# Application of Taguchi Method for Optimization of $Fe^{2+}$ Removal from Contaminated Synthetic Groundwater using Rotating Packed Bed Contactor

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# Application of Taguchi Method for Optimization of $Fe^{2+}$ Removal from Contaminated Synthetic Groundwater using Rotating Packed Bed Contactor

**Abstract:** Malaysia contains elevated levels of iron in shallow groundwater in the range of 3-7 mg Fe/L compared to USEPA safe limit of 0.3 mg Fe/L. Air Kelantan Sdn Bhd in Malaysia uses "River Bank Filtration" (RBF) technology to harvest hyporheic water. RBF treatment removes turbidity of the river water through river bed acting as a filter, but is unable to remove the Fe from the harvested water. This work proposes a technology to reduce Fe concentration in the extracted water using granular activated carbon in a laboratory scale rotating packed bed contactor (RPBC). Taguchi method was used for optimizing operating conditions for adsorption of Fe onto activated carbon in RPBC system. Taguchi optimisation results showed that a removal efficiency of 87% Fe from a 50 mg Fe/L concentration could be achieved by RPBC at initial pH of 6.5, feed rate of 40 L/h, rotating speed of 1600 rpm and packing density of 357 $kg/m^3$.

**Keywords:** $Fe^{2+}$ removal, Higee contactor, Rotating packed bed, GAC, Adsorption, Optimization, Taguchi

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## 1. INTRODUCTION

Iron is one of the most common elements on Earth and can often be easily dissolved by the groundwater from mineral bearing rocks (Ityel, 2011). Despite the fact that iron is an essential mineral for human body, its presence in concentrations above a certain limit can render the groundwater unsuitable for human consumption. Fe precipitates in contact with air, causes corrosion in the water distribution system and also gives a metallic taste to the water (Das et al., 2007). Hence, Fe still remains a major problem for the water distribution companies. Many Fe removal technologies are available to the water treatment companies both physicochemical and biological. Most widely used treatment method is the use of filter media (Birm, Pyrolox, hydroanthracite, Everzit, granulated activated carbon) and chemical oxidants (ozone, chlorine, hydrogen peroxide, oxygen, and potassium permanganate) (Munter et al., 2005). Another effective method is the process of aeration or chemical oxidation followed by rapid sand filtration (Sharma et al., 2005). However, these methods are often slow and regular backwash of the filters are required. The guideline value of iron in drinking water is 0.3 mg Fe/L (WHO, 2008). Malaysia, which is being affected by rapid industrialization and climate change, is in dire need of clean water especially during the events of flood and drought. However, shallow groundwater is often contaminated with high levels of iron, in the scale of 3.1 mg Fe/L and 7.8 mg Fe/L in the states of Kelantan and Terengganu, respectively (Hussin et al., 2010).

River bank filtration (RBF) is a process that can produce clean water from muddy river water and this is currently being used by Air Kelantan Sdn Bhd - the water supply company of the Malaysian state of Kelantan. The water harvested through RBF system is from Hyporheic Zone and is primarily river water, mixed to some extent with shallow groundwater. RBF uses the sandy river banks which have excellent natural mechanism for filtering out particulate matter, such as sediment, leaves, soil, bugs, dissolved chemicals and gases (Woodside et al., 2011). RBF is induced by pumping from a system of connected lateral or vertical wells within the riverbank sediments, and provides a means of obtaining clean public water supplies. The success of such schemes is dependent on the microbial activity and chemical transformations that are commonly enhanced in the colmation layer within the river bank compared to those that take place in surface or ground waters (Hiscock and Grischek, 2002). Research has also shown that the removal efficiency depends not only on the contaminant, but also on the "hydraulic and chemical characteristics of the bottom sediment and the aquifer, the local recharge-discharge conditions, and biochemical processes". However, in this case the water obtained from the RBF was found to contain dissolved minerals such iron and manganese, at concentrations higher than the permissible drinking water limit. This work proposes a method to remove the excess iron found in the water obtained from RBF using granular activated carbon in a Rotating Packed Bed Contactor (RPBC).

Adsorption using activated carbon is an effective method for heavy metals and organic compounds removal in the water treatment process  (Babel and Kurniawan, 2003, Bhatnagar et al., 2013). However, the separation of the treated water from the activated carbon by gravity filtration is a slow process and space required for the filtration unit is large. In this laboratory study, we are proposing the use of a RPBC which has enhanced volumetric mass transfer coefficient which considerably reduces the time of treatment and space required (Das et al., 2008). RPBC phases out the need for slow gravity filtration process. The RPBC consists of a doughnut shaped bed which is spun at high speed to achieve high centrifugal force. Due to the elevated force, very thin liquid films and/or tiny droplets are created in the packing area. Thus, gas-liquid or liquid-liquid or solid-liquid interfaces are constantly renewed, which aid in considerable intensification of mass transfer between the phases (Wang et al., 2008). In this work, the water obtained from the RBF unit, containing high iron concentration, was treated using activated carbon in this RPBC. Earlier, RPBC has been used by researchers for removal of heavy metals and dyes from wastewater (Lin and Liu, 1999, Kundu et al., 2015b, Kundu et al., 2016). Granular activated carbon (GAC) is a industry standard as adsorption media and has been widely used in experimental works (Kundu et al., 2015a, Bhatnagar et al., 2013). Therefore, GAC was used in this study and process optimisation was performed using Taguchi method to obtain a robust process design at low cost. Orthogonal arrays are used to determine optimum design points by performing minimum number of experiments (Durán-Jiménez et al., 2014, Taguchi, 1986). The ‘log’ functions of desired output, defined by Taguchi as Signal-to-Noise ratios (S/N), serve as the objective function for data analysis and optimization(Apte, 2000). Taguchi method also considers the effects of noise factors, which are often inconvenient to control.  In this method, the optimum condition will be insensitive to the noise factor.

The main objective of this work is to apply RPBC for the removal of iron from the water obtained from RBF system. Synthetic groundwater mimicking the RBF has been used in the study. The aim of this study is to propose a new equipment for iron removal from supply water, which can treat large volumes of water within a shorter time frame.

## 2. METHODOLOGY

### *2.1 Chemicals*

The granular activated carbon was purchased from Sigma-Aldrich and used as received. The properties of the activated carbon are presented in Section 3.1. All other chemicals used were of reagent grade and were supplied by R&M Chemicals.

### *2.2 Rotating Packed Bed Contactor (RPBC)*

The schematic diagram of a RPBC based on Higee technology is shown in Figure 1. RPB is a contactor in which a doughnut shaped packed bed is rotated at high speed using an electric motor. The contactor can accommodate 2.5 cm as the height of the bed. The inner radius of the rotor is 2 cm and the outer radius is 3.2 cm, giving the annular space 1.2 cm wide. The inner and outer walls are made of stainless steel mesh of size 50 µm. The rotor is driven by a 0.5 HP electric motor at a speed ranging from 100 to 2000 rpm, thereby generating 0.3 to 112 times the gravitational force on the basis of the arithmetic mean radius.

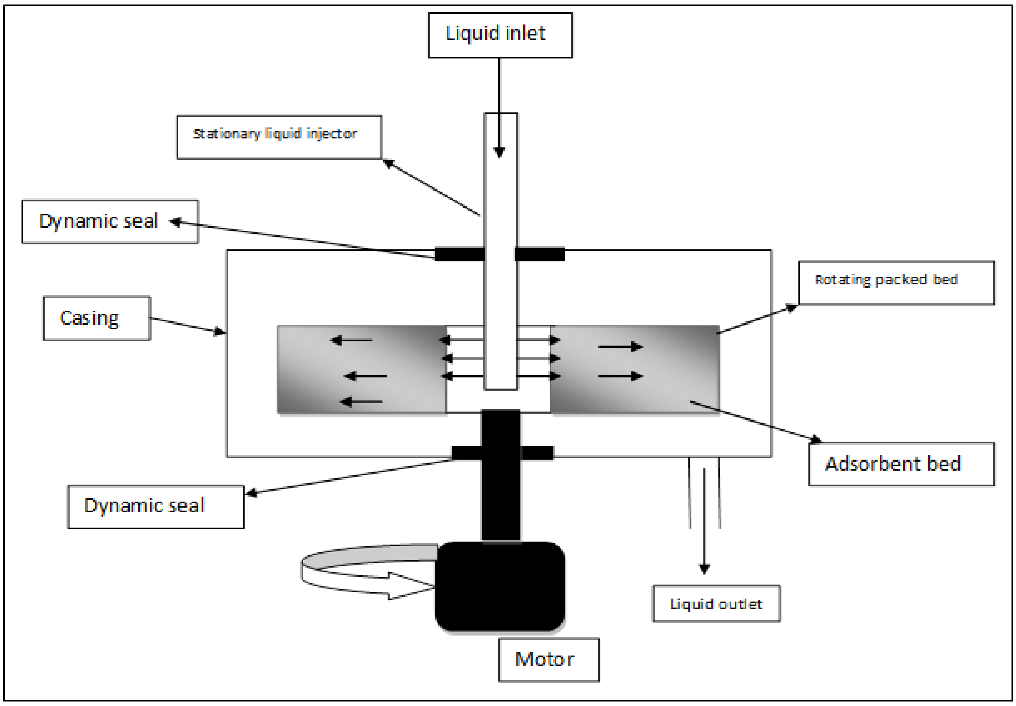

Figure 1. Schematic diagram of RPBC

### *2.3 Taguchi Method*

Taguchi method is an experimental algorithm in which a set of response variables are tested against a set of control parameters or independent variables by experiments arranged in “orthogonal array” to obtain the most suitable combination of the control parameters. Orthogonal arrays provide a best set of well-balanced but minimum number of experiments required to identify the optimum conditions. The log functions of desired output known as the signal to noise ratios (S/N), serve as the objective functions for optimization. In the present work, the algorithm for larger-the-better S/N ratio was chosen as the removal of Fe should be as high as possible at the optimum condition.  The S/N ratio is calculated using equation (1), where n is the number of replication for each experiment and R is the Fe removal efficiency for the experiment (Taguchi, 1986).

$$\frac{S}{N} = -10\log\left(\frac{1}{n}\sum_{i=1}^{n}\frac{1}{R_i^2}\right) \tag{1}$$

### *2.4 Experimental Procedures*

*2.4.1 Preparation of synthetic groundwater:*

Synthetic groundwater mimicking the water obtained from RBF installed at Pasir Tumboh site in Kelantan (Lat: 6.0573, Long: 102.2808; Table 1) was prepared by mixing $MgSO_4{\cdot}7H_2O$ (20 mg/L), $NH_4Cl$ (2 mg/L), $Na_2SO_4$(10 mg/L), $K_2HPO_4$ (0.10 mg/L), $CaCl_2{\cdot}2H_2O$ (0.67 mg/L), $NaHCO_3$(8 mg/L) (Ito et al., 2012). It can be observed from Table 1 that the concentration of Fe in the river water  increased from 3.6 to 13.6 mg/L at the RBF production well. This may be due to the fact that in the Hyporheic Zone, while passing through the river bank soil layer it comes in contact with sediments rich in Fe (Hussin et al., 2014, Hiscock and Grischek, 2002). This synthetic groundwater was contaminated by dissolving various amounts of $FeSO_4.7H_2O$. The pH of the solution was adjusted using 0.1N HCl and NaOH prior to the addition of Fe in the water.

**Table 1.** Characteristics of water obtained from RBF at Pasir Tumboh site in Kelantan state

| Parameters | River water characteristics | Collection well atPasir Tumbuh RBF unit (Kelantan) | | | Average | Standard deviation |
|---|---|---|---|---|---|---|
| | | Sample 1 | Sample 2 | Sample 3 | | |
| pH | 6.7 | 6.1 | 6.08 | 6.05 | 6.07 | 0.025 |
| Turbidity (NTU) | 85 | 0.3 | 0.5 | 0.5 | 0.4 | 0.087 |
| Fe (mg $L^{-1}$) | 3.6 | 13.6 | 13.6 | 13.7 | 13.6 | 0.031 |

*2.4.2 Rotating Packed Bed Experiments:*

For each experiment, varying amounts of GAC was packed into the annular space of the rotating packed bed contactor, depending of the packing density. Then, 1L of Fe-dosed synthetic groundwater of desired concentration was taken in a 2L beaker serving as the reservoir. On starting the motor, the contactor starts rotating and when it reaches a a stable speed, the solution is circulated from the reservoir through the RPB by a pump at a specific flow rate for the adsorption process. The experiment was conducted for one hour and the aqueous samples were withdrawn from the reservoir after the experiment. The concentrations of Fe in the samples were analysed using ICP-OES (Perkin Elmer ICP OES Optima 8000). The removal efficiency was calculated using Equation (2), where, $C_0$ (mg $L^{-1}$) is the initial Fe concentration, $C_t$ (mg $L^{-1}$) is the Fe concentration after the experiment.

$$\text{Removal efficiency, } \% = \frac{C_0 - C_t}{C_0} \times 100 \tag{2}$$

**Table 2:** Experimental range and level of the RPBC control factors

| Parameters | Level | | | |
|---|---|---|---|---|
| | 1 | 2 | 3 | 4 |
| Rotating speed, rpm | 400 | 800 | 1200 | 1600 |
| Feed rate, L/h | 20 | 30 | 40 | 50 |
| Packing density, kg/m$^3$ | 153 | 255 | 357 | 510 |
| Initial Fe concentration, mg Fe/L | 10 | 30 | 50 | 70 |
| Initial pH | 3.5 | 4.5 | 5.5 | 6.5 |

The study was carried out in two stages using Taguchi optimisation method. At the initial stage, five control factors are identified and the full possible ranges of these factors on the removal of Fe are investigated at four levels, as shown in Table 2. An experimental design matrix of L16 array is suggested by Taguchi Method, and is shown in Table A1. Based on the experimental results, the significance and contribution of the factors on Fe removal were identified and the factors that had less significance in Fe removal were screened out. In the second stage, optimisation of the significant factors obtained from the initial stage was carried out. The levels of the factors were selected at a range that was close to the initial optimum points. Hence, an accurate optimum point could be determined.

## 3. Results and Discussion

### *3.1 Characterization Study for Activated Carbon*

The granular activated carbon used was of commercial type with average size of 420 – 850 µm (20-40 mesh). Figure 2a shows that the surface morphology of the activated carbon was rough and porous. The BET nitrogen adsorption/desorption test identified that the activated carbon was mesoporous and microporous adsorbent (monolayer adsorption only) with specific surface area of 583 m$^2$ g$^{-1}$, as shown in Figure 2b. The average pore diameter of the activated carbon was found to be 22 Å. The FTIR data of the activated carbon shown in Figure 2c reveals O-H stretching of alcohol group or phenol group is available, as a peak is observed in between 3500-3200 cm$^{-1}$. The other functional groups present on the activated carbon were: (i) N-H bend of primary amines and C-C stretch of aromatic ring (complex peaks around 1580-1650 cm$^{-1}$), and (ii) C-O stretch for alcohol group, carboxylic acid group, ester group (peaks at the bands of 1300-1000 cm$^{-1}$).

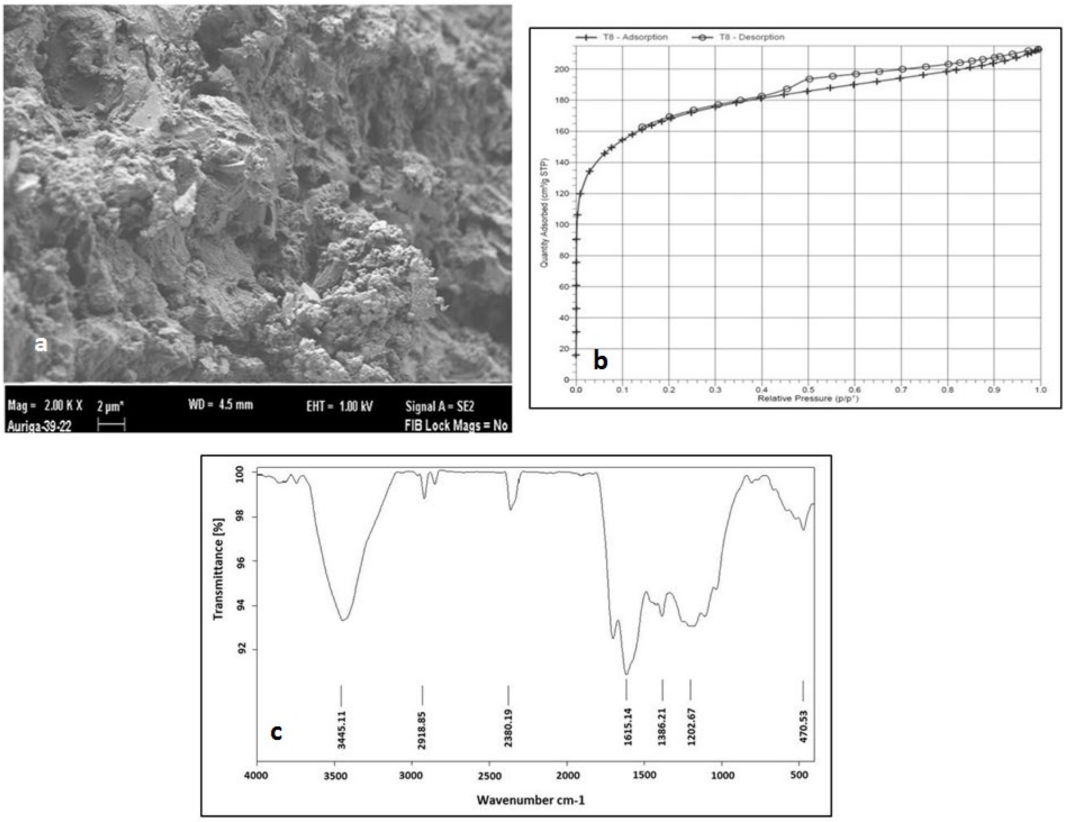

**Figure 2:**(a) SEM image of the activated carbon; (b) Nitrogen adsorption/desorption plot for the activated carbon; (c) FT-IR spectra for the activated carbon

### *3.2 Effect of Control Factors on Fe removal: Taguchi method*

The effect of control factors on Fe removal was analyzed using the Signal-to-Noise (S/N) ratio. In this study, the S/N ratio was analyzed in the form of “the larger the better”, considering that higher S/N ratio indicated that higher Fe removal efficiency. The control factors that are significant to the removal efficiency were determined by the S/N ranking.

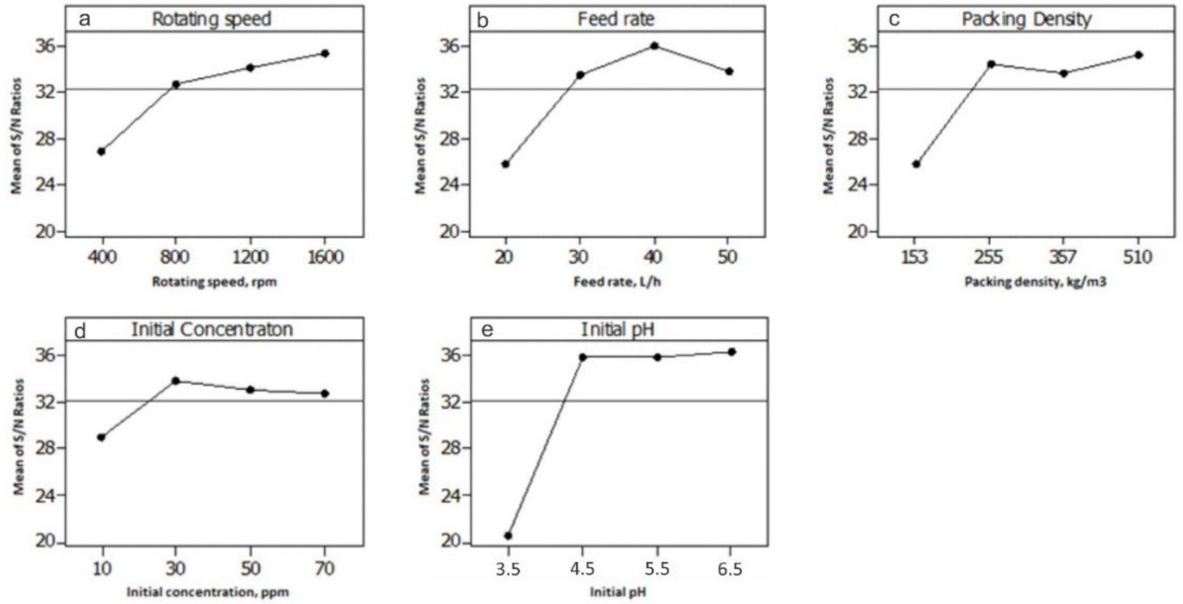

**Figure 3:**Mean data plots for S/N ratios at different control factors

The main effect plots for the S/N ratio for all the control factors are shown in Figure 3a-e. Figure 3a shows that when the rotor speed was increased from 400-1600 rpm, the S/N ratio was found to be higher, indicating higher removal efficiency was achieved. Increase in rotating speed increased the centrifugal force in the contactor, thus achieving higher mass transfer rate (Das et al., 2008). The increase in the feed rate from 20-40 L/h improved the S/N ratio, as shown in Figure 3b. As the flow rate increased, the probability of contact between the activated carbon and the Fe solution increased aiding the adsorption process. Figure 4b also reveals that a further increase of feed rate from 40 L/h to 50 L/h decreases the S/N ratio, as a result of the reduction in the contact time between the Fe and the activated carbon (Kundu et al., 2015b). Results showed that the increase of packing density from 153 kg/m$^3$ to 255 kg/m$^3$ improved the S/N ratio (Figure 3c). At low packing density, the Fe solution flowed through the loose packing so that adsorption was not facilitated. In contrast, higher packing density provided larger adsorption sites improving Fe removal. However, it is worth noting that further increase in packing density from 255 kgm$^{-3}$ to 510 kgm$^{-3}$ in this study did not improve the S/N ratio, due to decreasing in permeability. At low initial concentration, the S/N ratio was low at 28.9 (Figure 3d). This is because low concentration of $Fe^{2+}$ was available for the contact with the activated carbon. Thus, the adsorption was low due to lower mass transfer driving force and the S/N ratio decreased (Tan et al., 2009). However, when the initial concentration was too high; the adsorption sites were insufficient to provide the adsorption needed. Therefore, the S/N ratio decreased at higher initial concentration. Initial pH played an important role in the adsorption process. The results show that increase of pH from 3.5 to 4.5 improved the S/N ratio significantly from 20.5 to 35.9 (Figure 3e). This could be due to the presence of excessive $H^+$ ions in the solution at pH 3.5. The $H^+$ ions competed for the adsorption sites with $Fe^{2+}$ and instigated the activated carbon surface to become positive charge. This eventually caused the $Fe^{2+}$ ions to repel from the activated carbon surface and hence, the adsorption was low. With the increase in pH, the ratio of $H^+$ to $Fe^{2+}$ ions decreased and therefore, Fe adsorption was more effective and this improved the S/N ratio (Amuda et al., 2007). The effect of pH is insignificant when the pH of the solution is increased beyond 4.5.

S/N ratio values were ranked to select the most influential parameters. This rank is measured by the difference of the highest and lowest S/N ratio across the levels denoted by delta values. The higher the delta value, the more significant is that factor. Table A2 shows the S/N ranking for the control factors according to delta values. The study showed that initial pH was the most significant factor in determining the adsorption of Fe, followed by feed rate, packing density, rotating speed and initial concentration had the least impact. The best setting is rotating speed of 1600rpm, feed rate of 40 L/h, packing density of 510 kg/m$^3$, initial concentration of 30 mg Fe/L, and pH of 6.5.

### *3.3 Optimization Study by Taguchi method*

An optimization study was carried out in order to determine the best combination for the operating parameters in rotating packed bed operation. In this study, initial pH, feed rate and rotating speed were chosen as the significant control factors based on the S/N ranking analysis as discussed in Section 3.2. It is worth noting that packing density was not considered in optimization as the S/N ratio had less variation beyond 255 kg/m$^3$. Thus, the rotating speed, which was at 4th rank, was taken as the third control factor in this study.

**Table 3.**Experimental range and level of the control factors for optimisation study

| Parameters | Level | | |
|---|---|---|---|
| | 1 | 2 | 3 |
| Initial pH | 5.5 | 6.5 | 7.5 |
| Feed rate, L/h | 35 | 40 | 45 |
| Rotating speed, rpm | 1500 | 1600 | 1700 |

The effect of the control factors was investigated at three levels, as shown in Table 3. An L9 array experiment design was suggested by Taguchi Method. The experimental sequence, Fe removal efficiency, and the S/N ratio are as shown in Table A3.

### *3.3.1 ANOVA Analysis:*

ANOVA was performed on the data obtained using F-test, which describes the significance of a control factor on the quality characteristic, which is the Fe adsorption in this study. The F-value for the control factors was determined via the ratio of the mean of squared deviations to the mean of squared error. In general, the control factors have significant impact on the quality characteristic when the F-value is > 4 (Barman et al., 2011). ANOVA result is shown in Table A4. The F-value of 9.2 for the initial pH suggests that it contributes significantly to the Fe removal, with 41% of contribution. Rotating speed was also identified as a significant control factor which contributed 36.5% on the Fe removal efficiency, with an F-value of 8.2. In contrast, the effect of feed rate from 35 L/h to 45 L/h was relatively less significant, as it only had 17.7% of contribution on the removal, with an F-value of < 4. Therefore, the initial pH and rotating speed of the rotor are the most significant control factors in the rotating packed bed process.

### *3.3.2 Effect plots for S/N ratios and the S/N ranking:*

The results as shown in Figure 4a-c show that highest S/N ratio are observed at level 2 for all the factors. As the higher S/N ratio indicated better Fe removal efficiency, it was therefore suggested that the optimised best setting for the Fe removal in a rotating packed bed was pH of 6.5, feed rate of 40 L/h and rotating speed of 1600 rpm. The experiments were run at this optimum setting. The predicted removal was 89% and actual removal was 87%, which was highly satisfactory.

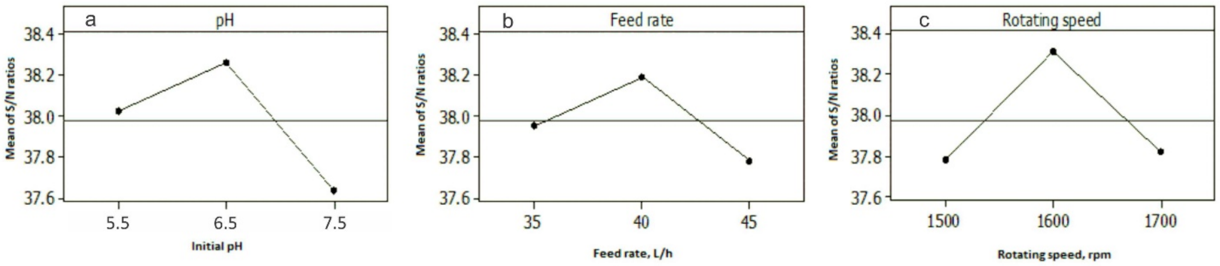

Figure 4: Main effect plots for S/N ratios

## 4. Conclusions

A rotating packed bed contactor (RPBC) has been used to treat Fe-dosed synthetic groundwater mimicking the River Bank Filtered (RBF) well water in laboratory scale trials for removal of iron by adsorption onto activated carbon. The process is fast and uses low energy for operation. Taguchi optimisation method was applied in this study to determine the optimum operating conditions for the adsorption of Fe onto commercially available activated carbon in an RPBC system. The experiment was conducted in two parts. Initially, the effect of control factors on Fe removal in rotating packed bed was investigated over a broad range for the five experimental parameters, namely: rotating speed, feed rate, packing density, initial concentration and initial solution pH. The results showed that the initial pH, feed rate and rotating speed were the most significant factors based on the variation in their Signal to Noise (S/N) ratio across the levels. On the other hand, the effects of packing density and initial Fe concentration were less significant when they were beyond level 2. An optimisation study was carried out for the three most significant parameters at a smaller range which was in the near vicinity of the initial best settings and the other two parameters, i.e. packing density and initial concentration were fixed at 357 kg/m$^3$ and 50 mg Fe/L, respectively. The optimisation results showed that a removal efficiency of 87% can be achieved by rotating packed bed contactor at initial pH of 6.5, feed rate of 40 L/h and rotating speed of 1600 rpm, using a Fe-dosed synthetic groundwater.

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## Appendix

**Table A1.** Experimental sequence and results based on L16 array design matrix

| Rotating speed, rpm | Feed rate, L/h | Packing density, kg/m$^3$ | Initial Fe concentration, mg Fe/L | Initial pH | Removal efficiency, % | | | S/N ratio |
|---|---|---|---|---|---|---|---|---|
| | | | | | 1st | 2nd | 3rd | |
| 400 | 20 | 153 | 10 | 3.5 | 1.4 | 0.7 | 2.2 | -1 |
| 400 | 30 | 255 | 30 | 4.5 | 59 | 62 | 63 | 35.7 |
| 400 | 40 | 357 | 50 | 5.5 | 66 | 65 | 64 | 36.3 |
| 400 | 50 | 510 | 70 | 6.5 | 63 | 63 | 65 | 36 |
| 800 | 20 | 255 | 50 | 6.5 | 47 | 48 | 48 | 33.5 |
| 800 | 30 | 153 | 70 | 5.5 | 37 | 41 | 42 | 31.8 |
| 800 | 40 | 510 | 10 | 4.5 | 98 | 98 | 98 | 39.8 |
| 800 | 50 | 357 | 30 | 3.5 | 18 | 20 | 29 | 25.5 |
| 1200 | 20 | 357 | 70 | 4.5 | 46 | 47 | 48 | 33.4 |
| 1200 | 30 | 510 | 50 | 3.5 | 22 | 25 | 26 | 27.4 |
| 1200 | 40 | 153 | 30 | 6.5 | 71 | 72 | 73 | 37.1 |
| 1200 | 50 | 255 | 10 | 5.5 | 81 | 83 | 83 | 38.3 |
| 1600 | 20 | 510 | 30 | 5.5 | 78 | 68 | 73 | 37.2 |
| 1600 | 30 | 357 | 10 | 6.5 | 84 | 88 | 88 | 38.7 |
| 1600 | 40 | 255 | 70 | 3.5 | 34 | 30 | 33 | 30.2 |
| 1600 | 50 | 153 | 50 | 4.5 | 56 | 55 | 55 | 34.9 |

**Table A2.** The S/N ranking for the control factors

| Level | Rotating speed | Feed rate | Packing density | Initial concentration | Initial pH |
|---|---|---|---|---|---|
| 1 | 26.7 | 25.8 | 25.7 | 28.9 | 20.5 |
| 2 | 32.7 | 33.4 | 34.4 | 33.9 | 35.9 |
| 3 | 34.1 | 35.9 | 33.5 | 33 | 35.9 |
| 4 | 35.2 | 33.7 | 35.1 | 32.8 | 36.3 |
| Delta | 8.5 | 10.1 | 9.4 | 5.0 | 15.8 |
| Rank | 4 | 2 | 3 | 5 | 1 |

**Table A3.** Experimental sequence and results based on L9 array design matrix

| Initial pH | Feed rate, L/h | Rotating speed, rpm | Removal efficiency, % | | S/N ratio |
|---|---|---|---|---|---|
| | | | 1st | 2nd | |
| 5.5 | 35 | 1500 | 78.7 | 78.7 | 37.9 |
| 5.5 | 40 | 1600 | 83.9 | 84.2 | 38.5 |
| 5.5 | 45 | 1700 | 76.4 | 76.5 | 37.7 |
| 6.5 | 35 | 1600 | 84.9 | 84.7 | 38.6 |
| 6.5 | 40 | 1700 | 83.5 | 83.7 | 38.4 |
| 6.5 | 45 | 1500 | 77.5 | 77.4 | 37.8 |
| 7.5 | 35 | 1700 | 73.9 | 73.7 | 37.4 |
| 7.5 | 40 | 1500 | 76.4 | 76.2 | 37.7 |
| 7.5 | 45 | 1600 | 78.1 | 78.9 | 37.9 |

**Table A4.** ANOVA for the S/N ratio

| Parameters | DF | Seq SS | Adj SS | Adj MS | F | P | Contribution, % |
|---|---|---|---|---|---|---|---|
| Initial pH | 2 | 0.61 | 0.61 | 0.30 | 9.24 | 0.10 | 41.29 |
| Feed rate | 2 | 0.26 | 0.26 | 0.13 | 3.97 | 0.20 | 17.73 |
| Rotating speed | 2 | 0.54 | 0.54 | 0.27 | 8.17 | 0.11 | 36.51 |
| Residual error | 2 | 0.07 | 0.07 | 0.03 | | | |
| Total | 8 | 1.47 | | | | | |