A laboratory study for the treatment of arsenic, iron, and manganese bearing ground water using Fe^{3+} impregnated activated carbon: Effects of shaking time, pH and temperature

Prasenjit Mondal, Chandrajit Balomajumder*, Bikash Mohanty

Department of Chemical Engineering, Indian Institute of Technology Roorkee, Roorkee 247667, Uttranchal, India

Abstract

This paper deals with the experimental investigation related to removal of arsenic from a simulated contaminated ground water by the adsorption onto Fe^{3+} impregnated granular activated carbon (GAC-Fe) in presence of Fe^{2+}, Fe^{3+}, and Mn^{2+}. Similar study has also been done with granular activated carbon (GAC) for comparison. The effects of shaking time, pH, and temperature on the percentage removal of As(T), As(III), As(V), Fe^{2+}, Fe^{3+}, and Mn have been discussed. The shaking time for optimum removal of arsenic species has been noted as 8 h for GAC-Fe and 12 h for GAC, respectively. As(T) removal was less affected by the change in pH within the pH range of 2–11. Maximum removal of As(V) and As(III) was observed in the pH range of 5–7 and 9–11, respectively, for both the adsorbents. Under the experimental conditions at $30\,^\circ C$, the optimum removal of As(T), As(III), As(V), Fe, and Mn are 95.5%, 93%, 98%, 100%, and 41%, respectively, when GAC-Fe is used. For GAC these values are 56%, 41%, 71%, 99%, and 98%. The adsorbent dose (AD) and its particle size (PS) for both GAC and GAC-Fe were 30 g/l and 125–150 μm, respectively. The initial arsenic concentration in the synthetic water sample was 200 ppb.
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Keywords: Arsenic; GAC; Iron-impregnated activated carbon; Surface-modified adsorbent; Ground water

1. Introduction

Arsenic, the world's most hazardous chemical [1] is found to exist within the shallow zones of ground water of many countries like Argentina, Bangladesh, India, Pakistan, Mexico, Mongolia, Germany, Thailand, China, Chile, USA, Canada, Hungary, Romania, Vietnam, Nepal, Myanmar, Cambodia, etc. in various concentrations. In some places in Bangladesh its concentration is as high as 1000 $\mu g/l$ [2].

Arsenic contamination in water has posed severe health problems around the world. Considering the lethal impact of arsenic on human health, environmental authorities have taken a more stringent attitude towards the presence of arsenic in water. World Health Organization (WHO) in 1993 and National Health and Medical Research Committee (NHMRC), Australia, in 1996 had recommended maximum contaminant level (MCL) of arsenic in

drinking water as 10 and 7 $\mu g/l$, respectively [2,3]. The MCL of arsenic in drinking water has also been reduced from 50 to 10 $\mu g/l$ by European Commission in 2003 [4].

EPA has decided to move forward in implementing the same MCL of arsenic that is recommended by WHO for drinking water in 1993 [5]. Japan and Canada has reduced the MCL for arsenic in drinking water to 10 and 25 $\mu g/l$, respectively. The MCL for arsenic in countries like India, Bangladesh, Taiwan, China, Vietnam, etc. is also 50 $\mu g/l$ [6].

As the diagnosis and medication of the arsenic related diseases are difficult the treatment of contaminated water as a preventive measure appears to be an effective alternative to combat arsenic poisoning.

Use of surface-modified adsorbents is becoming a recent research field for the development of cheaper arsenic removal technique. Recently, some adsorbents like Cu-impregnated coconut husk carbon, iron oxide coated polymeric materials, iron oxide coated sand, iron oxide coated cement, bead cellulose loaded with iron oxy hydroxide, etc. have been reported [2] for effective adsorption. Relatively very little information is

* Corresponding author. Tel.: +91 1332 270492; fax: +91 1332 276535.
E-mail address: chandfch@iitr.ernet.in (C. Balomajumder).

published on the adsorption of arsenic species by activated carbon; however, the use of iron-impregnated GAC has been reported by some researches [7–10] for removing arsenic from water. In these reports the complete account on the removal of total arsenic As(T), As(III), and As(V) are not mentioned. The optimization of the process parameters, the comparison between the adsorption efficiency of the GAC and iron-impregnated GAC, and the effect of other metal ions, which are frequently present in contaminated ground water, on the removal of arsenic species have rarely been discussed. Wide range of shaking time (10 min to 72 h) has been considered in these studies. Although the effect of pH on the percentage removal of As(III) and As(V) in absence of other metals is reported by some researchers, its effect on the removal of other metals like Fe and Mn is hardly reported. The effect of temperature is also rarely reported.

In this paper attempts have been taken to explore the possibility of the use of the ferric chloride-impregnated GAC to remove arsenic species in presence of iron and manganese ions, which are available frequently in ground water. The effects of shaking time, pH, and temperature on the removal of arsenic species along with Fe and Mn have been reported. The adsorption capacity of GAC and iron-impregnated GAC for the removal of Fe and Mn has also been compared.

2. Materials and methods

All the chemicals were of reagent grade and solutions were prepared by Milli-Q water (Q-H_2O, Millipore Corp. with resistivity of $18.2\,M\Omega\,cm$). The stock solutions of 100 ppm As(V) and As(III) were prepared by dissolving $Na_2HAsO_4 \cdot 7H_2O$ and $NaAsO_2$ in water and filtered through a $0.45\,\mu m$ membrane.

2.1. Procedure

Fifty millilitre of the synthetic water sample containing 200 ppb As_0(T), 2.8 ppm Fe (Fe^{2+}:Fe^{3+} = 1:1) and 0.6 ppm Mn was added in 100 ml vessel containing 1.5 g adsorbent, PS 125–150 mm, which was followed by shaking at 180 rpm at $30\,^\circ C$. The pH of the media was 7.1 and the shaking time was varied from 0 to 24 h. Optimum shaking time was used for studying the effect of pH and temperature on the percentage removal. In each case, after the reaction the solutions were filtered through $0.45\,\mu m$ membrane filter. The filtrate was analyzed for total arsenic by a Perkin-Elmer ICP-MS model ELAN-DRC-e. Arsenic speciation was done by Edwards ion exchange method [11] with correction due to Fe and Mn. The strong base anion resin AGX8 was purchased from Bio Rad. The analysis of iron and Mn were done by atomic absorption spectroscopy (AAS), GBC, Avanta, Australia. Separate experiments were conducted with GAC-Fe and GAC. SEM photograph was taken by an electron microscope, LEO Electron Microscopy Ltd., England. X-ray diffraction pattern was taken from a Cu target X-ray diffractometer model D8 Advance, BRUKER aXS with $2\theta = 5$ to 100°. IR spectra of the adsorbents have been taken by a Thermo FTIR, model AVATR 370 csl coupled with EZOMNIC software version 6.2.

a. Surface charge of GAC at lower pH ($<pH_{ZPC}$) (NPC= $4\delta+$)

b. Surface charge of GAC at pH_{ZPC} (NPC=0)

c. Surface charge of GAC at higher pH ($>pH_{ZPC}$) (NPC=$4\delta-$)
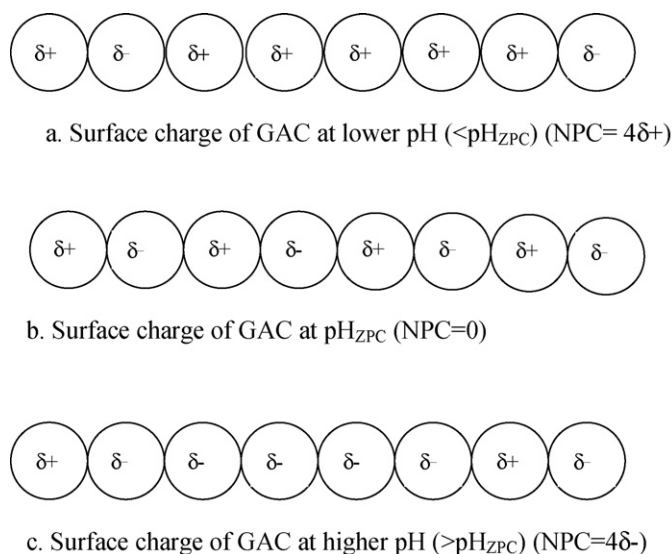
Fig. 1. Schematic presentation of changes in NPC of GAC surface with pH.

2.2. GAC-Fe preparation

One hundred grams of GAC was washed with Millipore water and dried at about $110\,^\circ C$ for 24 h. Then, 20 g of the dried GAC was mixed with 200 ml of ferric chloride solution containing 2.5% Fe^{3+} and pH was adjusted to ~12 by the addition of NaOH solution. The impregnation was carried out at $70\,^\circ C$ on a water bath till the complete evaporation of water and dried at $120\,^\circ C$ for 24 h [12]. The dried material was washed with Millipore water till the washing liquid became free from iron and dried to constant weight. No colour change of wash liquid due to the addition of silver nitrate and KSCN solution indicated absence of chloride and iron in the wash liquid, respectively.

Degree of impregnation of Fe^{3+} onto the GAC surface increases with the decrease in the net positive charge (NPC) of GAC. It will be maximum when net charge of GAC surface becomes negative. Therefore, pH has been increased to 12 for the impregnation of GAC. At this pH maximum attachment of Fe^{3+} is possible which leads to the maximum NPC on the GAC surface. Fig. 1 schematically represents the change in NPC of GAC with pH.

From the above discussion it seems that the NPC of produced GAC-Fe may depend on the concentration of Fe^{3+} ions in the impregnating solution. Initially, NPC of the produced GAC-Fe will increase with the increase in the Fe^{3+} concentration in impregnating solution and an optimum Fe^{3+} concentration will be obtained beyond which NPC will change negligibly. It has been reported by Zu et al. [9] that the optimum concentration for the production of GAC-Fe is equal to 2.34% Fe solution. Accordingly in the present investigation it has been taken as 2.5% Fe^{3+} solution. When this GAC-Fe comes in contact with aqueous media the entrapped Fe^{3+} ions form complex with hydroxyl ions. The release of Fe^{3+} from GAC-Fe during the experiment was found negligible throughout the whole pH range (2–13). There are some reports on the removal of arsenic using iron-impregnated activated carbon [7,10] where no evidence of iron release into the treated solution has been mentioned within this
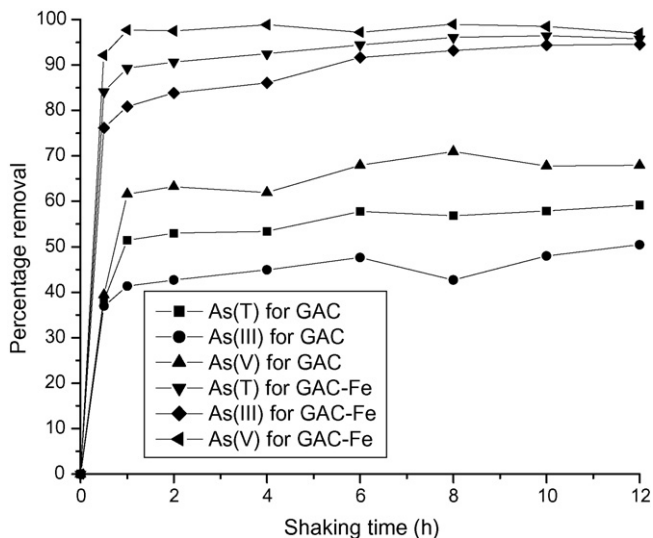
Fig. 2. Effect of shaking time on the percentage removal of arsenic species.

pH range. However, Huang and Vane [8] have found desorption of iron along with adsorbed arsenic using 5 N H_2SO_4 which has pH less than one. They have also reported 7 and 100% removal of As^{5+} with this acid stripped carbon and iron-impregnated activated carbon at a pH of 2.75, respectively, which indirectly indicates the nonrelease of iron at pH 2.75.

3. Results and discussions

Removal of arsenic species, iron, and manganese by GAC and GAC-Fe are discussed in the subsequent sections.

3.1. Effect of shaking time on percentage removal of arsenic

The effect of shaking time on the removal of arsenic species by the adsorption on GAC and GAC-Fe are shown in Fig. 2. From this graph it is evident that after 12 h of shaking the increase in percentage removal with time is very less when GAC is used. However, it increases slightly even after 24 h.

For GAC-Fe, after 8 h of shaking the changes in the percentage removal of the arsenic species is very less. Therefore, it seems that majority of the arsenic species are adsorbed within shaking times of 12 and 8 h for GAC and GAC-Fe, respectively. It is obvious that, before equilibrium the greater the shaking time the greater will be the adsorption and consequently, the greater will be the percentage removal. For GAC the difference between the adsorption of As(III) and As(V) is more than that of GAC-Fe. This can be explained as follows: with time As(III) is converted to As(V), which is then adsorbed onto the surface of GAC or GAC-Fe. The greater peak area at λ value of 860 cm^{-1} than that at λ value of 780 cm^{-1} in the FTIR spectra of GAC-Fe$_{(AA)}$ (Fig. 3) supports the partial conversion of As(III) to As(V). Similar observation on the As(III) and As(V) adsorption by amorphous iron oxide has been reported by Goldberg and Johnston [13]. The addition of bands at the wave number of 825 and 860 cm^{-1} in the spectrum of GAC-Fe$_{(AA)}$ indicates the adsorption of As(V) onto the GAC-Fe [14].

The presence of Fe^{3+} in GAC-Fe accelerates the oxidation of As(III) to As(V) as Fe^{3+} is capable of oxidizing As(III) [15]. Hence, the difference between the percentage removal between As(III) and As(V) is decreased in case of GAC-Fe. The extra peak on the XRD of GAC-Fe at $2\theta = 12°$ and higher peak at $2\theta = 28°$ in Fig. 4 indicates the presence of Fe_2O_3 (Fe^{3+}/OH^-) and β-FeOOH onto the GAC-Fe, respectively [16,17].

The percentage removal is slightly increased with time even after sufficient shaking time (24 h) for both GAC and GAC-Fe. This may be explained in the light of the As(V) adsorption mechanism. Arsenic(V) is specifically adsorbed onto an oxide surface via ligand exchange mechanism and exists as an inner sphere surface complex [18]. For systems with low As(V) surface coverages, monodentate surface complexes are considered
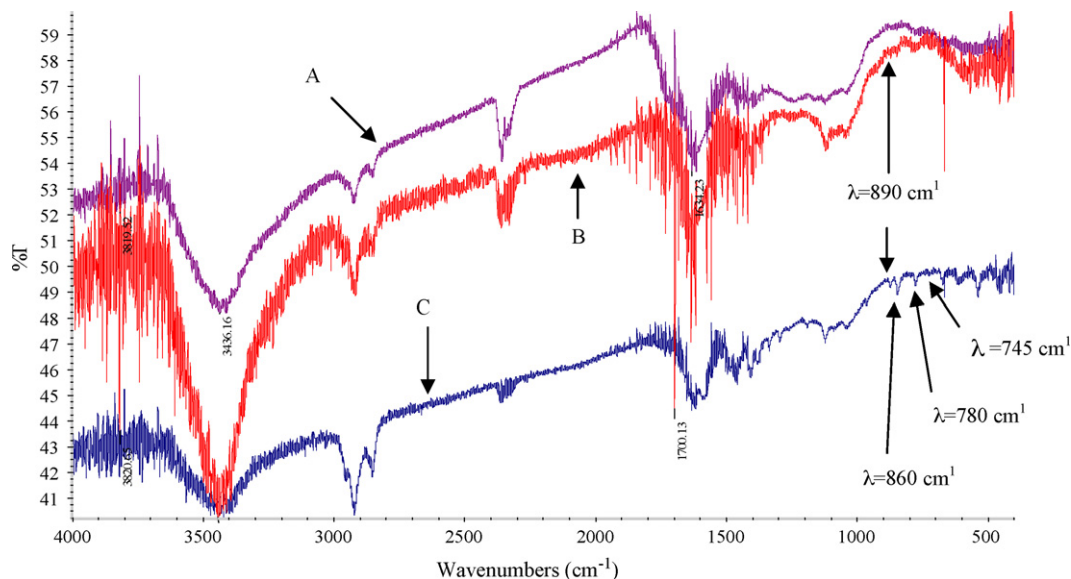

Fig. 3. FTIR spectra of GAC (A), GAC-Fe before adsorption (B) and GAC-Fe after adsorption (C).
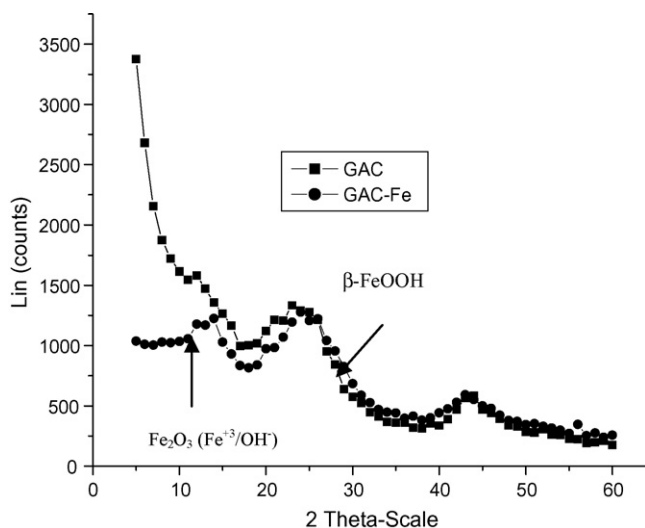
Fig. 4. X-ray diffraction pattern of GAC and GAC-Fe.

Fig. 5. Effect of shaking time on the percentage removal of Fe and Mn.

to be predominant over bidentate complexes [10]. It is possible that the formation of bidentate complexes at high surface coverage is slower than that of the predominantly monodentate reactions at low surface coverages [19]. Therefore, slow adsorption proceeds with time [20]. Fuller at al. [21] have also observed a period of rapid As(V) uptake followed by continual adsorption. Mcgeehan et al. [22] have shown rapid adsorption followed by a plateau phase.

Recently, the shaking time has been optimized to 4 h for the removal of As(III) by copper-impregnated coconut husk carbon [23]. Optimized shaking time of 4 h has also been reported for adsorption of As(V) by ferrihydrite. However, optimum-shaking time has been reported at 15 h for the removal of arsenic species by GAC impregnated with Fe and Mn separately. The greater shaking time in that report is due to the absence of Fe and Mn in the water sample along with arsenic species. The presence of these metal ions in the water sample of the present experiment activates the oxidation of As(III) to As(V) and hence, the shaking time is reduced. From this observation it seems that the As(III) is oxidized before its adsorption.

3.2. Effect of shaking time on percentage removal of Fe and Mn

Fig. 5 shows the effect of shaking time on the percentage removal of Fe and Mn. For GAC after 12 h of shaking 100% removal of Fe and Mn are obtained. Fe removal reaches 100% after 2 h of shaking when GAC-Fe is used. But complete removal of Mn is not possible using GAC-Fe. Mn removal reaches its maximum value within the first hour of shaking and decreases with shaking time. At 8 h of shaking time the percentage removal of Mn by GAC-Fe is around 41%. This decrease in percentage removal with shaking is due to the release of Mn in the water. The source of Mn may be $FeCl_3$ used for the impregnation that contains 0.15% Mn. Therefore, if $FeCl_3$ free from Mn is used then the Mn removal may reach to 100%.
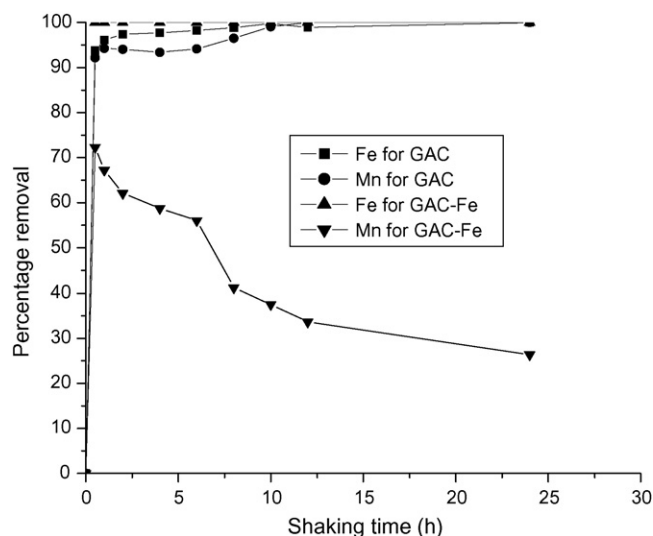
3.3. Effect of pH on percentage removal of arsenic

pH is one of the most important parameter that influences the percentage removal of a compound by adsorption. Fig. 6 shows the effect of pH on the percentage removal of arsenic species by adsorption onto the surface of GAC and GAC-Fe. It is evident that for GAC the removal of total arsenic is less dependent on the pH within the range of pH 2–11 and for GAC-Fe this is also true within the range of pH 2–7. Beyond pH 11 the percentage removal of total arsenic declines sharply. For As(V) the percentage removal is maximum in the pH range of 5–7 and for As(III) it is maximum in the pH range of 9–11 for both GAC and GAC-Fe. It is also evident that the GAC-Fe has more percentage removal for all the arsenic species than that of GAC within the pH range of 2–11. At pH above 11 the percentage removal of all the arsenic species declines for GAC-Fe. It is also true for GAC except for the removal of As(III), which does
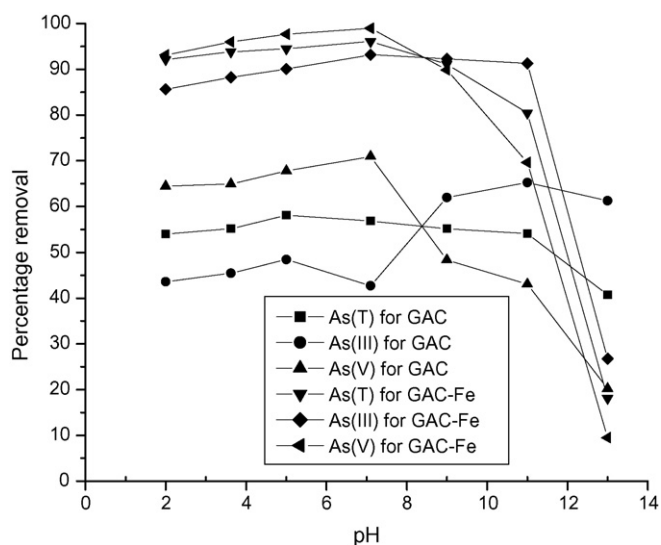

Fig. 6. Effect of pH on the percentage removal of arsenic species.

Table 1
Arsenic species in aqueous solution at various pH

pH	As(III)
0–9	H_3AsO_3
10–12	$H_2AsO_3^{-1}$
13	$HAsO_3^{-2}$
14	AsO_3^{-3}

pH	As(V)
0–2	H_3AsO_4
3–6	$H_2AsO_4^{-1}$
7–11	$HAsO_4^{-2}$
12–14	AsO_4^{-3}

Fig. 7. Effect of pH on the percentage removal of Fe and Mn.

not decline sharply at pH 11. The above observation may be explained as follows: the adsorption of arsenic species is guided by the following factors [10]:

(1) The adsorbent surface chemistry;
(2) Aqueous phase chemistry.

GAC contains oxides of aluminium, calcium and silicon, those are responsible for the development of charges on the adsorbent surface when GAC comes in contact with water. The change in chemical environment of the adsorbent surface according to the pH of the solution is as follows:

$$M\text{-}OH + H^+ \rightarrow M\text{-}OH_2^+$$

$$M\text{-}OH + OH^- \rightarrow M\text{-}O^- + H_2O$$
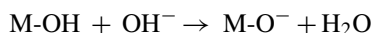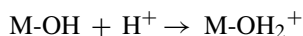
where, M stands for Al, Ca or Si.

The zero point charge of SiO_2, Al_2O_3, and CaO are 2.2, 8.3, and 11.0, respectively, hence a positive charge exists on the surface at lower pH, which gradually decreases and attains negative value at higher pH (around 11.0).

Again, the ionic character of the arsenic species also varies with pH, which is shown in Table 1 [24].

At pH below 6.5 a significantly high electrostatic attraction exists between the positively charged surface of the adsorbent and the negatively charged $H_2AsO_4^{-1}$ ions. Hence, the As(V) removal is maximum in the pH range of 5–7 for both the adsorbents. At higher pH range the positive charge on the adsorbent surface is reduced, that is why at higher pH (pH > 11) the arsenic removal is declined. The negatively charged arsenic ion and positively charged adsorbent surface favours the arsenic adsorption. At lower pH the As(III) species is present in non-ionic form but GAC surface is positively charged and at higher pH both As(III) and As(V) are negatively charged but the positive charge intensity onto the GAC surface is reduced. Therefore, overall effect of pH on the arsenic removal from a mixture of As(III) and As(V) within the pH range of 2–11 is not so prominent. For As(III) although the negative charge increases with increase of pH the GAC surface also reduces positive nature, hence, the As(III) removal is also reduced at higher pH.

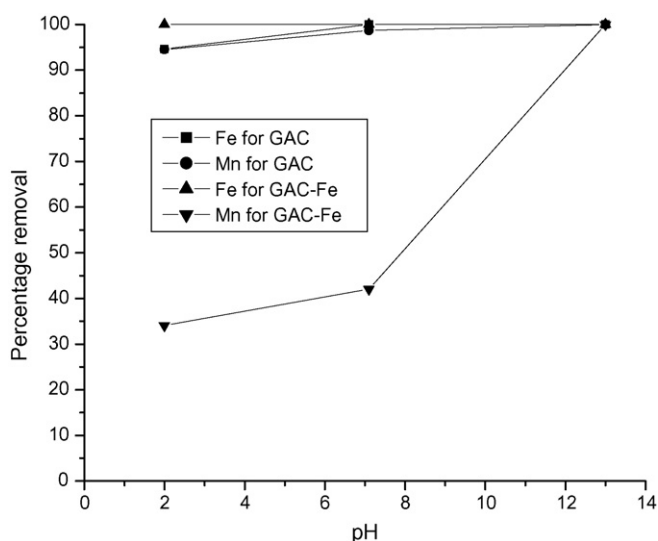For GAC-Fe the Fe^{3+} ion is incorporated onto the surface of GAC. Amorphous Fe oxide has zero point charge of 8 [25]. Due to the impregnation of more Fe the relative amount of Al, Ca or Si is decreased. As a result the positive charge intensity of the GAC-Fe may be less than that of GAC after pH 9. That is why the removal of total arsenic starts to reduce after pH 9 and declines sharply beyond pH 11. The greater percentage removal of any arsenic species by GAC-Fe within the pH range of 2–11 is due to the inclusion of Fe onto the surface of GAC-Fe. At higher pH (pH > 11) the GAC-Fe surface gets negatively charged as the pH value crosses the zero point charges of all metal oxides present in it [10].

Zu et al. have also reported the little influence of pH on the removal of As(V) by GAC within pH 4.4–9.0 by their experiment using 24 h shaking time [9]. They have also reported the decrease in percentage removal of As(V) with the increase of pH above 11.

3.4. Effect of pH on percentage removal of Fe and Mn

The effect of pH on the removal of Fe and Mn is shown in the Fig. 7. It is evident that for GAC the percentage removal of both Fe and Mn is almost 100% at pH above two. For GAC-Fe the Fe removal is also 100% within the whole pH range whereas the removal of Mn is minimum at pH 2 and increases with the increase of pH. At pH 13, 100% removal of Mn is possible. Both Fe^{2+} and Fe^{3+} can be adsorbed by both GAC and GAC-Fe within the whole pH range due to the amphoteric nature of iron in aqueous solution.

The percentage removal of Mn^{2+} is minimum at pH 2 because of the positive nature of both Mn^{2+} and adsorbent surfaces. As the positive nature of GAC-Fe is more than that of GAC therefore, Mn^{2+} removal by GAC-Fe at lower pH range is less than that of GAC. With the increase in pH, the NPC of GAC-Fe decreases, consequently percentage removal of Mn increases. At pH above 11 the GAC-Fe becomes negatively charged, as a result almost 100% removal of Mn is obtained at pH above 11. At pH 13 GAC-Fe gives 100% removal of Mn.
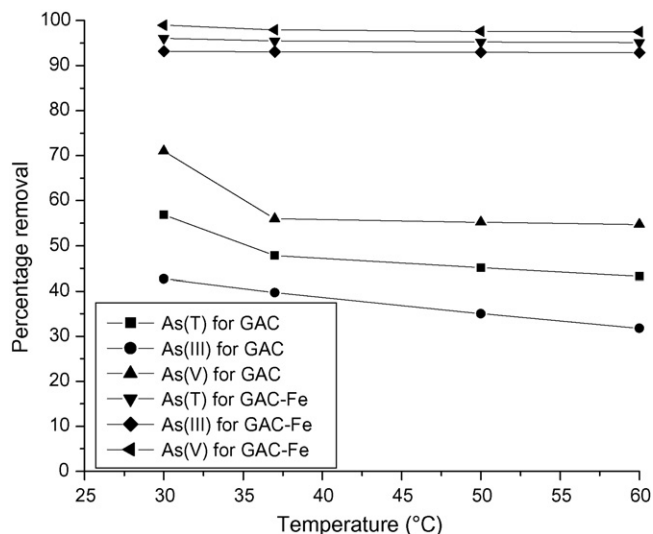
Fig. 8. Effect of temperature on the percentage removal of arsenic species.

Fig. 9. Effect of temperature on the percentage removal of Fe and Mn.

3.5. Effect of temperature on percentage removal of arsenic

Fig. 8 shows the effect of temperature on the percentage removal of arsenic species. It is evident that with the increase in temperature the percentage removal of all the arsenic species are decreased. This agrees with the exothermic nature of adsorption process. It seems that the diffusion process (intra particle transport-pore diffusion) has less control over the adsorption of arsenites and arsenates as the diffusion-controlled adsorption is endothermic in nature [26]. Fuller et al. [21] have also reported that the time dependence of arsenate adsorption on ferrihydrite could be described by a general model for diffusion into a sphere if a subset of surface sites located near the exterior or aggregates is assumed to attain equilibrium rapidly. Generally, it is believed that arsenate forms inner sphere complexes on iron oxide surfaces. However, more investigation is required on the exact structure of such surface complexes. Recent publications support intensely surface precipitation as an additional and simultaneously acting removal mechanism for arsenate on iron oxides [24,25,27,28]. With the increase in temperature the mobility of ions will increase, as a result surface precipitation will decrease. Again, with the increase in temperature the stability of the bonds between the active sites of GAC and arsenic moiety decreases. Due to these reasons the percentage removal decreases with the increase in temperature. As arsenic is more strongly bound with the active sites in case of GAC-Fe, hence, the percentage removal of all arsenic species is greater in case of GAC-Fe.

3.6. Effect of temperature on percentage removal of Fe and Mn

The effect of temperature on the percentage removal of Fe and Mn is shown in Fig. 9. It is evident that the Fe removal by both GAC and GAC-Fe is not affected by the temperature. This indicates that both Fe^{2+} and Fe^{3+} form stable bonds with the active sites of GAC and GAC-Fe. Percentage removal of Mn is
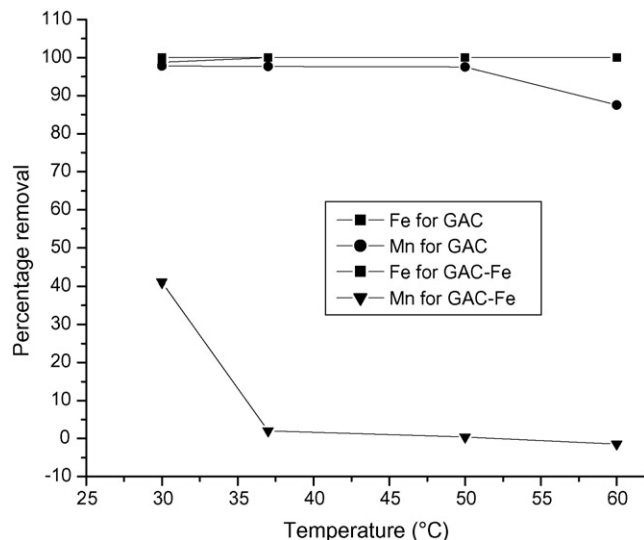
highly affected by the temperature for both GAC and GAC-Fe which indicates less stability of the bonds between Mn and active sites of GAC and GAC-Fe. For GAC-Fe the greater decrease is due to the release of Mn from iron-impregnated onto the surface of the GAC. At a temperature of $50\,^\circ$C the percentage removal of Mn is 0% and at $60\,^\circ$C it is -1.4%. This confirms the release of Mn from the Fe impregnated onto the GAC.

Fig. 10 shows the concentration of the As(T) in the treated water at various As_0 for both GAC and GAC-Fe. It is evident that by using GAC-Fe the total arsenic content in the treated water may be reduced below 10 ppb if the As_0 value lies within 200 ppb. It is also evident that the As(T) in the treated water is below 50 ppb when As_0 value is 520 ppb or less. However, GAC cannot reduce the As(T) below 50 ppb even if the As_0 value is 200 ppb. In India and Bangladesh the arsenic content in ground water varies from 50 to 300 ppb. Therefore, we believe that GAC-Fe can be used to treat the contaminated water in this region. Shaking time, pH and temperature for optimum removal of the pollutants are shown in Table 2.
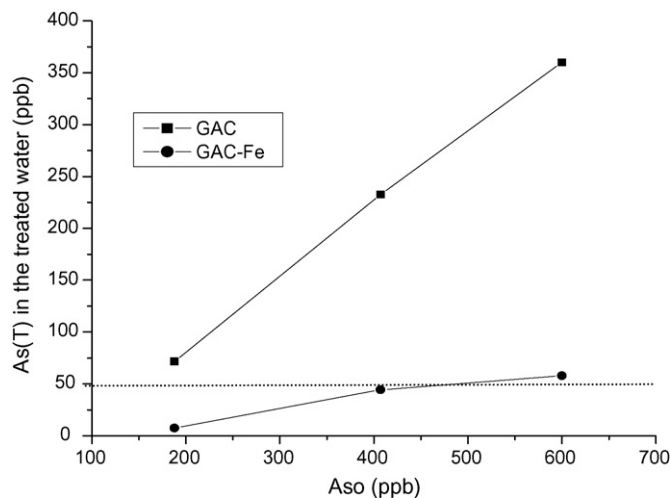

Fig. 10. As(T) in the treated water at various As_0.

Table 2
Shaking time, pH and temperature for optimum removal of the elements by GAC and GAC-Fe

Parameters	Adsorbents	
	GAC	GAC-Fe
Shaking time (h) (most of the arsenic species is removed)	12	8
pH	7.1	7.1
Temperature (°C)	30	30

4. Conclusion

From the above discussions the following conclusions are made:

(1) The GAC-Fe gives more percentage removal of all types of arsenic species than that of GAC.
(2) The effect of pH is not significant on the percentage removal of total arsenic within the range of 2–11 for GAC and 2–9 for GAC-Fe. However, maximum removal of As(III) and As(V) are obtained in the pH range of 9–11 and 5–7, respectively, for both the adsorbents.
(3) Percentage removal of arsenic species on GAC and GAC-Fe decreases with the increase in temperature.
(4) Mn is released from the iron of GAC-Fe. This rate is increased with the increase in temperature. However, at higher pH (pH > 11), the release of Mn is negligible from the GAC-Fe.
(5) Fe can be removed almost completely at the pH when removal of arsenic species is maximum.
(6) Using GAC-Fe the arsenic concentration in the treated water can be reduced below 10 and 50 ppb from arsenic solutions containing maximum As_0 value of 250 and 520 ppb, respectively. Hence, GAC-Fe may be used to treat the arsenic contaminated ground water in India and Bangladesh as the treated water satisfies the national standard of these countries and the contamination level in ground water also varies from 50 to 300 ppb. At the same time it may be used for treating arsenic contaminated ground water of developed countries where MCL value of arsenic in drinking water is 10 ppb.

Acknowledgements

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