



Activated charcoal as a capture material for silver nanoparticles in environmental water samples

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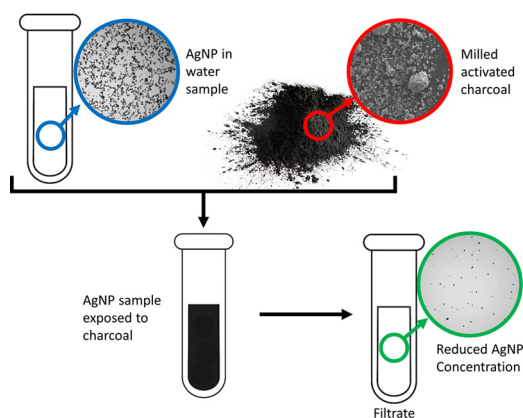
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HIGHLIGHTS

- Activated charcoal is a suitable material for the capture of silver nanoparticles from water samples
- Milled activated charcoal successfully captured 63.6% of the silver in 100 ppb 25 nm AgNP samples
- AgNP capture increased with decreasing concentration and increasing the time that the sample was exposed to the charcoal
- Changing the size and coating of the AgNPs used to 10 nm citrate AgNPs did not inhibit the capture by the milled charcoal
- An average of 94.8% of the Ag captured by the charcoal was recovered through the development of an HCl leaching procedure

GRAPHICAL ABSTRACT



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ABSTRACT

Silver nanoparticles (AgNPs), due to their antibacterial activity, have been incorporated into numerous consumer products. Their environmental impact however, is currently unclear. Uncertainties surround the concentration, fate, and effects of AgNPs in aquatic environments. This study examined the suitability of activated charcoal as a capture material for AgNPs from water. Samples of 100 ppb AgNPs were initially generated and exposed to activated charcoal for 24 h to examine the ability of charcoal to capture AgNPs. The decrease in Ag concentration was measured using ICP-MS. Following initial investigations, the surface area of the charcoal was increased firstly with a pestle and mortar and secondly by milling the charcoal using a ball mill. The increased surface area of the milled charcoal increased the capture of the AgNPs from 11.9% to 63.6% for the 100 ppb samples. Further investigations were carried out examining the effect on the capture of AgNP concentration (with concentration ranging from 10 to 100 ppb), particle coating and the effect of exposure time to the activated charcoal. The capture of AgNP increased with decreasing concentration. A hydrochloric acid (HCl) leaching procedure was also developed which successfully removed the captured silver allowing the fraction captured by the charcoal to be quantified with an average of 94.8% recovery. The results show that milled activated charcoal, can successfully capture

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AgNPs from water samples, and that therefore, activated charcoal may prove to be a cost effective material for the remediation of waters impacted by AgNP or other nano-wastes.

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1. Introduction

Silver nanoparticles (AgNP) and their potential impact on the environment, has become an area of emerging concern in recent years, due to the proliferation of their use in consumer products (Foss Hansen, 2017; McGillicuddy et al., 2017). While several elements and compounds are used in nano-functionalised consumer products, silver is of particular interest due to it being among the most commonly incorporated engineered nanomaterials (ENM) in consumer products (Vance et al., 2015). These nano-functionalised products have led to an increased probability of AgNPs entering the environment (Shevlin et al., 2017). Examples of the products AgNP have been incorporated into include paints, clothing, electrical appliances, cosmetics, food contact materials, water filters, medical and medicinal products and nano-functionalised plastics (Benn and Westerhoff, 2008; Blaser et al., 2008; Cushen et al., 2012; Dubas et al., 2006; Etheridge et al., 2013; Farkas et al., 2011; Jain and Pradeep, 2005; Kumar et al., 2008; Maneerung et al., 2008; McGillicuddy et al., 2017; Silver et al., 2006). Many of these products are listed in various inventories of nano-functionalised products (ANEC/BEUC, 2013; Hansen et al., 2016; Vance et al., 2015).

A number of studies have found that these consumer products can release their incorporated AgNPs. The release of AgNPs has been observed from nano-functionalized textiles and fabrics (Benn and Westerhoff, 2008; Geranio et al., 2009; Kulthong et al., 2010; Lorenz et al., 2012), toothbrushes (Mackevica et al., 2017), medical devices (Sussman et al., 2015), outdoor paints (Kaegi et al., 2010), washing machines (Farkas et al., 2011) and food packaging materials (Cushen et al., 2014; Hannon et al., 2015). The release of AgNP from these products will impact on the environment including the aqueous environment.

In the environment the ultimate fate of AgNPs is complicated, AgNPs can undergo numerous different physical and chemical interactions which will impact on their fate (Shevlin et al., 2017; Zhang et al., 2016). In environmental waters nanoparticles may; aggregate or agglomerate, remain suspended, dissolve, sediment, or react with the numerous species present in the aquatic system (Luoma, 2008; McGillicuddy et al., 2017; SCENIHR, 2014; Shevlin et al., 2017). The fate of AgNPs is influenced by numerous intrinsic and extrinsic effects, such as: particle size, particle shape, particle coating, temperature, ionic strength, pH, dissolved oxygen levels and the presence of ligands (He et al., 2013; Liu and Hurt, 2010; Luoma, 2008; McGillicuddy et al., 2017; Shevlin et al., 2017; Zhang et al., 2011, 2016).

Currently the concentrations of AgNPs in the aquatic environment have primarily been estimated through modelling with predicted environmental concentrations (PEC) in the ng/L range (Blaser et al., 2008). PECs ranges of between 0.09 and 0.43 ng/L and between 0.59 and 2.16 ng/L have been predicted in US and European surface waters respectively (Gottschalk et al., 2009). More recently studies in Germany using cloud point extraction (CPE) (L. Li et al., 2016b) and The Netherlands using Single Particle Inductively Coupled Plasma Mass Spectrometry (SP-ICPMS) found that AgNPs are now present in the environment in the ng/L range, therefore, techniques need to be developed in order to capture and detect these AgNP in the aquatic environment.

Numerous methods have been investigated for the capture of AgNPs from aqueous samples including; ultracentrifugation (Kennedy et al., 2010), tangential (Trefry et al., 2010) and cross-flow ultrafiltration (Farmen et al., 2012), hydrodynamic chromatography (Tiede et al., 2009), nanofiber filtration membranes (Liang et al., 2010), nanostructured poly(amic acid) [n-PAA] nanofilters (Okello et al., 2011), surface

functionalised magnetic iron nanoparticles (Mwilu et al., 2014), CPE (Hartmann et al., 2013; Liu et al., 2009) and tangential flow filtration (Maurer et al., 2014).

A previous study investigated the ability of activated carbon to capture AgNPs from suspension (Gicheva and Yordanov, 2013), successfully removing 60 nm citrate coated AgNPs from aqueous dispersions using mesoporous activated carbon (Norit® CA1). In their study Gicheva and Yordanov, (2013) found that the AgNPs capture by the activated carbon increased in the presence of electrolytes (such as NaCl, KCl, NaNO₃, KNO₃) with complete removal observed when the electrolytes were present above the critical coagulation concentration. While this method was suggested as a suitable for nanoparticle removal from water samples the volumes of electrolytes required may act as a hindrance to its applicability (Gicheva and Yordanov, 2013).

The present study investigated the suitability of activated charcoal to act as a capture material for AgNP in aqueous sample, without the addition of chemical aids just the physical alteration of the activated charcoal itself, by milling. Further to the capture experiments, a leaching procedure was developed to allow for the silver captured by the charcoal to be quantified and recovered.

2. Methodology

2.1. Materials and chemicals

Samples of AgNPs were generated using commercially available AgNPs dissolved in Milli-Q water (18.3 MΩ·cm, Millipore, Bedford, USA). Polyvinylpyrrolidone (PVP) coated 25 nm 5 mg/mL Econix Silver Nanospheres were purchased from nanoComposix Europe (Prague, Czech Republic). In addition, 10 nm 0.02 mg/mL citrate coated AgNPs (Sigma-Aldrich, USA) were used to investigate the impact of NP size and coating on capture by the charcoal. All acids used in the experiments were of trace metal grade, HCl (Assay 34–37%) and HNO₃ (Assay 67–69%) [SpA grade, Romil™, Cambridge, UK]. Powdered activated carbon (Norit® CA1, Norit N.V. Amersfoort, The Netherlands) which has previously been used to examine AgNP removal from aqueous samples, (Gicheva and Yordanov, 2013) was purchased from Sigma-Aldrich. The activated carbon samples were freeze dried (Freezone 12, Labconco, Kansas City, USA) at –50 °C (Healy et al., 2016b; Ratcliff et al., 2016). The activated carbon was initially used as received (Fig. 2(a)) and was subsequently pulverised, following grinding with a ceramic pestle and mortar, in an agate ball mill (Fritsch™ Pulverisette 6 Planetary Mono Mill) with a rotational speed of 500 rpm for 5 min (repeated three times) using an 80 mL agate vial and balls (Ø 10 mm) (Healy et al., 2016a; McEneff et al., 2017), in order to increase the surface area exposure to AgNPs. The granulometry of the processed activated charcoal was determined for both the ground (pestle and mortar) and milled (agate ball mill) fractions using laser particle sizing (LPS) by introducing samples of the activated charcoal to the Hydro-G dispersion unit of a Malvern Mastersizer 2000 as outlined previously for sediments (Walls et al., 2017). The charcoal had initially been made into a slurry with isopropyl alcohol to limit the possibility of the charcoal clumping in the dispersion unit. The LPS analysis measured the ground fraction to have a mean diameter 341.150 µm (with a 10th percentile diameter of 27.139 µm and a 90th percentile of 1061.639 µm) and the milled fraction as having a mean diameter 22.134 µm (with a 10th percentile diameter of 6.555 µm and a 90th percentile of 159.724 µm).

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