



Biosorptive removal of bare-, citrate-, and PVP-coated silver nanoparticles from aqueous solution by activated sludge



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ABSTRACT

The removal of AgNPs released from nano-consumer products or effluent of wastewater from treatment plants is important to reduce the potential risk of AgNPs. In this work, biosorptive removal of bare-, citrate-, and polyvinylpyrrolidone-AgNPs were tested using biological floc in activated sludge. The results of isotherms and kinetics test showed that zeta potential of AgNPs depended on the hydrodynamic diameter and then finally maximum uptake capacity. Herein, biological floc in activated sludge could be used as effective biosorbents for removal of AgNPs in aqueous solutions.

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Introduction

The rapidly increasing nanotechnology industry is producing nanomaterials that are incorporated into nano-consumer products. In particular, silver nanoparticles (AgNPs) are used in various fields of applications such as disinfecting sprays, cosmetics, fabrics, and household appliances [1–5]. While Ag⁺ released from AgNPs, AgNP surface-associated ionic Ag and AgNPs are all likely to contribute to observed toxicity, ionic Ag generally exhibits the strongest toxic effects [6]. As a proof-of-Ag⁺ release from AgNPs contained in nano-consumer products, few studies are available on the leaching of silver from clothing into water [1–5,7,8]. For example, Benn and Westerhoff investigated the silver released from commercial socks into water, and found socks that contained up to 1.36 mg-Ag/g-sock leached as much as 0.65 mg of silver in 0.5 L of distilled water [3]. Nowack et al. [1] also determined the amount and form of silver released during washing from nine fabrics with different methods of silver incorporation into the fibers. Impellitter et al. [7] have shown that bleach has strong effects on the speciation of silver in textiles and that significant transformation of elemental Ag to AgCl occurred in the presence of bleach. Farkas et al. [8] provided evidence that AgNPs of about

10 nm diameter are present in the effluent of washing machines that release Ag into the washwater.

The silver released into sewer systems can be partitioned onto wastewater biomass and be removed at the wastewater treatment plant (WTP). Based on the pilot scaled WTP, about 90–95% spiked AgNPs are efficiently reduced by biological treatment and accumulated in activated sludge or biosolids [9]. Namely, it is possible for 5–10% of silver to remain in the treated water. Although many studies focus on the removal of Ag⁺ from aqueous solutions [10], few studies have been carried out that investigate the elimination of AgNPs using adsorbents or resins. Khan et al. [11] reported the adsorptive removal of AgNPs by bacteria resistant to AgNP, isolated from the sewage environment. The removal of AgNPs might differ from that of Ag⁺ in aqueous solutions. In addition, the activity of microorganisms in active sludge was influenced by AgNPs as well as its ions. In previous report [12], we found that AgNPs inactivated biofilms in a biosorption-dependent manner, although the activity of AgNPs against planktonic cells was ca. 10% that of silver ions. The presence of both a synthetic wastewater component and extracellular polymeric substances (EPS) significantly enhanced the AgNPs removal efficiency [13]. AgNPs appeared disperse within the EPS matrix, while in the absence of EPS, the AgNPs were adsorbed to the surface of the microorganisms. AgNPs entrapment in the EPS matrix was one of the major mechanisms of AgNPs removal, while, regardless of the presence of EPS, the removal of AgNPs are only affected by

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AgNPs adsorption onto microorganisms which can be explained by the electrical double layer compression of both AgNPs and microorganisms.

In this work the adsorptive features of different type of AgNPs in aqueous phase using activated sludge is investigated. Herein (i) the influence of stabilizer type of nanoparticles for adsorption of AgNPs on activated sludge, (ii) the adsorption isotherms and kinetics of adsorption in de-ionized water (DW) and synthetic sewage water (SSW) and (iii) desorption feature of AgNPs from activated sludge is elucidated. Three different AgNPs, i.e., bare, citrate, and polyvinylpyrrolidone (PVP) coated, are used as target adsorbates.

Experimental

Materials

Except for citrate-AgNP, all chemicals, and bare-AgNPs and PVP-AgNPs were purchased from Sigma–Aldrich. Citrate-AgNP was prepared using a method previously reported [14]. AgNPs were dispersed in de-ionized water (DW) and synthetic sewage water (SSW) using ultrasonic horn with a frequency of 200 kHz (UIh700S, ULSSO Hi-Tech). Based on the modified OECD TG (Organization for Economic Co-operation and Development, Test Guidance) 303A [15], SSW stock with 138 mM ionic strength was prepared using a mixture of the following compositions: glucose 200 mg/L, yeast extract 10 mg/L, bactopectone 10 mg/L, $(\text{NH}_4)_2\text{SO}_4$ 100 mg/L, K_2HPO_4 30 mg/L, KH_2PO_4 30 mg/L, MgSO_4 1.8 mg/L, FeCl_3 0.04 mg/L, NaCl 1.4 mg/L, CaCl_2 0.04 mg/L, CoCl_2 0.48 mg/L, and NaHCO_3 60 mg/L. Activated sludge for use as biosorbents was obtained from the Joong-Rang sewage treatment plant (STP) in Seoul, Korea.

Characterization of AgNPs

The morphology of AgNPs dispersed in SSW was observed using transmission electron microscopy (TEM, JEM-200CX Jeol). Hydrodynamic diameter (HDD) and zeta potential was analyzed by dynamic light scattering spectroscopy (DLS, ELS-Z Photal). The adsorption capacities were measured by inductively coupled plasma-atomic emission spectroscopy (ICP-AES, Shimadzu ICPQ 1000).

Adsorption test for isotherm and kinetics

The equilibrium concentrations for three different AgNPs were controlled at 5–200 ppm. Approximately 200 mg/L of activated sludge was added to 40 mL of a solution prepared with a predetermined AgNPs concentration in DW and SSW, followed by shaking at room temperature for 24 h. The samples were separated by filtration, and the adsorption capacities were calculated from the difference between the initial and the final concentrations. A kinetic study was carried out at different time intervals according to the above procedure to determine the removal rate of AgNPs by activated sludge at a concentration of 200 ppm. The rate constants were calculated using the conventional rate expression.

Results and discussion

Characterization of AgNPs

Because bare-AgNP was powder type but citrate- and PVP-AgNPs were colloidal suspensions, the three AgNPs samples have different particle properties in solution. While bare- and citrate-AgNPs are stabilized by electrostatic repulsion mechanism, PVP-AgNP is stabilized by the steric hindrance of PVP. Although bare-AgNP was dispersed in DW or SSW using tip-type ultrasonic, TEM images showed this particle in an aggregated form (Fig. 1). Whereas, individually separated particle distribution is observed in colloidal-type AgNPs (citrate- and PVP-), as shown in Fig. 1.

Citrate with three carboxyl groups (COO^-) immobilized on the surface of AgNP revealed a strong negative charge of the AgNP. Thus, citrate-AgNP was stabilized by electrostatic repulsion, reducing the aggregation between neighboring citrate-AgNPs. PVP with the pyrrolidone group (cyclic organic) immobilized on the surface of AgNPs induced steric hindrance and maintained an appropriate separating distance between neighboring PVP-AgNPs [16]. In addition, carbon and oxygen double bonds in pyrrolidone induced a negative charge, causing PVP-AgNPs to show a weak negative surface charge. As a result, PVP-AgNPs were stabilized by a combination of steric hindrance and electrostatic repulsion.

As summarized in Table 1, the primary particle size of bare-, citrate-, and PVP-AgNP was <120, >20, and <150 nm, respectively. When these materials were dispersed in DW and SSW, the zeta potential was affected by the ionic strength of the solution and

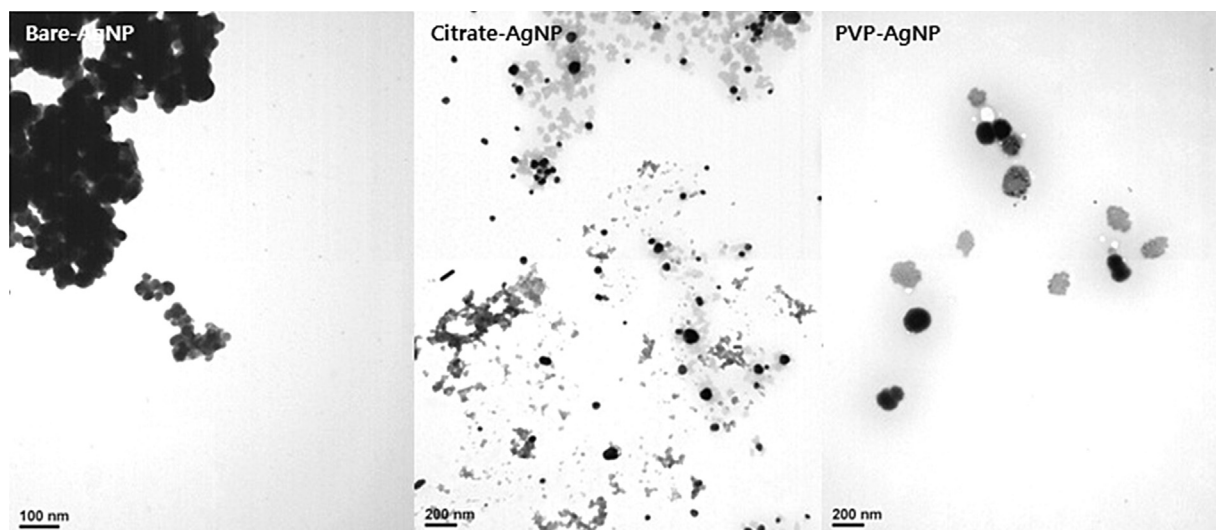


Fig. 1. TEM images of bare-, citrate-, and PVP-AgNP.

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