REMOVAL OF ARSENIC FROM GROUNDWATER VIA ADSORPTION

by

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APPROVAL SHEET

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DEDICATED

TO

OUR LOVING PARENTS AND FAMILIES

For their constant Support, Love and Encouragement

They have been a source of inspiration and have continually encouraged us and provided moral, spiritual and emotional support.

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ABSTRACT

Groundwater contaminated with arsenic, poses a serious risk to human health. Approximately 11 million people in Pakistan are exposed to Arsenic poisoning through drinking water (Shahid et al., 2018). With the population of Pakistan growing even faster than projected; the intensity of water usage is increasing exponentially, resulting in the shortage of clean surface drinking water and increased reliance on arsenic contaminated groundwater. The goal is to utilize the same groundwater while eliminating its impacts on human health, ensuring cost efficiency.The affinity of iron to adsorb arsenic on its surface was observed experimentally. Simultaneously residual iron testing was also carried out to make sure that the iron content in the treated water is well within limits. Maximum adsorption capacity of arsenic in batch experiments came out to be 86.6 µg arsenic/g iron nails.

Batch experiments were performed to determine the viability of recycled iron nails as an adsorbent for arsenic treatment in groundwater as affected by dose, contact time (15-120 minutes), pH (2-11), temperature (25-45^{o}C), and varying initial arsenic concentration (100-500ppb). The above mentioned parameters were optimized against maximum removal efficiency. Adsorption isothermal modeling was performed to assess the adsorption behaviour. The acquired data was translated to a flow-through system. The column filter was designed and optimized to ensure that the concentration of arsenic as well as iron in the effluent is below the permissible limits defined by the NSDWQ and the WHO.The designed filter proved to be highly efficient with a removal efficiency of 98.2% for treating real groundwater, obscured from Lahore, Pakistan.

The results suggest that iron nails proved to be a suitable adsorbent for the treatment of groundwater contaminated with arsenic.

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INTRODUCTION

1.1 Heavy Metals Pollution

Heavy metals are elements that are naturally presentin the earth’s crust [1]. Due to the toxic nature of heavy metals, they have received a supreme attention to environmental chemists among all the pollutants. Heavy metals concentrations in water bodies are on the rise, especially in Pakistan, where freshwater resources are already scarce. This can be linked to the release of unregulated effluents from a vast number of industries. This is not only a problem to aquatic life but also causes harm to human health and environment.

Heavy metals can be emitted by both anthropogenic and natural procedures and end up in different environmental bodies. Natural processes include sprays of sea-salt, forest fires, volcanic eruptions, weathering of rocks and wind-borne particles of soil. Agricultural practices, industrial emissions and wastewater andwaste from mining and metallurgical procedures also result in the release of pollutants to soil, air and water. The most commonly found heavy metals are chromium (Cr), cadmium (Cd), nickel (Ni), lead (Pb), mercury (Hg), copper (Cu) zinc (Zn) and arsenic (As) [2]. Because of their highly toxic nature, heavy metals even in very minute quantities can cause serious health effects. Exposure pathways include ingestion, dermal contact, inhalation, oral intake or direct intake from potable water sources. High concentration of these toxic metals may also lead to fatalities.

In different areas of Pakistan, the by-products of different industries including but not limited to cement, pesticides, fabric dye chemicals, textile, fertilizers, petrochemical, power, leather tanning, steel mills, food processing etc. are the main sources of the ground and surface water pollution [3].

1.2 Heavy Metals Removal Strategies

Heavy metals contamination is taking place on a global scale and some common removal techniques and processes include foam flotation, chemical precipitation, complexation, coagulation, adsorption using activate carbon, solvent extraction, ion exchange, electro-deposition membrane operations and cementation [4]. The above-mentioned techniques are rather poor methods and have numerous disadvantages which include production of sludge, high energy demand and cost and inefficient removal [5]. Adsorption is an emerging removal mechanism technique as it provides a low-cost alternative to the currently adopted methods and is highly effective due to high ratio of adsorbent's surface area to the volume of influent or untreated solvent. We opted for iron nails as an adsorbent to eliminate arsenic from groundwater, as they are easily available, have no energy demand, provide easy separation, are environmentally friendly and are cost effective and highly efficient.

1.3 Arsenic Pollution

Arsenic, also known as the king of poisons, is a member of a group of highly toxic metals, that is acquired from the natural environment. In humans, arsenic poisoning occurs due to ingestion of groundwater contaminated with arsenic. Arsenic is naturally present in underground rocks and leaches down to the groundwater hence, contaminating it. It is extensively used in industrial processes and is well renowned for being employed as an alloying agent, and in the processing and manufacturing of paper, glass, textiles, pigments, ammunition and wood preservatives. The water acquired for drinking purposes from groundwater sources contains varying levels of arsenic in many industrialized and less industrialized countries [6].

Contamination of groundwater due to arsenic is a global issue and there are many areas where arsenic concentration levels are well above the WHO limit. Approximately a population of 140 million in is exposed to and is drinking water with arsenic concentrations exceeding the WHO provisional guideline value of 10 µg/L; in 500 different countries [7]. Arsenic is present in two oxidation states: the first is arsenite [As_2O_3; As (III)] and the other is arsenate [As_2O_5; As (V)]. It is relatively more difficult to remove As III from water because it is 60 times more toxic than As V. Toxicity subsides in Inorganic arsenic only whileorganic arsenic is non-toxic. Arsenic may enter the food network or chain of the plants from soil on which irrigation is done by using water contaminated from arsenic or from agricultural products.

Punjab and Sindh, the two most densely populated provinces of Pakistan are the most vulnerable to arsenic contamination in groundwater. A recent study conducted by Sanjrani and his research fellows in 2017 concluded that 16% of the Sindh's population was exposed to arsenic concentration exceeding 50 µg/L via drinking water, and 36% of the population is facing an exposure of concentrations higher than 10 µg/L. The situation in Sindh was much worse than that in Punjab, as shown by the results that more than 3% and 20%of the population is susceptible to water containing arsenic higher than 50 µg/L and 10 µg/L respectively, following the Pak-EPA standards and the WHO guidelines [8]. Contamination of groundwater due to the presence of arsenic places a severe risk on human health. Various industrial and naturalsources are held responsible for Arsenic contamination of the groundwater. The figure shows whether natural or industrial aspects are the cause of increased arsenic level in different areas of Punjab and Sindh.

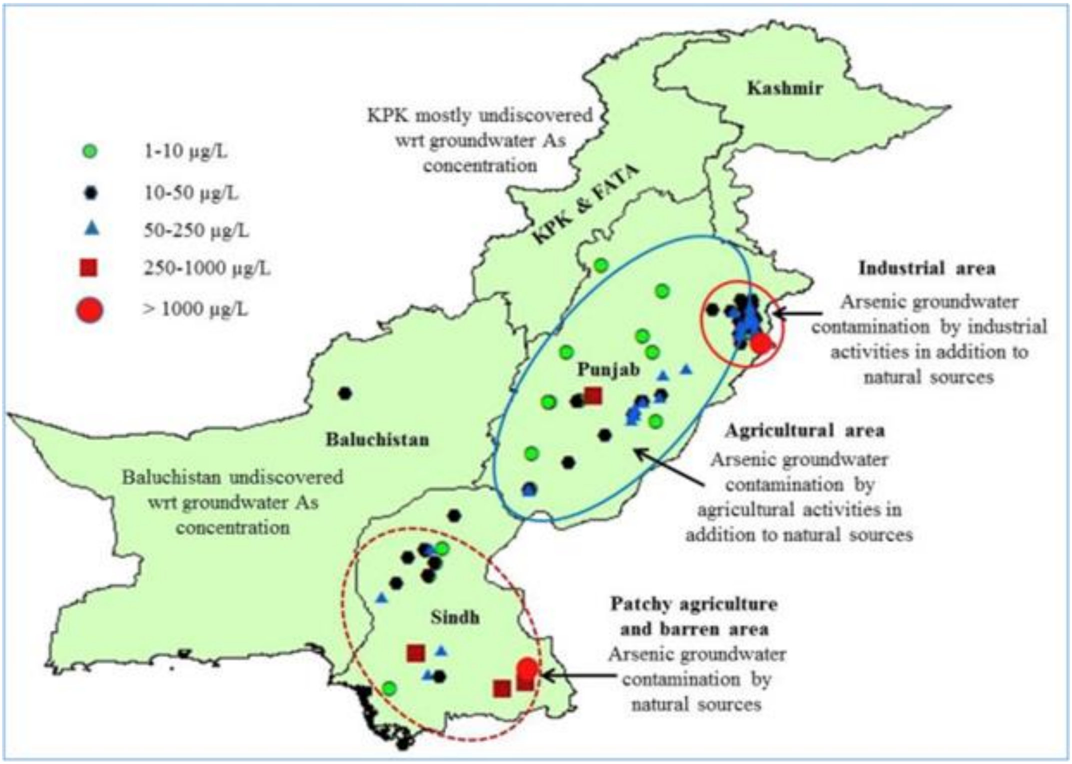


Figure 1.3-1 - Source: (Shahid et al., 2018)

1.4 Objectives

Our research comprises of following objectives:

1. Optimization of the iron nails as an adsorbent on batch scale against varying parameters, stated as follows:
    i. **Dosage optimization:** Range for the dose was 1,5,25,50 and 100 grams per liter solution.
    ii. **Contact Time optimization:** Contact time was ranged from 15 mins - 120 mins.
    iii. **pH optimization:** pH was varied from 2 to 11.
    iv. **Initial Concentration optimization:** Initial Concentration of Arsenic was kept between 100-500 µg/L.
    v. **Temperature optimization:** Temperature ranged between 15°C -35°C.

And also, the application of isotherm studies to the results, namely Freundlich and Langmuir.

2. The design and optimization of a flow-through filter so that the system can be applied as a commercial product at household level.

    A cost efficient and highly effective filter for arsenic removal was designed. The aim was to target and provide a solution to the rural communities being affected by arsenic contamination.

3. Real groundwater testing to investigate ifanychanges in the removal efficiencies of synthetic water and real water occur; and the regeneration of the designed filter.

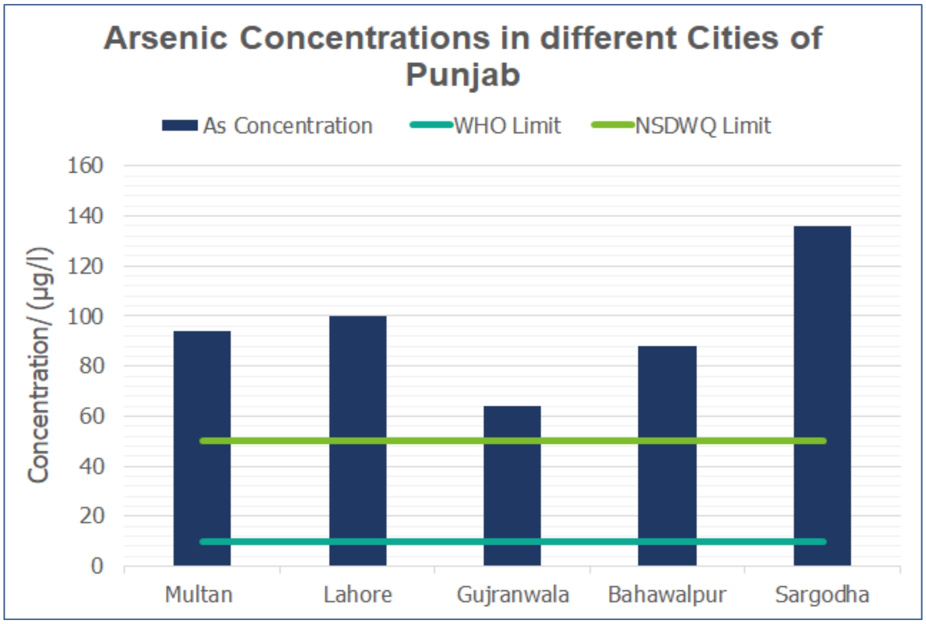
    The filter was tested against synthetic samples and then to ensure its application in real life scenarios, it was also tested against real groundwater samples.

LITERATURE REVIEW

2.1 Background

Pakistan stands third in the list of countries of the world that are facing an acute shortage of water and currently the availability of water is lower than 1000 m^3 per capita, as stated by a report recently presented by the International Monetary Fund (IMF).Due to the shortage of clean surface water resources there has been a rise in the reliance on groundwater sources for potable purposes due to which a sizable percentage of the population is being exposed to numerous pollutants. Arsenic is one of the leading contaminants in Pakistan's groundwater and present in the following different forms i.e. arsenite and arsenate. Arsenic has been known to be significantly toxic to all life forms[9]. The World Health Organization classified it as a group 1 human carcinogenic substance [10].

A mean Arsenic concentration of 14.9 µg/L, and a peak concentration of 350 µg/L was found in the potable water from wells in two hundred and sixteen villages, selected at random, in Sindh along the Indus River [11].



Graph 1 - Source: (Sanjrani, Mek et al. 2017)

The graph shows the average arsenic concentration observed in the samples taken from the different cities of Punjab. The minimum concentrations were found in Gujranwala being 1.28 times higher than the NSDWQ limit of 50 mg/L and the maximum concentrations were found in Sargodha, being 2.72 times higher than the defined limit. [8]

2.2 Arsenic Occurrence

Arsenic was rated as the most toxic substance by The United States' Agency for Toxic Substances and Disease Registry in its Priority List of Hazardous Substances at Superfund sites(2001) [12]. Arsenic in natural system is mostly exist in two valence states +3 and +5. It can also be present in an extremely toxic state that is -3. The toxic state of -3 can be created under very reducing conditions, which is why it is relatively rarely found in nature. Whilst organic and inorganic species of arsenic both abundantly exist in the natural environment, with the inorganic species being more commonly present in the freshwater bodies. It is a highly significant inorganic pollutant that is being added to the environment through different anthropogenic or natural activities and harmful to not only the humans but also the environment. United States Environmental Protection Agency (USEPA) has strictly established an action level of 10µg/L for total arsenic present in the water used for drinking purposes. Susceptibility to high arsenic concentrations in human beings can cause health problems such as arsenicosis and various cancers.[8]

2.3 Sources of Arsenic

2.3.1 Natural

Arsenopyrite is the most common mineral specie among 200 others in which arsenic is present.
Approximately 1/3rd of the arsenic entering the atmosphere has been found to be due to natural sources. A major source being volcanic activity, followed by low-temperature volatilization.
Long-term geochemical variations cause the arsenic-containing soils and rocks to be eroded which are then washed out resulting in high arsenic concentrations in the environment.
In various parts of the world, inorganic arsenic is used as drinking- and is found in groundwater.
Compounds such asarsenocholine, arsenobetaine, tetramethylarsonium salts and arsenosugars are primarily found in marine species however some of these compounds of the organic nature have been found in terrestrial species [9].

2.3.2 Anthropogenic

Charcoal reduces arsenic trioxide (As_2O_3) to produce elemental arsenic. While metal smelting processes produce As_2O_3as their by-product; 22% and 70% of the world's arsenic production have been found to be used in agricultural chemicals and in timber treatment as copper chrome arsenate (CCA), respectively. The remaining arsenic is used in the production of pharmaceuticals, glass and non-ferrous alloys.

Major contamination of the environment i.e. land, air and water, due to arsenic is because of the following industrial processes; smelting of non-ferrous metals, mining and burning of fossil fuels. In olden times pesticides that contained arsenic were used on agricultural fields, which has left large fractions of land contaminated. The preservation of timber requires arsenic, that is another industrial process that pollutes the environment with arsenic [10].

2.4 Toxicity of arsenic

2.4.1 Impact on Plants

Arsenic is present in natural waters and all soils; thus, arsenic ions being there caused the plants to grow. It could therefore be assumed that for plant growth arsenic is an important element, but it has not been proved. The beneficial effects of arsenic on plants are not well authenticated. Arsenic can substitute for phosphorus as an important nutrient for plants because they are chemically alike, however the toxicity of arsenic in the soil has been found to be increased by the application of phosphates-containing fertilizer [9]

2.4.2 Impacts on Human Health

Because of the toxic nature, arsenic has both acute as well as chronic health effects. Acute effects include abdominal pain, vomiting, muscle cramping diarrhea, and in some cases death. Chronic health effects of arsenic poisoning are related to developmental effects, skin lesions, cancers of bladder and lungs, diabetes and cardiovascular diseases [8].

1. Skin Illness

Long term exposure causes numerous skin changes [13]. Initial diagnosis of arsenic poisoning is generally based on blisters, skin lesions, hyperpigmentation and solar keratosis. In a non-melanin pigmented skin, arsenic may cause a basal cell carcinoma [14].

2. Gastrointestinal Disease

Diarrhoea is known to be an early and major occurring symptom for acute arsenic poisoning. This symptom appears sporadically, and it may be related to chronic toxicity with vomiting. The suspicion of arsenic ingestion is increased, if neuropathy and skin changes are also present [15].

3. Cardiovascular Disease

Due to high arsenic exposure, the risk of contracting a cardiovascular disease increases. Myocardial injury, cardiac arrhythmias, and cardiomyopathy are the known effects of arsenic exposure. Another disease which can be contracted due to exposure to arsenic for a longer period is called Black foot disease, which is related to peripheral vascular disease [16].

4. Neurological Disorder

The neurological system is the most targeted area for harmful upsets related to a few known metals. Lead, arsenic and mercury are the identified heavy metals that have toxic effects. The most frequently occurring conclusion is a peripheral neuropathy which mimics the Guillain-Barré syndrome having similar electromyographic results. [17]

Changes in confusion, memory loss and behavior are also related to the effect of arsenic poisoning [18]. Due to high exposure to arsenic, there is an increased rate of occurrence of cerebrovascular disease, especially cerebral infarction [6].

5. Genitourinary Illness

Arsenic present in drinking water can cause deaths and prostate and nephritis cancer. Drinking water having high arsenic concentrations lead to urethral cancers and transitional cell carcinomas of the ureter, kidney and bladder. [19]

6. Endocrine and Hematological Systems

The risk of contracting diabetes mellitus is increased as the exposure to drinking water with high arsenic concentration is increased. Chronic arsenic toxicity is linked with neutropenia [6].

7. Malignant Disease

Arsenic and malignancy are interrelated, and this is of concern as millions of people are exposed to arsenic and are potential victims. Lung, kidney, skin, liver and bladder cancers are the known effects of arsenic. Arsenic exposure is the leading cause for malignancies of the colon, skin, bone, liver, lung, bladder, kidney, liver, nasal cavity, larynx and stomach. [16].

Arsenic poisoning has an unfavorable effect on activation of the proto-oncogene c-myc, methylation of DNA, DNA repair and increased free radical formation. Under certain circumstances, arsenic can behave as a tumor progressor, tumor supporter and co-carcinogen [6].

2.5 Removal Strategies

Various state of the art technologies are currently being used to reduce and ultimately remove arsenic ions from the water which include:

2.5.1 Reverse Osmosis

Reverse osmosis (RO), is a promising practice because of the flexibility and modularity of the system in concern. Concentration of arsenic was brought down to 10 microgram/L using reverse osmosis. However, this system is highly energy intensive which will make it an expensive process. Additionally, a liquid with high concentration of arsenic is produced, which needs careful measures to dispose [20].

2.5.2 Coagulation

For arsenic removal from surface or ground water, the coagulation process is an effective method. The advantage of this method being that it does not call for any additional pre-treatment or conditioning, except in some cases. At higher concentrations of FeCl3, the removal efficiencies of arsenite and arsenate were enhanced. However, this leads to the content of residual iron in the drinking water to exceed the contaminant level [21].

2.5.3 Ion Exchange

This method is used to treat water on a larger scale. Soluble ions from the liquid phase are attracted towards the solid phase in this process. This method is very cost effective and also works for low concentrations of heavy metals [22]. Cations and anions are separated by using an ion exchanger.

2.5.4 Adsorption

For heavy metals' removal, adsorption is extensively used. This is the simplest method involving low-cost and non-toxic adsorbents. Some of them are activated carbon, alumina and iron oxide. Many other adsorbents are being synthesized and functionalized with suitable functional groups [23]

The reason we chose natural iron oxide by using iron nails is because it outweighs all the cons of the methods mentioned. The natural iron oxide seems to be an effective adsorbent as it has high removal efficiencies while ensuring cost-effectiveness and it requires no chemical modification. Iron oxide is widely used for the removal of heavy toxic metals as known to give removals of up to 90 -99 % [24]. Hence, we decided to test its effectiveness against arsenic laden groundwater.

MATERIALS & METHODOLOGY

3.1 Materials

All reagents used were of analytical reagent grade and were utilized without further purging.

- Arsenic standard solution (1000 mg/L)
- 15% nitric acid solution
- Hydrochloric acid
- Sodium hydroxide
- Recycled iron nails
- Sand
- Gravel
- Distilled water

3.2 Methodology

3.2.1 Preparation of Stock Arsenic Solution

For performing experiments, standard arsenic solution with a concentration of 1000 mg/L was serially diluted into different standard solutions. 200 µg/L of arsenic stock solution was prepared by adding 0.6 ml of the standard solution in 3 liters of distilled water.

3.2.2 Spectrophotometric Detection of Arsenic

Arsenic analysis was conducted via a Spectro Genesis Inductively Coupled Plasma-Optical Emission Spectroscopy (ICP-OES) GBC 932B PLUS at 189.042nm in accordance with the USEPA method 2005. In adsorption test, the iron concentration of iron was analyzed simultaneously by the ICP-OES to monitor the residual iron leaching into the effluent. Although arsenic and iron interfere with each other in ICP analysis, because in all isotherm tests the concentration of iron was found very small (<0.3 mg/L), the interference caused by iron at 189.042nm was negligible.

Figure 3.2-1- ICP-OES

3.2.3 Working principle of Spectrophotometer

The elements shift into an excited state after the plasma energy is provided to the sample that is needed to be analyzed. The excited atoms come back to low energy positions, emission rays are released and the emission rays that correspond to the photon wavelength are measured. The element type is determined by the location of photon rays, and the intensity of rays are used to determine the content of each element [25]

3.3 Arsenic Adsorption Studies

The bearing of the iron nails as arsenic adsorbents was evaluated by batch adsorption tests. Optimized dose of iron nails was added into 100 ml solution containing arsenic concentration of 200 µg/L. Sodium hydroxide and hydrochloric acid were used to adjust the pH to 7, followed by shaking in for 30 minutes at 175 rpm. After shaking, the solution was filtered by using 40 microns Whattman filter paper. After filtration absorbance of each sample was measured to ascertain the concentration of remaining arsenic present in the solution using ICP-OES.

To calculate the percentage removal, following formula was used

$$\% \, removal = \frac{C_o - C_t}{C_o} \times 100$$

Where;

- C_o = Initial concentration of arsenic
- C_t = Residual arsenic concentration

3.3.1 Working Range of Parameters

Working range of various parameters is described as follows:

1. **Dose**: For optimization of dose, experiments were conducted on arsenic concentrations of 200 µg/L. Range for the dose was 1,5,25,50 and 100 grams per liter solution.
2. **Contact time**: Effect of time was investigated from 0-120 minutes at 30°C
3. **pH**: pH was varied from 2 – 11
4. **Temperature**: Temperature was ranged between 15 – 35°C.
5. **Initial Concentration**: Effect of initial concentration on arsenic adsorption was determined from 100-500 µg/L.

3.3.2 Isotherm Models

The amount of solute that an adsorbent can adsorb onto their surface can be predicted by isotherm models. The following two well established and popular models were used:

3.3.3 Langmuir Isotherm

Langmuir Isotherm predicts the relationship between change in adsorption and equilibrium concentration. The following are the assumptions for this isotherm:

1. The monolayer adsorption takes place on the homogenous surface.
2. The adsorbate molecules donot interact with each other and majority of the sites have been occupied by the adsorbate [26].

The equation is given as:

$$\boldsymbol{\frac{C_e}{q_e} = \frac{1}{Q_m} \,. b. C_e + \frac{1}{Q_m}}$$

Where C_e is the equilibrium concentration of solution (mg/L), Q_m is the adsorption capacity of monolayer, b is the Langmuir constant (L/mg) which is related to the affinity of binding sites.

3.3.4 Freundlich Isotherm

Freundlich Isotherm anticipates the relationship between

1. The concentration of solute which is in contact with the adsorbent.
2. The concentration of solute adsorbed on the surface of an adsorbent and;

In this Isotherm, surface is assumed to be heterogeneous for occurrence of adsorption phenomenon [27].

Its equation is given below:

$$\boldsymbol{Q_e = K_f . C_e . \frac{1}{n}}$$

Where K_f is the amount of solute adsorbed on the adsorbent surface, n is the deviation from linearity of the adsorption.

3.4 Design of Filter Column

The filter media was arranged in the following manner:

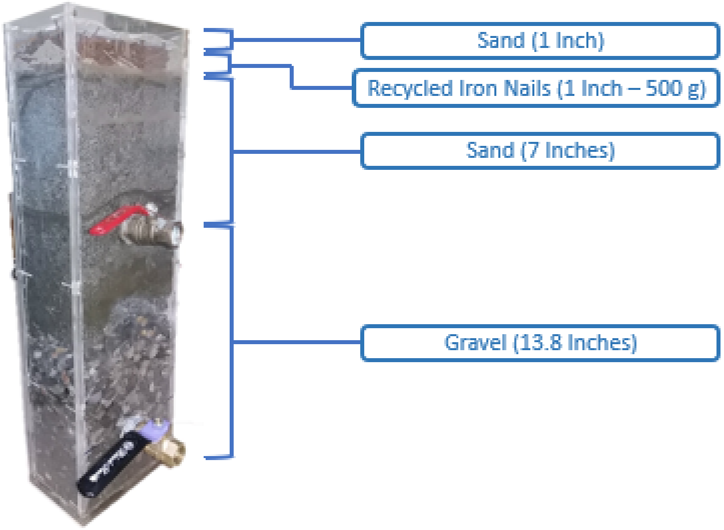


Figure 3.2-2 – Design of Filter

Figure 3.2-3 – Pipe with Six Outlets

The depth of each layer was determined and calculated on the basis of column dimensions and literature and so was the flow rate which came out to be 3.6 L/hr. A pipe with six outlets was used to supply water to the filter; this was to ensure uniform flow of water to the entirety of the filter column.

3.5 Real Groundwater Testing

The samples were collected from a village known as Khudpur Village, near Lahore [28]. For the sole purpose of determining the efficiency of the column filter against real ground water, containing other ions.

3.5.1 Sampling

The water samples were collected in PET bottles for the purpose of testing real groundwater on the column-based household filter. Arsenic testing kit was used to determine if the source water was contaminated with arsenic, before it was sampled.

3.5.2 Testing

The samples collected from Khudpur Village were tested against the various drinking water quality parameters to determine the composition of water or to determine what other ions are present in the groundwater. This was done so that a comparison of Initial composition can be made with the final composition of the water, after it has been passed through the column filter.

- Total suspended solids: The total suspended solids in the sample were determined by gravimetric analysis. A well-mixed, measured volume of a water sample was passes through a pre-weighed filter. The filter was heated at constant temperature of 105º C and then weighed. The mass increased divided by the water volume filtered is equal to the TSS in mg/L.

- Total dissolved solids: Total dissolved solids were measured by using conductivity meter. Total dissolved solids and electrical conductivity are correlated and usually expressed by a simple equation:

$$\boldsymbol{TDS = k.EC}$$

; where k is a conversion factor [29]

- Turbidity: Turbidity meter was used to test the turbidity of water samples. According to World Health Organization guidelines, the water is safe for drinking if the turbidity is below 5 NTU.

- Hardness: Hardness of water can be determined by EDTA titrimetric method. A sample of water was analyzed to measure the amount of hardness by performing titrations. An EDTA (ethylene diamine tetra-acetic acid) solution was used as a titrant, which capture or react with the calcium ions in the water. To visualize the end point Erichrome black T was used as metal ion indicator. The end point was indicated when the original red solution turned to blue. The mg/L of calcium carbonate was calculated by using the following formula: [30]

$$\boldsymbol{mg/L\ CaCO_3 = (ml\ of\ EDTA\ used\ in\ titration/ml\ of\ water\ sample)\ \times 1000\ mg/L}$$

RESULTS & DISCUSSIONS

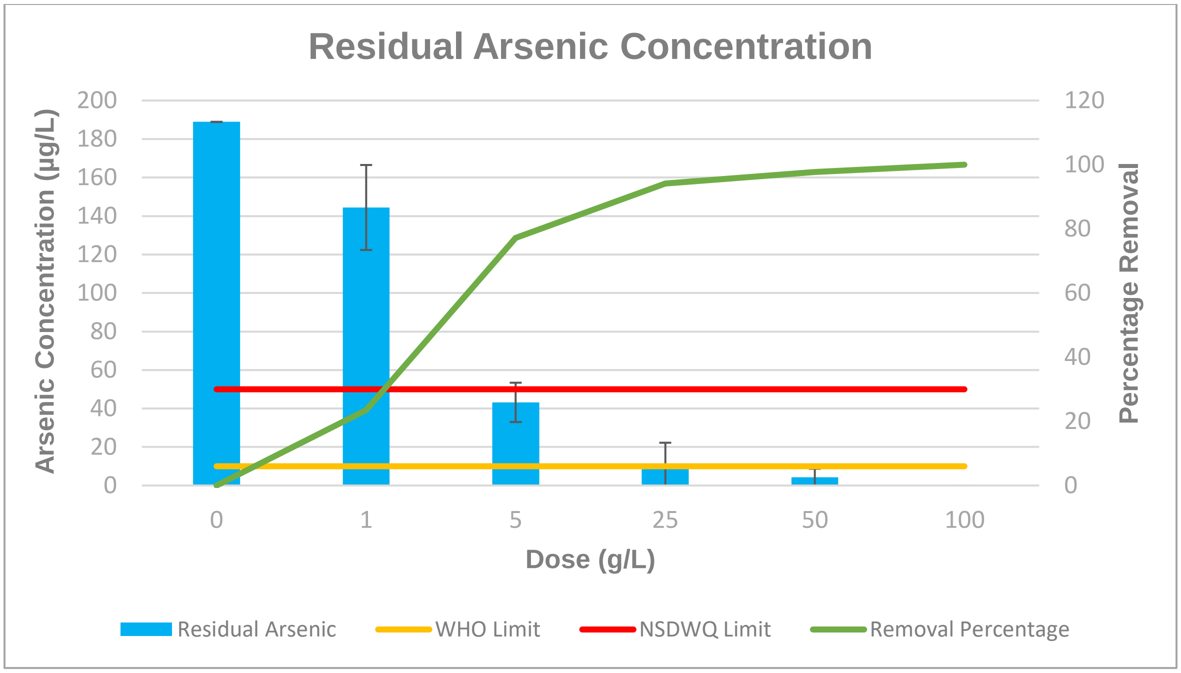
4.1 Optimization of Different Parameters

Results of optimization of parameters including contact time, dose of iron nails, temperature, pH and initial concentration are as follows:

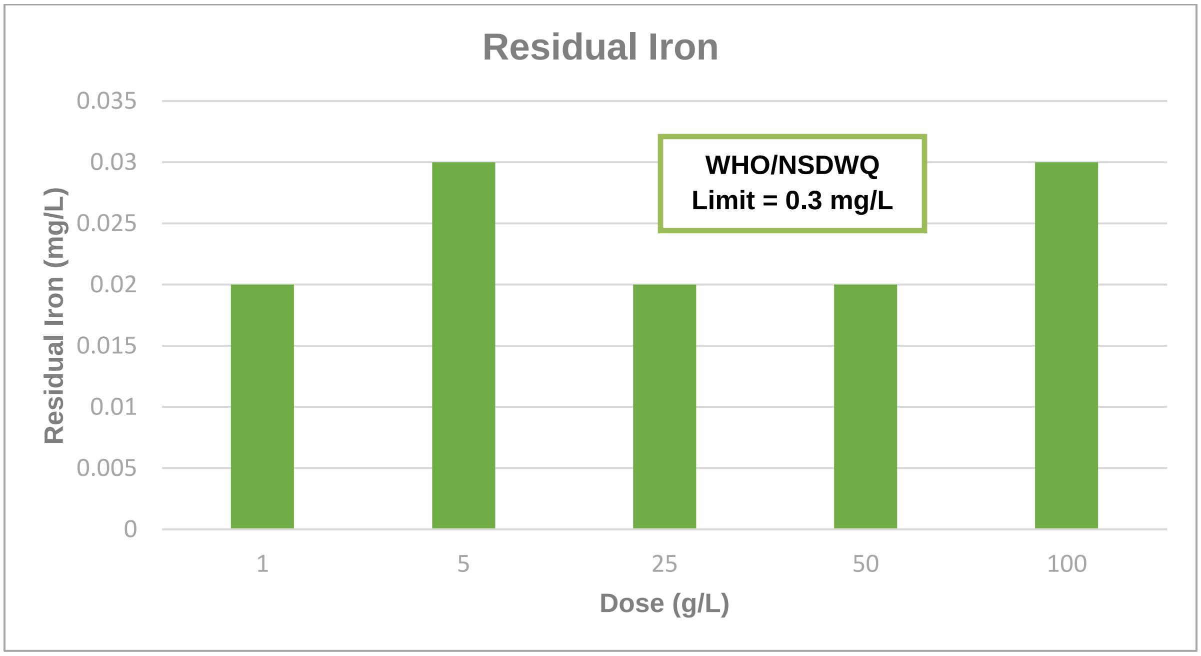
4.1.1 Effect of Dose

For optimization of dose, experiments were conducted on arsenic (As) concentration of 200 µg/L. The dose was varied from 0-100 g/L for iron nails. The experiments were performed at a constant temperature of 30°C and shaking was done for 60 mins at a speed of 175 rpm. The graph 2 shows the final concentration after removal of arsenic using iron nails as an adsorbent. The removal efficiency was observed to increase with an increase in dose with a maximum removal efficiency of 100% observed at the quantity of 100 g/L and a removal efficiency of 97.7% was observed at a dose of 50 g/L. The dose of **50 grams of iron per 1 liter** of water was taken as the optimum dose for further experiments, because at this dose the residual concentration of arsenic was well below the NSDWQ and WHO limits. The residual iron concentration at each of the doses was also observed to ensure that the iron present in the effluent water due to leaching of the iron nails is below the maximum permissible limit defined by the NSDWQ and WHO. The graph 3 shows that the residual iron concentration at the optimized dose of 50 g/L was found to be 0.02 mg/L which is well below the defined limit of 0.3 mg/L.

There is less significant increase in the removal efficiency as the dose is increased from 50 g/L to 100 g/L. This may be attributed to the fact that as the dosage increases the number of available active sites for adsorption increases. The results observed are in line with the work done by Chen etal. and Samad et al. [31, 32].



Graph 2– Effect of dose on removal

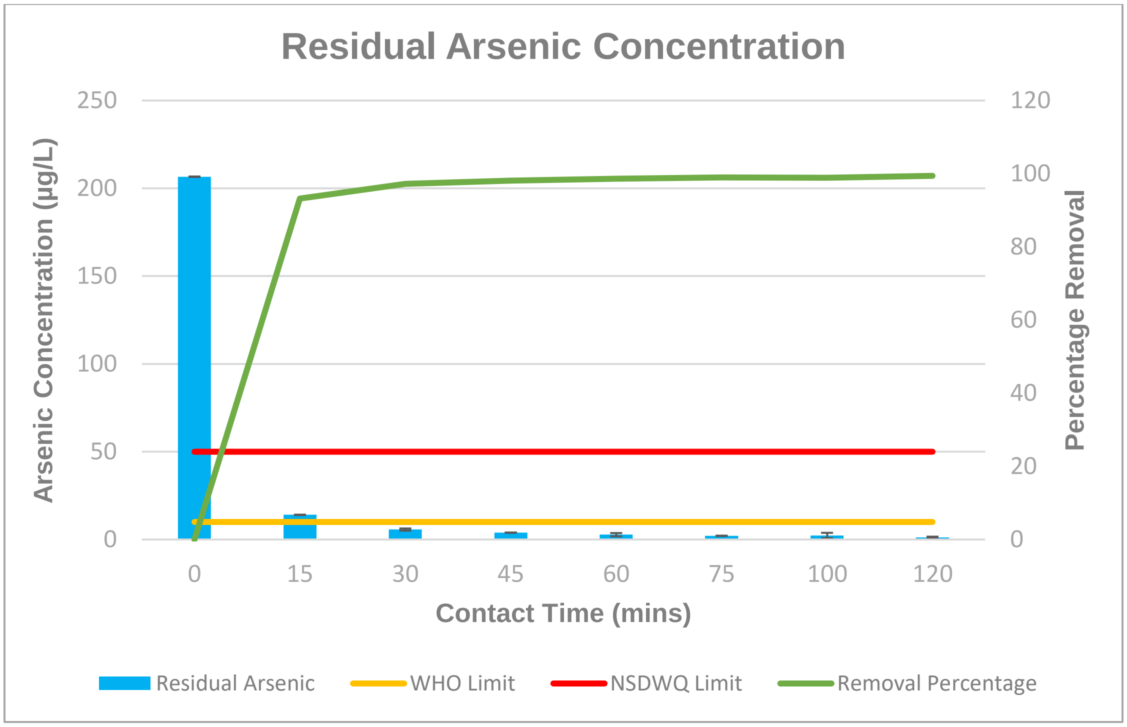


Graph 3 – Effect of dose onresidual iron

4.1.2 Effect of Contact Time

For optimization of contact time, experiments were conducted on arsenic (As) concentration of 200 µg/L. The contact time was varied from 15 minutes to 120 minutes. The experiments were performed at a constant temperature of 30°C and shaking was done at a speed of 175 rpm. The dose of adsorbent used was 50 g/L as optimized earlier. Graph 4 shows that the removal rate increased with increasing contact time with the maximum removal efficiency being achieved at 120 minutes of contact time and whilst the residual arsenic concentration at 15 minutes is below the NSDWQ defined limit for arsenic, it is not within the limits defined by WHO hence the contact time was optimized at **30 minutes** being the most feasible and achieving the required arsenic removal. The residual iron concentration was also within limits throughout the range of contact times and found to be as low as 0.05 mg/L at the optimized time of 30 minutes.

Before equilibrium was attained, as the contact time increased the removal efficiency increased and then the efficiency became constant, similar observations were made by Mondal et al. [33] Equilibrium was reached after 30 mins which indicates that the adsorption occurred on the surface of the adsorbent, and swiftly reached the saturation point within the fast adsorption time period, as was determined by Lin et al. [34].



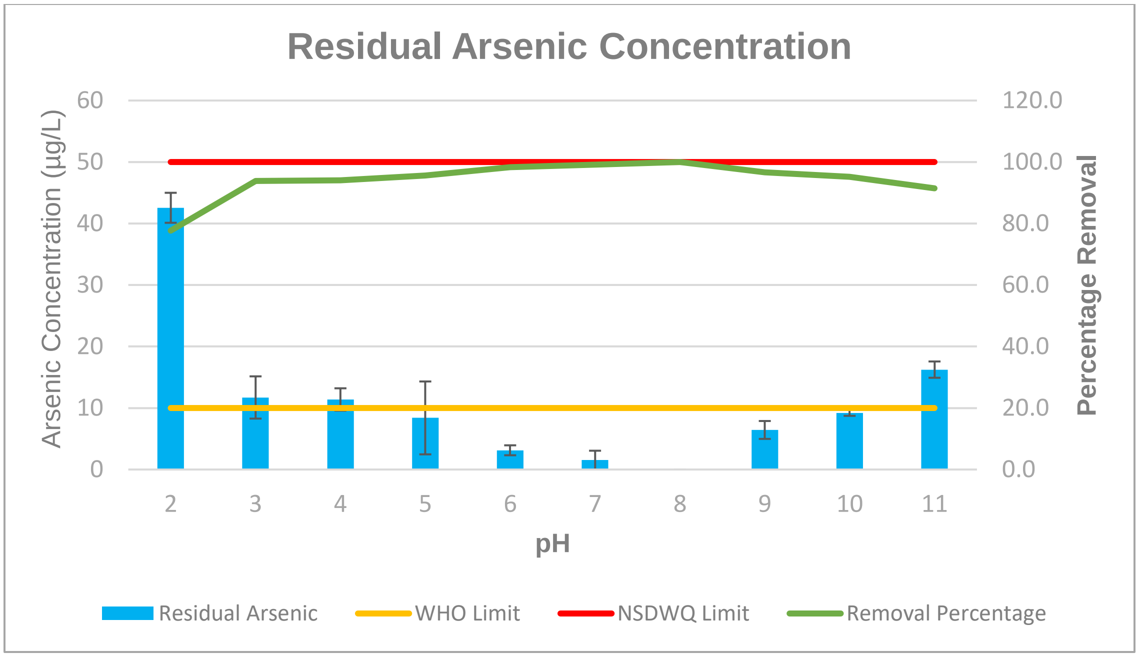
Graph 4 – Effect of contact time on removal

Graph 5 – Effect of contact time on residual Iron

4.1.3 Effect of pH

pH of the aqueous solution is the most important factor affecting arsenic adsorption process because it affects the chemical properties of adsorbate, speciation of arsenic and surface characteristics of the sorbent [35]. The effect of solution pH on the adsorption of arsenic by using an iron nails dose of 50 g/L, placed in the shaker at 30°C for the optimized contact time of 30 minutes at a speed of 175 rpm, is illustrated in the graph 6. The graph shows a decrease in residual arsenic concentration from pH 2-8 and then concentration begins to increase after pH 8. Hydroxyl groups on the surface of the adsorbent due to iron rusting attract the arsenic particles and promote adsorption. Even though the highest removal rate was observed at pH 8, the **pH was optimized at7** since it is more economically feasible, and the residual arsenic concentration acquired meets both the NSDWQ and WHO limits. The residual iron is also within limits at the neutral ph7.

Changing the pH from acidic to basic can vary the net charge on the adsorbent surface, which affects the adsorption process. The surface charge is zero at zero point of charge pHpzc. The surface of iron nails is positively charged below pHpzc which favors anion adsorption by electrostatic attraction. The surface is negatively charged above the pHpzc which favors cation adsorption. The negatively charged arsenic ions and positively charged surface of the adsorbent favor the adsorption of arsenic hence adsorption takes place in the pH range of 2-8. The removal efficiency may be higher near the neutral range because the arsenic species are mobile in this pH range. The trend observed in our study is in line with the studies performed by Samad et al. and Mondal et al. [32, 33].



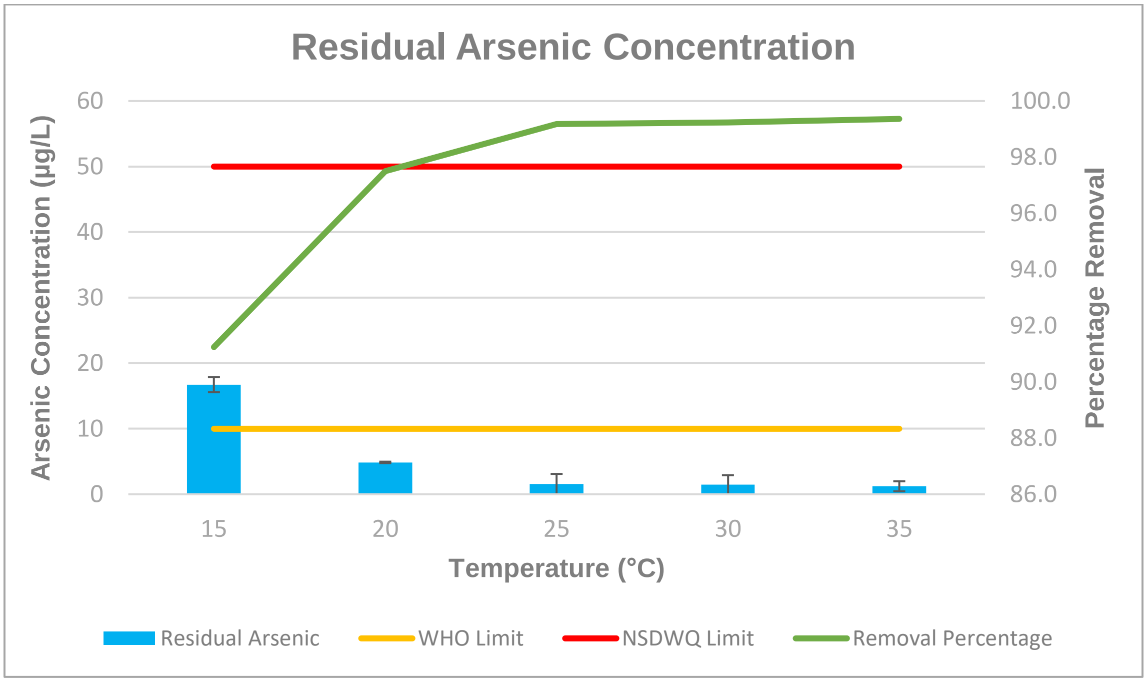
Graph 6 – Effect of pH on removal

Graph 7 – Effect of pH on residual iron

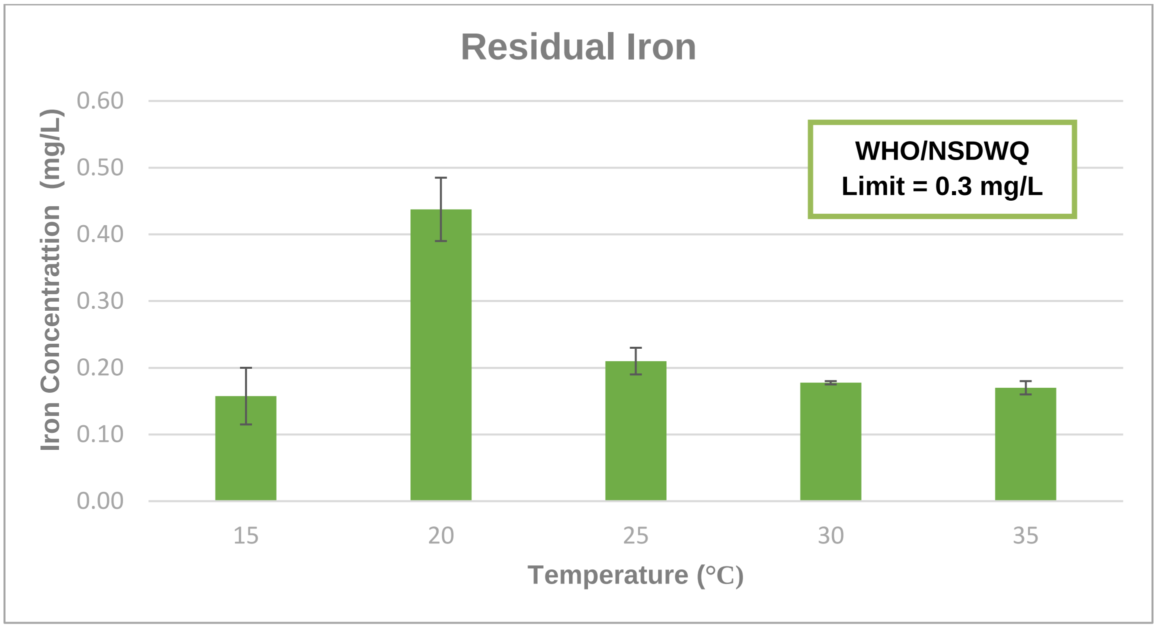
4.1.4 Effect of Temperature

For optimization of temperature experiments were carried on synthetic water sample with initial concentration of 200 µg/L whilst keeping all other parameters constant. Graph 8 shows that as temperature increases the removal efficiency increases. The maximum removal efficiency is achieved at 35 °C but 25 °C is an economically viable option and it lowers the residual arsenic concentrations well below the limits defined in the WHO guidelines. Hence the temperature was optimized at **25 °C**. The residual iron concentration at this optimized value is also within limits as shown in graph 9.

It can be seen on the graph that the removal efficiency increases significantly from 15 °C to 25 °C, after which there is a negligible increase in efficiency this may be due to the exothermic nature of the process of adsorption, and also because as the temperature increases the movement of ions also increases which in turn decreases the rate of surface precipitation, as determined in the study done by Mondal et al and Lin et al. [33, 34].



Graph 8 – Effect of temperature on removal

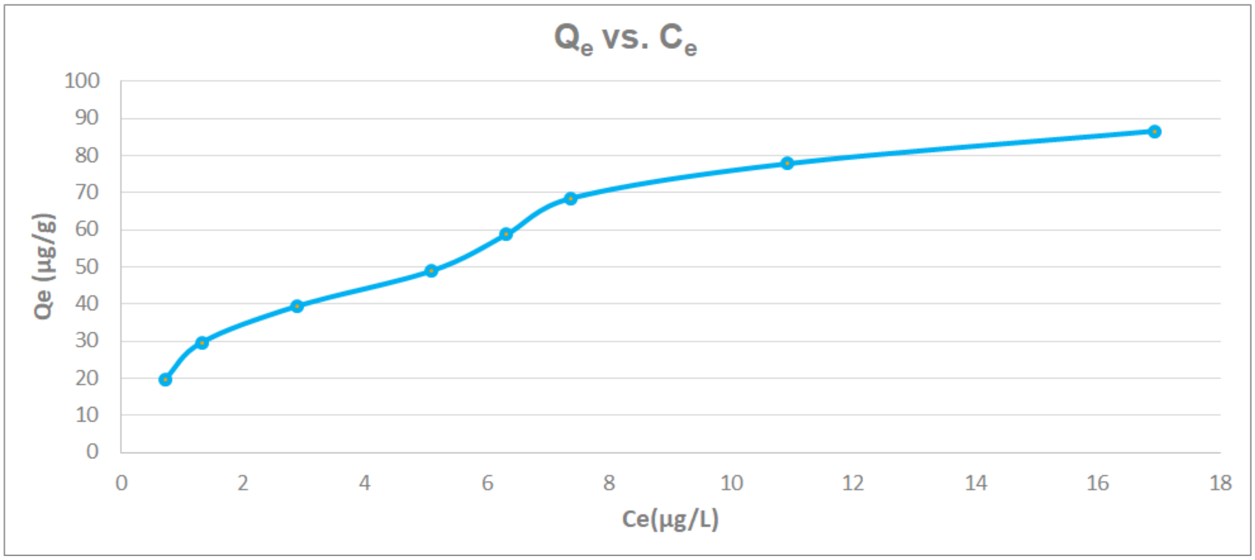


Graph 9 – Effect of temperature on residual iron

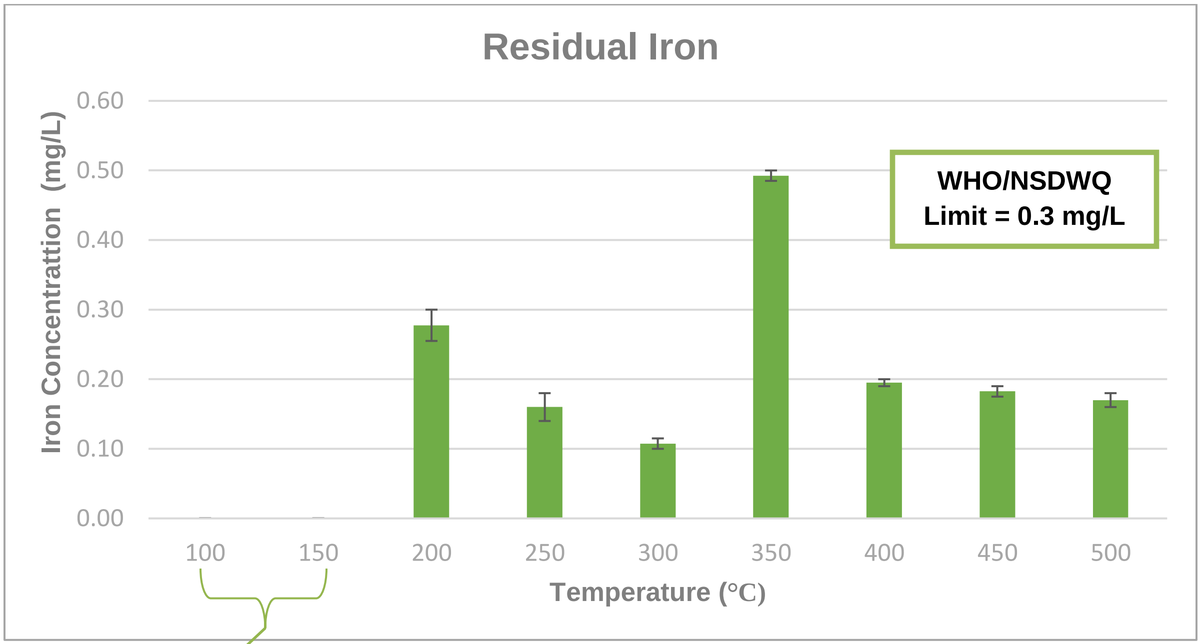
4.1.5 Effect of Initial Concentration

The effect of varying the initial concentration of arsenic on its removal was investigated from 100 - 500 µg/L. All other parameters were kept constant at the optimized values determined earlier. The graph illustrates that the arsenic removal does not increase significantly after 450 mg/L. The **adsorption capacity** came out to be **86.6 µg/g**, as shown by the graph. The residual arsenic was also observed to be within limits at all concentrations except350 µg/L; this anomaly in the trend may be due to variation in the level of rusting on the iron nails.

This trend can be explained from the fact that the adsorption has reached at its maximum value. All the active sites have been occupied by arsenic molecules and adsorption will not increase further. The removal efficiency decreased with increasing concentration, which means that arsenic uptake increased with increasing initial concentration; this trend has been verified by the work done by Samad et al. [32].



Graph 10 – Adsorption capacity of the adsorbent



Graph 11 – Effect of increasing concentration on residual iron

Negligible concentrations observed

4.2 Isotherm Models

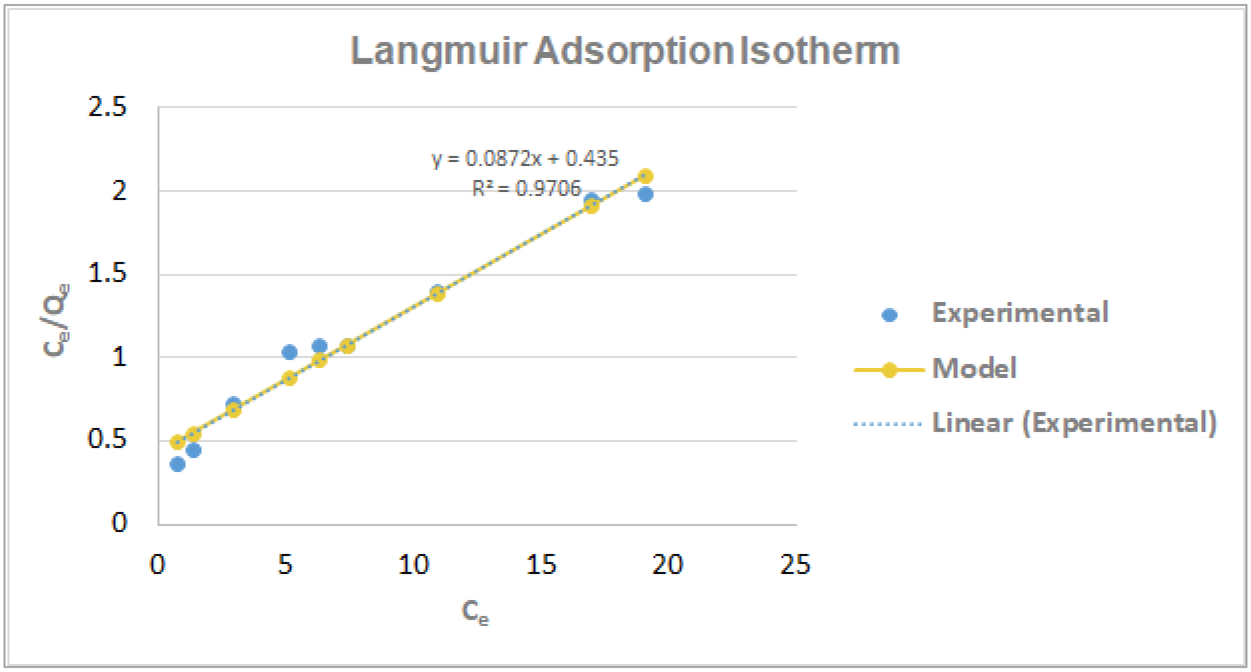
The process of Adsorption is usually studied through graphs know as adsorption isotherms. It is the graph between the amounts of adsorbate (x) adsorbed on the surface of adsorbent (m). The most commonly used and valid till date isotherms are Langmuir and Freundlich Isotherm models.

4.2.1 Langmuir Isotherm

Langmuir Isotherm predicts the relationship between the quantity adsorbed and equilibrium concentration. The Langmuir Isotherm assumes a single layer adsorption on a homogenous surface with a finite number of identical sites [37], which means that no interaction between adsorbed molecules occurs and majority of the sites have been occupied by the adsorbate. The adsorption capacity came out to be 11.5 µg/g. The values of constants obtained from trend lines are summarized in the table below.

Langmuir Isotherm Parameters	
Q_e (µg/g)	11.5
b (L/mg)	0.2
R^2	0.97

R_L values indicate the favorability of adsorption. The R_L values for our results came out to be within 0 - 1, hence suggesting favorable adsorption. The graph shows that our results have little deviation from the modeled results. It can also be observed on the graph below that the Langmuir Isotherm effectively describes the adsorption data since the value of $R^2 = 0.97$.



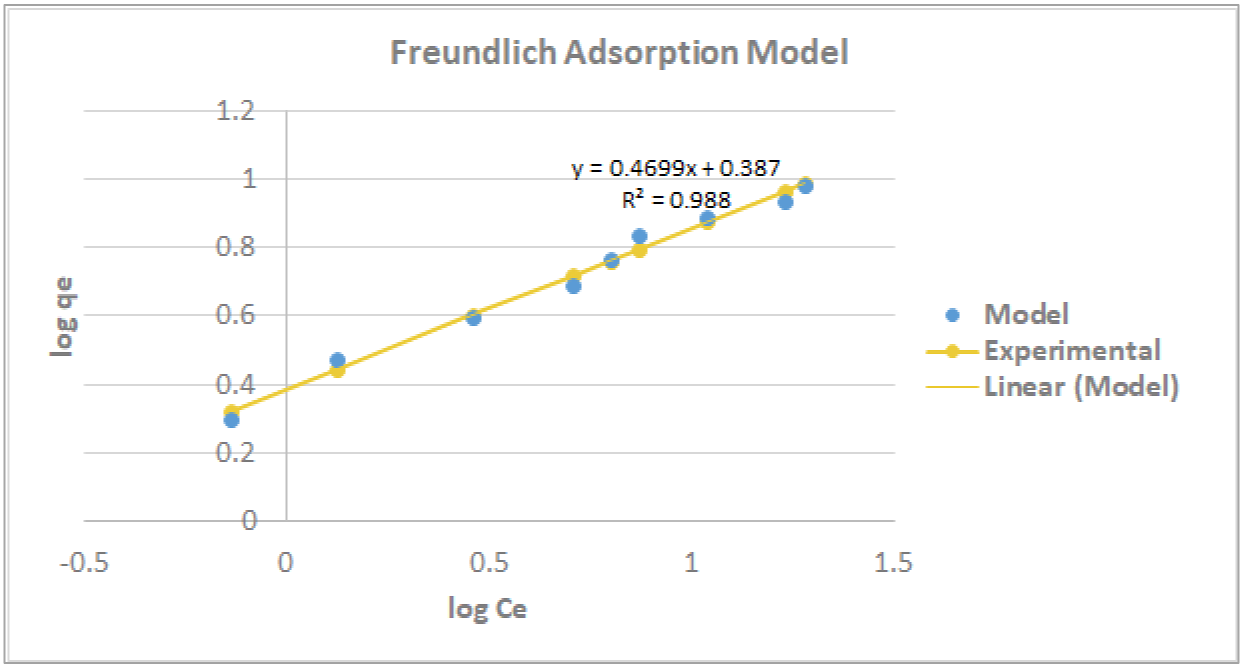
Graph 12 – Langmuir Isotherm

4.2.2 Freundlich Isotherm

Freundlich Isotherm predicts the relationship between the concentration of solute adsorbed on the surface of an adsorbent and the concentration of solute which is in contact with the adsorbent. Freundlich Isotherm assumes adsorption to occur on a heterogeneous surface.

Freundlich Isotherm Parameters	
n	2.13
K_f	2.44
R^2	0.99

It can be seen from the graph given below that Freundlich Isotherm more effectively describes the adsorption data with R^2 values equal to 0.99, being higher than Langmuir Isotherm hence more accurate.



Graph 13 – Freundlich Isotherm

Conclusions from Isotherms

In Langmuir the parameter b is related to the net enthalpy of adsorption that reflects the strength of binding of the adsorbate to the adsorbent or in other words the affinity of the adsorbate for the adsorbent [37]. The initial steep slope of Langmuir isotherms indicates the high values of b which show high affinity of Arsenic.

The results however illustrate that the Freundlich isotherm fits more perfectly to the adsorption data as observed in other studies as well. Therefore, the following conclusions were derived from adsorption isotherms:

- Freundlich isotherm more effectively describes the adsorption data relative to the Langmuir isotherm.
- Freundlich isotherm indicates adsorption on a heterogeneous surface.

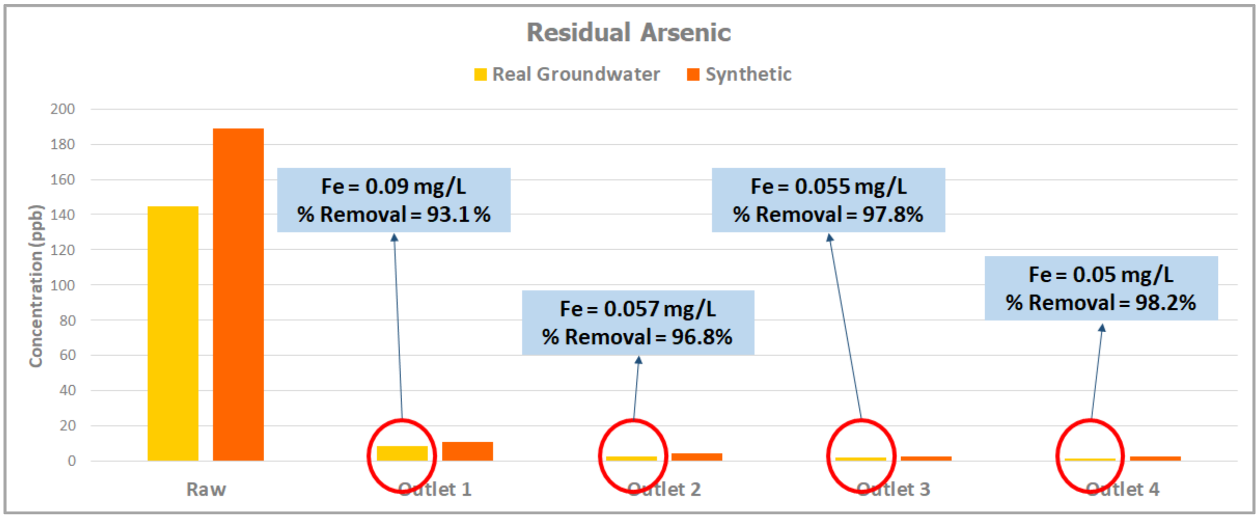
The results obtained correspond with the work done by Lin et al. and Chang et al., hence they can be verified [34, 36].

4.3 Real Groundwater Testing

The samples collected from Khudpur village were tested against the various drinking water quality parameters to determine the composition of water or to determine what other ions are present in the groundwater. This was done so that a comparison of Initial composition can be made with the final composition of the water, after it has been passed through the column filter, to determine if any variations in efficiency occur.

4.3.1 Arsenic Testing - Synthetic vs. Real Groundwater

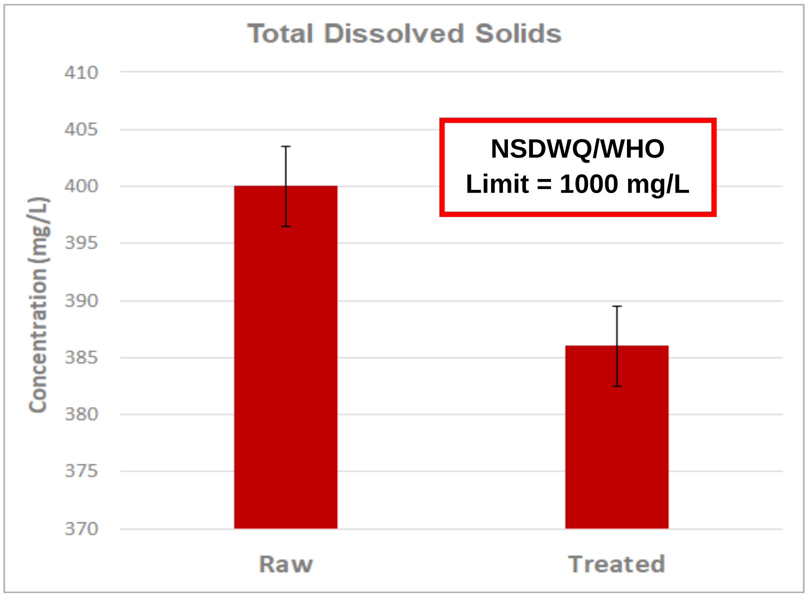
The main purpose of the filter was to remove arsenic from the groundwater, but all the other parameters for drinking water quality were also tested. Synthetic water with an arsenic concentration of 190 µg/L and the real groundwater having an arsenic concentration that was determined to be 145 µg/L were passed through the designed column filter. The effluent was collected at each of the four outlets, to check for the efficiency of each layer which is displayed on the graph as follows. The residual arsenic concentrations as well as the residual iron concentrations were found to be decreasing along with the depth of the column filter. A removal efficiency of 98.2 % was observed in the treated groundwater collected at the bottom of the column, from outlet 4 and the residual iron concentration for this treated water was 0.05 mg/L which is well below the defined limit of 0.3 mg/L.



Graph 14 – Arsenic removal from real ground water vs. synthetic water using the filter

4.3.2 Total Dissolved Solids

The Total dissolved solids were determined for the groundwater sample and the value acquired was found to be within the permissible limits as defined by the National Standards for Drinking Water Quality, i.e. below 1000 mg/L. The column had no significant role in decreasing the dissolved solids, but since they were below the defined limit this was not a matter of concern.



Graph 15 – Total Dissolved Solids in raw vs. treated groundwater

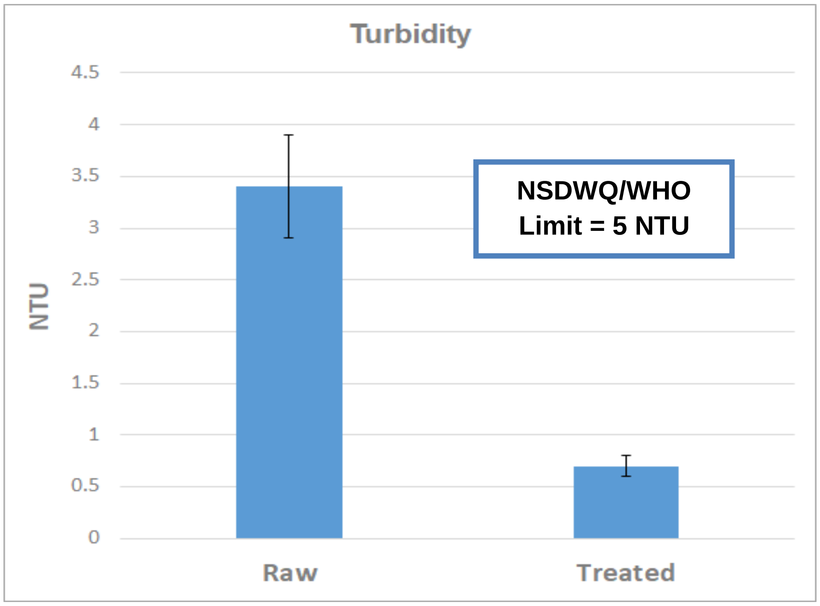
4.3.3 Total Suspended Solids

Gravimetric analysis was used to determine the total suspended solids in the sample. While the obtained sample already had low levels of suspended solids the column filter further reduced the total suspended solids to very low levels, as can be seen in the graph.

Graph 16 – Total Suspended Solids in raw vs. treated ground water

4.3.4 Turbidity

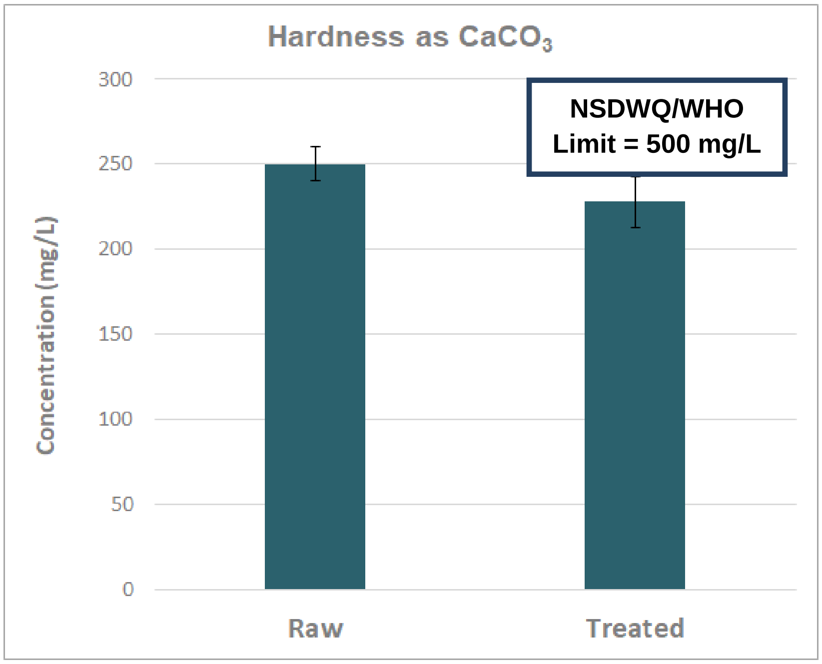
According to the limit stated by the NSDWQ as well as the WHO guidelines, the water is suitable for drinking if the turbidity levels are below 5 NTU. While the groundwater already contained low levels of turbidity, the filter further reduced them to insignificant levels.



Graph 17 – Turbidity in raw vs. treated ground water

4.3.5 Hardness (as CaCO3)

The filter had no substantial effect on the removal of hardness from the groundwater, since hardness is a component of the dissolved solids and unless the ions precipitate, they cannot be filtered using this column. But nonetheless the amount of Hardness found in the groundwater was below the defined limit of 500 mg/L.



Graph 18 – Hardness in raw vs. treated groundwater

4.3.6 Total Phosphorus & Chemical Oxygen Demand

The chemical oxygen demand and total phosphorus were found to be negligible in the groundwater sample.

Conclusion& Recommendations

5.1 Conclusion

Batch adsorption studies showed that the iron nails as an adsorbent were effective for the arsenic removal. The following conclusions were derived from our study:

- The optimized dose for iron nails adsorbent was 50 g/L.
- The adsorption capacity at pH 7.0 was 86.6µg/g.
- Freundlich Isotherm more effectively described the adsorption data.
- The column filter had a removal efficiency of 98.2% for real groundwater from Khudpur Village; Lahore, bringing the arsenic levels to 2.63 µg/L i.e. well below the WHO limit of 10 µg/L.

5.2 Recommendations

- Identifying whether other forms of waste iron like iron fillings can act as good adsorbents for removal of arsenic from water.
- Determination of the removal capacity of iron for other heavy metals found in the groundwater such as Cd, Mn, Cr, Pb etc.

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Appendices

Appendix – A

Batch Experiments

1.1 Dose

Initial Concentration (µg/L)	Volume (ml)	Dose/100 ml (g/100ml)	Residual Arsenic (µg/L)	Residual Arsenic (µg/L)	% Removal (mean)
200	100	0.1	166.52	122.38	23.5
200	100	0.5	53.41	32.98	77.1
200	100	2.5	22.23	0	94.1
200	100	5.0	8.72	0	97.7
200	100	10.0	0	0	100

1.2 Contact Time

Initial Concentration (µg/L)	Volume (ml)	Contact Time (mins)	Residual Arsenic (µg/L)	Residual Arsenic (µg/L)	% Removal (mean)
200	100	15	13.97	14.13	93.2
200	100	30	5.24	6.12	97.3
200	100	45	3.89	3.94	98.1
200	100	60	3.41	2.08	98.7
200	100	75	2.11	2.1	99.0
200	100	100	3.38	1.24	98.9
200	100	120	1.5	0.9	99.4

1.3 pH

Initial Concentration (µg/L)	Volume (ml)	pH	Residual Arsenic (µg/L)	Residual Arsenic (µg/L)	% Removal (mean)
200	100	2	40.12	45	77.7
200	100	3	8.28	15.15	93.9
200	100	4	9.58	13.21	94.0
200	100	5	2.47	14.32	95.6
200	100	6	2.32	3.94	98.4
200	100	7	0	3.06	99.2
200	100	8	0	0	100.0
200	100	9	4.98	7.89	96.6
200	100	10	9.69	8.73	95.2
200	100	11	17.57	14.92	91.5

1.4 Temperature

Initial Concentration (µg/L)	Volume (ml)	Temperature (°C)	Residual Arsenic (µg/L)	Residual Arsenic (µg/L)	% Removal (mean)
200	100	15	15.56	17.86	91.2
200	100	20	4.98	4.75	97.5
200	100	25	3.11	0	99.2
200	100	30	2.91	0	99.2
200	100	35	1.98	0.45	99.4

1.5 Initial Concentration

Initial Concentration (μg/L)	Volume (ml)	Residual Arsenic (μg/L)	Residual Arsenic (μg/L)	% Removal (mean)
100	100	1.45	0	99.6
150	100	2.65	0	99.3
200	100	3.13	2.62	98.5
250	100	4.95	5.23	97.3
300	100	6.2	6.43	96.7
350	100	6.89	7.86	96.1
400	100	11.11	10.73	94.3
450	100	17.11	16.76	91.1
500	100	18.89	19.25	90.0

1.6 Langmuir Isotherm

				Experimental	**Model**	
Initial Conc.	C_e	m	q_e	C_e/q_e	C_e/q_e	Residuals
μg/L	μg/L	g	μg/g	g/L	g/L	-
100	0.73	5	1.9855	0.365147	0.49822	0.133073
150	1.33	5	2.9735	0.445603	0.55054	0.104937
200	2.88	5	3.9425	0.729233	0.6857	-0.04353
250	5.09	5	4.8982	1.039157	0.878848	-0.16031
300	6.32	5	5.8737	1.075132	0.985668	-0.08946
350	7.38	5	6.8525	1.07625	1.0781	0.00185
400	10.92	5	7.7816	1.40331	1.387224	-0.01609
450	16.94	5	8.6613	1.955249	1.911732	-0.04352
500	19.07	5	9.6186	1.982617	2.097904	0.115287

1.7 Freundlich Isotherm

			Experimental	**Experimental**	**Model**	**Model**	
Initial Conc	C_e	Log C_e	q_e	Log q_e	Log q_e	q_e	Residuals
µg/L	µg/L	-	µg/g	-	-	µg/g	-
100	0.73	-0.13966	1.9855	0.297869891	0.321371	2.095901662	0.110402
150	1.33	0.122216	2.9735	0.473267943	0.444427	2.782449648	-0.19105
200	2.88	0.458638	3.9425	0.595771702	0.602512	4.004165302	0.061665
250	5.09	0.706718	4.8982	0.690036514	0.719085	5.237026345	0.338826
300	6.32	0.800373	5.8737	0.768911761	0.763094	5.795534723	-0.07817
350	7.38	0.867762	6.8525	0.835849044	0.794759	6.233894499	-0.61861
400	10.92	1.038223	7.7816	0.891068903	0.874859	7.496505958	-0.28509
450	16.94	1.228785	8.6613	0.937583081	0.964404	9.213067194	0.551767
500	19.07	1.280351	9.6186	0.983111864	0.988635	9.741702517	0.123103

Appendix – B

Residual Iron

2.1 Dose

Dose/100ml (g/100ml)	Volume (ml)	Residual Iron (mg/L)
0.1	100	0.02
0.5	100	0.03
2.5	100	0.02
5.0	100	0.02
10.0	100	0.03

2.2 Contact Time

Contact Time (mins)	Volume (ml)	Residual Iron (mg/L)
15	100	0.08
30	100	0.05
45	100	0.09
60	100	0.22
75	100	0.10
100	100	0.07
120	100	0.08

2.3 pH

pH	Volume (ml)	Residual Iron (mg/L)	Residual Iron (mg/L)	Mean (mg/L)
2	100	196.95	215	196.95
3	100	92	101.57	96.785
4	100	62.12	42.75	52.435
5	100	6.33	24	15.165
6	100	1.94	0.41	1.175
7	100	0.05	0.19	0.12
8	100	0.05	0.01	0.03
9	100	0.04	0	0.02
10	100	0.12	0.01	0.065
11	100	0	0	0

2.4 Temperature

Temperature (°C)	Volume (ml)	Residual Iron (mg/L)	Residual Iron (mg/L)	Mean (mg/L)
15	100	0.115	0.2	0.16
20	100	0.485	0.39	0.44
25	100	0.19	0.23	0.21
30	100	0.175	0.18	0.18
35	100	0.18	0.16	0.17

2.5 Initial Concentration

Initial Concentration (µg/L)	Volume (ml)	Residual Iron (mg/L)	Residual Iron (mg/L)	Mean (mg/L)
100	100	0	0	0.00
150	100	0	0	0.00
200	100	0.255	0.3	0.28
250	100	0.14	0.18	0.16
300	100	0.115	0.1	0.11
350	100	0.485	0.5	0.49
400	100	0.19	0.2	0.20
450	100	0.175	0.19	0.18
500	100	0.18	0.16	0.17