RESEARCH ARTICLE

Effect of fullerol nanoparticles on the transport and release of copper ions in saturated porous media



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Abstract

While the application and discharge of carbon nanomaterials (CNMs) increased rapidly, the research on the environmental safety of CNMs is also increasing. The high dispersity and mobility of modified CNMs in environmental media may have impacts on the environmental behavior of heavy metals. This work mainly studied the effect of fullerol nanoparticles ($C_{60}(OH)_n$) on Cu^{2+} transport, sorption, and release in water-saturated porous media. The results showed that due to the strong adsorption capacity of $C_{60}(OH)_n$ for Cu^{2+} , the transport of Cu^{2+} could be facilitated. However, with the pre-existence of $C_{60}(OH)_n$ in porous media, the transport of Cu^{2+} was also slightly enhanced. In addition, when loaded into the pre-contaminated porous medium, the $C_{60}(OH)_n$ also enhanced the release of retained Cu^{2+} , which implies a high environmental risk of $C_{60}(OH)_n$.

Keywords Environmental behavior · Copper ions · Fullerol nanoparticles · Facilitate

Introduction

Carbon nanomaterials (CNMs) take up a unique position in nanoscience and technology. Because of their excellent properties such as high chemical stability, mechanical strength, specific surface area, and electrical conductivity, CNMs have been applied in multiple areas (Choi et al. 2010; Eissa et al. 2017; Jariwala et al. 2013; Hou et al. 2016; Kato et al. 2010; Liu et al. 2009). The big amount of production and utilization of CNMs will inevitably give rise to the release of them into the environment, resulting in potential risks to human health and the ecological environment (Hou et al. 2017; Pérez et al. 2009; Selck et al. 2016; Wang et al. 2010).

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Most kinds of CNMs show low stability and mobility in aqueous environment because of their high hydrophobicity. Therefore, in practical applications, to prepare uniform and well-dispersed CNMs, popular surface modification techniques have been widely applied (Zhu et al. 2003). For example, carbon nanotubes could be modified with various functional groups on their surfaces via covalent or non-covalent methods (Bolong et al. 2009; Bougeard et al. 2010; Chowdhury et al. 2011; Tian et al. 2013) to improve their hydrophilicity and dispersity in aqueous media (Ahmed et al. 2012; Saleh et al. 2008; Treviñocordero et al. 2013).

Numerous studies have shown that modified CNMs had high stability and mobility, and particularly, the surface modification could considerably enhance the adsorption ability (Rao et al. 2007). In an aquatic environment, the presence of colloidal modified CNMs might increase the risk of colloidfacilitated transport of adsorbed environmental pollutants (Vendelboe et al. 2012). For example, Johnson et al. (2009) have shown that nanoparticles have a strong adsorption capacity for soil organic matters and can promote their mobility in porous media. Zhang et al. (2011) reported that fullerene nanoparticles could significantly enhance the transport and mobility of organic contaminants polychlorinated biphenyl (PCB) and phenanthrene.

Furthermore, heavy metals, as a group of the typical environment contaminants, could also interact with CNMs in an aqueous environment (Li et al. 2013; Rao et al. 2007; Yuan and Peng 2017). Studies proved that CNMs had strong adsorption ability for heavy metals, and the mechanism was mainly the electrostatics attraction, complexation between heavy metal ions and the surface functional groups of CNMs, and the sorption-precipitation (Gupta and Saleh 2013). Yuan and Peng (2017) found that in the saturated aqueous environment, C₆₀(OH)_n facilitated Cu²⁺ transport mainly by decreasing the retention factor of Cu²⁺ on porous media, and pH and C₆₀(OH)_n concentration had significant effects on it. Moreover, Newman et al. (1993) found that the association with mobile colloids could facilitate the transport of metals through soil-packed columns, and alterations in eluent chemistry had apparent influence on it, such as pH and ionic strength. Most natural organic matters were found to significantly increase the metal transport by complexation in aqueous system (Schmitt et al. 2003). Bradford and Kim (2010) reported that the transport of Cu²⁺ was dramatically enhanced by adsorption on clay colloidal particles that were released following cation exchange and ionic strength reduction in porous media.

However, to date, the research on the influence of CNMs on the transport and release of heavy metals in environmental media is still limited. In this study, fullerol nanoparticles and Cu^{2+} were respectively selected as representative materials of CNMs and heavy metals; co-transport experiments of Cu^{2+} and fullerols in water-saturated porous media were conducted to investigate the environmental risks of the fullerols. However, in some cases, the existing order of fullerols and Cu^{2+} in the environmental media was different; therefore, the transport experiments of Cu^{2+} in fullerol-existed porous media and the release experiments of Cu^{2+} under the effect of fullerols in pre-contaminated porous media were conducted respectively to further clarify the environmental risks of fullerols.

Methods

Materials and characterization

Solid fullerols was purchased from Nanjing XFNANO Materials Tech Co. Ltd. (Nanjing, China). It was claimed to have 18 to 28 hydroxy functional groups by the producer. The fullerol dispersion utilized in this research was prepared by simply weighing a predetermined amount of solid fullerols and adding into a certain amount of DI water. Other chemicals of guaranteed reagent (GR) grade, including copper nitrate, nitric acid, sodium hydroxide, and sodium chloride, were provided by Sinopharm Chemical Reagent Co. Ltd. Quartz sand was bought from Beijing Shuangxiangda Instrument Sales Department. The average particle size of the quartz sand was measured by a Laser Diffraction Particle Size Analyzer (Mastersizer 2000, Malvern Instruments. Ltd. UK) to be 523 μ m. The UV-visible Spectrophotometer (DR5000, Hach Company, USA) was used to determine the concentration of fullerol dispersion. The measurement was conducted at 342 nm with 10-mm quartz cells, where a good correlation between the absorbance and the fullerol concentration was observed. The particle morphology of fullerol dispersion was observed by TEM (H-7500, Hitachi Ltd. Japan). The data used in each figure were taken from the average of three replicate experiments, and the standard deviation of each group of data was within $\pm 5\%$.

Copper transport experiments

Batch column experiments were employed to study the cotransport of fullerol nanoparticles and Cu²⁺. The experimental setup is shown in Fig. 1. Polymethyl methacrylate (PMMA) columns with a height of 12 cm and the inner diameter of 3 cm were employed in the column experiments. Cleaned dry quartz sand was used as the porous media for convenient operation, and it was dry-packed into the column at several times. For each time, 1–2-cm height of sand was packed with slight patting on the column side face. After that, the porosity of the packed column was measured to be 0.38 ± 0.01 . For each column, DI water was input upward for at least 24 h at a relatively fast groundwater flow velocity of 60 ± 0.5 cm/h by a peristaltic pump to avoid bubbles and stabilize the flow velocity. Then a certain amount of influent solution was loaded into the column upward.

Two different influent solutions were used in the experiment of Cu^{2+} transport with fullerol nanoparticles. One was pure Cu^{2+} solution with a concentration measured to be 10.01 mg/L, and another was mixed Cu^{2+} solution and fullerol dispersion where the Cu^{2+} concentration was 9.89 mg/L and the fullerols concentration was 100 mg/L. The concentration of Cu^{2+} and fullerol



Fig. 1 The apparatus of the column experiments

was selected based on a typical contaminative concentration. Before being input into the column, the mixed solution was stirred at 25 °C, 170 rpm for at least 24 h for complete adsorption. After the column was flushed by DI water, the influent was input upward and the effluent was sampled at a fixed interval by a fraction collector (SBS-100, Shanghai Qing Pu-Hu xi Instruments Factory, China). The Cu²⁺ concentration of samples was analyzed by an Inductively Coupled Plasma Optical Emission Spectrometer (ICP-OES) (ICPE-9800, Shimadzu Corporation, Japan).

The column setup was the same as mentioned above when studying Cu²⁺ transport in fullerol-existed porous media. After the flow velocity was stable, 100 mg/L fullerol dispersion or DI water was loaded upward into the column at the same velocity for about 3.5 h. Effluent samples of fullerol dispersion were collected to calculate the fullerol retention rate. After that, 10.01 mg/L (C₀) of Cu²⁺ solution was input as influent, and the effluent samples were collected at a fixed interval. Cu²⁺ and fullerol concentration were measured by the methods mentioned above.

Copper release experiments

The Cu²⁺ release from the pre-contaminated porous media was investigated by column experiments. The column setup was the same as mentioned above and the porosity was measured to be about 0.39. Firstly, the column was flushed with DI water at about an average flow velocity of 12 cm/h for at least 24 h. After flushing the column, 10.01 mg/L Cu²⁺ solution was loaded upward into the column at the same velocity for about 16 h to make sure that the media were completely contaminated. Measurement of the effluent showed that no Cu²⁺ was detected in the effluent, which means that the Cu²⁺ was most retained in the column and the porous media had already been contaminated. Then 100 mg/L fullerol dispersion or DI water was loaded into the column upward to flush the column for about 20 h. Effluent samples were collected to plot the release curve.

Fig. 2 TEM images of fullerol nanoparticles. $\mathbf{a} \times 60,000$; $\mathbf{b} \times 300,000$

Adsorption isotherm

The adsorption isotherms of Cu²⁺ on fullerols and porous media (quartz sand) were studied. Firstly, a certain amount of Cu^{2+} solutions was added into a series of 50-mL centrifuge tubes containing 50 mg/L C₆₀(OH)_n dispersion. The tubes were shaken at 25 °C, 170 rpm for 24 h and then sampled with an injector. To remove $C_{60}(OH)_n$ completely, samples were filtered using a 3 KD Millipore Ultrafiltration Device centrifuged at 10,000 rpm for 20 min. Then the Cu²⁺ concentration was analyzed to obtain the adsorption isotherm of Cu²⁺ on fullerols. Secondly, a certain amount of Cu²⁺ solutions was added into a series of 50-mL centrifuge tube and then 5 g cleaned and dried quartz sand was added into the tubes. The tubes were shaken at 25 °C, 170 rpm for 24 h and then sampled with an injector. The sample was filtrated using a 0.22-µm PES filter to completely remove quartz sand, and then the Cu²⁺ concentration was analyzed to obtain the adsorption isotherms of Cu²⁺ on quartz sand.

Results and discussion

Morphological properties of fullerol nanoparticles

Fullerol nanoparticles used in this study are a derivative of fullerene C_{60} . Fullerols have the same basic structure as fullerenes, which in this case are the closed carbon cages formed by 12 pentagons and 20 hexagons (Li et al. 1993). However, compared to the highly hydrophobic fullerene particles, fullerols are partly hydrophilic because of its abundant hydroxy groups on the surface of the carbon structure, which makes fullerol easily to disperse in an aqueous environment and to form very fine particles.

The morphology of fullerols in an aqueous environment was observed using TEM and the image is given in Fig. 2. When dispersed into water, fullerols will form nano-size particles whose diameters are about 90–150 nm, and these nano-particles may gather to form bigger aggregates with diameters of hundreds of nanometers. The good dispersity in water as



well as the nano-scale particles formed by fullerols indicates that fullerol nanoparticles may have high possibility to interact with copper ions in water solutions when existing at the same time.

Transport of copper ions with fullerol nanoparticles

The effect of fullerol nanoparticles on Cu²⁺ transport is shown in Fig. 3. Obviously, the existence of fullerol nanoparticles enhanced the transport behavior of Cu²⁺ in saturated porous media. Without fullerol nanoparticles in the influent solution, the breakthrough of Cu^{2+} started at 9 PV, and the C/C₀ value reached 0.48 at 14 PV. However, when transported with fullerol nanoparticles, Cu²⁺ was detected in the effluent at 2 PV, and then the breakthrough curve experienced a quick increase from 2 PV to 4 PV and after 14 PV, the C/C₀ value reached 0.79. The transport of Cu²⁺ with fullerol nanoparticles showed similar breakthrough pattern as fullerols transport which was studied in previous research (Yuan and Peng 2017), which the C/C₀ value reached over 0.8 within 10 PV, and from 2 PV to 4 PV, the fullerols breakthrough curve rapidly increased. Considering the high dispersity and mobility of fullerol nanoparticles, it is reasonable that fullerols can enhance the mobility of Cu²⁺ when transported together in porous media. However, this phenomenon also indicates that when released into the environment, fullerol nanoparticles may interact with the heavy metal ions and further enhance their aqueous mobility, which could be harmful to the environment.

Transport of copper ions in fullerol-existed porous media

Given that in the realistic aqueous environment, there is usually not enough time for the nanoparticles to adsorb heavy



Fig. 3 Transport of Cu^{2+} with fullerol nanoparticles

metal ions until reaching to the adsorption equilibrium, the experiment of Cu^{2+} transport in fullerol-existed porous media was also conducted.

Figure 4 shows the influent and effluent concentration curves of fullerol nanoparticles. By integrating the two curves, the retention rate of fullerol nanoparticles in the porous media was calculated to be 15.24%. At the same time, since the average flow velocity applied in this experiment is relatively high (60 cm/h), the time for Cu^{2+} to react with fullerol nanoparticles is really limited.

Figure 5 shows the comparison of breakthrough curves of Cu²⁺ in the porous media with and without fullerol nanoparticles, which reflects the effect of the pre-existence of fullerol nanoparticles on Cu²⁺ transport through the porous media. It is surprisingly found that the pre-existence of fullerol nanoparticles could slightly enhance the transport of Cu²⁺ since breakthrough starts at around 2 pore volumes earlier, and the final C/C_0 value (at about 14 pore volumes) reached 0.73, which is higher than that without fullerol pre-existence (0.48). This is contrary to the hypothesis that the retained fullerol nanoparticles in porous media would adsorb Cu²⁺ and thus resulted in a higher retention of Cu²⁺ in porous media. In fact, results in accordance with this hypothesis have been found in other studies. A study found that under relatively high ion strength, the average percent elution of gold nanospheres is lower for the coated sand by pluronic acid modified single-walled carbon nanotubes, but this effect is very small at low ion strength (Afrooz et al. 2016). Besides, it was reported that graphene oxide (GO) enabled the sand column to increase the metal removal efficiency of Cu and Pb, and higher GO amount led to a higher removal efficiency (Ding et al. 2014). However, in this study, the presence of fullerol nanoparticles in the column did not improve the deposition of Cu²⁺ but enhanced its transport instead. This may indicate that although fullerol



Fig. 4 The influent and effluent concentration curves of fullerol nanoparticles



Fig. 5 Transport of Cu^{2+} with and without fullerols pre-existed in columns



ΡV

0.08

0.06

0.05

0.03

0.0

10 20

nanoparticles partly retained in the column, they did not closely combine with the quartz sand surface. Therefore, when Cu^{2+} solution was loaded into the column, the retained fullerol nanoparticles may be released from the column and at the same time, carrying some Cu^{2+} to transport. Besides, based on some similar study results (Han et al. 2013; Li et al. 2013; Wu et al. 2016, 2017; Yang et al. 2012), the competition for deposition site on the quartz sand surface by Cu^{2+} and fullerol nanoparticles could be another important reason result in the enhanced transport of Cu^{2+} . However, these hypotheses need further experiments to verify.

Release of copper ions in pre-contaminated porous media

To further determine the environmental risk of fullerol nanoparticles, the impact of fullerol nanoparticles on Cu^{2+} release from the porous media was studied by comparing the Cu^{2+} release rate from columns flushed by fullerol dispersion and by DI water. Figure 6 gives the release curves of Cu^{2+} from the pre-contaminated porous media with and without fullerol nanoparticles; the inset figure in Fig. 6 is the magnification of the release curve of Cu^{2+} from 0 PV to 10 PV when the column was flushed by DI water.

When the influent was fullerol dispersion, the presence of Cu^{2+} was detected in the samples from 2 pore volumes, and the highest Cu^{2+} concentration measured was 10.5 mg/L whereas the concentration of Cu^{2+} solution used to contaminate the column was only 10.01 mg/L. By integrating the release curve, the total Cu^{2+} release rate was up to 73.8% when flushed by fullerol nanoparticles. However, when the influent was DI water, only a few Cu^{2+} were detected at around 3 pore volumes, and the release rate of Cu^{2+} was only 0.4%. The results imply that fullerol nanoparticles could significantly enhance the release of Cu^{2+} from the contaminated porous media and further promote its

transport, which was mainly achieved in two steps. Firstly, due to the strong adsorption capacity of fullerol nanoparticles for copper ions, when fullerol dispersion was loaded in, the most copper ions retained in the column were released. Secondly, because fullerol nanoparticles have a very high transport ability in porous media, it could carry the released copper ion transport. Therefore, fullerol nanoparticles should be regarded as having environmental risks.

Adsorption isotherms

12

10

4

2

04

Cu²⁺ Concentration (mg/L)

Figure 7 shows the adsorption isotherm of Cu^{2+} on fullerol nanoparticles. The maximum adsorption capability is almost up to 200 mg/g, which shows that the fullerol nanoparticles have a very high adsorption ability on Cu^{2+} . Figure 8 shows the adsorption isotherm of Cu^{2+} on quartz sand. The maximum adsorption capability is only 0.0911 mg/g. These results indicate that Cu^{2+} is more easily to be adsorbed by fullerol



Fig. 7 Adsorption isotherm of copper ions on fullerols

- input DI water

Þ١

30

- input fullerol solution

40



Fig. 8 Adsorption isotherm of copper ion on quartz sand

nanoparticles other than by the porous media. Thus, the existence of fullerols can have significant influence on Cu²⁺ transport and release due to the adsorption effect.

Conclusions

In this study, based on the high dispersity and mobility of fullerol nanoparticles in water-saturated porous media and the strong adsorption behavior for Cu^{2+} , the effect of fullerol nanoparticles on the transport and release of Cu^{2+} was studied in saturated aquatic environment. The following can be concluded.

- Fullerol nanoparticles can facilitate the transport of Cu²⁺ in saturated porous media.
- (2) With the pre-existence of fullerol nanoparticles in saturated porous media, the transport of Cu²⁺ is slightly increased, which may be because the release of retained fullerols when Cu²⁺ solution is loaded into the column and the competition for deposition site on the quartz sand surface between fullerols with Cu²⁺.
- (3) When loaded into the pre-contaminated porous media, the fullerol nanoparticles can increase the release of retained Cu²⁺.

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