

Competition of Various Ions Present in Shallow Aquifer Water in Respect of Arsenic Removal by Hydrated Ferric Oxide

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Abstract: Iron-oxides have long been used as adsorbent in the arsenic filter materials. Little is known about the arsenic removal efficiency of these materials in the presence of other naturally occurring ions. Hydrated ferric oxide (HFO) based mesoporous material was prepared freshly in our laboratory by co-precipitating silicic acid and ferric oxyhydroxide from silicate and iron salt solutions. We carried out detailed laboratory experiments to understand how the effect of other ions such as iron, bicarbonate, ammonium and phosphate influence the adsorption of arsenic on HFO at neutral pH. It was found that arsenate and phosphate were in direct competition and the efficiency of the material decreased in the presence of aqueous phosphate. Also, the material can remove carbonate/bicarbonate effectively but arsenic removal efficiency decreases to nearly the same extent as observed for phosphate. The presence of iron in the solution interestingly increases the life time of HFO and hence the efficiency for the removal of arsenic. The results from our experiments suggest that it is essential to know the concentration of other ions along with arsenic in the contaminated tubewell water before it is subjected to adsorption filtration using HFO.

Key words: Arsenic, Ferric oxide, Bangladesh, aquifer.

Introduction

Drinking water arsenic problem emerged out as an environmental and health issue for the last few decades in many parts of the world (Clarke, 2001; Cullen, 2008; McArthur et al., 2001; Safiullah, 2007; Smelly and Kinniburgh, 2002). There are a number of possible ways that arsenic can enter into the groundwater (McArthur et al., 2001; Safiullah, 2007; Smedley and Kinniburgh, 2002). Though the sources of arsenic are mostly natural, some anthropogenic activities are also involved for the contamination of arsenic in subsurface. Natural sources

include mainly the weathering of arsenic-rich rocks and minerals, volcanic activity, and biogeochemical transformations of arsenicals. Among the anthropogenic sources, mining, combustion of fossil fuels, use of organoarsenicals as herbicides and in poultry feed industries, and in wood preservatives are the most common (Smedley and Kinniburgh, 2002). Since the discovery of groundwater arsenic as a potential health hazard, thousands of research articles, reports and news have been published. Those studies include geochemical mapping, biogeochemical cycling, mobilization

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mechanism, and the cleanup measures against arsenic pollution. Health effects in humans associated with the exposure of high level of arsenic motivated the health personnel to study the fate of arsenic compounds in living cells (Bentley and Chasteen, 2002; Cohen et al., 2006; Tchounwou et al., 2002).

As the mobilization of arsenic in groundwater from the natural sources is beyond the human hands, it is only possible to control or to reduce arsenic pollution that occurs from anthropogenic contributions. There are two possible ways to minimize the potential risk of arsenic exposure. They are: (i) the preventive measures by drinking arsenic-free water, and (ii) the health treatment of the people exposed to arsenic. The research presented herein was aimed to make up a cost effective and user friendly arsenic removal technology considering the needs for the underdeveloped communities of third world countries such as Bangladesh. There are several existing methods for the removal of arsenic from the contaminated water (Kartinen et al., 1995). These are: coagulation-precipitation (Boccelli et al., 2005; Pallier et al., 2010; Wang et al., 2014), ion-exchange (Dominguez-Ramos et al., 2014; Pillewan, 2014), reverse osmosis (Boels et al., 2012; Lin et al., 2014; Ning, 2005; Xu et al., 2013), and adsorption-filtration (Mohan and Pittman Jr, 2007; Safiullah, 2007; Singh et al., 2015). In coagulation-precipitation, contaminated water is treated with a ferric salt in the presence of an oxidant and simple aeration is sufficient on most of the occasions. The method, in principle, is simple and efficient but not risk-free. This is because of the possibility of arsenic-rich micro flocks being injected to cause more harm than good in the long run. Besides this, sludge disposal is another problem.

Both reverse osmosis and ion exchange resin can remove arsenic from medium to low concentration level. However, under the economic realities of Bangladesh, these methods are highly expensive. From the studies on the mobilization of arsenic in geochemical environment, it has been seen that the major sink of arsenic in aquatic environment is the iron-rich sediment (McArthur et al., 2001; Safiullah, 2007; Smedley and Kinniburgh, 2002). The adsorption of arsenic on some other mineral surfaces like silicate, Al-oxides, titanium dioxide have also been reported (Smedley and Kinniburgh, 2002). The competencies of these materials have been studied in laboratory for the removal of arsenic and other harmful water soluble species. Most of the studies have used these materials in pure form and the treatments were done in small scales. There are some arsenic removal filters available in market but these, in most of

the cases, are not accessible for economically insolvent groups who are in real need (Johnston et al., 2010).

From the available literatures, it is believed that Himalayan arsenic minerals are the main contributor of arsenic pollution in the Bengal basin (Ahmed et al., 2004; Harvey et al., 2006; McArthur et al., 2004; Nickson et al., 2000; Safiullah, 2007; Tareq et al., 2003). The known anthropogenic arsenic sources are not well known in Bangladesh. The discovery of arsenic in the groundwater of Bangladesh was first reported in 1992 (DPHE, 1993). Since then, our group has been exploring arsenic issues with research and by creating public consciousness at local and national levels. At the same time, we have been trying to develop adsorption-based low cost arsenic removal technology in our laboratory. We already have designed and developed mesoporous HFO based arsenic filter (Safiullah et al., 2003; Safiullah et al., 2004). After intensive laboratory studies, we served the filters in some arsenic contaminated areas on a voluntary basis and still now those are in use locally. The occurrence of arsenic in groundwater is highly associated with the concentration of some other ions such as phosphate, bicarbonate, ammonium and iron (Mahamud-Ul-Hoque et al., 2014, 2002; Safiullah, 2007; Smedley and Kinniburgh, 2002). Most of the research on arsenic removal technologies ignores the combined effect of these ions. The present research focuses how the presence of these ions affects the performance of the HFO based arsenic removing bed filter developed by us. The research is expected to add valuable information in the field of arsenic removing technologies.

Materials and Methods

Preparation of HFO

NM₄, a new adsorption filtration material, was prepared by co-precipitating silicic acid and ferric oxyhydroxide from silicate and trace amount of manganese salt solutions. Resulting precipitate was carefully dried at 80 °C to give rise to porous NM₄ material (Figure 1).

Brief Description of Domestic and Community Filters Based on NM₄

Cylindrical containers with perforation at the bottom were fitted with synthetic wool filters. Filtering materials were placed and another synthetic wool filter with wire mesh at the top was placed and a space for passing in water was left. Two filtration chambers were placed on top of each other and this assembly was placed on top of a collection chamber fitted with a tap (Figure 2).

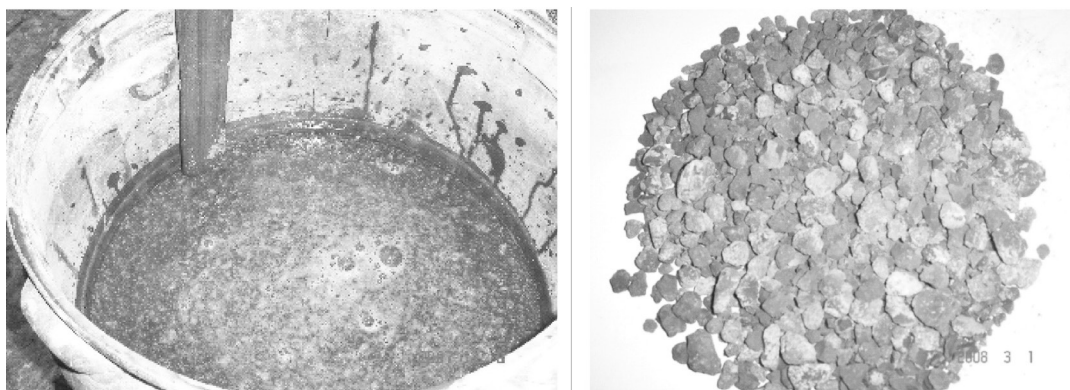


Figure 1: Preparation of (a) HFO based adsorbing material (NM₄); and (b) prepared NM₄ material before grinding.



Figure 2: Setup of arsenic filter prepared using NM₄ (left) and a domestic filter (right) in use.

In case of domestic filter, 5 kg of the material and a collection chamber of 10 L capacity were used. For the community filter 8 kg of filter material and a collection chamber of 60 L were used. Domestic filter has a filtration capacity of 500 L/D; while community filter is of 1500 L/D. Both the filters have expected lifetime of five years and need washing every six months. The iron-oxide based filtration materials, after its life time has expired, are safe to dispose in the environment because no leaching of arsenic is expected at near-neutral pH (Safiullah et al., 2003; Safiullah et al., 2004).

Solution Preparation and Analytical Methods

Two arsenic solutions were prepared initially; a solution of only arsenic and another was a mixture of arsenic and other naturally occurring species. The second type is termed herein as synthetic tubewell water which contained 10 mg/L arsenic, 5 mg/L each of iron(II), bicarbonate and ammonium, and 10 mg/L phosphate by dissolving respective chemicals. To observe the effect of each of the species in arsenic adsorption, controlled

experiments with each of these species were completed. pH of the solutions was adjusted to 7.0 by using dilute HCl and NaOH. The concentration used for these ions in individual solution was same as was in the synthetic tubewell water. Arsenic has pronounced tendency to be absorbed and desorbed by glass; therefore plastic container was used for the sample storage. Also, iron(II) in contact with air may transform into ferric hydroxide and may get precipitated scavenging solution arsenic with it. To overcome this situation, a few drops of nitric acid were added to the prepared samples to suppress the co-precipitation (Safiullah, 2007; Safiullah et al., 2013). The prepared solutions were passed through a series of columns separately at a flow rate ~ 2 mL/min. Each of the columns (1.04 cm inner diameter) was packed with 5.0 g of the filter material. The height of the material in the columns was ~ 10 cm. The effluents obtained for each of the solutions were analyzed for the above mentioned ions.

For the measurement of total arsenic, spectrophotometric method (M-390, Taiwan) was used based on

the arsine (AsH_3) generation by an acid-zinc reaction. The produced $\text{AsH}_3(\text{g})$ was then scrubbed through lead acetate impregnated glass wool and finally allowed to absorb by silver diethyldithiocarbamate (SDDC) solution prepared in pyridine. The colour developed by the AsH_3 -SDDC complexation was measured photometrically at 535 nm. Concentrations of arsenic were determined from standard curve produced by Aldrich arsenic standard. Total iron concentration in water samples was measured spectrophotometrically using 1, 10-phenanthroline at 510 nm against a reagent blank as usual. The concentrations of ammonium and phosphate were analyzed with the respective test kits (E-Merck, Germany) and the HCO_3^- concentration was determined by titrimetric method. Because of the high concentrations of these ions, the samples were diluted to the desired level as required for the determination. All the experiments were conducted in the Department of Chemistry at Jahangirnagar University in Bangladesh.

Results and Discussion

The removal of arsenic from aqueous solution by adsorption filtration depends on the nature of the adsorbent, pH, concentration of arsenic, and the presence of other ions. The efficiency of an adsorbent for the removal of bulk contaminants gradually diminishes with attaining its breakthrough volume. We conducted a comprehensive study on a number of adsorbents for the removal of arsenic. The results show that mesoporous HFO deposited on porous silicate material had the highest removal efficiency for arsenic (Safiullah, 2007). After an extensive research, an HFO based bed filter, NM_4 , has been developed. The HFO material (5 kg) used in each bed filter showed a breakthrough after passing 100,000 L at a flow rate of 700 L/D for the water with 1.6 mg As/L. Along with arsenic, tubewell water of Bangladesh contains significant concentration of other ions such as $\text{Fe}^{2+}/\text{Fe}^{3+}$, NH_4^+ , $\text{HCO}_3^-/\text{CO}_3^{2-}$, and PO_4^{3-} (Mahamud-Ul-Hoque et al., 2014). The presence of these ions in the adsorption/desorption nature of arsenic at the water/sediment interface plays an important role. The efficiencies of HFO based filters could significantly be improved if the influence of the other ions on the arsenic removal is known individually. With this in mind, it was considered worthwhile to study the competitive mode of ions in the adsorption process of arsenic.

Before the start of the experiments, synthesized NM_4 was treated at various temperatures and were investigated with Fourier transform infrared (FT-IR)

spectroscopy. The heat-treated adsorbents were then used for removal of arsenic in our laboratory. From the breakthrough volumes observed for NM_4 treated at various temperatures, in respect arsenic removal, the material showed greater efficiency when heated within 180 to $<250^\circ\text{C}$. The removal efficiency of the material falls off dramatically when heated $\geq 250^\circ\text{C}$. This is expected since HFO could be converted to Fe_2O_3 at $\geq 250^\circ\text{C}$ (Saraswat et al., 1980). This hypothesis is supported by IR spectra collected for NM_4 between 180 and 600°C (Figure 2). At 600°C , the complete disappearance of the surface -OH groups of HFO in the region $3000\text{--}3500\text{ cm}^{-1}$ is observed. The IR spectral features on the thermal transformation of HFO have also been reported elsewhere (Saraswat et al., 1980; Rajendran et al., 1982). The IR bands within $1600\text{--}1700\text{ cm}^{-1}$, for the bending mode of adsorbed water, disappeared completely in the samples heated over 350°C . The role of -OH groups present in HFO for the adsorption of arsenate through ligand exchange reactions were reported previously (Arts et al., 2013; Sabur et al., 2015).

The gradual disappearance of the IR signatures for the surface -OH groups at high temperatures (Figure 3) clearly indicate the damage of porous and mesoporous architecture of HFO in thermally treated NM_4 . The presence and the nature of the -OH groups are very important in the determination of the arsenic adsorption efficiency of HFO (Dzombak and Morel, 1990).

The performance of NM_4 was studied thoroughly for the competitive adsorption of arsenic, iron, bicarbonate, ammonium and phosphate at the initial solution of pH 7.0. It was found that the material exhibits a breakthrough ($>0.05\text{ mg As/L}$) volume $\sim 2500\text{ mL}$ when arsenic is the only species present in solution. The breakthrough volume for arsenic on this material appeared around 2000 mL in presence of other competitive ions. Now, the question is what individual or integrated roles were played by the other ions to reduce the efficiency of the material for arsenic removal. To address this issue, we conducted another series of experiments for the adsorption of each of iron, bicarbonate, ammonium, and phosphate.

The initial flow rates for the solutions were maintained at $\sim 2\text{ mL/min}$ during the adsorption experiments. For all the solutions flow rates started decreasing after passing $\sim 1500\text{ mL}$ of solution and became low ($\sim 1.5\text{ mL/min}$) around 2000 mL . For the combined solution, when the flow rate became low

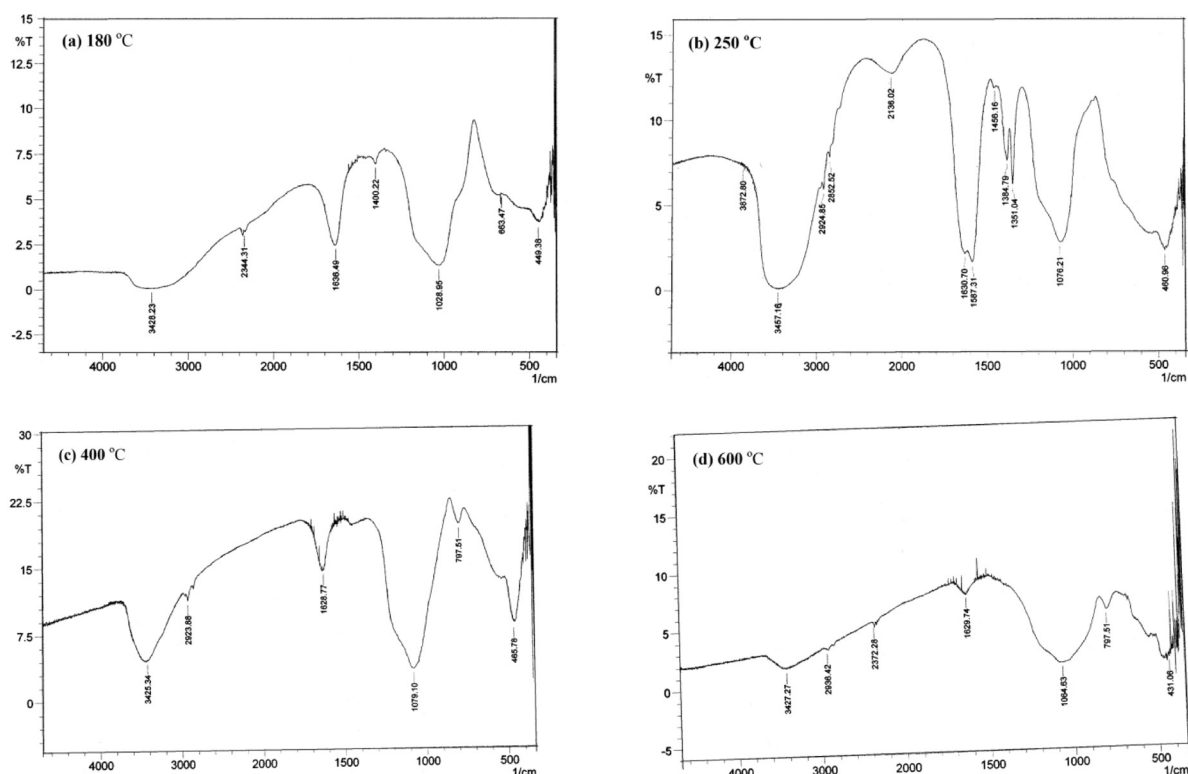


Figure 3: Infrared spectra collected for NM_4 before the adsorption experiments and heated at (a) 180; (b) 250; (c) 400 and (d) 600 °C.

after passing 2000 mL, the material was taken out of the column. After washing with distilled water, NM_4 was then put back into the column. Flow rate improved, but arsenic removal was greatly diminished probably due to loss of fine particles of HFO. The concentrations

of arsenic and other ions in the effluent of the synthetic tubewell water are shown in Figure 4.

Removal of arsenic from contaminated water by adsorption filtration has been proved to be an efficient, cost effective, and user-friendly method (Giles et al.,

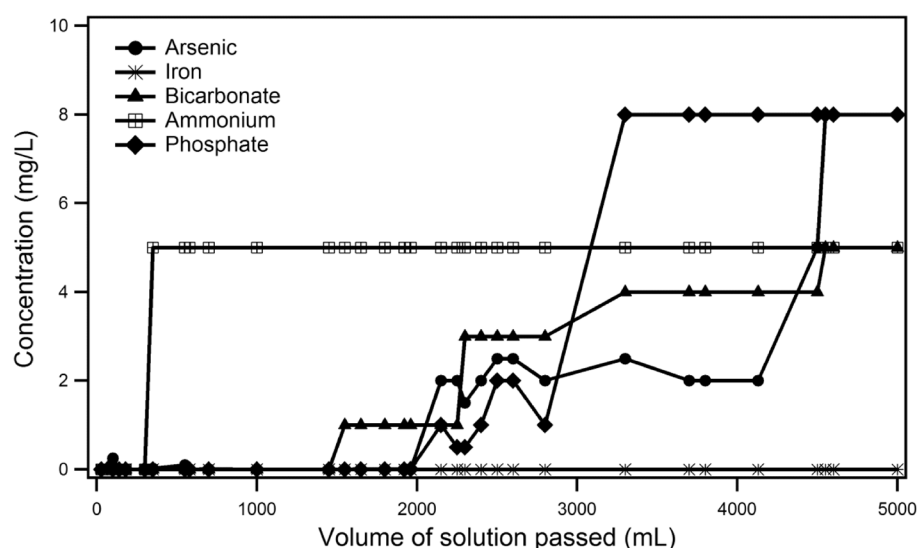


Figure 4: The results of the column adsorption experiment using a standard solution containing arsenic (10 mg/L), iron (5 mg/L), bicarbonate (5 mg/L), ammonium (5 mg/L) and phosphate (10 mg/L) ions on 5 g of NM_4 at 2 mL/min and pH 7.

2011; Kartinen et al., 1995; Mohan and Pittman Jr., 2007; Singh et al., 2015). But endeavour in this respect has been restricted by the adsorption process by which arsenic is removed. Materials like activated charcoal and alumina although remove arsenic during initial runs, ultimately give out adsorbed arsenic in cascade because arsenic is retained in these materials mostly by physical adsorption (Safiullah et al., 2003). The adsorbing material in filters like 3-Kalshi and Sono used in Bangladesh market is actually a very thin layer of HFO deposited on iron fillings (Hussam and Munir, 2007; Neumann et al., 2013). The removal capacities of these filters are easily exhausted at high tubewell arsenic concentration. It was observed that iron fillings used in those filters are irregular shaped solid mass. Congealing of the fillings could be possible due to the chemisorption of carbonate/bicarbonate on the HFO surface. On the other hand, NM_4 manufacturing process in the present study is tailored to produce maximum amount of HFO with high porosity. Previously, we proved that porous and mesoporous HFO can remove arsenic through the process of diffusion-controlled chemisorption (Safiullah et al., 2004).

Arsenic has many competing ions such as iron, bicarbonate, ammonium and phosphate in actual tubewell water in respect of its removal by HFO. From Figure 4, it reveals that HFO has very high affinity for all the ions including arsenic, present in tubewell water. Therefore, when arsenic contaminated water is subjected to HFO based adsorption filtration, arsenic is put in a very competitive situation with respect to other ions present in tubewell water. Arsenic and phosphorous are present in the same group on the periodic table. Both elements are present in the aquatic environment in anionic form with almost similar chemical structure at pH ~7 (Sabur, 2014). A number of studies have been reported on the relative binding affinity of arsenate and phosphate from the adsorption/desorption kinetic and thermodynamic experiments. Most of the studies show that arsenate and phosphate adsorb on the iron-oxide surface in a bidentate fashion. Also, it is possible to remove most of surface arsenicals by desorbing with aqueous phosphate at the same concentration used for arsenate (Arts et al., 2013; Tofan-Lazar and Al-Abadleh, 2012). Previous literatures and our experimental results suggest that arsenate and phosphate are in direct competition for the same binding sites on iron-oxide. Assuming the chemical similarities of PO_4^{3-} and AsO_4^{3-} , it is surmised that the mode of removal of PO_4^{3-} by HFO

is also diffusion-controlled chemisorptions. Therefore, PO_4^{3-} stands in direct competition with AsO_4^{3-} .

The increased concentration of CO_3^{2-}/HCO_3^- is caused by higher arsenic concentration in groundwater (Anawar et al., 2004; Mahamud-Ul-Hoque et al., 2014). Also, some other studies have reported that carbonate binds with the iron-oxide surface through normal chemisorptions (Appelo et al., 2002; Su and Suarez, 1997). In our experiment, concentration of bicarbonate was almost similar up to passing 1500 mL, individually and as the synthetic tubewell water. The concentration of bicarbonate in the effluent started decreasing after passing ~2000 mL for both solutions and became same in the effluent as the influent concentration after passing ~4500 mL. It is surprising that HFO effectively removes arsenic in the presence of HCO_3^- . This is possible if it is assumed that the mode of removal of CO_3^{2-}/HCO_3^- is simple chemisorptions.

Added to these complexities, is the role of adsorbed iron. Figure 4 shows that HFO has an extraordinary affinity for iron. The concentration of iron was still 0 mg/L even after passing 5000 mL of the combined solution. Now, question is what the role of iron in arsenic removal was once it was absorbed by HFO. We hypothesized that the adsorbed iron increased the efficiency of HFO material for the removal of incoming aqueous arsenic. Thus the presence of iron in tubewell water can be expected to prolong the life of HFO as an arsenic removal material. Adsorption filtration based on HFO has a feedback loop in respect of iron present in tubewell water. Similar observation during the preparation of HFO, in the absence of adsorbate ions, has been reported in Mercer and Tobiason (2008).

There is very little indirect evidence on how the presence of NH_4^+ affects the adsorption of arsenic on iron-oxides or oxyhydroxides. The gas phase ammonia can be chemisorbed on the surface of HFO (αFe_2O_3) through the formation of coordination bonds with the vacant sites of the material (Blyholder and Richadson, 1962). But this is unlikely in the case of ammonium ion; the positively charged ammonium being present in solution is not likely to interact with the surface of NM_4 material at pH ~7.0. However, the results of some field-based works indicate that there is a significant correlation between arsenic and ammonium where the mobilization of arsenic takes place through the reductive dissolution of iron minerals (Mahamud-Ul-Hoque et al., 2014). In that case, it only provides indication about the reducing geochemical condition. It

is possible that pysisorption of ammonium on HFO can take place through formation of H-bond with the surface –OH groups. But this physisorbed species should not hamper the chemisorptions of arsenic on HFO. From the results of our experiment it can be inferred that in the presence of other ions, ammonium ion somehow chemisorb on HFO which can be explained by the sharp breakthrough volume of ~350 mL. The breakthrough volume for NH_4^+ in the absence of other ions in the same experimental conditions was ~1000 mL. This is likely that the chemisorptions of ammonium ions could be influenced by HFO surface when other ions were present. But it was beyond our experimental facilities to provide clear evidence.

Our experimental results demonstrate that except the cases of iron and ammonium there is an inflection. This means that the removal efficiency is not exhausted all at once but proceeds with lower efficiency for another interval till the influent and effluent concentrations become equal. Whereas in the case of ammonium, effluent water immediately attains the concentration of the ions of influent water as soon as the breakthrough volume is achieved. In case of iron, removal efficiency of the material remained all through at 100% so as to give rise to a straight line at zero iron concentration in the effluent. These observations clearly indicate that iron and ammonium behave differently in their adsorption process on HFO.

Conclusion

Thus from the present work, it is evident for the first time that removal of arsenic from tubewell water by porous HFO is not a simple process but involves complex processes which may be summarized in the following:

- AsO_4^{3-} and PO_4^{3-} are in direct competition.
- $\text{CO}_3^{2-}/\text{HCO}_3^-$ is removed by chemisorptions on HFO which may disturb the porous architecture of HFO and thereby decrease the arsenic removal efficiency of HFO to some extent.
- Iron adsorbed by HFO further increases the efficiency and life of HFO.
- The role of adsorbed NH_4^+ is not as yet clear.

In our research, we could not come up with a clear concept how iron and ammonium are behaving in presence of arsenic on HFO. In future, it needs to be investigated the pattern how iron improves the efficiency of HFO and the role of ammonium as well. The research is significant in improving the effective iron-oxide based arsenic removal materials.

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