

## Iron removal from groundwater using granular activated carbon filters by oxidation coupled with the adsorption process

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### ABSTRACT

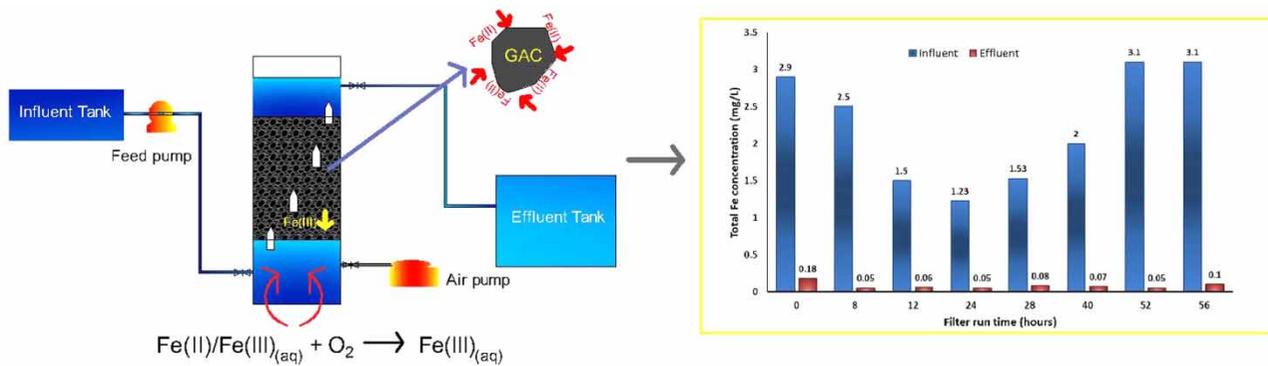
Granular activated carbon (GAC) is commonly used as an adsorbent for the removal of organic micropollutants. However, little is known about the inorganic pollutant removal using GAC. In this study, GAC was examined for the removal of iron from synthetic groundwater (SGW) using oxidation coupled with an adsorption mechanism. Batch experiments including kinetic and isotherm studies were carried out to find out the adsorption capacity of GAC. A column test was conducted at different phases to identify the influence of the initial concentration of iron, hydraulic retention time (HRT), and air supply to the system. It was observed that the experimental results for the kinetic experiment best fit the pseudo-first-order kinetics model with 88% maximum removal efficiency achieved in 240 min. A reduction in adsorption capacity was observed with the concomitant increase in the weight of GAC, whereas the maximum adsorption capacity of GAC was found to be 0.17 mg Fe/g GAC. Column experiments confirmed that the effluent iron concentration reduced from 0.05 to 0.03 mg/L when the iron concentration in the influent increased from 1 to 3 mg/L. The reduction in HRT from 7 to 3.5 h did not significantly affect the iron removal efficiency. The air supply to the system with the flow rate of 3.5 L/min did not show any improvement in the iron removal efficiency due to the increased airflow rate. In all cases, the iron level in the effluent satisfied the drinking water quality standard recommended by the World Health Organization (WHO) which affirms that GAC filters are more suitable for iron removal from groundwater.

**Key words:** adsorption, granular activated carbon, iron removal, oxidation, synthetic groundwater

### HIGHLIGHTS

- The Freundlich isotherm model best described the iron adsorption process, and the maximum adsorption capacity of GAC is 0.17 mg/g.
- The key iron removal mechanism in filter is oxidation coupled with adsorption.
- The results from the column experiment showed that at 7 h HRT and 3 mg/L inlet iron concentration, the optimal percentage of iron removal is 99%.
- Aeration with a rate of 3.5 L/min did not influence the filter performance.

## GRAPHICAL ABSTRACT



## INTRODUCTION

Groundwater resources play a crucial role in the production of clean and adequate drinking water supplies all around the world. The comprehensive use of groundwater can be explained by the ease of access, better protection from pollution sources, higher water purity as compared to surface water (Chen *et al.* 2016; Mohammadi *et al.* 2020), less subject to seasonal and perennial changes, uniformly spreading over large areas (Zhou *et al.* 2020), and also lower capital cost of production (Katsanou & Karapanagioti 2017). These benefits have resulted in groundwater use for water supply at a larger scale. Most groundwater and surface water sources that are used to supply public water systems contain a variety of organic and inorganic chemicals. Inorganic contaminants are usually present at much higher concentrations than their organic counterparts. Iron is the fourth most ample element in the earth's crust and a common inorganic constituent in the groundwater (Usman *et al.* 2019).

The high concentration of iron in groundwater leads to clogging of distribution systems, the shortened lifespan of major appliances, and more energy requirements for pumping water through clogged pipes and for heating water in industries (Moser *et al.* 2021). The World Health Organization has approved the removal of iron when the concentration is higher than 0.3 mg/L (Shahin *et al.* 2019). Therefore, there is a need to carry out a study that endeavors to alleviate the problem of high iron concentration in groundwater. The study area has been selected as Vallipuram coastal area which is located in Jaffna Peninsula in Northern Province, Sri Lanka. Groundwater supply through collector wells in Vallipuram water treatment plant which has higher iron concentration affects the treatment plant process and the population in Point Pedro where the groundwater is distributed for drinking purposes.

Different mechanisms (physical, chemical, and biological) may contribute to the removal of iron in filters but the treatment method domains the removal mechanism depends on water quality and process conditions applied. Under the commonly applied treatment conditions in iron removal plants, the oxidation-floc formation mechanism (floc filtration) is commonly believed to be dominant. In this floc filtration mode, commonly applied in many groundwater treatment plants, soluble  $Fe^{2+}$  is first oxidized to insoluble  $Fe^{3+}$  by aeration or chemical oxidation, and the flocs formed are subsequently removed in a rapid sand filter (Khatri *et al.* 2017). In several conventional plants, however, oxidation is only partially achieved before filtration and, as a consequence, besides oxidation-floc formation, adsorption-oxidation plays a role as well.

Based on the low efficiency of the iron removal plants, research was done to identify the appropriate filter media and mechanism that can achieve improved water quality of the treatment plants. Granular activated carbon (GAC) prepared using charcoal, a porous form of carbon made from hardwood, as the base material has been identified, investigated, and found capable of achieving a substantial iron and manganese removal from sample groundwater sources in a batch test (Siabi & Ghana 2003). The choice of carbon as a filter medium for water treatment is increasingly becoming important as a result of the new knowledge acquired through research regarding its high capacity for removing manganese and iron, and other minerals through the filtration process (Ismail *et al.* 2017; Thinojah *et al.* 2020). Recently, the study of oxidation coupled with adsorption for pollutant removal from groundwater using oxidizing adsorbents is gaining momentum as an integrated treatment system (Wei *et al.* 2018). However, oxidizing the pollutant ions by providing an air supply could reduce the chemical usage and cost of the treatment system for the oxidation coupled with the adsorption process. Meanwhile, there were very

few studies done previously to evaluate the iron removal efficiency of GAC with the oxidation process where the removal could take place in a single step.

This study aimed to investigate an alternative cost-effective and easily operatable conventional treatment technique for the total iron removal from groundwater using oxidation coupled with the adsorptive removal mechanism using GAC made of coconut shell as an adsorption media with the aeration system. While oxidized iron flocs can precipitate/adsorbed on the filter media the dissolved iron can also be adsorbed throughout the up-flow filtration process by GAC so that the removal performance of the filter can be increased. In the meantime, the adsorbed ferrous iron, on GAC media could also be oxidized with the continuous supply of aeration to the filter. The batch and column experiments with different operational conditions were done to find out the adsorption and filtration properties of GAC at different operating conditions.

## METHODS

### Sample collection

The study area has been selected as Vallipuram coastal area which is located in Point Pedro in Jaffna, Sri Lanka. Groundwater is the only available prime natural water source for domestic, agricultural, and industrial purposes in the Jaffna Peninsula because it has a low percentage of surface water sources due to its karstic nature and flat terrain (Ray & Ray 2019). Groundwater quality plays a crucial role in the quality and the supply of water in the Peninsula and also it can pose a serious threat to human health.

The physical–chemical properties of groundwater from the collector well located in Vallipuram were tested in October 2018 by collecting samples from top and bottom using a standard sampler made of stainless steel having 300 mL capacity in the daytime. The top sample was collected approximately 10 cm from the top surface which was opened to the atmosphere. The pH, Total dissolved solids (TDS), Electrical conductivity (EC) and salinity were measured using a PC2700 multimeter while dissolved oxygen (DO), turbidity, and ion concentrations were measured using CyberScan DO 600 meter, turbidity meter, and atomic absorption spectrophotometer, respectively. The measured parameters which were used to prepare iron contaminated SGW are given in Table 1. The highest iron concentration was found at the bottom of the collector well than on the top surface due to the settlement of oxidized iron from the top surface.

### Preparation of synthetic groundwater

Synthetic groundwater (SGW) was used in this study due to the seasonal variation of iron concentration throughout the year which was not suitable for the experimental study. Laboratory reagent  $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$  was used to prepare SGW. The required amount of reagent was mixed with 500 mL distilled water and then added with groundwater for the batch and column test.

### GAC preparation

A coconut shell-based GAC was used as a filter media with the product type of PUC-PAC having the mesh size of  $8 \times 20$  which is purchased from Enviro Equipments Pvt. Ltd. The ash content, moisture content, and Iodine number of the GAC are 2%, 6%, and 600 mg/gmin, respectively as per the specifications. The pH of GAC varies between 9 and 11. 1 kg of GAC particles was sieved through a sieve size range of 2.36–0.8 mm using a sieve shaker. The particle size distribution curve used to identify the mean size is given in Figure 1. According to the curve, the mean size was found as 1.2 mm which was used to carry out the batch experiment. The sieved particles were separately rinsed several times with a constant amount of distilled water until getting a clear color and constant pH. Finally, the pH of washed water was found to be nearly 8.5. Then the samples were dried at 105 °C for 24 h using the oven. Then it was stored safely to avoid contamination from the environment.

### Kinetic study

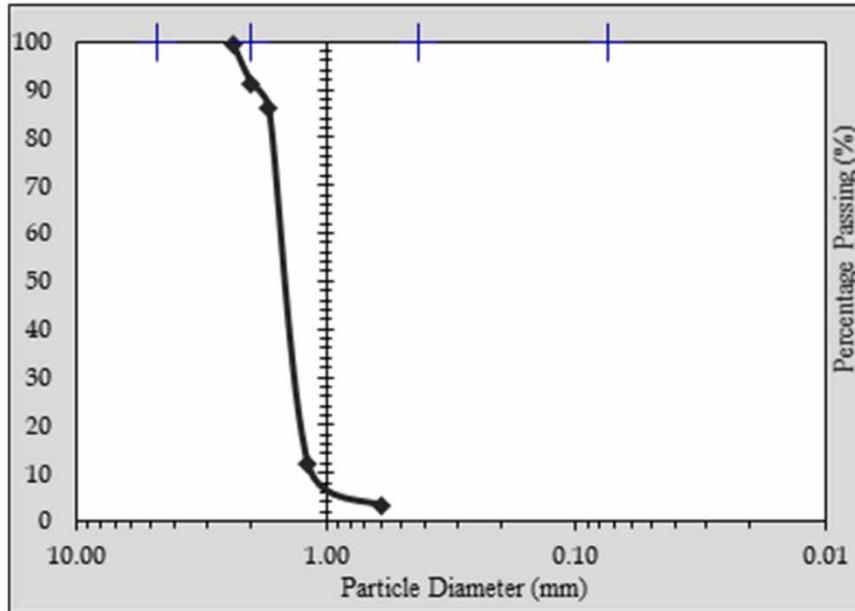
The kinetic experiment was conducted for 100 mL SGW with 5 g GAC for the time range of 10–300 min then the equilibrium iron concentration was measured. The initial concentration of iron was set to 1 mg/L. The sorption kinetics was studied using pseudo-first-order and pseudo-second-order kinetic models as mentioned in Equations (1) and (2) respectively, and the best fit values were calculated using the linear plots.

$$\ln(Q_e - Q_t) = \ln Q_e - Kt \quad (1)$$

where  $Q_e$  (mg/g) is the mass of iron adsorbed at equilibrium,  $Q_t$  (mg/g) is the mass of iron adsorbed at time  $t$  (min), and  $K$  is the first-order reaction rate constant ( $\text{min}^{-1}$ ). The pseudo-first-order considers the rate of occupation of adsorption sites to be

**Table 1** | Groundwater collector well properties at 32 °C

Parameter	pH	TDS	EC	Turbidity	Salinity	DO	Total Fe	Ca <sup>2+</sup>	Mg <sup>2+</sup>
Unit	–	mg/L	μS	NTU	mg/L	mg/L	mg/L	mg/L	mg/L
Top sample	7.85	1,200	1,812	1.27	840	5.3	0.04	60.26	2.85
Bottom sample	7.8	1,700	2,390	47.6	1,230	<0.01	0.2	31.14	2.18

**Figure 1** | Particle size distribution of GAC.

proportional to the number of unoccupied sites.

$$\frac{t}{Q_t} = \frac{1}{(K_2 \times Q_e^2)} + \frac{t}{Q_e} \quad (2)$$

where  $t$  is the reaction time (min),  $Q_t$  is the mass of iron adsorbed at time  $t$  (mg/g),  $Q_e$  is the mass of iron adsorbed at equilibrium (mg/g), and  $K_2$  is the second-order reaction rate constant ( $\text{g mg}^{-1} \text{min}^{-1}$ ).

### Isotherm study

Since GAC has the adsorption ability, it was needed to find out the adsorption capacity of GAC on iron removal. GAC particles passed through 1.7 mm and retained in a 1.18 mm sieve was used in this experiment. An equilibrium isotherm experiment was conducted using a series of conical flasks containing 100 mL SGW with 1 mg/L iron concentration and GAC doses of 0.5, 1, 2, 3, 4, 5, 10, and 15 g by mixing in a sterilizer with 150 rpm for 180 min. The amount of iron adsorbed ( $Q$ ) per unit mass of GAC was calculated using the Equation (3):

$$Q = \frac{(C_o - C_e) \times V}{W} \quad (3)$$

where  $Q$  is the amount of iron removed from SGW (mg/g) at time  $t$ ,  $C_o$  is the initial concentration of iron (mg/L),  $C_e$  is the equilibrium concentration of iron (mg/L),  $V$  is the volume of SGW (L), and  $W$  is the weight of GAC used (g).

Temkin and Freundlich isotherm models were used to analyze the adsorption behavior of GAC on iron removal as given by Equations (4) and (5), respectively.

$$Q_e = \frac{RT}{b} \ln K_T + \frac{RT}{b} \ln C_e \quad (4)$$

where  $b$  is the Temkin constant which is related to the heat of sorption (J/mol) and  $K_T$  is the Temkin isotherm constant (L/g).  $R$  is the universal gas constant (8.314 J/mol K) and  $T$  is the temperature (K).

$$\log Q_e = \log K_f + \frac{1}{n} \log C_e \quad (5)$$

where  $Q_e$  is the amount of iron adsorbed (mg/g),  $C_e$  is the equilibrium concentration of iron in solution (mg/L), and  $K_f$  and  $n$  are constant integrals, the factors affecting the adsorption capacity, and intensity of adsorption, respectively.

### Column experimental study

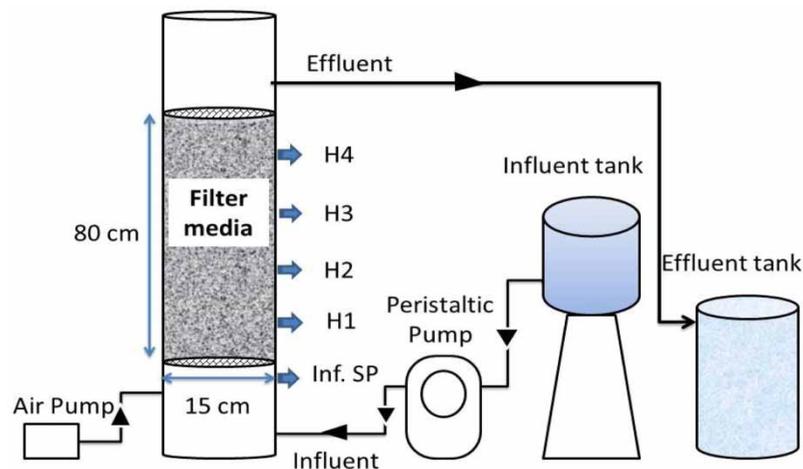
The column having a height of 1.5 m and 15 cm diameter was designed using an acrylic tube for its transparency. The diameter was selected greater than 50 times the mean media size to avoid the channeling effect. The smaller diameter was not used to avoid excess wall effects. The column height was selected according to the column bed/height ratio of  $\cong 1/10$ . For the column test, the GAC particles were placed according to their particle size range where the largest to smaller in size were packed from the bottom to top. The schematic diagram of the experimental setup is given in Figure 2.

The sampling ports were placed throughout the column height with a 20 cm interval for the sample collection. The air supply and SGW were given concurrently from the bottom. The aquarium air pump was used to supply a 3.5 L/min air flow rate to the column from the bottom. The widely used up-flow configuration was used to supply influent SGW in this study. The peristaltic pump was used to supply SGW. The filter media were packed partially for 80 cm height starting 40 cm from the bottom.

### Column operational conditions

The column was operated in three different phases to identify the effect of the initial concentration of iron in SGW, HRT, and air supply into the system on iron removal efficiency. Phase 1: iron concentration increased approximately from 1 to 3 mg/L at 7 h HRT, phase 2: HRT was reduced from 7 to 3.5 h for the initial iron concentration of 3 mg/L, and phase 3: with the air supply of 3.5 L/min to the column having 3 mg/L iron and 7 h HRT.

The porosity of the GAC filter was measured using the volumetric method. Initially, the distilled water was sent up to the filter media height. Then the system was allowed to get saturated for 20 min. Finally, the distilled water was drained from the



**Figure 2** | Schematic diagram of the experimental setup. Sampling ports: Inf.SP, H1, H2, H3, H4, Effluent.

bottom and the porous volume was measured. The procedure was continued three times to get the average porous volume. Finally, the porosity was measured as 0.28 from which the flow rate was calculated according to the HRT. The column was operated continuously and the samples were collected from the sampling ports with 4-h time intervals for the measurement. The column was backwashed at the end of each phase using clean water with a flow rate of 1.2 L/min for 30 min. The parameters pH, Temperature, and TDS were measured using PC2700 multimeter, DO was measured using CyberScan DO 600 m, and the total iron concentration was measured using DR900 calorimeter.

At the end of phase 1, the filter material from the top and bottom of the column was collected to analyze the adsorbed and oxidized iron concentration during the column experiment. The bound iron ions were extracted by dissolving 5 g of GAC in 0.5 M HCl of 50 mL for 1 h. The extracted HCl sample was filtered through 0.45  $\mu\text{m}$  filter paper and the dissolved iron concentration was measured.

## RESULTS AND DISCUSSION

### Batch experimental results

Laboratory batch studies are useful in obtaining and providing fundamental equilibrium adsorption and kinetic data for potential adsorbents. The importance of obtaining isotherms and kinetics curves lies in developing a model which accurately represents the results obtained and could be used for design purposes. Moreover, the models applied to describe the breakthrough behavior have a key role in the scale-up procedure from laboratory experiments through pilot plants to industrial scale.

To find out the mechanism of iron adsorption and the potential rate-controlling steps, like diffusion mass transport and chemical reaction process, it is required to characterize the adsorption mechanism. To understand the kinetics of iron removal using GAC as an adsorbent pseudo-first-order and pseudo-second-order models were plotted with the experimental data.

Figure 3 shows the pseudo-first-order and second-order models that describe the sorption kinetics with  $R^2$  values of 0.987 and 0.963, respectively. It is observed that the pseudo-first-order model agreed well with experimental values compared to the pseudo-second-order model. The parameters  $Q_e$  and  $K$  were calculated as  $8.54 \times 10^{-5}$  mg/g and  $0.019 \text{ min}^{-1}$ , respectively from the linear plot results of the pseudo-first-order model. Results from the kinetic study indicated that the rate of iron removal was relatively fast at the beginning due to the greater availability of the potential adsorption sites on the GAC surface. It was also evident from the results that iron adsorption occurred in two steps. In the first step, 50% of sorption occurred within 40 min followed by very slow adsorption between 60 and 120 min in the second step. After 240 min, the removal percentage remained relatively constant at 88%. According to Doke & Khan (2012), this can be attributed to the diffusion process where the adsorbate species are most probably transported from the bulk of the solution into the solid phase through intra-particle diffusion which is often the rate-limiting step in many adsorption processes.

The results of adsorption capacity and Freundlich isotherm tested for iron contaminated groundwater (1 mg/L) by GAC at 25 °C are shown in Figure 4. An equilibrium contact time of 240 min was maintained in this case. At equilibrium, a nonlinear

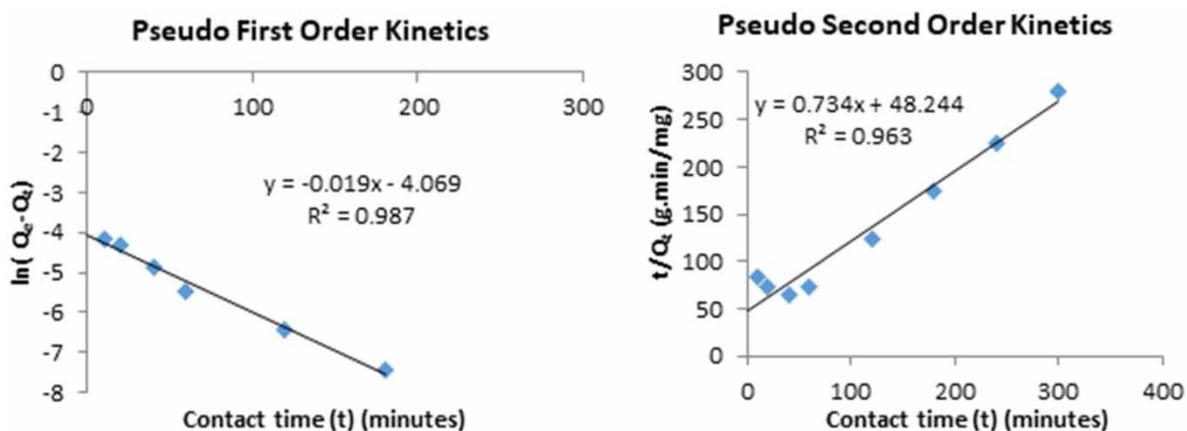
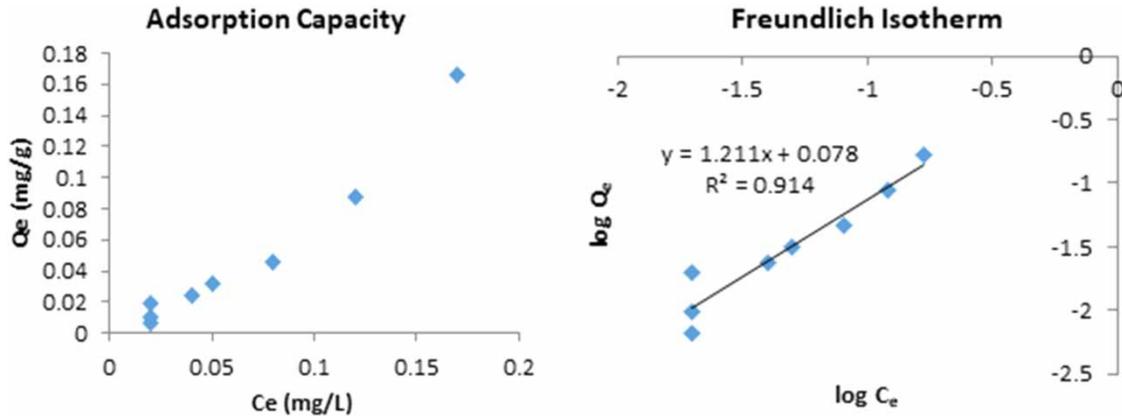


Figure 3 | Pseudo-first-order and second-order kinetics model for iron-contaminated groundwater.



**Figure 4** | Adsorption capacity diagram and the Freundlich isotherm model.

relationship was observed between the amount of iron adsorbed on GAC and iron contained in the solution. A maximum  $Q_e$  value of 0.17 mg/g was obtained with respect to the  $C_e$  value of 0.17 mg/L.

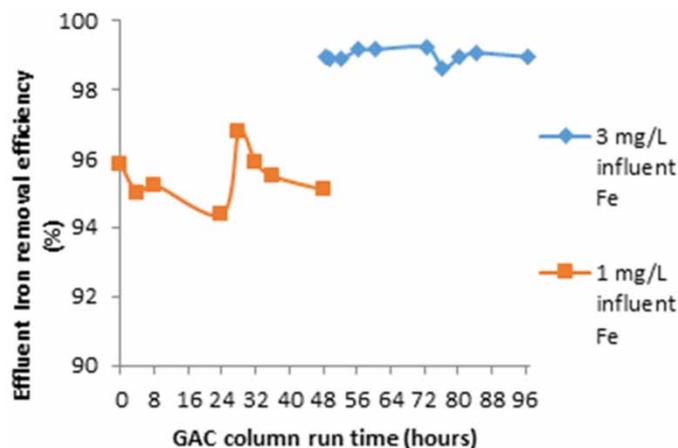
The values of the correlation coefficient ( $R^2$ ) revealed that the Freundlich model follows the best fit of 0.914 compared to the Temkin model ( $R^2 = 0.783$ ). The Freundlich constants  $K_f$  and  $n$  were calculated as 1.198 and 0.825, respectively from the linear plot. In a previous study, Okeola *et al.* (2011) found the best fit to the Freundlich model for iron removal using GAC media while confirming the heterogeneous adsorption process. The GAC adsorbent has a heterogeneous surface with adsorption sites that have different energies of adsorption. The adsorption parameters  $b$  and  $K_T$  obtained from the Temkin model were  $1.9 \times 10^4$  J/mol and 50.19 L/g, respectively. Here, the constant related to the heat of sorption ( $b$ ) has a positive value which indicates that the adsorption process is endothermic where the heat energy was absorbed from the surrounding during the isotherm experiment.

### Column experimental results

The results of each phase in the column experiment are discussed below. In all phases, 68% of iron entering the filter bed was found to be in a dissolved form.

#### Phase 1: influence of initial iron concentration on GAC filter run

The initial iron concentration was increased from 1 to 3 mg/L at 7 h HRT to study the removal efficiency of GAC on iron removal. As shown in Figure 5, an experimental lag time of 48 h with 30 min backwash time was maintained in this case. Increasing influent iron concentration from 1 to 3 mg/L resulted in an increase in removal efficiency from 95 to 98.9% for the GAC column. When the initial iron concentration was increased, the percentage of oxidized iron particles entering



**Figure 5** | Effluent iron removal efficiency of GAC at 1 and 3 mg/L influent iron with 7-h HRT.

the filter also increased from 32 to 85%. This was due to the higher DO level (5 mg/L) in SGW at the inlet. The coexistence of oxidation and adsorption mechanisms was the key factor enhancing iron removal in the presence of oxygen. As a result, the total iron removal efficiency was increased with a concomitant increase in initial iron concentration.

The results of the kinetic study with 5 g of GAC confirmed the best fit first-order adsorption constant,  $Q_e$  of  $8.54 \times 10^{-5}$  mg/g at equilibrium. This implies that 5 g of GAC was able to adsorb up to  $4.27 \times 10^{-4}$  mg of iron. However, total mass of iron recovered from 5 g of GAC at the end of phase 1 column experiment was approximately 1.95 mg. This indicates that the precipitation of insoluble iron inside the filter predominantly influences the removal of iron as a result of iron oxidation. In most test conditions in iron removal filters, the oxidation-floc formation mechanism is believed to be dominant due to the increase in the rate of  $\text{Fe}^{2+}$  ion oxidation by air. In fact, 90% conversion may be achieved in a few minutes at a pH of 7 (Chaturvedi & Dave 2012).

### Phase 2: influence of HRT on GAC filter run

The iron removal efficiency (influent iron concentration = 3 mg/L) was reduced from 99 to 98.4% when the HRT was reduced from 7 to 3.5 h. Figure 6 compares the trend in the iron removal efficiency in phase 2 and HRT did not seem to have any significant effect on iron removal efficiency. However, Tchobanoglous *et al.* (2003) reported that the increase in filtration velocity results in a factor of 4 higher Reynolds number compared to normal conditions. Consequently, turbulence within the hydraulic pores may disturb and accelerate the precipitated iron towards the outlet and reduced the iron removal efficiency. It is also mentioned by Gude *et al.* (2018) that increasing the filtration velocity by reducing the HRT can force precipitated iron flocs into the outlet, as it, among other mechanisms, increases the hydraulic shear forces within the pore channels.

### Phase 3: influence of air supply on iron removal using GAC filter media

Aeration did not appear to significantly influence the iron removal efficiency (94–98%) as shown in Figure 7. Effluents in both conditions reached the Environmental Protection Agency (EPA) drinking water standards of 0.3 mg/L (EPA 2018). Oxygen transfer efficiency is the main factor determining the effective performance of the filter which in turn depends on the air to water ratio. In the present study, the air to water ratio was set as 100:1. A previous study reported that 2–10:1 of air to water ratio led to efficient removal of pollutants for aerated filters. Furthermore, increasing the air to water ratio can push down the air to water interface, where the uneven distribution of air is also possible (Kamarden *et al.* 2014). It was evident from the present study that a high airflow rate washed out the precipitated iron particles from GAC media bed due to the effect of air binding. Consequently, this caused the fluctuation of iron concentration throughout the column height.

Moran (2018) pointed out that air scour must be distributed evenly across the base of the filter if it is to work effectively without air binding, otherwise, it could change the liquid flow rate and affect the removal process. However, an influent DO level of 6.5 mg/L was observed in phase 3 at the beginning and reduced to nearly 6 mg/L with time. The sudden reduction of DO from the influent port to H1 height and the unstable reduction throughout the media height were also observed during the column operation. The reduction of DO level conforms to the oxidation of iron ions during the removal process.

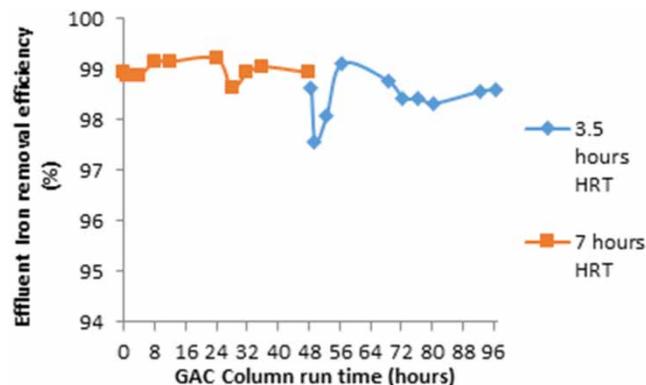
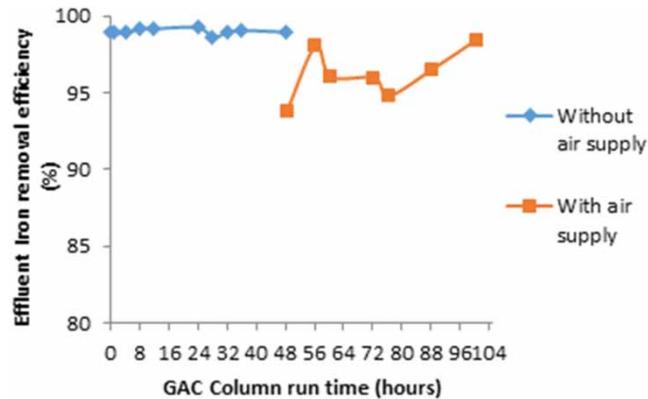


Figure 6 | Effluent iron removal efficiency at phase 2 for GAC filter media.



**Figure 7** | Effluent iron removal efficiency for GAC media at phase 3.

Overall, the results from the column experimental study confirmed that the precipitation of oxidized iron has taken place prior to adsorption. However, the adsorption capacity of GAC on iron removal can play a significant role when the oxygen concentration is insufficient. Future studies can be done in a more analytical way for deoxidized iron contaminated water to evaluate the adsorption properties of GAC before the oxidation has taken place. Moreover, the experimental study can be done on-site with raw collector well water to understand the removal performance more realistically with fluctuating influent iron concentration.

Finally, at some point, GAC becomes exhausted and we would need to re-bed the media. Concerning the disposal of the iron saturated GAC to the environment, the regeneration of saturated GAC shall be done with thermal regeneration. Reactivation can be done by heating the spent carbon in a high-temperature furnace under inert conditions followed by the gasification of residual carbon at a temperature of around 800 °C in the presence of a mildly oxidizing atmosphere such as steam or carbon dioxide (Guo & Du 2012).

## CONCLUSIONS

In this research, combined use of batch and column experiments were performed to evaluate the oxidation coupled with adsorption mechanism by providing aeration inside the column filter and thus reducing the operating space with a single-step iron removal mechanism. The following conclusions were reached:

- The Fe adsorption on GAC was heterogeneous and the maximum uptake was 0.17 mg Fe/g of GAC.
- The precipitation of insoluble iron inside the filter predominantly influences the removal of iron as a result of iron oxidation.
- It can be concluded from the present study that the aeration is not essential as the 99% removal was achieved without air supply due to the higher oxygen level in SGW.
- From the analysis of column experimental results, it can be suggested that the air:water flow ratio can be reduced to improve the iron removal efficiency in phase 3.

The study shall be continued to analyze the possibilities of widely used thermal regeneration of GAC to find the regeneration period of spent GAC.

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## CONFLICT OF INTEREST

The authors are not affiliated with or involved with any organization or entity with any financial interest or non-financial interest in the subject matter or materials discussed in this paper.

## DATA AVAILABILITY STATEMENT

All relevant data are included in the paper or its Supplementary Information.

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