterials in the environment (LEOPOLD *et al.*, 2016).

The detection of nanoparticles in aquatic systems is difficult and scarce. This situation results from the lack of sensitivity and selectivity of analytical methods capable of detecting and characterizing these materials, especially in complex natural matrices in which traditional methodologies must be modified in an attempt to detect nanoparticles (VON DER KAMMER *et al.*, 2012). Von der Kammer *et al*. (2012) conducted an extensive review regarding this issue.

However, the analysis of nanomaterials in the environment can be quantified based on their mass, volume, or particle number. Qualitative analysis can sometimes identify the difference between engineered and natural nanoparticles according to their chemical composition and, along with the determination of particle size distribution, is very important for data interpretation (LEOPOLD *et al.*, 2016). Natural nanoparticles are formed by natural processes through chemical, photo-chemical, mechanical, thermal, and biological pathways. Human activities such as mining can also generate them spontaneously. Engineered as well as natural nanoparticles are formed by the same synthetic principles, which can occur by bottom-up or top-down approaches (SHARMA *et al.*, 2015). The bottom-up principle consists of obtaining a final material through its construction from smaller particles (AGHARKAR *et al.*, 2014). On the other hand, the top-down principle involves making a small final material from something larger (TOUR, 2014). Also, other parameters are relevant to analyze, such as metal speciation, particle shape, surface area, surface charge, surface functionality, nature, stability, and coating structure (LEOPOLD *et al.*, 2016).

Single particle inductively coupled plasma mass spectrometry (SP-ICP-MS) has proven to be a reliable method for detecting nanoparticles in aquatic media. Its advantages include the high sensitivity for environmental nanoparticles in relation to their size, size distribution, and dissolved element concentration (DONOVAN *et al.*, 2016).

However, analytical methods for detecting nanoparticles in water are sometimes difficult to reproduce. Based on this information, some authors (MUELLER & NOWACK, 2008; GOTTSCHALK *et al.*, 2009; DUMONT *et al.*, 2015) created a probabilistic method to determine the concentration of a certain nanoparticle in the environment. This modeling of predicted environmental concentrations (PEC) is usually necessary and a valuable replacement for measurement studies (GOTTSCHALK *et al.*, 2009). The modeling performed by Gottschalk *et al*. (2009) was developed based on a probabilistic material flow analysis approach. They used different compartments to calculate better the probable concentration of a certain nanoparticle, including:

- environmental: water, air, soil, sediment, and groundwater;
- technical: production, manufacturing and consumption, sewage treatment plant (STP), waste incineration plant (WIP), landfill, and recycling processes.

The derivations of the sizes of air, water, soil, and sediment were also used to calculate the concentrations of engineered nanoparticles in these compartments.

This same study took into account the life cycle and the different release pathways of engineered nanoparticles and grouped similar life cycles together. Release pathways depend on the engineered nanoparticle-containing product, including the following assumptions:

- glass and ceramic have all their nanoparticles released into the environment;
- cosmetics, coatings, and cleaning agents, as well as dietary supplements present major release of nanoparticles into the environment;
- paints have their nanoparticles disposed of in the sewage treatment plant (STP), landfill, soil, and/or surface waters (GOTTSCHALK *et al.*, 2009).

In a similar study, Dumont *et al*. (2015) developed the Global Water Availability Assessment (GWAVA) model, analyzing whether this model was capable of simulating the concentrations of nano silver (Ag-nano) and nano zinc oxide (ZnO-nano) released into surface waters. Unlike the Gottschalk model, Dumont's also considered space and time; for example, the spatial variability in population density and temporal variability in river discharge. GWAVA simulates the river discharge and the number of some hydrological conditions, such as lake water volumes and human water abstractions. One of the equations includes the area-specific load of engineered nanoparticles in surface water through sewage effluent, assuming that households are the only source of Ag-nano and ZnO-nano in the sewage effluent. However, the authors concluded that the estimated concentrations were lower than those of other studies found in the literature, which can be justified by the differences in modeled regions, assumed production volumes, and market penetration factors.

PRESENCE OF NANOPARTICLES IN WATER

Population growth and waste disposal from industries have caused a major problem in the aquatic systems (COSTA *et al*., 2014). The anthropogenic materials, which include nanoparticles, released into aquatic environments depend on the volume of industrial production and on how these materials are used (TROESTER; BRAUCH; HOFMANN, 2016). Engineered nanomaterials can contaminate the environment in any stage of their life cycle, such as production, use, and disposal (PETERS *et al*., 2018). ZnO and cerium dioxide (CeO_2) are two of the most used nanomaterials, being present in items such as personal care products, paints, and catalysts. Consequently, they are released into river basins through wastewater or runoff (DONOVAN *et al*., 2016). Table 1 presents some results of analytical and model determinations of nanoparticle concentrations in rivers.

The potential for environmental and human exposure to engineered nanoparticles depends on the amount of these materials in the environment, which in turn have their effect based on their behavior and fate regarding the adsorption, accumulation, persistence, aggregation, and mobility in different environmental media (GAO *et al*., 2013). The fate of nanomaterials in aqueous systems is subject to their solubility or dispersibility, interactions between the nanomaterial and natural or

Table 1 – Summary of nanoparticles analyzed in the environment and the predicted environmental concentration (PEC) found in the literature.

Location	n-Ag (µg/L)	n-CeO_2 (µg/L)	n-TiO_2 (µg/L)	n-ZnO (µg/L)	Method	Matrix	Reference
Netherlands	0.025	0.052			Analytical	Surface water	PETERS *et al*., 2018
Netherlands			0.6		Analytical	Sludge – WWTP	MARKUS *et al*., 2018
Netherlands			0.13		Analytical	Influent – WWTP	MARKUS *et al*., 2018
USA		< 0.10*		1.11	Analytical	Source water – DWTP	DONOVAN *et al*., 2016
Europe	0.58 – 2.16		0.012 – 0.057	0.008 – 0.055	Model	Surface water	GOTTSCHALK *et al*., 2009
USA	0.088 – 0.42		0.002 – 0.010	0.001 – 0.003	Model	Surface water	GOTTSCHALK *et al*., 2009
Switzerland	0.555 – 2.63		0.016 – 0.085	0.011 – 0.058	Model	Surface water	GOTTSCHALK *et al*., 2009
Switzerland	0.0023			0.36	Model	Surface water	DUMONT *et al*., 2015

µg/L: concentration of nanoparticles in aqueous media; WWTP: wastewater treatment plant; DWTP: drinking water treatment plant; *below the detection limit.

anthropogenic chemicals in this environment, and biological and abiotic processes (BRAR *et al*., 2010).

Aggregation and dissolution are related to nanoparticle stability in aqueous media and must be considered. Some factors, such as ionic strength, pH, and organic matters, can affect the aggregation and dissolution of nanoparticles (DONOVAN *et al*., 2016). The bioavailability and transportation efficiency of nanoparticle aggregates are associated with aggregation and sedimentation when released into the environment. Also, water chemistry strongly influences the stability of nanoparticles (PENG *et al*., 2017).

However, nanoparticles can also be found in different types of water besides river basins. In a study performed by Graca *et al*. (2018), they were able to detect different nanomaterials in seawater from natural sources. They also investigated the influence of seasons on the number of nanoparticles in seawater. The authors identified environmental silica nanofibers of 15 nm, probably from remains of flagellates; manganese and iron oxide nanofibers, possibly from microbes; and pyrite nanospheres of 55 nm, potentially formed in anoxic sediments. Nanoparticles increased in water samples in June compared to November. This fact can be explained by the seasonal variation of flagellates found in the study, in which Summer (June) presents the highest concentration of flagellates in comparison to Autumn (November) (GRACA *et al*., 2018). The finding demonstrates the effects that different seasons can have on the concentration of nanoparticles.

POSSIBLE TOXICITY TO AQUATIC BIOTA

Water is an important transfer and fate medium for engineered nanoparticles. Human health is related to water safety, and the potential human impact of metallic nanoparticles leaching into aquatic environments is attracting attention (GAO *et al*., 2013). The toxicity to aquatic ecosystems is mainly due to changes in water quantity and quality, as well as in the physical habitat and biological components, the so-called pressures. Chemicals with nanoparticle size are some of the materials responsible for the toxicity of aquatic organisms (GRIZZETTI *et al*., 2016). The properties of nanoparticles, such as the high surface area to volume ratio and small size, give them unique characteristics and applications when compared to bulk materials. For this reason, their bioavailability and, consequently, their toxicity can increase (SOUSA; CORNICIUC; TEIXEIRA, 2017). Due to the small particle size and corresponding enhanced activity, organisms can have more interaction with engineered nanoparticles than large particles (GOSWAMI *et al*., 2017).

The release of nanoparticles into the environment through water can be very concerning given the potential for contamination, as they are capable of cotransporting sorbed contaminants into surface and groundwater, and also because they are nanoparticles themselves (CHEKLI *et al*., 2015). Some properties, such as the charge of different metal ions (Ag^{+}, Cu^{2+}, and Al^{3-}) and the adsorption efficiency of engineered nanoparticle, can affect the bioavailability of these materials and their consequent eco-toxicological effects (GOSWAMI *et al*., 2017).

An important question concerning nanoparticle toxicity is whether this type of material is more dangerous to organisms than the corresponding bulk material. In order to evaluate this toxicity, Xiong *et al*. (2011) analyzed the acute toxicity of ZnO-nano and TiO_2-nano on zebrafish (*Danio rerio*) and compared it to the effects caused by the corresponding bulk materials. The acute toxicity of TiO_2-nano, ZnO-nano, and bulk ZnO demonstrates a dose dependency. The highest concentration of TiO_2-nano studied (300 mg/L) was able to cause 100% mortality. However, bulk TiO_2 showed no acute toxicity to zebrafish. The concentration of 30 mg/L of ZnO-nano and bulk ZnO led to 100% mortality. Their results suggest that TiO_2 toxicity is subject to particle size; however, ZnO does not exhibit this characteristic, demonstrating that ZnO depends on chemical composition.

PRESENCE AND REMOVAL OF NANOPARTICLES IN WATER TREATMENT PLANTS

Anthropogenic activities are some of the main pressure generators. These pressures can affect the biodiversity and the status of aquatic systems. Any change in these systems can alter their economic value.

The relationship between these activities and the ecological status needs to be understood in order to devise cost-effective measures aimed at achieving good ecological status for water bodies (GRIZZETTI *et al*., 2016).

Conventional water treatment consists of coagulation, flocculation, sedimentation, filtration, and disinfection (SOUSA; CORNICIUC; TEIXEIRA, 2017). With the purpose of analyzing the removal of TiO_2-nano with conventional drinking water treatment, Sousa, Corniciuc and Teixeira (2017) evaluated four synthetic waters and different concentrations of TiO_2-nano. They were able to prove that the sedimentation of TiO_2-nano depends on pH, as at a pH of 5.4, TiO_2-nano settled faster than in waters with a different pH. This study also revealed that titanium removal efficiency was around 80% when coagulant was not added to water. In conclusion, they proved that TiO_2-nano can be removed from surface water through conventional water treatment.

Nanoparticles and biofilms can interact through three different processes: transportation of nanoparticles to the vicinity of the biofilm; deposition of the nanoparticle in the biofilm surface; and migration of nanoparticles in the inner area of the biofilm. Nonetheless, different characteristics can interfere with these interactions, such as nanoparticle characteristics, physicochemical and biological composition of the biofilm, and environmental parameters, including water chemistry, flow, and temperature (IKUMA; DECHO; LAU, 2015). Besides, different weather conditions can affect the status of nanoparticles in wastewater treatment. For example, during dry water conditions, fulvic acids can promote the uptake and bioaccumulation of silver nanoparticles in biofilms, and the sewer biofilm can act as a temporary sink to these nanoparticles and accumulate them. In contrast, during rainy conditions, this biofilm can work as a source of Ag-nano and release it into the environment. Therefore, during these weather conditions, the nanoparticles can bypass the wastewater treatment plant and be released directly into aquatic systems during stormwater discharge (KAEGI *et al*., 2013).

Also, seasons can affect nanoparticles regarding their release into municipal wastewater streams, given that they can be incorporated into functionalized products, which subsequently have their use related to different seasons and their disposal dependent on climate conditions. For instance, sunscreen and cosmetics with sun protection factor are used during diurnal solar radiation, especially in Summer (CHOI *et al*., 2018).

Season-related changes led Choi *et al*. (2018) to study the concentration of engineered nanoparticles (TiO_2-nano and ZnO-nano) in a wastewater treatment plant, which included primary clarifier, aeration basin, secondary clarifier, and chlorination, during twelve months aiming at analyzing the relationship between the consumption of nanoparticle-containing products and the concentration of nanoparticles in wastewater. They collected wastewater samples from influent, effluent, sludge, and sedimentation tanks. The results revealed a higher inflow of TiO_2-nano and Zn-nano concentration during Summer and Winter, probably due to the use of personal care products under high or low temperatures. Also, the general inflow of TiO_2-nano was higher than that of ZnO-nano, indicating greater use of TiO_2-nano-related products in comparison to ZnO-nano-related products. In conclusion, the findings demonstrated that nanoparticle concentrations vary seasonally, and that temperature is an important factor for the engineered nanoparticle sorption into sludge particulates.

Sometimes, wastewater treatment plants do not fully remove TiO_2-nano; thus, a great amount of this substance can reach the environment and natural waters (CHEKLI *et al*., 2015). However, in the research performed by Wang, Westerhoff, and Hristovski (2012), they analyzed the TiO_2-nano removal from a wastewater treatment based on sequencing batch reactors with aerated and mixed samples. The reactors were seeded with bacteria culture from the sludge of an urban wastewater treatment plant, which had a retention time of approximately six days. The nanomaterials were added to the feed solution and subsequently to the sequencing batch reactor. The aeration time was approximately 8 hours. They were able to remove around 70% of TiO_2-nano from wastewater with the presence of biomass. Therefore, in the absence of biomass, these nanoparticles were not removed due to aggregation and sedimentation, factors that belong to the abiotic mechanisms mentioned above. Briefly, they were able to remove TiO_2-nano using a biological wastewater treatment plant in lab scale.

Another highly studied nanomaterial is Ag-nano. Numerous products have this substance, such as clothing, paints, bandages, and food containers. The consumption of these products results in the release of these

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nanomaterials into sewer systems and, consequently, into municipal wastewater treatment plants. For this reason, Hou *et al*. (2012) evaluated the removal of Ag-nano in a wastewater treatment plant from Beijing that uses an activated sludge process involving primary clarification, aeration, secondary clarification, and treatment. The reactors were operated for 15 days, with a hydraulic residence time of 12 hours, and 10 hours of aeration followed by 2 hours of settling. The results demonstrated that, in the primary clarification process with an influent concentration of 269 mg/L of suspended solids, most of Ag-nano (94%) remained in the upper layer of wastewater, which means that the first clarification was not able to remove Ag-nano. However, when aeration and secondary clarification processes were implemented, the Ag-nano was completely removed from the wastewater.

In a similar study performed in field-scale, Kaegi *et al*. (2013) evaluated the fate of Ag-nano in an urban wastewater system. They found that Ag-nano was transported through the entire distance of 5 km in a sewer system without deposition. When evaluating efficiency, they verified that nanoparticle removal was around 99%, suggesting that they could be incorporated/attached to flocs of activated sludge. With this result, the authors assumed that a great number of nanoparticles that enter the wastewater treatment plant would be incorporated in the sludge and, consequently, removed from the wastewater stream. Nevertheless, the wastewater sludge can still contain nanoparticles after treatment, and if spread to agricultural lands to be used as biosolids, it can potentially release nanoparticles into groundwater, subsurface waters, and soil (BRAR *et al*., 2010).

This scenario reveals the anthropogenic contamination of nanomaterials into sewage, which, if not properly treated, can be released into rivers basins and contaminate aquatic organisms as well as humans, affecting their health in proportions that sometimes cannot be measured.

CONCLUSIONS

The production of nanomaterials is growing together with the release of these materials in aquatic environments. Nanomaterials are being detected in rivers, which can result in toxic effects on the biota and human health. However, conventional water and sewage treatments have proven to be effective in removing these nanomaterials. In conclusion, the application of nanotechnology in daily products is increasing the presence of nanomaterials in different sources of water, so water treatments should improve their removal processes to reduce the consequences for the health of animals and humans.

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