The role and influence of hydrogeochemistry in the behaviour and fate of silver nanoparticles in freshwater systems

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Abstract

This paper reports on the occurrence, fate and influence of selected hydrogeochemical parameters in the distribution profile of Ag NPs in freshwater from selected dams in South Africa. Single particle-inductively coupled plasma mass spectrometry (SP-ICPMS) was used to characterize, detect, and quantify Ag NPs in aqueous samples. The same technique was applied to determine particle size, particle number concentration and dissolved concentration of Ag NPs in water samples collected from the selected dams. The mean particle number concentration ranged from 6.92×10^3 parts/mL to 1.04×10^5 parts/mL. The mean dissolved Ag ranged from concentration of 0.18 µg/L to 0.27 µg/L, while the size of Ag ranged from 12.52 to 20.01 nm. From this work, it was proved that, SP-ICPMS is an effective technique for detection and characterization of nanoparticles in aqueous environment. Moreover, this study has revealed that hydrogeochemical properties of water as well as dissolved organic carbon (DOC), affect the presence and physical properties of Ag NPs in aqueous environment.

Keywords Nanoparticles · Ag NPs · Environmental waters · SP-ICPMS · Water chemistry

1 Introduction

Nanomaterials (NMs) in the modern world have shown great potentials for application in many technological and industrial sector including food safety, information technology, pharmaceutical, energy, storage, cosmetics and agriculture [34]. Silver nanoparticles (Ag NPs) are the most commonly used nanomaterials and they are found in many products such as paints, nano-agrochemicals [3, 40], water treatment devices, coatings, foods, and cosmetics, packing materials [34]. Due to the widespread application of Ag NPs there is a growing concern about their ecological and environmental impact due to the potential release of engineered nanoparticles (ENPs) into the environment.

Once released to the environment, silver may remain as either Ag NPs or oxidized to Ag ion (Ag^+) [10].

The release of silver nanoparticles into the environment has been reported to occur during the process of their manufacturing and incorporation into products such as textile (Blankets, clothing), in antimicrobial agents [4], and also during the use of commercial products that contain NPs. Commercial products that contain NPs are many and they include agrochemicals, paints and during the disposal of products containing Ag NPs [23]. However, Ag NPs are also known to be produced in the environment by a variety of natural geochemical processes [30].

Of concern is that, several research publications have reported on the toxicity of Ag NPs in aquatic organisms [17], human cells [33], *Daphnia* [8], and algae [36].

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Moreover, reports of acute toxicity of Ag NPs in zebra fish have also been published [6]. The accumulation of Ag NPs in fish tissues (gills, liver) has as well been reported to affect fish survival at low oxygen levels and have been implicated to cause oxidative stress [39]. The toxicity of NPs is attributed to factors such as particle surface properties, particle concentration, particle size and nature of the environment [14, 27].

In the aquatic environments, water chemistry/hydrochemical parameters (conductivity (EC), pH, temperature), nanoparticle concentration and natural organic matter (NOM) potentially alter the surface properties that lead to either the aggregation/agglomeration [47] or stabilization of Ag NPs [19, 21]. In order to understand the potential environmental fate and effects of Ag NPs [42], evaluating the effect of hydrochemical parameters on the properties and bioavailability Ag NPs is essential [20]. However, information on the effect of hydrochemical parameters on bioavailability of NPs in natural waters is still limited [18].

Apart from the modelling studies, there is little experimental information available in the literature on the occurrence of Ag NPs into aquatic environment. Of the few available studies reported in the literature have found that nanomaterials from consumable products could accumulate in surface waters [5, 22]. A report by Gottschalk et al. [15] have given predictions that the levels of Ag NPs in surface waters are expected to increase due to the increase in their application). Moreover, modelling approaches have shown that the predicted information concerning the presence, accumulation and concentration of ENPs in the environment due to the increased usage of consumer products containing nanomaterials [32]. The use of modelling methods, has given the predictions of the annual exponential increase of environmental concentrations of ENPs in surface waters, in sewage sludge and sewage treatment plant effluents [32]. However, there is still scanty scientific data on the occurrence, distribution and fate of Ag NPs in aquatic environment to validate the predictions as provided by these models. Moreover, it is possible that Ag NPs concentrations may increase in an environment to levels which could be greater or equal to those of dissolved Ag found in contaminated waters [28].

Moreover, few studies have reported the use of SP-ICPMS for the analysis of Ag NPs in drinking water [11], soil [29], plastic food containers [35], environmental waters [43], and domestic wash waters [31]. Despite the enhanced sensitivity of the SP-ICP-MS in the detection and quantification of Ag NPs in complex environmental samples, the techniques is yet to enjoy a wide and universal use thus far.

This technique can prove to be highly useful in the study of Ag NPs in South African freshwater systems where the status of these NPs in water system is not fully known due to insufficient data available [1]. Therefore, this

research aimed at providing data on the occurrence and distribution of Ag NPs as well as interactions between Ag NPs and physicochemical parameters in selected water system in south African Provinces.

2 Materials and methods

2.1 Standard and reagents

Gold (Au) nanoparticles (carboxylic acid-capped 50, 100 nm) in pure water were purchased from PerkinElmer, Johannesburg South Africa/Shelton, Connecticut, USA). The Ag NPs (60 nm, 0.02 mg/mL) in aqueous buffer, with sodium citrate as stabilizer were purchased from Sigma-Aldrich, Johannesburg, South Africa. Au (1000 mg/L) and Ag (1000 mg/L) standard was purchased from Sigma-Aldrich, Johannesburg, South Africa. Pure water used (18.2 MΩ cm) was produced using a Milli-Q water purification system (Millipore S.A.S made in France) and Multi-element tune standard solution was purchased from PerkinElmer, Johannesburg South Africa.

2.2 Water sample collection

The samples were collected between 19 May and 7 July 2018. Fresh water samples were collected about 10–30 cm depth below the water surface using pre-cleaned polypropylene bottles and amber glass bottles with Teflon caps. After sample collection the bottles were placed on ice in a container to lower the sample temperature to at least 4 °C and to prevent the samples from sunlight. Thereafter the samples were transferred to the laboratory and stored in dark at 4 °C prior to analysis. The pH, turbidity, total dissolved solids (TDS) and conductivity (EC) were measured at the sampling point and as the samples got into the laboratory.

2.3 Standard and water sample preparation

2.3.1 Water sample preparation

Prior to the analysis for SP-ICP-MS samples were filtered using 0.2 µm membranes (cellulose acetate) to remove dust and interfering particles. Water samples for total organic carbon analyzer (TELEDYNE TEKMAR, TOC Torch, made in USA) were filtered with acrodisc® syringe filters with GHP membrane (0.45 µm, 25 mm).

2.3.2 Standard solution preparation

For size calibration and transport efficiency determination, spherical Au–Np suspension (50, 100 nm) and

Ag NP suspension (60 nm), NexION® 350D (PerkinElmer, Johannesburg South Africa/Shelton, Connecticut, USA) was utilized. Prior to dilution, all standards were sonicated for 10 min. Since the Au NP are only stable at temperature within the range of 4–25 °C, small ice cubes were added to the ultrasonic bath to prevent any possibility of temperature rise that could facilitate the dissolution of NPs [25]. The mixture was then diluted in Milli-Q water (18.2 MΩ cm) to a final nominal concentration between 50,000 and 100,000 parts/mL. After preparation, all standards were ultrasonicated for five minutes before analysis to make sure that all the particles were dispersed as described previously [44]. Dissolved calibration standards for Au and Ag consisting 1, 5, and 10 µg/L were made in Milli-Q water from 1000 mg/L stock standard solutions (Sigma-Aldrich, Johannesburg, South Africa).

2.4 The SP-ICP-MS analysis

Analysis for Ag NPs was performed on a PerkinElmer NexION® 350D SP-ICP-MS using the nano module application, present in PerkinElmer Syngistix™ software. The sample introduction system involved a quartz cyclonic spray chamber, quartz bore injector and concentric glass nebulizer. Data acquisition time was set at 60 s and dwell time length was set at 50 µs. The analyte 106.905 Ag was observed. The sample uptake rate was measured every day and the values ranges between 0.278 and 0.335 mL/min. The transport efficiency (TE) was calculated every day based on nanoparticle size calibration analysis of the aspirated solution [2, 9]. Working suspensions of Ag NPs were prepared daily by diluting the stock solutions with ultrapure water (Milli-Q®).

The Au NP suspensions (50 nm and 100 nm) was utilised for particle calibration and determination of transport efficiency. The flow rate was established by weighing the amount of ultrapure water aspirated in the system for 5 min. PerkinElmer Syngistix software with the Nano Application Module was used for data collection and processing [2, 3]. Prior to measurements of the dam water samples, Silver (Ag) nanoparticle suspension (60 nm) was characterized in ultrapure water to check the performance of the optimized methodology. During data acquisition, ultrapure water was analysed between replicates to check memory effects. Detection limit (LOD) of the dissolved Ag NPs was determined using Linear regression analysis [46]. The Ag nanoparticle diameter limit of detections (DLs) were determined by three times the standard deviation above the background when measuring ultrapure water [26, 45]. Particle number concentration (LOD_{NP}) was determined as described previously [24].

2.5 DOC measurements

Total organic carbon Analyzer (TELEDYNE TEKMAR, TOC Fusion, made in USA) was used to determine dissolved organic carbon (DOC) in dam water samples. The TOC analyser was calibrated using potassium hydrogen phthalate (KHP) standard solutions. During data acquisition, standards (KHP) were analysed between samples to check instrument performance. The DOC measurements were carried out in triplicates.

2.6 Principal component analysis

Principal component analysis (PCA) was performed using XLSTAT software, while the principal components (PCs) were processed using the Varimax rotation with Kaiser Normalization. The numbers of PCs to retain was based on the eigenvalues, where only PCs with eigenvalues > 1 were retained while the parameters were retained if their *p* value < 0.05 at 95% confidence level. The value of a factor scores for a parameter represented the importance of that factor at the sampled site. A factor score > + 1 reflected a sampling area significantly influenced by the parameter highly loaded in a PC. Factor scores < − 1 reflected sampling areas virtually unaffected by parameters highly loaded in a PC, whereas near-zero scores reflected areas moderately influenced by parameters loaded in a particular PC.

2.7 Statistical data analysis

XLSTAT software was used to compute the PCA, descriptive statistics and the correlation tests. The PCA and the correlation tests were used to determine the variations between observations and variables at 95% confidence level. Analysis of variance (ANOVA) was performed using IBM SPSS statistics version 25.0 to determine variations in the test parameters with respect to between groups and within groups sampling sites at 95% confidence level. The hierarchical cluster analysis (HCA) was used to group sampling areas into distinct clusters according to data for test parameters.

3 Results and discussion

3.1 The SP-ICP-MS analysis

The results of the size distribution of Ag NPs in ultrapure water as shown in Fig. 1, are in agreement with values reported on the certificate obtained from the manufacturer. Thus, the obtained results confirm and validate the performance of the methods. Results obtained from the

Fig. 1 Size distribution histogram of 60 nm Ag prepared in ultrapure water from Syngistix nano application module for SP-ICP-MS

analysis of Dam water samples by SP-ICP-MS are summarised in Table 1. The parameters (mean size, particle concentration and dissolved concentration) registered statistically significant variations with respect to sampling sites, p -value < 0.05 at 95% confidence level (Table 2). Variations between tested parameters may be associated with hydrogeochemical characteristics and physicochemical

composition of the sampling site. Dissolved silver concentration were found to be low in concentration in all water samples (Table 1), which might be due to formation of complexes with various ligands (chloride, sulphate) and dissolved organic carbon [18].

In our study, the concentrations of Ag NP in water samples was found to be ranging from 0.18 $\mu\text{g/L}$ to 0.27 $\mu\text{g/L}$ in which is falling within the predicted concentrations estimated range by the previous modelling studies [7, 12, 16, 41]. In their report, Dumont et al. [12] provided predictions of the concentration of Ag NPs in European rivers to be approximately ranging from 0.002 to 18 ng/. In another report, Boxall et al. [7] predicted that the concentrations of the Ag NPs in natural waters expected to be in the range 1 to 10 $\mu\text{g/L}$ [7]. Likewise, Gottschalk et al. [16] predicted that the concentration of Ag NPs in surface waters ranged from 0.01 $\mu\text{g/L}$ to 0.1 $\mu\text{g/L}$ [16]. Moreover, the summarized modelling studies mostly estimate Ag NPs concentrations in surface waters ranged from low ng/L to low $\mu\text{g/L}$ [12, 41]. Additionally, as increasing the usage of various products containing Ag NPs, it is expected that in the near future, their occurrence in surface waters will increase accordingly as predicted by the modelling studies.

3.2 Physicochemical water quality parameters

Table 3 presents the results that indicates the descriptive statistics for water quality parameters which were

Table 1 Mean values for all environmental variables investigated

Sampling point	Province	Mean size (nm)	Part. conc. (parts/mL)	Dissolved conc. ($\mu\text{g/L}$)	pH	Conductivity (μS)	Turbidity (NTU)	TDS (mg/L)	DOC (mg/L)
NW Dam	North-West	12.52	104,260.89	0.26	8.37	540.67	5.80	268.10	9.8093
WC1 Dam	Western Cape	15.68	37,188.82	0.27	7.53	50.53	32.67	25.03	8.3081
FS Dam	Free state	20.01	6679.38	0.27	7.45	131.65	158.70	20.70	7.1186
WC2 Dam	Western Cape	15.39	15,091.70	0.18	8.70	42.20	43.40	20.70	4.4251
NC Dam	Northern Cape	18.85	6922.89	0.26	8.10	150.50	66.40	76.00	3.88

Table 2 Analysis of variance (ANOVA)

		Sum of squares	df	Mean square	F	Sig.
Mean size (nm)	Between groups	71.268	4	17.817	5.432	0.046
	Within groups	16.4	5	3.28		
	Total	87.667	9			
Part. conc. (Parts/mL)	Between groups	13,567,732,794	4	3.392E+09	250.061	0
	Within groups	67,822,103.72	5	13,564,421		
	Total	13,635,554,898	9			
Dissolved conc. ($\mu\text{g/L}$)	Between groups	0.013	4	0.003	258.721	0
	Within groups	0	5	0		
	Total	0.013	9			

Table 3 Descriptive statistics for all the environmental variables investigated

Statistic	Mean size (nm)	Part. conc. (parts/mL)	Dissolved conc. ($\mu\text{g/L}$)	pH	Conductivity ($\mu\text{S/cm}$)	Turbidity (NTU)	TDS (mg/L)	DOC (mg/L)
Nbr. of observations	5	5	5	5	5	5	5	5
Minimum	12.516	6679.376	0.176	7.450	42.200	5.800	20.700	3.884
Maximum	20.012	104,260.885	0.274	8.700	540.667	158.700	268.100	9.809
Range	7.497	97,581.509	0.097	1.250	498.467	152.900	247.400	5.925
1st Quartile	15.392	6922.895	0.259	7.533	50.533	32.667	20.700	4.425
Median	15.680	15,091.699	0.259	8.100	131.650	43.400	25.033	7.119
3rd Quartile	18.850	37,188.818	0.268	8.367	150.500	66.400	76.000	8.308
Mean	16.490	34,028.735	0.247	8.030	183.110	61.393	82.107	6.709
Variance (n)	7.127	1,356,773,279.288	0.001	0.230	33,798.103	2746.651	9085.947	5.107
Variance (n-1)	8.908	1,695,966,599.110	0.002	0.288	42,247.629	3433.314	11,357.434	6.383
Standard deviation (n)	2.670	36,834.403	0.036	0.480	183.843	52.408	95.320	2.260
Standard deviation (n-1)	2.985	41,182.115	0.040	0.536	205.542	58.594	106.571	2.527
Variation coefficient	0.162	1.082	0.145	0.060	1.004	0.854	1.161	0.337

investigated. It was observed that the pH of the dam waters ranged between 7.5 and 8.9 with a mean value of 8.030, which was slightly above the neutral to the alkaline. It was observed that all water samples collected remained within the alkaline nature. The levels of ionic strength (expressed as conductivity) of water samples was found to vary between 42.2 $\mu\text{S/cm}$ and 540 $\mu\text{S/cm}$ with mean value of 183.110 $\mu\text{S/cm}$. Water samples from NW dam was found to have high levels of ionic strength while WC1 and WC2 dams showed low levels of ionic strength (Table 1). The turbidity values ranged from 5.8 NTU to 158.7 NTU with the mean value of 61.393. The water samples from NW dam were found to be less turbid as compared to water samples from other dams (Table 1). Dissolved organic matter (DOM) concentration in water samples (expressed as dissolved organic carbon, DOC) was found to vary from 9.809 to 3.884 mg/L with the mean value of 6.709. NW dam registered higher concentrations of DOM compared to NC, WC1, WC2 and FS Dams (Table 1). The levels of total dissolved solids TDS measured from water sample ranged between 20.7 and 268.1 mg/L with a mean value of 82.107. Also, levels of TDS were found to be relatively higher for water samples collected from NW dam compared to those sampled from other dams (Table 1). The total dissolved solids (TDS) in aquatic environment may be an indication of the occurrence of inorganic salts and dissolved organic matter content [38].

3.3 Effects of water chemistry on the occurrence of Ag nanoparticles in water samples

Table 4 presents the Principal Components and variable loadings which were generated by the PCA model. The first two PCs, accounted for 87.335% of the total variation in the data. PC1 explained 57.349% of the variation in the original dataset for the occurrences of Ag NPs in water samples from the study area while PC 2 explained 29.986% of the variation of the original dataset for the occurrences of Ag NPs. This indicates that more than 87% from the total variable can be explained with the first two PCs. PC 1 registered strong positive correlation with particle

Table 4 Correlations between variables and factors extracted using the PCA

Variable	Principal component 1	Principal component 2
Mean size (nm)	-0.897	0.304
Part. conc. (parts/mL)	0.984	0.159
Dissolved conc. ($\mu\text{g/L}$)	0.051	0.953
pH	0.423	-0.861
Conductivity ($\mu\text{S/cm}$)	0.872	0.252
Turbidity (NTU)	-0.751	0.429
TDS (mg/L)	0.924	0.133
DOC (mg/L)	0.676	0.606
Variability (%)	57.349	29.986
Cumulative %	57.349	87.335

concentration, total dissolved solids (TDS), conductivity, and DOC and high negative correlation with mean size and turbidity. This suggests that the concentration and occurrence of Ag NPs in water are influenced by the levels of conductivity, TDS, DOC, and virtually unaffected by mean size and turbidity. This also suggests that the concentration and the occurrence of Ag nanoparticles increase proportionally with conductivity, TDS, DOC, and the decreases in mean size and turbidity. Based on the strong positive correlation of 0.984, PC 1 is principally a measure of the particle concentration. On the other hand, PC 2 registered strong positive correlation with dissolved concentration and DOC, and high negative correlation with pH. This suggests that dissolved concentration of Ag in water is influenced by the levels of DOC and weakly affected by water pH. This also suggests that dissolved concentration of Ag increases with decrease in water pH. In addition, based on the strong positive correlation of 0.953, principal component 2 is primarily a measure of the dissolved concentration.

It was also observed that there was a significant correlation between particle concentrations of Ag NPs and TDS ($r^2 = 0.905$, p -value = 0.035) at the 95% confidence level (Table 5). This suggests that the particle concentrations of the Ag NPs in dam water samples are strongly influenced by the level of total dissolved solids in water. This means particle concentration of Ag NPs increases with the increase of TDS in water (Table 5). The TDS in aquatic environment denotes the existence of inorganic metals or salts and dissolved organic matter content [37]. Similarly, was observed that there was a statistically significant correlation between TDS and conductivity ($r^2 = 0.982$, p -value = 0.003) at the 95% confidence level (Table 5), suggesting that, TDS in aquatic environment pronounces the levels of conductivity in water. This phenomenon has also been reported by Sawyer et al. [38]. Nevertheless, there was a significant correlation between mean size of Ag NPs

and turbidity content ($r^2 = 0.884$, p -value = 0.046) at the 95% confidence level (Table 5), suggesting that the levels of turbidity increase with the increase of Ag NPs in water.

Figure 2 shows the correlation between the investigated environmental variables and active observations (sampling areas) of the extracted PCs. It was observed that, the occurrence of the Ag NPs in water samples collected from different dams in South Africa, is influenced by the physicochemical water quality parameters loaded in a particular principal component Fig. 2. The water from NW dam was significantly influenced by the parameters highly loaded in PC 1 (F1) and moderately influenced by the parameters highly loaded in PC 2 (F2). The water samples from WC1 dam seems to be moderately influenced by the parameters highly loaded in both PC1 (F1) and PC2 (F2). The water from FS dam the are virtually unaffected by the parameters highly loaded in both PC2 (F2) and significantly influenced by the parameters highly loaded in PC 1 (F1). The water sample from WC2 dam was found to be moderately influenced by the parameters highly loaded in PC 1 (F1) and virtually unaffected by the parameters highly loaded in both PC2 (F2). The water collected from NC dam was virtually unaffected by the parameters highly loaded in both PC1 (F1) and moderately influenced by the parameters highly loaded in PC 2 (F2).

Figure 3 shows that the observations (sampling areas) using PCA can be divided into two main groups (1 and 2) based on their physicochemical characteristics of water. Group 1 comprises of WC2, FS, WC1 and NC dams sampling areas. Group 2 comprised of NW dam sampling area. This was also observed with the hierarchical cluster analysis (HCA), where the dendrogram using Ward method presented two major clusters that identify groups of sampling areas with similar hydrogeochemical characteristics. Cluster 1 comprised of WC2, FS, WC1 and NC dams. Cluster 2 comprised of NW dam sampling area (Fig. 4). Table 1 shows that the occurrence of Ag NPs in water sample

Table 5 Correlation matrix (Pearson) studies for all the environmental parameters investigated

Variables	Mean size (nm)	Part. conc. (parts/mL)	Dissolved conc. (µg/L)	pH	Conductivity (µS)	Turbidity (NTU)	TDS (mg/L)	DOC (mg/L)
Mean size (nm)	1	−0.857	0.281	−0.554	−0.577	0.884	−0.673	−0.541
Part. conc. (parts/mL)	−0.857	1	0.181	0.264	0.876	−0.668	0.905	0.796
Dissolved conc. (µg/L)	0.281	0.181	1	−0.793	0.295	0.280	0.216	0.515
pH	−0.554	0.264	−0.793	1	0.271	−0.578	0.372	−0.317
Conductivity (µS)	−0.577	0.876	0.295	0.271	1	−0.388	0.982	0.621
Turbidity (NTU)	0.884	−0.668	0.280	−0.578	−0.388	1	−0.545	−0.256
TDS (mg/L)	−0.673	0.905	0.216	0.372	0.982	−0.545	1	0.574
DOC (mg/L)	−0.541	0.796	0.515	−0.317	0.621	−0.256	0.574	1

Values in bold are different from 0 with a significance level $\alpha = 0.05$

Fig. 2 Biplot showing correlation between variables and observations (sampling sites)

Fig. 3 Related Observation in PCA. The sampling areas are grouped based on their characteristics and uniqueness at 95% confidence intervals

characterized from group 1 and 2 is affected by the physicochemical characteristics of water. It was observed that the presence of Ag particle concentration was higher in

water from group 2 than water from group 1, even though the EC was higher in group 2 (Table 1). This result was attributed to the influence of DOM on the properties of

Fig. 4 The resulting dendrogram using ward method showing the two major clusters used to identify groups of water with similar hydrogeochemical characteristics

nanoparticles. High concentration of DOM in water from NW dam (Table 1) may result into stabilization of Ag NPs in water, most likely due to the adsorption of DOM on the surface of the nanoparticles [13].

4 Conclusion

This study indicated that the occurrence of Ag NPs in dam water samples from North West, Western Cape, Free State, Western Cape, and Northern Cape Provinces in South Africa is basically influenced by the physicochemical and hydrogeochemical properties. It was also observed that the occurrence of Ag NPs varies significantly with respect to the sampling sites and were a function of two principal components which accounted for 87.335% of the of the variation in the original dataset. This study contributes significantly to the body of knowledge on distribution and occurrence of Ag NPs in selected dam waters in South Africa. Understanding levels and occurrence of Ag NPs in water, will provide an insight towards assessing the effect and environmental risk assessment of the Ag nanoparticles in water as well as to the establishment of guidelines and regulation of the nanoparticles.

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Compliance with ethical standards

Conflict of interest On behalf of all authors, the corresponding author states that there is no conflict of interest.

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