CHARACTERIZATION OF NANOPARTICLES IN THE LEACHATE OF CONSTRUCTION AND DEMOLITION WASTE LANDFILLS

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The past two decades have been characterized by the rapid emergence of nanotechnologically-enabled materials and products. The production, use and disposal of these products will definitely lead to the release of engineered nanomaterials into air, soil and aquatic systems.

The presence of nanomaterials in the environment can have significant environmental and toxicological consequences. Therefore, the waste industry is expected to establish new practices to limit uncontrolled release and dispersion of these materials. For this to happen, it is essential to study the fate and behaviour of these materials in complex systems, particularly waste disposal systems.

In this context, this work aimed to detect manufactured silica nanoparticles and characterize the structures found in the simulated leachate of construction and demolition (C&D) waste landfills using scanning electron microscopy (SEM) coupled to energy-dispersive X-ray spectroscopy (EDS) for elemental analysis.

Unfortunately, the manufactured silica nanoparticles added to the experimental C&D waste columns were not detected in the SEM images. This might be the result of changes in their size and shape during sample handling, of the heterogeneity of the samples, or of the absence of these particles from the analysed sample spots. Moreover, the analyses revealed the presence of several different structures. However, these analyses did not allow the identification of these structures and of their origin.

Hence, the results from this experimental work indicate that there is a need to revise the protocol used to prepare the samples and also to investigate other methods of detecting specific engineered nanoparticles in such heterogeneous samples.

Keywords: engineered nanomaterials; nanoparticles; construction and demolition waste landfills; leachate; scanning electron microscopy; energy-dispersive X-ray spectroscopy

INTRODUCTION

The past two decades have been characterized by the rapid emergence of nanotechnologically-enabled materials and products [1,2]. The fact that more than 1300 consumer products containing nanomaterials (NMs) and nanoparticles (NPs) are already available on the market [3] is an evidence of this trend. Furthermore, this number seems to be growing steadily [4]. In fact, estimates show that, by 2014, more than 15% of all products in the global market will be to some extent nanotechnologically enabled [1].

At the moment, applications of NMs and NPs in consumer products include paints and sunscreens with TiO$_2$ and ZnO NPs for their sterilizing and anti-fouling properties, textiles and paints with Ag NPs due to their antibacterial properties, and concrete with SiO$_2$ and Fe$_2$O$_3$ NPs to increase its strength and durability, among many others [1]. Furthermore, nanotechnologies hold great potential in reducing the volume of waste, in using resources more sparingly, in the reduction of industrial contamination, in the treatment of water to provide potable water and in improving the efficiency of energy production and usage [5,6]. However, the production, use
and disposal of engineered nanoparticles (ENPs) will definitely lead to their release into air, soil and aquatic systems [5]. The presence of nanomaterials in the environment may have significant environmental consequences [4]. Studies on the toxicity of NMs suggest that these materials pose varied levels of potential risks both to humans and to the environment [2]. In this sense, the nanotechnology boom raises an important question: what is being done to address the environmental and toxicological risks of nanomaterials in the environment [1]?

The waste industry plays an important role on preventing contaminants from reaching the environment. Hence, it is expected that this industry will establish new and sound waste management protocols and practices to limit uncontrolled release and widespread dispersion of ENPs into ecological systems. For this to happen, it is essential to have data on the fate, behaviour and interactions of NMs in complex systems, particularly waste disposal systems. Yet, only a few studies have focused on this topic and so there is paucity of scientific data. Moreover, there is also a lack of data on the actual concentrations of NMs being produced and released, making it difficult to do accurate quantitative risk assessments. This poses as a challenge for industries, waste management specialists and regulators to adequately manage waste streams containing NMs [2].

In order to understand the environmental fate and behaviour, as well as toxicity of ENPs, it is important to first be able to detect and characterize them in complex matrices. However, even though there are numerous techniques available for this purpose, these tools have severe limitations, especially when applied to more complex samples. Therefore, new techniques should be developed and existing ones should be improved in order to acquire reliable data [7,8].

In this context, this study aims to help in finding answers to some of the problems underlined above.

CASE STUDY
Considering the applications of ENPs in construction materials, it can be concluded that SiO₂, TiO₂ and Fe₂O₃ NPs from concrete applications and ZnO, TiO₂ and Ag NPs used in paints will likely end up in construction and demolition (C&D) waste landfills. Although NPs in concrete are in the interior of the bulk material, the demolition and crushing of the construction materials is likely to expose the nanoparticles at the surface of the obtained fragments [2]. These fragments are then likely to suffer further mechanical stress and abrasion during the deposition of the materials and subsequent rearrangement of the waste in the landfill, which will probably lead to the release of NPs from the waste materials [2,9]. Consequently, NPs can possibly be washed out of landfills either by the generated landfill gases or by the water that percolates through the waste (leachate).

Once NPs are released into the leachate, the leachate composition will significantly influence their fate. C&D waste landfill leachate is composed of dissolved organic matter (much lower concentration than municipal solid waste landfill leachate), inorganics, heavy metals and other trace compounds [9,10]. The composition of the leachate has been shown to significantly influence the aggregation and deposition of nanoparticles and, consequently, their mobility in the waste matrix. Furthermore, the composition of the leachate changes over time as a result of physical, chemical and biological reactions/transformations [9]. Hence, there is a need to investigate the fate and behaviour of ENPs in the leachate of C&D waste landfills.

In this sense, this work aimed to characterize the NPs found in the leachate of C&D waste landfills, as well as understanding the behaviour of manufactured SiO₂ nanoparticles in the waste matrix.

Silica (SiO₂) was chosen for the purpose of this study since it is greatly used in the construction industry to improve the strength and durability of concrete and in fire-resistance glass.

In silica materials, each silicon atom is in tetrahedral coordination with 4 oxygen atoms. In amorphous silica, the tetrahedra are randomly interconnected. The structure of the silica nanoparticles depend on the synthesis methods used for their preparation, as do the physico-chemical properties [11]. For the purpose of this work, amorphous silica
nanoparticles were produced by the Stöber method. This method consists of hydrolysis and condensation reactions of tetraethylorthosilicate (TEOS). The intermediate silicon tetrahydroxide is generated by hydrolysis first, followed by the condensation reaction to gradually form a three-dimensional net structure [12]. The silica nanoparticles produced by this method have OH groups at the surface, which make them polar and hydrophilic [13]. Thus, a coating of the particles with silane was performed in order to expand their stability and increase their lifetime in water samples. The silanization reaction converts Si-OH groups into non-polar (hydrophobic) Si-O-SiR groups. These groups are stable towards hydrolysis and will work as an organic protective coating for the particles, protecting their surface against water [13].

MATERIALS AND METHODS

Synthesis and coating of SiO$_2$ nanoparticles

To prepare SiO$_2$ nanoparticles with a diameter of 100 nm using the Stöber method, 93.755 mL of denatured ethanol with a purity of 99.8% acquired from Carl Roth were mixed with 15.12 mL of Milli-Q water (18.2 MΩ cm, TOC <3 ppb) and 1.597 mL of ammonium hydroxide solution (30-33% NH$_3$ in H$_2$O), provided by Sigma-Aldrich. Agitation was then started by means of a magnetic stirrer and 6.245 mL of tetraethylorthosilicate (TEOS: C$_5$H$_{12}$O$_5$Si) from Sigma-Aldrich were added. Mixing was ceased after approximately 2 hours.

45 mL of the resulting suspension were centrifuged at 5000 rpm for 60 minutes in a 5804R centrifuge from Eppendorf and the pellet was washed three times with 45 mL of ethanol. The final supernatant was removed and the deposited particles were frozen by immersion in an ethanol bath for 30 minutes and subsequently dried overnight in a lyophilizer (Labconco, model freezone6) at 51 ºC. The obtained powder was then kept in a cold room for approximately 24 hours to remove the moisture from the SiO$_2$ nanoparticles. Washing steps were repeated as above. As before, the moisture from the SiO$_2$ nanoparticles was removed overnight in the lyophilizer.

Experimental set-up and test description

Column experiments, based on the European standard up-flow percolation test (CEN/TS 14405) [14], were performed in order to obtain the leachate from a simplified inorganic construction and demolition waste matrix. For the purpose of these experiments, seven columns with 9.4 cm of diameter and 15 cm of working height were used. Also, a MasterFlex peristaltic pump from Cole-Parmer was used to pump the water through the columns. The waste matrix was composed of 49% bricks, 49% cement and 2% gypsum. The components of the matrix were crushed and sieved, in order to have a waste sample with fragment sizes between 4 and 10 mm as required by the CEN standard. After the saturation, the columns were packed with the sample, making sure to compact the waste well during this stage. Approximately, 1.34 kg were added to each column, measured using a precision balance. Afterwards, according to the CEN, the columns must be filled with deionized water using hydrostatic pressure and left equilibrating for a period of three days.

For the purpose of this study, two of the seven columns (columns 1 and 2) were equilibrated with deionized water, to act as control samples, and five columns were equilibrated with a suspension of 100 mg L$^{-1}$ of SiO$_2$ nanoparticles to ensure that the particles would be inside the sample. After the equilibration period, the multichannel pump was connected to the seven columns. The collection of the leachate was also done according to the CEN standard, respecting the liquid to solid ratios (L/S) given in Table 1. The leachate was recovered in 1 L glass bottles and then transferred to plastic collection flasks as soon as the quantity of deionized water defined by the table was passed through. The leachate samples were then stored at 4 ºC while waiting for handling and analysis. The volumes to be collected from each column...
according to the mean value of dry mass of waste found in the columns can also be observed on Table 1.

Table 1 – Table for collection of samples according to the liquid to solid ratios of CEN/TS 14405 [14].

<table>
<thead>
<tr>
<th>Fraction</th>
<th>Fraction volume (L)*</th>
<th>Volume to be collected (mL)</th>
</tr>
</thead>
<tbody>
<tr>
<td>F1</td>
<td>(0.1 ± 0.02) \times m₀</td>
<td>113.1 ± 22.6</td>
</tr>
<tr>
<td>F2</td>
<td>(0.1 ± 0.02) \times m₀</td>
<td>113.1 ± 22.6</td>
</tr>
<tr>
<td>F3</td>
<td>(0.3 ± 0.05) \times m₀</td>
<td>339.5 ± 56.6</td>
</tr>
<tr>
<td>F4</td>
<td>(0.5 ± 0.10) \times m₀</td>
<td>565.9 ± 113.2</td>
</tr>
</tbody>
</table>

* m₀ is the dry mass of sample in the column.

After the starting of the pump, it was realised that two of the columns were exhibiting unstable flow, probably due to problems related to the pump or the set-up of the columns. Therefore, the leachate fractions for columns 1 and 5 were not collected.

Additionally, the effect of the equilibration time on the particles was tested on two extra columns that were prepared with the same matrix, one with deionized water and the other with the nanoparticle suspension. After the three days of equilibration, the water was removed from the columns and solid samples were collected from the columns, in order to check whether the nanoparticles had adsorbed to the matrix. From this point forth, these will be referred to as equilibration tests.

Preparation of samples for characterization

Since the volume of the sample fractions recovered from the column was fairly large, the particles were too diluted in the liquid and, therefore, there was a need to pre-concentrate the samples. Also, these samples contained larger particles released from the matrix that had to be filtered out before the pre-concentration steps.

For this reason, the solutions were first filtered through a 3 μm cellulose nitrate filter from Sartorius to remove larger particles, mainly dust released from the brick fragments. Afterwards, the filtered solution was sonicated in an ultrasonic bath for 5 minutes, which was followed by a second filtration through a Durapore membrane with 0.45 μm pore size from Milipore. Both filtrations were performed in a Milipore filtration unit with the help of vacuum suction.

Following the filtration steps, the samples were placed on a VMS-C7 hot plate stirrer from VWR for evaporation of the excessive water. Both the stirring and a temperature of 80 ºC were kept constant during the process. The samples were concentrate to a final volume of 15 mL for fractions 1 and 2, and to 25 and 50 mL, for fractions 3 and 4, respectively.

This process took approximately 2 to 10 hours, depending on the volume of the samples.

This step revealed the presence of a substantial amount of gypsum that had been dispersed in the solution and, thus, had not been visible. Hence, in order to dissolve the gypsum, a solution of 8% of ethylenediamine tetraacetic acid (EDTA) disodium salt (Na₂C₁₀H₁₄N₂O₈·2H₂O, ≥99%) from GERBU Biotechnik GmbH in Milli-Q water was prepared. This solution was then mixed with the concentrated samples and the gypsum was dissolved immediately. The amount of EDTA added was just enough to achieve ready dissolution of the gypsum.

The mix was then centrifuged at 5000 rpm for 1 hour, the supernatant was discarded and the pellet was resuspended in 1.5 mL of the EDTA solution and centrifuged at 15000 rpm for 15 minutes in a 5424 centrifuge from Eppendorf. The pellet was then washed in 1.5 mL of water for three times using the same centrifuge in order to remove any traces of EDTA solution. Finally it was washed another three times with the same amount of ethanol. After the final washing, the pellet was resuspended in 20 μL of ethanol for the subsequent SEM analysis.

Analytical and characterization techniques

FTIR

FTIR spectra were recorded with an Alpha-P spectrometer from Bruker. All samples were analysed in triplicate.

pH and conductivity

Conductivity and pH were measured for every fraction immediately after the collection from the columns. The LF 318 conductivity meter from Wissenschaftlich-Technische Werkstaten GmbH (WTW) was calibrated for 25 ºC. Hence, there was a need to convert all the values to 20 ºC. The pH values were measured using a pH 531 meter also from WTW.

SEM and EDX

The actual size of the obtained nanoparticles was determined using a scanning electron microscope (SEM). For this purpose, a droplet of the nanoparticles suspension was directly deposited in the SEM sample holder, which was covered by a thin carbon layer to improve conductivity.

The samples in the SEM holders were sputter coated with a gold layer in order to minimize the charging effects and enhance the resolution. The imaging was conducted using a FEI Inspect S50
instrument operating at 10 and 15 kV in high vacuum mode. The liquid samples from both the equilibration and column tests were analysed in the same way. For the examination of the solid samples from the equilibration tests, pieces of brick and cement were directly placed on the holder using conductive carbon cement from Plano GmbH to glue the samples. Since these samples were slightly moist, low vacuum mode was chosen for this analysis. While performing the visual characterization of the samples in the SEM, the elemental analysis of the samples was also obtained through energy-dispersive X-ray spectroscopy (EDS) coupled to the SEM. The software EDAX Genesis from EDAX was used to perform this analysis.

RESULTS AND DISCUSSION

Synthesis and coating of \( \text{SiO}_2 \) nanoparticles

As mentioned, silica nanoparticles were manufactured by the Stöber method and coated by silanization in order to improve their stability in water samples. FTIR spectra for both modified and unmodified nanoparticles were obtained, as well as for the pure silane used for the coating. Figure 1 shows the FTIR spectrum window from 3800 to 2600 cm\(^{-1}\), which gives a clear evidence of the surface modification.

![FTIR spectrum](image)

**Figure 1** – FTIR spectrum (2600-3800 cm\(^{-1}\)) for pure silane and for unmodified and modified silica nanoparticles.

A new peak is observed on the spectrum of the modified particles. This peak at 2990 cm\(^{-1}\) represents the C-H stretching of the \(-\text{CH}_2\) bonds and reveals that the silane functional groups were anchored to the silica after the silanization reaction. Finally, after confirming that the coating had been successful, the particles were observed under the SEM again to determine the final diameter of the particles, which was approximately 192 nm.

Equilibration tests

As mentioned before, two columns were set to equilibrate over a period of three days, one being equilibrated with deionized water and the other with a suspension of 100 mg L\(^{-1}\) of silica nanoparticles. After the equilibration period, the water was removed from the columns and brick and cement fragments from three different points in the column (bottom, middle and top) were collected in order to investigate whether these particles had adsorbed to the matrix over the equilibration period. Several silica particles were found on the surface of the cement fragment taken from the bottom of the column. In reality, as it can be seen in Figure 2A, a rather big cluster of aggregated particles was found. This results from the deposition of a cluster of nanoparticles that was not totally dispersed by sonication of the suspension added to the column. Nevertheless, since this aggregate was not washed out from the fragment when the water was removed from the column, it can be assumed that the silica particles can adsorb to the cement matrix.

Some spherical particles were also detected on the surface of the brick fragment taken from the bottom of the column (Figure 2B). These results suggest that the silica nanoparticles will also adsorb to this type of matrix.
**Column tests**

*Overview of pH and conductivity of fractions*

In the column leaching tests, the collection of the leachate fractions was done according to the CEN standard, respecting the liquid to solid ratios given in Table 1. Moreover, the pH and conductivity measurements are then illustrated in the graphics (Figures 3 and 4).

The pH values shown for fractions 1, 2 and 3 are typical for construction and demolition waste leachates [10] and are due to an equilibrium between calcite in the leachate and atmospheric CO₂. Regarding the decrease in pH values for fraction 4, it is possible that some biodegradation of traces of organics present in the matrix occurred during the collection and storage of the leachate, causing the formation of CO₂ and, consequently, this decrease in the pH value [15].

As for the values of conductivity of the first fraction, these are consistent with the values for leachates from pure gypsum. Therefore, the gypsum in the matrix is probably responsible for the initial conductivity of the samples. Moreover, the decrease in conductivity in the following fractions is probably due to dilution of the gypsum. Again, chemical analysis is needed to confirm if there is reduction in the concentration of gypsum in the samples.

Subsequently, the SEM-EDS analyses results for column 2, the control column, and column 3 are presented and discussed.

**SEM-EDS: Fractions 1, 2 and 4**

Figure 5, 6 and 7 show the SEM and EDS analyses for columns 2 and 3, for fractions 1, 2 and 4, respectively. These analyses show that mainly silica constitutes the samples. Nonetheless, the images show structures other than particles and the spectra suggest the presence of other components.
Figure 5 – SEM images and EDS spectra of samples for fraction 1 from columns 2 (left) and 3 (right).

Figure 6 – SEM images and EDS spectra of samples for fraction 2 from columns 2 (left) and 3 (right).

Figure 7 – SEM images and EDS spectra of samples for fraction 4 from columns 2 (left) and 3 (right).

**SEM-EDS: Fraction 3**

The images and spectra obtained for fraction 3 show very distinct results from the other fractions (Figure 8). During the preparation of this fraction for visualization under the SEM, the samples were left overnight in the EDTA solution. This led to the precipitation of magnesium carbonate (\(\text{MgCO}_3\)), as it can be observed in the SEM image and in the spectrum. Therefore, it was concluded that the EDTA solution had to be washed out of the samples as soon as the gypsum was dissolved.

Figure 8 – SEM image and EDS spectrum for fraction 3.
Discussion

The analysis of the liquid samples from the column tests failed to find the manufactured silica nanoparticles added to the columns. This might have resulted from several different reasons, which are presented below.

First, several studies have proven that pH 7 and the presence of magnesium and calcium ions affect the stability of aqueous silica nanoparticles dispersions, favouring the aggregation and even agglomeration (fusion) of particles [16]. Hence, considering that the leachate presented a pH value of 7 and a high concentration of calcium and magnesium ions, big aggregates and even some agglomeration of the silica particles might be expected.

Considering the facts above, it is possible that the aggregates/agglomerates deposited at the bottom of the column during the equilibration period and that they were not leached out in the collected fractions. Nevertheless, these aggregates would probably be carried in the water up-flow and would leach out in later fractions.

In the case that the aggregates did leach out in the collected fractions, it might have happened that the aggregates were not redispersed in the sonication step. As it was mentioned before, these aggregates were probably several micrometres wide, therefore, if they did not redisperse, it is very likely that they were filtered out during the sample preparation.

Secondly, the sample handling procedures, namely the sonication, might have affected the size and shape of the particles, meaning that they would no longer be recognizable in the heterogeneous samples obtained.

Thirdly, as stated by Dudkiewicz et al. (2011), location of individual nanoparticles in real environmental samples by electron microscopy “is very much like finding a needle in a haystack, due to time-consuming analysis of small sample volumes and the issues of representativeness and reproducibility, and probably also the wide range of natural nanostructures present in the sample”. In this sense, it is also possible that the desired nanoparticles were hidden by the other structures leached out from the column and that these particles were not on the spots of the sample that were analysed.

Finally, the results from the analysis of the solid samples from the equilibration tests proved that silica particles can adsorb to the components of the waste matrix. Unfortunately, these results did not show to what extent these particles adsorbed to the matrix. Nonetheless, the number of particles being leached out and, consequently, the chances of detection of these particles certainly decreased.

Although the manufactured silica particles were not found, results showed that particles of silica are naturally released from the waste matrix. This was first proven by the equilibration tests, where the sample from the control column showed a substantial amount of silica. In fact, since the concentration was so high, it can be concluded that silica particles and other structures are very easily released from the bricks and concrete matrix.

The spectra obtained for the samples from the column tests are in accordance with the results from the equilibration tests. Mainly silica is leached out in the fractions collected and the majority of the structures observed seem to be spherical particles. In general, these particles appeared in clusters and, again, it is hard to determine whether they were aggregated or fused together. Furthermore, it is not possible to establish if this phenomenon is just an artifact of the drying of the samples in the SEM holder.

A wide size range of these spherical nanoparticles was observed. Nanoparticles as small as 45 nm were found, together with particles with several hundred nanometres. As previously revealed, the bigger particles seem to have leached out mainly in the last fraction, which leads one to believe that these particles settled at the bottom of the column during the equilibration period. To some extent this supports the theory stated above that the aggregated manufactured nanoparticles might have deposited and that they would be leached out in later fractions.

Moreover, since most of the nanoparticles found seem to be originated from erosion of the matrix, it is possible that they will not pose a toxicological risk. Besides, as it was also mentioned, it is very hard to determine whether the silica particles found are just interacting with each other by weak, breakable bonds or are fused together. The difference in interaction will
determine the future behaviour and fate of the particles in the environment. Since fusion is irreversible, it is likely that these particles will remain aggregated once they enter the environment and will sediment. Although this poses a lower risk for microbial uptake, the aggregates can still be up taken by plants or ingested by animals. On the other hand, if the nanoparticles are not fused together, it is possible that they will separate due to chemical changes or to mechanical stress in the environment, and pose significant toxicological risks. In this sense, further testing on these particles is needed to understand the risks they may pose.

As highlighted above, the SEM and EDS analysis revealed some structures other than simple silica particles. The identification of these structures, as well as the determination of their origin, based on the analyses performed is not possible. Nonetheless, some studies have shown the presence of phyllosilicates in the leachate from waste landfills, originated from the waste matrix [17]. Some of the most usual phyllosilicates found are pyrophyllite \([\text{Al}_2\text{Si}_3\text{O}_{10}(\text{OH})_2] \) and biotite \([\text{K(Mg,Fe)}_2\text{AlSi}_3\text{O}_{10}(\text{OH})_2] \). Even though some EDS spectra show compositions that resemble these (Figure 7), it is not possible to determine the exact composition of these structures using only the EDS. Probably an XDR analysis would have to be performed for this purpose.

Furthermore, it is possible that the mineral structures which were found precipitated from solution in the samples, which would explain the presence of structures bigger than 450 nm [17]. However, a chemical analysis of the liquid samples, followed by speciation analyses, should be performed in order to determine which species would precipitate [18]. Nonetheless, this last analysis must be performed keeping in mind that precipitation can be an artifact of the sample drying.

Comparing the amount of material deposited on the SEM holder, it is possible to conclude that the samples from column 3 were more concentrated than the ones from column 2. This occurrence can also be observed by comparing the images from the SEM. This difference between the control column and the columns with the added silica particles leads to believe that the manufactured silica nanoparticles are, indeed, in the leachate samples analysed under the microscope. However, as it was stated before, the particles were not found in the images taken with the SEM. Furthermore, this disparity between columns was also perceived in the samples from the equilibration tests.

Considering the arguments posed initially in this chapter, the observed difference might be a result of changes in particle size and shape, due to chemical and physical stress during sample handling, or of the heterogeneity of the sample. Moreover, since the volumes of sample analysed at a time are quite small, it is possible that the particles were not present on the analysed spots of the samples.

The amount of silica particles in the samples also seems to decrease with the increase of the liquid to solid ratio – fraction 1 shows bigger amounts of silica than fraction 4 for all columns. This decrease of the concentration of silica is in accordance with the results obtained by Delay et al. (2007) on the study of the leachate of construction and demolition waste landfills. Also, it is possible to observe that in the last fraction, where less silica nanoparticles are present, more of the other structures are visible. In a way, this supports the hypothesis that the salts in solution precipitate in the samples.

**Conclusion**

Detection and quantification of ENPs in the environment, as well as their distinction from naturally occurring particles, is still very difficult to achieve and remains one of the highest priorities for the waste management industry.

In this context, this work aimed to detect manufactured silica nanoparticles in the simulated leachate of a construction and demolition waste landfill using a scanning electron microscope coupled with energy-dispersive X-ray spectroscopy for elemental analysis. In addition, the experiments performed were expected to provide information on the particles and colloids found in the size fraction <0.45 µm. However, the work failed to achieve its goals.

The manufactured SiO$_2$ nanoparticles added to the columns were not detected in the SEM images. Nonetheless, comparing the SEM images from control and test columns, it is
possible to conclude that the samples from the column with added nanoparticles were more concentrated than the ones from the control column. This difference between the samples leads to believe that the manufactured silica nanoparticles are indeed present in the leachate samples analysed under the microscope, only they could not be positively identified.

It is possible that the manufactured particles were not detected in the sample as a result of changes in their particle size and shape, due to chemical and physical stress during sample handling, or as a result of the heterogeneity of the sample. Moreover, since the volumes of sample analysed at a time are quite small, it is possible that the particles were not present on the analysed spots of the samples.

Furthermore, the SEM and EDS analyses revealed some structures other than simple silica particles. However, the identification of these structures and of their origin, considering only the analyses performed is not possible.

Therefore, there is a need to improve the protocol applied and explore the use of different techniques in order to be able to detect specific manufactured nanoparticles and distinguish them from the particles released from the matrix.

References


