



Research article

Retention of silver nano-particles and silver ions in calcareous soils: Influence of soil properties



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ABSTRACT

The rapid production and application of silver nanoparticles (AgNPs) have led to significant release of AgNPs into the terrestrial environments. Once released into the soil, AgNPs could enter into different interactions with soil particles which play key roles in controlling the fate and transport of these nanoparticles. In spite of that, experimental studies on the retention of AgNPs in soils are very scarce. Hence, the key objective of this research was to find out the retention behavior of AgNPs and Ag(I) ions in a range of calcareous soils. A second objective was to determine the extent to which the physico-chemical properties of the soils influence the Ag retention parameters. To this end, isothermal batch experiments were used to determine the retention of Poly(vinylpyrrolidone)-capped AgNPs (PVP-AgNPs) and Ag(I) ions by nine calcareous soils with a diversity of physico-chemical properties. The results revealed that the retention data for both PVP-AgNPs and Ag(I) ions were well described by the classical Freundlich and Langmuir isothermal equations. The retention of PVP-AgNPs and Ag(I) ions was positively correlated to clay and organic carbon (OC) contents as well as electrical conductivity (EC), pH, and cation exchange capacity (CEC) of the soils. Due to multicollinearity among the soil properties, principal component analysis (PCA) was used to group the soil properties which affect the retention of PVP-AgNPs and Ag(I) ions. Accordingly, we identified two groups of soil properties controlling retention of PVP-AgNPs and Ag(I) ions in the calcareous soils. The first group comprised soil solid phase parameters like clay, OC, and CEC, which generally control hetero-aggregation and adsorption reactions and the second group included soil solution variables such as EC and pH as well as Cl⁻ and Ca²⁺ concentrations, which are supposed to mainly affect homo-aggregation and precipitation reactions.

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1. Introduction

The widespread and ever increasing production and use of manufactured nanoparticles (MNPs) in various industrial, medical, and consumer products have led to potential release of MNPs into the environment. Silver nanoparticles (AgNPs) are currently the most widely commercialized MNPs because they exhibit distinctive properties such as broad range of antibacterial activity and unique electrical and thermal conductivities (Fabrega et al., 2011). Hence, AgNPs are being increasingly exploited in hundreds of commercially available products like deodorants, textiles, biomedical devices, food packaging materials, electronics, cosmetics, and

pesticides (Bergeson, 2010).

A large fraction of the AgNPs used in consumer products eventually end up in soils, through improper disposal of household, industrial, and medical wastes, as well as land application of sewage sludge, biosolids, and pesticides (Anjum et al., 2013). Bioavailability, mobility, and toxicity of AgNPs released into soils are considerably controlled by their retention by soil constituents (Anjum et al., 2013). Association of AgNPs with mobile soil colloids can facilitate AgNPs transport. On the contrary, retention of AgNPs onto soil immobile solid phase may result in reduced AgNPs bioavailability and potential risk from off-site transport (Cornelis et al., 2013; Pachapur et al., 2015).

Although there is still much unknown about the retention mechanisms of AgNPs in soils, several studies have intended to predict how soil properties control deposition and mobility of AgNPs in terrestrial ecosystems. In these studies, it has been shown

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that physico-chemical properties of soils are very important when determining the retention and mobility of AgNPs (Benoit et al., 2013). For instance, Hoppe et al. (2014) implied that in clay-rich soils a large fraction of the added AgNPs was retained, whereas in soils with lower clay contents AgNPs remained partially colloidal stable. Retention of AgNPs has been found to be dependent on soil clay content in other studies as well (Cornelis et al., 2012; Oromieh, 2011).

Various organic components in soil system may also affect retention behavior of AgNPs in soil. It has recently been shown that humic substances in soil may have an enhancing effect on the mobility of AgNPs (Manoharan et al., 2014). This can be due to the prevention of AgNPs retention onto the soil solid phase by the steric layer of humic molecules which are already sorbed onto the AgNPs and soil surfaces. Similar results were reported by Furman et al. (2013) showing a decrease in AgNPs deposition in the presence of humic acid. On the contrary, Pachapur et al. (2015) reported that organic matter increases aggregation and agglomeration, and therefore reduces mobility of AgNPs in natural soils. The role of organic matter on the fate of AgNPs in soil thus remains controversial and needs further investigation.

Several studies have reported the influence of solution chemistry such as ionic strength (IS), ionic composition, and pH on aggregation and retention of MNPs in aqueous solutions and porous media (Saleh et al., 2008). Hotze et al. (2010) suggested that as IS increases, the electrostatic repulsion decreases and van der Waals forces become dominant between the AgNPs and soil particles. Also, more retention of AgNPs in the presence of Ca^{2+} compared with K^{+} has been reported (Hotze et al., 2010). El Badawy et al. (2010) showed that in environments that are acidic and/or with high IS and in particular those containing divalent cations, AgNPs aggregation and settling may occur. Furthermore, surface complexes of AgNPs with soluble ligands such as chloride and phosphate have been shown to change their dissolution and mobility in the soil (Klitzke et al., 2015; Li et al., 2010). Soil solution pH can also affect retention of Ag ions and AgNPs (Oromieh, 2011). Nonetheless, further studies are required to better understand the retention of AgNPs under conditions that are typical of calcareous soils.

In addition to soil characteristics, aggregation and retention of AgNPs in soil are considerably controlled by surface properties of the AgNPs. Many commercially available AgNPs are coated with capping agents such as borohydride, citrate, and polyvinylpyrrolidone (PVP) (El Badawy et al., 2010). Surface modification of AgNPs with capping agents increases colloidal stability and prevents aggregation by either enhancing their surface charge or introducing steric or electrosteric repulsion (Benoit et al., 2013). El Badawy et al. (2010), for example, showed that PVP modification of AgNPs prevents aggregation of the nanoparticles and increases the stability of the suspension, which in turn increases the mobility of AgNPs in water. Cornelis et al. (2013), however, indicated that PVP-capped AgNPs rapidly attached to the surfaces of soil natural colloids and become immobilized in soil columns. Unfortunately, reactivity of capped AgNPs in different soil types, which vary in their properties, has not been extensively investigated.

Quantification of the interactions between MNPs and soils is critical for developing practical risk assessment and mitigation strategies. Park et al. (2008) studied retention of quantum dot nanoparticles onto model surfaces and concluded that batch isotherm experiments can provide a promising approach for quantifying nanoparticle-surface interactions. They expressed the need of future studies to assess whether the interaction of other nanoparticles with surfaces can be described by classical retention equations. However, to our knowledge, very few systematic studies have been performed to model retention of MNPs in soils. Moreover, the quantitative contribution levels of each reactive

component in soils are not clear. In order to build models for predicting the fate and transport of MNPs in soils, there is a need to relate retention properties of the MNPs with specific soil parameter(s). In the present study, we investigated the utility of classical isothermal and partitioning techniques in providing a useful characterization of the Ag(I) ions and PVP-capped AgNPs retention in a range of calcareous soils. Additionally, relationships between soil properties and Ag retention parameters were determined to identify the major factors responsible for retention of the AgNPs and Ag(I) ions. Any research findings in these areas should be of interest to risk assessors to regulate the commercial and industrial usages, applications, and productions of AgNPs and Ag(I) ions.

2. Materials and methods

2.1. Synthesis of AgNPs

All the chemicals reagents used in our experiments were of analytical grade and were used as received without further purification. A sonochemical method was applied for preparing AgNPs in aqueous polyvinylpyrrolidone (PVP) solutions. In a typical preparation, 0.05 g of PVP was added to 100 mL of aqueous solution containing 0.1 g of AgNO_3 . The PVP acts as both reducing and capping agent. The mixture was stirred for complete dissolution and agitated under sonication. Ultrasound irradiation was carried out with a multiwave ultrasonic generator (Sonicator 3000; Bandeline, MS 72, Germany), equipped with a converter/transducer and titanium oscillator (horn), 12.5 mm in diameter, operating at 20 kHz with a maximum power output of 60 W, was used for the ultrasonic irradiation. The operating condition was at 5 s pulse on and 5 s pulse off time with amplitude of 72% at 25 °C for 20 min.

2.2. Characterization of AgNPs

Transmission electron microscope (TEM) images of the synthesized AgNPs were obtained on a Philips CM30 instrument at an accelerating voltage of 150 kV. Average hydrodynamic diameter and surface charge characteristics of the AgNPs were determined by dynamic light scattering (DLS) with a Malvern ZEN 3600 Nano ZS Zetasizer (Malvern Instruments, UK). Zeta potential was measured with the same equipment between -200 and $+200$ mV. X-ray powder diffraction (XRD) pattern was recorded by a Philips X'Pert Pro diffractometer using $\text{CuK}\alpha$ radiation to determine the crystallite size of the AgNPs. The crystallite size (D) of AgNPs was calculated using Scherrer equation (Moore and Reynolds, 1997):

$$D = \frac{k\lambda}{\beta \cos \theta} \quad (1)$$

where, k is the so-called shape factor, and usually takes a value of about 0.9, λ depicts the wavelength of $\text{CuK}\alpha$ radiation and β is the breadth of the observed diffraction line at it is half intensity maximum (FWHM).

2.3. Soil sampling and characterization

Nine surface soils (i.e., 0–10 cm) with different physical and chemical properties were collected from Isfahan Province, Iran (Table 1). Soil samples were then air-dried, sieved (<2 mm), and stored before analyses. Soil pH and EC values were determined in 1:2 soil:water extracts with a digital pH meter (Cyberscan 2100 model) and an electrical conductivity meter (Elmetron CC-501 model), respectively. The amounts of calcium carbonate equivalent (CCE) (Loeppert and Suarez, 1996), organic carbon (OC) (Nelson and Sommers, 1996), total nitrogen contents (Bremner, 1996) and

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