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RESEARCH ARTICLE



Removal of Arsenate in Drinking Water Sources by Combined Coagulation Process

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Abstract: The objective of this study is to examine arsenate (As(V)) removal from drinking water sources with combined coagulation processes using Single-Walled Carbon Nanotubes (SWCNTs) and Multiwalled Carbon Nanotubes (MWCNTs). Ulutan Lake Water (ULW) in Zonguldak-Turkey, was used as drinking water source. Conventional coagulation experiments was conducted on using aluminum sulfate (Alum) and ferric chloride (FeCl₃). Water samples were synthesized by spiking 300 µg/L As(V) into ULW samples and also all arsenic removal tests were performed with As(V). The maximum removal percentages of As(V) (97%) was observed with combined SWCNTs and FeCl₃ in ULW. Similar to that of SWCNTs, the removal of As(V) (92%) during the coagulation processes occurred at MWCNT with the addition of FeCl₃. Compared to SWCNTs, the removal percentage of As(V) was slightly lower when using only MWCNTs (76%). This result demonstrated that SWCNTs were generally more powerful than MWCNTs for removing the As(V). The presence of humic acid (HA) increased As(V) removal with related the solution pH. On the other hand, the changing of As(V) residual concentrations in ULW was observed as a function of pH and the removal of As(V) increases in the acidic pH levels whereas decreases alkaline pH levels. While As(V) removal efficiency was remained constant at acidic pH values, it decreased about 10% at pH 6, 7 and 8 as a result of the competitive adsorption between As(V) and HA. It was observed that the As(V) removal efficiency increased both low and high pH with monovalent electrolyte (NaCl) whereas di-valent ions (Ca⁺² and Mg⁺²) improved As(V) removal only at pH 9 and 10 during the coagulation processes in ULW samples. The results of this study display that combined coagulation process is more effective than conventional coagulation alone for the As(V) removal.

Keywords: Arsenate removal, carbon nanotubes, water treatment, combined coagulation.

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INTRODUCTION

The shortage in freshwater resources due to the increased human population and industrial activities is one of the most challenging issue existing in the world in the field of water supply (1,2). According to many studies about toxicity of arsenic, which is a well-known poison, it is carcinogenic for humans (3-5). In other words, arsenic creates a risk of pulmonary, dermal, bladder, and hepatic cancer in humans (6-8). Due to its negative effects on human health, the WHO has set the maximum contaminant

level for arsenic in drinking water as 10 μg L $^{-1}$ (9, 10).

Arsenic exists in aqueous systems in both inorganic and organic forms. Major treatment techniques have included chemical coagulation-flocculation, ion exchange and adsorption, and membrane processes (11-14). Coagulation and filtration known as conventional treatment technology is widely used for removing arsenic in surface water sources because of its economy and simplicity. In water treatment processes, adsorption processes are generally applied due to the simple design and operation as well as the small space requirement. In the literature, it has been reported that many adsorbents such as iron oxide, aluminum oxide, and activated carbon are effective for arsenic removal (15). CNTs have some distinctive features such as high reactivity, small size, easy separation, catalytic potential, large surface area, and large number of active sites that facilitate better arsenic removal efficiency compared to other available adsorbents (16). Though nano adsorbents give better results compared to other adsorbents for arsenic removal, there are two main challenges in usina nanomaterials for water purification; one is the nonavailability of CNTs at economically affordable prices and the other is the toxicity and the environmental fate of nanomaterials. Additionally, research has shown that CNTs can enter the body through the skin, respiratory tract, or gastrointestinal tract. The objective of the present study is to investigate the removal of arsenic as As(V) in drinking water sources through a combination of coagulation with CNTs. SWCNTs and MWCNTs were used as CNTs for determining to the removal efficiency of arsenic in the coagulation process at the presence of Alum and FeCl₃. ULW, an important drinking water source that provides nearly 35,000 m³ of raw water to the drinking-water treatment plant of Zonguldak city, Turkey. SWCNTs and MWCNTs are also used as coagulant materials to remove arsenic in ULW by a new water treatment technique involving a novel

combined coagulation process. On the other hand, very few studies on the use of combined coagulation process with CNTs have been reported for arsenic removal at the range of world. In addition, the novelty of study is the first attempt in Turkey to examine the removal of arsenic from drinking water sources by a combined coagulation process using CNTs.

EXPERIMENTAL

Source water and sampling

The water samples used in this study were collected from raw water entering ULW, Zonguldak- Turkey at four different times. ULW is the water reservoir that contributes significantly to Zonguldak's drinking water treatment plant. The some important chemical properties of ULW are presented in Table 1. Collected samples were quickly sent to the laboratory. Raw water samples were passed through 0.45 µm membrane filter papers within 24 h and stored in a refrigerator at 4 °C. Water samples were synthesized by spiking a required amount of As(V) $(300 \ \mu g/L)$ into ULW prior to the coagulation experiments. The common form of arsenic found in surface waters is As(V). Therefore, arsenic removal tests were performed with As(V). Stock As(V) solutions was prepared with Na₂HAsO₄.7H₂O. The chemical coagulant stocks, 1 g/L ferric chloride (FeCl₃.6H₂O) and alum (Al₂(SO₄)₃.18H₂O) were prepared with deionized (DI) water, respectively.

Parameters	Unit	Range	Average
pH	-	7.21-7.85	7.53
Turbidity	NTU	4.41-7.55	5.98
Conductivity	μS/cm	432-513	473
Total hardness	mg CaCO₃/L	126-166	146
Alkalinity	mg CaCO ₃ /L	105-123	114
Temperature	°C	10-17	14
As(V)	µg/L	<10	<10
TOC	mg/L	4.05-4.81	4.43
SO4 ²⁻	mg/L	22.1-30.2	26.15
Na ⁺	mg/L	12.3-15.2	13.25
K+	mg/L	1.83-2.1	1.97
Mg ²⁺	mg/L	6.61-7.89	7.25
Ca ²⁺	mg/L	38.7-42.6	40.66
Iron	mg/L	0.05-0.08	0.07

Table 1: Physicochemical characteristics of ULW sam
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Coagulation procedure

Stock solutions containing 5,000 mg/L of the SWCNTs and MWCNTs were prepared by adding 1 g of the CNTs to 200 mL of DI water and stirring with a magnetic stirrer at 600 rpm. The ferric chloride and alum were consistently used for As(V) removal at similar dosages as coagulant. ULW was treated with coagulants in the ranges of 0–100 mg/L and 0–50 mg/L, respectively. However, based on economic

and engineering considerations, 80 mg/L was selected as the optimum coagulant dosage. As the coagulation combined was analvzed, and preliminary testing was applied to determine the optimal coagulant dose for raw water samples. The optimum combined coagulant dosage for ULW was determined as 40 mg/L. Additionally, after conventional combined coagulation and experiments, the treated water was taken with a

syringe and filtered through $0.45-\mu m$ membrane filter. For determining As(V), samples were preserved in dilute HNO₃ (final conc. 0.1%, pH < 2). Additionally, after conventional and combined coagulation experiments, the treated water was taken with filtered through 0.45- μm membrane filter.

Purified CNTs

One gram of raw CNTs was dispersed into a 100-mL flask containing 40 mL of mixed acid solutions (30 mL of HNO₃ +10 mL of H₂SO₄) for 24 h to remove metal catalysts (Ni nanoparticles). After cleaning, the CNTs were again dispersed in a 100-mL flask containing 40 mL of the mixed acid solutions, which were then shaken in an ultrasonic cleaning bath (Branson 3510 Ultrasonic Cleaner, Connecticut, USA) and heated at 80 °C in a water bath for 2 h to remove amorphous carbon. After cooling to room temperature, the mixture was filtered with a 0.45µm glass-fiber filter, and the solid was washed with deionized water until the pH of the filtrate was 7. The filtered solid was then dried at 80 °C for 2 h to obtain the purified CNTs. This procedure for purifying CNTs has been used by other researchers in previous CNT studies (17). After purifying the CNTs, a simple coagulation process with application of alum was used for precipitating CNTs from the solution and thus, CNTs particles were recollected. Then, the residual CNT waste was sealed carefully using double layers of polyethylene bags and transported to solid waste incineration plants.

Analytical methods

The experimental analysis such as TOC and UV and As(V) measuruments were performed according to in Standard Methods (18). TOC analyses were performed with a Shimadzu TOC-5000 analyzer equipped with an auto sampler77 according to the combustion–infrared method described in Standard Methods 3510-B (18). The sample was injected into a heated reaction chamber packed with a platinum-oxide catalyst oxidizer to oxidize organic carbon into carbon dioxide gas. UV_{254} absorbance

measurements were performed in accordance with Standard Methods 5910-B (18) using a Shimadzu 1608 UV-vis spectrophotometer at a wavelength of 254 nm with a 1-cm quartz cell. The samples were first passed through a 0.45-mm membrane filter to remove turbidity, which can interfere with the measurement. Distilled ultrafiltered water was used background correction the the as in spectrophotometer. As (V) analyses were performed with ICP-OES equipped with a manual hydride generator at 188.9 nm wavelength (PerkinElmer SIMAA 6000 AAS) according to the hidride atomic absorption method described in Standard Methods 3114-B (18). Analyses of Ca⁺², Mg⁺² and the other ions in water samples were carried out using the Dionex IC 3000 system (USA) equipped with an AS-19 analytical column and an AG-19 guard column as per USEPA method 300.1 and also its analytical detection limit is 0.001 ± 0.0001 mg/L.

RESULTS AND DISCUSSION

As(V) removal by with coagulation using CNTs Figure 1 (a) shows the removal of As(V) by combined coagulation proceses. Using SWCNTs as a coagulant without the addition of a conventional coagulant (Alum or FeCl₃), the highest As(V) removal was recorded as 77%. On the other hand, with the application of alum, the removal of As(V) increased nearly 10%. The higher arsenate removal (>80%) was observed with combined coagulation process (SWCNTs + Alum). In other words, the percentages of arsenate removal was not determined after the optimum SWCNT doses (40 mg/L) with addition of alum (Figure 1). This result can be explained that the electrostatic attraction plays a very important role in facilitating the coprecipitation and also the colloidal properties of the metal salt precipitation is more effective on As(V) removal by coagulation or combined coagulation processes with Al³⁺ and Fe³⁺ coagulants. Similar observations have been reported by some authors (19, 20).



Figure 1: Removal of As(V) by SWCNTs and combined coagulation (left). Optimum coagulant dose=40 mg/L. Removal of As(V) by MWCNTs and combined coagulation (right), Optimum coagulant dose=40 mg/L.

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The highest removal of arsenate (97%) is recorded with the application of $FeCl_3$ (SWCNTs + $FeCl_3$). Figure 1 (b) demonstrates the removal of As(V) as a result of the application of MWCNTs and conventional coagulants during the coagulation. Arsenate removal obtained using only MWCNT was determined as 76%. On the other hand, with the application of using only SWCNTs, removal percentage of As(V) was measured as 82%. This observation indicates that SWCNTs has larger surface area in adsorption processes, as compared to MWCNTs, resulting in more adsorption sites for chemicals. As shown in Figure 2, the addition of alum increases As(V) levels in combined coagulation process. Figure 2 compares the removal percentage of As(V) using conventional coagulation only and combined coagulation processes for ULW. The removal of As(V) also remained constant at MWCNT doses of 40 mg/L or greater (82%). As the

combined coagulation process is done, high As(V) removal percentages (>%95) was observed in ULW. Although the As(V) removal was 51% with only alum, higher As(V) removal was determined when using only $FeCl_3$ (63%) than that with alum. A significant increase was seen when $FeCl_3$ was combined with SWCNTs compared to the use of FeCl₃ only. The highest arsenate removal rate was achieved in coagulation with carbon nanotube types and $FeCl_3$ in ULW water samples. Another trend was observed for As(V) removal using alum, which produced the highest As(V) removal alone and combined with SWCNTs (81% and 88%) in ULW samples. Overall, compared to the As(V) removal achieved by employing only conventional coagulation, the combined coagulation is more effective treatment method for drinking water sources including arsenic.



Figure 2: Comparison of As(V) removal using conventional combined coagulation processes.

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Figure 3: The effect of pH on As(V) removal by conventional coagulants (left), CNTs (right).



Figure 4: The influence of humic acid (HA).

Factors affecting the As(V) removal

The arsenate residual concentrations in ULW during coagulation processes as a function of coagulant dose and pH are shown in Figure 3.

The As(V) residual concentrations in ULW due to aluminum and iron oxides/hydroxides generated in coagulation illustrated variable reductions between pH 7 and 10, while at pH 5 and pH 6 the concentrations were more consistent (Figure 3a). The As(V) residual concentrations at pH 5 and 6 were similar, and comparison of removal efficiencies at pH 8 and 10. It was shown that the As(V) removal decreased with increasing pH during the coagulation process. It was reported as based on some literature researches (21, 22). This result also can be revealed that during the coagulation, Al³⁺ and Fe³⁺ hydrolyzed and formed different cationic complexes at acidic pH. On the other hand, the As(V) changes of residual concentrations demonstrate a similar trend for SWCNTs and MWCNTs (Figure 3b).

The presence of HA in solution strongly influences As(V) removal by coagulation with CNTs (23). A concentration of 4.25 mg C /L HA was used to examine its effects on the As(V) removal as a function of pH. The results are presented in Figure 4.

As can be seen in Figure 4, the increasing pH value changed the influence of HA on arsenate removal

both conventional and combined coagulation. Similar studies were reported by many researches previously (24). At pH 5, with the addition of HA, As(V) removal efficiency about remained constant at the original maximum removal. Nevertheless, As(V) removal reduced by 5%, 10%, and 15% at pH 6, 7, 8 and 9, respectively. HA has high affinity for the surface of alum and iron oxides, which could modify their properties and block the adsorption sites for As (V). Further, HA was found to readily form both aqueous and surface inner-sphere complexes with cationic metals, which would in turn associate strongly with other anions like those of As (V) by metal-bridging mechanisms.

This trend results in to reduce the competitive adsorption between As(V) and HA on the coagulated iron and aluminum hydroxide and also increases to the As(V) removal. The findings of experimental study confirmed that the removal efficiency of As(V) were increased as the pH increased at the presence of HA and the combined coagulation treatment mechanism was very effective on As(V) and HA removal for studied pH levels in ULW samples.

Ca²⁺ and Mg²⁺ ions are widely available in surface waters. In this part of study, 0.58 mM Ca(OH)₂ and MgCl₂ were used combined coagulation experiments with SWCNTs+FeCl₃ in order to observe the effect of divalent ions (Ca²⁺ and Mg²⁺) on As(V) removal due to the different pH values. The results are presented in Figure 5. The addition Ca(OH)₂ and MgCl₂ in ULW

samples including As(V) caused some changes on coagulation process. Both Ca^{2+} and Mg^{2+} enhanced As(V) removal efficiency, especially at pH 9 and 10.

It has been reported that the presence of Ca^{2+} increased the adsorption of As(V) on hydrous alum and ferric oxide and SWCNTs at high pH.



Figure 5: The influence of divalent ions (Ca^{+2} and Mg^{+2}) and monovalent electrolyte (NaCl) on the removal of As(V).

However, Ca²⁺ and Mg²⁺ did not affect As(V) removal between pH 5 and 8. These findings with the results of similar coincide studies conducted by Qiao et al. (25) and Meng et al. (26) on arsenic removal. On the other hand, to investigate the change of mono-valent ions on the As(V) at this study, the level of 10 mmol/L NaCl was used in the combined coagulation with SWCNTs+FeCl₃.

The results also show in Figure 5 that at the high NaCl concentration of the coagulated suspension was significantly increased the As(V) removal both low and high pH values. Furthermore, monovalent electrolyte (NaCl) have very much stronger effects on As(V) removal than on As(V) removal effects of divalent ions.

Mechanisms of As(V) removal

SWCNTs and MWCNTs provide sufficient adsorption sites as well act as a good supporting material for other coagulants. Coprecipitation and adsorption are both active As(V) removal mechanisms. Large specific surface area, light mass density, purity, and strong interaction between CNTs and pollutant molecules made the CNTs extensively studied not only adsorbent but also coagulant coagulation data CNTs and conventional with for ULW samples at pH 6 are shown together in Figure 2. As seen in Figure 2, As(V) removal by combined coagulation was efficient than with only conventional more coagulants. In other words, higher As(V) removal percentages (>90%) were observed when using the combined coagulation whereas As(V) removal was recorded as 51% and 63% with only alum and FeCl₃, respectively. On the other hand, the differences in As(V) removal efficiency between combined and conventional coagulation were high. Moreover, especially for Alum and FeCl₃, the differences were more pronounced at low dosages than that at high dosage. The results from these experiments demonstrate that adsorption onto precipitated AI and Fe hydroxides are the main mechanism for As(V) removal by conventional

coagulants but precipitation/coprecipitation also plays specific roles at low dosages for CNTs. During coagulation process, As(V) removal depended on the coagulant dose and consequently on the number of active sites on the hydroxide surface. For instance; as As(V) removal was observed as 28%and 35% with the only alum and $FeCI_3$ dose of 20 mg/L, higher As(V) removals (65-72%) was determined with SWCNTs+Alum and SWCNTs+FeCl₃, respectively, at the same dose (Figure 2). Similar trends were determined for MWCNTs+Alum and MWCNTs+FeCl₃. This result shows that surface complexation and electrostatic attraction govern the coagulation behavior of As(V) on SWCNTs and MWCNTs and presence of functional groups on the surface are the other significant factors for higher As(V) removal during the combined coagulation compared to conventional coagulation. Also, the extraordinary properties of CNTs such as large surface area, uniformly distributed pores and presence of functional groups on the surface are the other significiant factors for higher As(V) removal. Similar results were reported by some researchers (27, 28).

CONCLUSION

The combined coagulation process using carbon nanomaterials to determine the removal efficiency As(V) for different pH levels and the presence of HA was investigated in this study. Experiments demonstrated that SWCNTs were more effective than MWCNTs in removing As(V) in ULW. This is probably because of the smaller diameter and the larger surface area of the SWCNTs as compared to MWCNTs. The highest removal percentage of (97%) arsenate was observed with the SWCNTs+FeCl_{3.} Also, the As(V) removal was lower when using only conventional coagulants in ULW. Compared to the As(V) removal achieved by employing only conventional coagulation, the combined coagulation treatment generally resulted in higher removal of As(V) in this study. The As(V)removal increased with decreasing pH whereas the decreasing of the As(V) removal for alkaline pH maximum As(V) removal levels. The was determined at pH 5. Additionally, the influence of HA on arsenate removal increased with solution pH. On the other hand, the highest As(V) removal efficiency was recorded at pH 5, with the addition of HA. During to increasing the concentration of the monovalent electrolyte, As(V) removal increases at low (5, 6) and high (8, 9, 10) pH with coagulation processes. As the divalent ions (Ca⁺² and Mg⁺²) were added, the removal efficiencies of As(V) were greatly improved at pH 9 and 10. Results from this investigation show that coagulation using CNTs can be effective in the removal of As(V) from various drinking water sources. Therefore, water treatment plant operators can use the CNTs as coagulants or aid-coagulant matter instead of conventional coagulants, such as those described in this paper, to effectively remove As(V).

CONFLICT OF INTEREST

The author declares that there is no conflict of interest.

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