



REMOVAL OF AS (III) FROM SALINE WATER MEDIUM VIA ADSORPTION ONTO POLYANILINE/Fe₃O₄ MAGNETIC NANOCOMPOSITE

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ABSTRACT

Since many years, research groups throughout the world paying attention on removal of heavy metals from water. Saline water and fresh water both are consumed in diverse human sectors. Arsenic is a common element that is found in small amounts throughout the environment. Acute and chronic poisoning can result from consuming water that contains arsenic. Acute arsenic poisoning has no distinct symptoms. Weariness, colitis, loss of reflexes, weight loss, weakness, anorexia, gastritis, hair loss, and anorexia are a few of the non-specific symptoms of chronic poisoning. As for the pathogenesis of conditions like cardiovascular disease (CVD), hyperkeratosis, disturbances of the nervous and peripheral vascular systems, hyperpigmentation, circulatory disorders, hepatic and kidney disorders, experimental evidence has linked prolonged arsenic poisoning to these conditions (Hall, 2002) [1]. Recent advances for air arsenic removal include using both laboratory-scale and pilot-scale methods, as well as numerical modelling to help interpret pilot-scale results. Utilizing a tiny packed-bed reactor, new sorbents and removal methods for arsenic removal from flue gas are being studied on a lab scale. New sorbents are tested in the reactor system to see if they can arsenic from gas streams. These unique sorbents' capacities are calculated as a function of temperature and gas composition, and the findings are contrasted with those obtained using activated carbons that are readily accessible in the market.

Keywords: Polyaniline/Fe₃O₄, nanocomposite; heavy metals; Basic blue 3 dye; FeCl₃·6H₂O; Bioconcentration Factor (BCF); Polymerization; Deprotonation; Protonation; N-methylpyrrolidone

INTRODUCTION

Arsenic (As)

Arsenic appears in three allotropic forms: yellow, black and grey; the stable form is a silver-grey, brittle crystalline solid. It tarnishes rapidly in air, and at high temperatures burns forming a white cloud of arsenic trioxide. Arsenic is a member of group Va of the periodic table, which combines readily with many elements. The metallic form is brittle, tarnishes and

when heated it rapidly oxidizes to arsenic trioxide, which has a garlic odour. The non-metallic form is less reactive but will dissolve when heated with strong oxidizing acids and alkalis [2].

Arsenic is one of the well-established toxic elements and has been recognized to have an adverse human health hazard such as possible carcinogenic activity [3]. It occurs often in human tissues, including the hair, skin, and nails [4]. For the majority of people, drinking water is a main source of arsenic [5]. Up to 0.4 mg per day is the suggested tolerance limit for its absorption [6]. Inorganic arsenic predominates in drinking water and is present as arsenite [(As (III))] and arsenate [As (V)] [7]. Different levels of toxicity for various arsenic compounds (organic and inorganic) have been reported by some investigators [8]. The major sources of arsenic in natural waters are also from its widespread use in herbicides, pesticides, fungicides and insecticides [9]. The allowed concentration of arsenic in drinking water reported by the US Environmental Protection Agency (USEPA) is 50 ppb [10]. Living organisms are exposed to the toxic arsenic species primarily from food and water. However, certain scientific data also support the idea that arsenic should be used in moderation. The toxicity of arsenic arises from its ability to react with the sulphhydryl groups of the enzyme system thereby blocking the action of the thiol groups of enzymes [11]. Inorganic arsenic predominates in drinking water and is present as arsenite [(As (III))] and arsenate [As(V)]. Arsenic occurs in the natural environment in four oxidation states: As(V), As (III), As (0) and As (-III) depending on pH and redox conditions [12]. Among the different oxidation states, because of the ability to complex with certain co-enzymes, arsenite, As (III) is more toxic to animal and plants than As(V) (~10 times) and methylated species (~70 times) [13]. The mobility and toxicity of arsenic are determined by its oxidation state. The pH of the water and the oxidation-reduction conditions affect the valence and species of inorganic arsenic [14].

Sources of Arsenic in environment

Arsenic is found widely in Earth's crust in oxidation states of -3, 0, +3 and +5, often as sulfides or metal arsenides or arsenates. In water, it is mostly present as arsenate (+5), but in anaerobic conditions, it is likely to be present as arsenite (+3). It often occurs at quantities of less than 1-2 g/l in natural waters. However, concentrations can be markedly increased in waterways, particularly groundwaters, where there are sulphide mineral deposits and sedimentary deposits derived from volcanic rocks [15].

Anthropogenic sources, which account for 82,000 metric tons/year globally [16], are yet another significant source of concern for arsenic. Arsenic is typically coupled with other metals; it is discharged into the environment during mining or smelting for industrial uses. Wood preservatives (chromated copper arsenate [CCA] chemicals), mining of arsenopyrite, electrical waste (semiconductors), insecticides, pesticides, weed control, disposal of industrial and sewage materials, and paint products are a few examples of anthropogenic industrial sources that release arsenic directly or indirectly into the environment [17]. Dietary sources of arsenic include fish and shellfish, where it is mostly present in its less deadly organic form. There are only limited data on the proportion of inorganic arsenic in food, but these indicate

that approximately 25% is present in the inorganic form, depending on the type of food [18]. The most significant exposure pathways, aside from occupational exposure, are food and drinking water, including drinks manufactured with drinking water. Where the concentration of arsenic in drinking-water is 10 µg/l or greater, this will be the dominant source of intake [19]. When soups or other similar foods are a regular part of the diet, the amount of drinking water used in food preparation will be considerably higher. In locations with natural sources, concentrations can be increased (up to 12 mg/l), while levels in natural streams typically range between 1 and 2 g/l [20].

Polyaniline (Fe₃O₄) Magnetic Nanocomposite (PANI)

The chemical substance "Polyaniline (PANI)" is an organic semiconductor and conducting polymer that belongs to the same family as semi-flexible rod polymers [21]. Electrical conductivity and mechanical properties of this compound has attracted the scientists since the 1980s [22]. Polyaniline is one of the most studied conducting polymers by scientists, researchers and chemists. It is a conducting polymer belongs to a peculiar family of compounds composed of monomer units with conjugated chemical bonds that, under certain conditions (doping), ensure the electron conductivity of the polymer [23]. From the commercial viewpoint, these polymers show promise for the production of materials for various applications; they can replace metals and semiconductors, because they feature conductivity, low density, and easy processability. Polyaniline (PANI)—a representatives from the family of conducting polymers—is distinguished by easy synthesis and high environmental stability [24,25]. Polyaniline consists of monomer units built from reduced (n) and oxidized (mx) blocks:

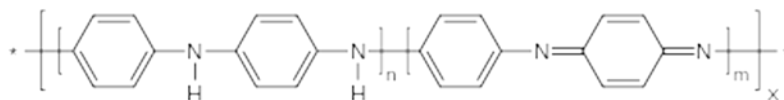


Figure 1.1. Structure of polyaniline

The protonation and deprotonation and various other physio-chemical properties of PANI can be said to be due to the presence of the -NH- group.

Green and Woodhead [26] were the first to depict PANI as a chain of aniline molecules coupled head-to-tail at the para position of the aromatic ring. They have proposed a linear octameric structure for PANI. Polyaniline, a typical phenylene based polymer, has a chemically flexible NH- group in the polymer chain flanked by phenyl rings on either side.

The diversity in physicochemical properties of PANI is traced to the -NH- group. Out of several possible oxidation states, the 50 % oxidized emeraldine state shows electrical conductivity. Because of its exciting physicochemical properties and doping-dedoping chemistry, polyaniline (PANI) has been as a potential adsorbent to remove heavy metals from saline media.

MATERIALS AND METHODOLOGY

Water sampling

Water sampling was done from five different sampling stations along the coast of Mumbai city, namely Manori, Versova, Bandra, Mahim and Worli.

The water samples were collected randomly during morning from five different sampling stations. The samples were collected and subsequently analysed during the month of October 2021. The sampling was done during morning between 06:00 a.m. to 08:00 a.m. Grab water samples were collected in 2.5 L and 2.0 L Polythene bottles. The bottles were thoroughly cleaned with hydrochloric acid, washed with fresh tap water and with distilled water twice to render free of acid, again rinsed with the water sample to be collected and then filled up the bottle with the sample leaving only a small air gap at the top. The sample bottles were stoppered and sealed with paraffin wax. Water samples (500 mL) were filtered using Whatman No. 41 (0.45 µm pore size) filter paper for estimation of dissolved Pb, As and Hg. Filtrate (500 mL) was preserved with 2 mL nitric acid prevention of metal precipitation. The samples were concentrated to tenfold on a water bath and subjected to nitric acid digestion using the microwave-assisted technique, setting pressure at 30 bars and power at 700 Watts [27, 28]. About 400 mL of the sample was transformed into clean glass separating funnel in which 10 mL of 2% ammonium pyrrolidine dithiocarbamate, 4 mL of 0.5 M HCl and 10 mL of methyl isobutyl ketone (MIBK) are added. The solution in separating funnel was shaken vigorously for 2 min and was left undisturbed for the phases to separate. The MIBK extract containing the desired metals was then diluted to give final volumes depending on the suspected level of the metals. The sample solution was then aspirated into air acetylene flame in an atomic absorption spectrophotometer [29].

Preparation of Polyaniline (Fe₃O₄) Magnetic Nanocomposite (PANI)

Aniline (Across) is vacuum-distilled before usage. Simple blue dye, FeCl₃·6H₂O (Sigma-Aldrich, St. Louis, MO, USA), FeSO₄·7H₂O, Na₂SO₄ and Dodecyl benzene sulphonic acid, DBSA (Across), have all been utilized in their original forms [30]. The grade of all substances should be analytical. The following substances were utilised as doping agents: aniline (ANI, 100%, Prolabo), hydrochloric acid (HCl, 37%, Carlo Erba), sulfuric acid (H₂SO₄, 98%, Sigma Aldrich), nitric acid (HNO₃, Cheminova, 60%), and phosphoric acid (H₃PO₄, 50%, Panreac). As oxidising agents for the chemical aniline polymerization, ammonium persulfate ((NH₄)₂S₂O₈, 98%, Biochem), potassium permanganate (KMnO₄, 99%, Fisher), and potassium dichromate ((K₂Cr₂O₇, 99%, Prolabo) were employed. The catalyst ink was created using isopropanol (99.5%, Sigma Aldrich) and Nafion® suspension (5 wt%) [31]. For the production of polyanilines or for electrochemical characterizations, all compounds were employed without additional purification, and electrolytic solutions were newly generated. The source of all referred to ultrapure water (MQ) is Milli-Q Millipore (18.2 M cm at 20 °C). Captive System S.r.l. created magnetite nanoparticle aggregates using a co-precipitation technique that has been described. It is important to note that the process

variables and chemicals employed to prepare the particles can have a significant impact on their aggregation characteristics, which rely on the pace at which the acidic and alkaline solutions mix and the thickness of the organic coating [31].

Synthesis of Fe₃O₄

Preparation of PANI

Numerous reports on the oxidative polymerization method of PANI manufacture have recently been published in research, as shown in Figure 1.2. This approach uses chemical or electrochemical processes to concurrently produce polymerization and doping.

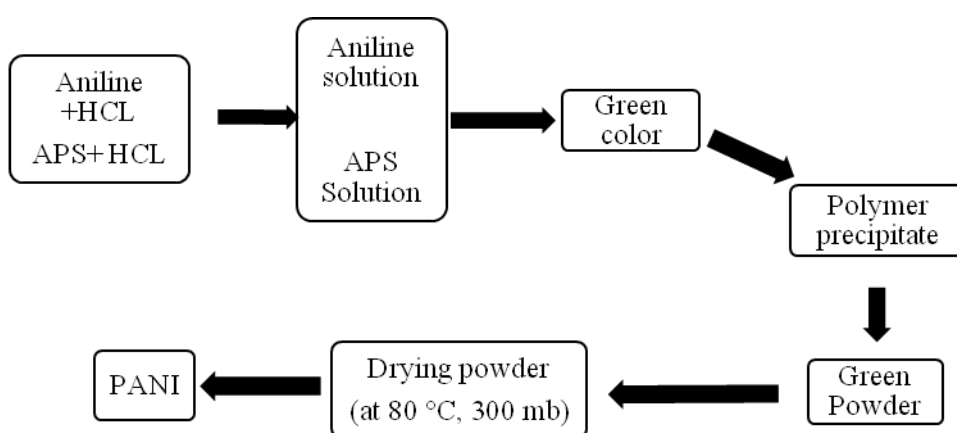


Figure 1.2. Preparation of PANI

Chemical Preparation (Oxidative Polymerization)

PANI is produced by chemical oxidation polymerization utilising ammonium persulfate (APS) as an oxidant and hydrochloric acid (HCl) or sulfuric acid (H₂SO₄) as a dopant in an aqueous environment. At this point, the oxidant can take a proton from the aniline monomer without forming a heavy bond or using the absolute product. At this point, the oxidant can take a proton from the aniline monomer without forming a heavy bond or with the absolute product.

Three PANI reactants are needed for chemical synthesis: aniline, an oxidant, and an acidic media. While ammonium sulphate ((NH₄)₂S₂O₈), hydrogen peroxide (H₂O₂), sodium vanadate (NaVO₃), cerium sulphate (Ce(SO₄)₂), potassium dichromate (K₂Cr₂O₇), potassium iodate (KIO₃), and potassium free cyanide (K₃ (iron (CN)₆)) may all be utilised as oxidizers in the manufacture of PANI, HCl and The most common way to make PANI is by oxidant polymerization, when aniline interacts with an acidic material to serve as a neutralizer before polymerizing when a drop of an oxidising agent, such as ammonium persulfate (APS), is

added at different temperatures. After realisation, an aqueous solution that had been filtered served as the mechanism for the 3-hour polymerization process. Rinsing the aforementioned solution five to six times with deionized water will provide pure PANI. The filter is subsequently made colourless by the addition of alcohol and acetone, which also guarantees that all n\|-reactive substances have been eliminated. The substance, termed as PANI polymeraldine salt, is slime green and unstable since swords are present in it. Thus, the polymeraldine salt transforms into the naturally stable at room temperature PANI-EB structure, allowing this precipitate to equilibrate with the proper quantity of NH₄OH [32]. Chemical polymerization is used to create camphor sulfonic acid (CSA), which is then rotated with a spin coater and doped with thin PANI films on plain glass [33]. Due to the organic solvent and undesirable organic matter, the thin film adheres to the glass very well. However, the FTIR data revealed the presence of a dopant and a change in their molecular structure for the thin film due to an outstanding CSA extraction fraction (1:8). It has been demonstrated that the amorphous aspect of these pure films changes as the amount of CSA added to the crystalline structure increases. SEM reveals a transition from smooth to rough morphology, as seen in the root of PANI thin films [33].

RESULT AND CONCLUSION

Characterization

Using scanning electron microscope (SEM, SIRION-100), morphology and particle size of synthetic m-\|gnetic (Fe₃O₄) nanoparticles were examined (SEM, SIRION-100), TEM an H-7100 electron microscope, Hitachi) and Almost 150 particles in Scanning Electron Microscope image were selected for determination of their particle size and distribution and it was done by s/tatistical analysis of at least 150 nanoparticles visible in Scanning Electron Microscope image. Chemical structure of samples was analysed by using Fourier-transform infrared spectra (FTIR, Nicolet 6700, Thermo Scientific, Boston, MA, USA). The Brunauer–Emmett–Teller (BET) was applied for specific surface area measurement. The Pb (II) ion concentration in saline water was measured by using UV–spectrophotometry (UV-2600) using xylenol orange as the complexing agent [34].

Adsorption Experiments

Using a batch method of experiment, the amount of arsenic (III) removal was investigated. Adsorbent and a set volume of arsenic (III) solution were combined in a 250 ml stopper glass container. Stoppers were employed to prevent concentration changes brought on by evaporation. Until equilibrium was reached, the mixture was shaken mechanically at a speed of 400 rpm in an incubator by Remi Equipment, Nagpur, India. The aqueous samples in each bottle were decanted after the predetermined contact time, centrifuged for five minutes at 4500 rpm, and then filtered through Whatman-42 filter paper (pore size: 2.5 mm). The arsenic concentration in the filtrate was then determined using an atomic adsorption spectrophotometer (AAS). Variable dosage (0.1-1.5 g/100 mL), pH (1-12), contact period (10-140 min), temperature (20-80 1C), and beginning concentration were used in the tests

(10–270 mg L⁻¹). The following equation was used to determine the arsenic absorption by the adsorbing medium when the best experimental circumstances were identified:

$$Q_e = (C_0 - C_e) V/W \dots \dots \dots (1)$$

where V is the volume of the solution, and Q_e is the equilibrium uptake (mg g⁻¹) (L). The starting concentration of arsenic (III) is C₀ (mg L⁻¹), the equilibrium concentration of arsenic (III) is C_e (mg L⁻¹), and the mass of the adsorbent material is W. (g). Anions were used in research to determine the impact of competing ions on the adsorption of arsenic (III) (bicarbonate, carbonate, sulphate and nitrate). These anions' sodium and potassium salts were used to create solutions of the anions. While the initial concentration of the other anions ranged from 50 mg L⁻¹ to 150 mg L⁻¹, arsenic (III) had a consistent beginning concentration of 50 mg L⁻¹. Software from Origin pro (version 9, origin lab corporation) and MS-Excel (version 2019, Microsoft 2019) were used to create the best fit models and analyse errors in the current experiments. Error bar plot, regression correlation coefficient (R²), standard deviation (SD), and chi-square analysis (2) were used to exclude the best fit [35].

Effect of adsorbent dose, pH, and initial arsenic concentration on the removal of arsenic (III)

The effect of adsorbent dose on removal of arsenic (III) was studied at pH 5, stirring speed of 25 r min⁻¹ at temperature 25 °C and represented in Fig. 1.3. As expected the removal of arsenic (III) increases from “49% to 97.62%, 45.41% to 95.81% and 43.50% to 93.99%” with increase in adsorbent dose from 1.1 to 1.5 g/100 ml respectively at a selected contact time of 1 hour keeping initial solute concentration at 50 mg L⁻¹, 100 mg L⁻¹ and 150 mg L⁻¹. It is found that after dosage of 10 g L⁻¹, there is no significant change in percentage removal of arsenic. The increase in percentage removal could be attributed to the availability of a greater number of adsorption sites at the solid phase. Availability of specific surface area and micropore volume plays a vital role for surface adsorption process [36].

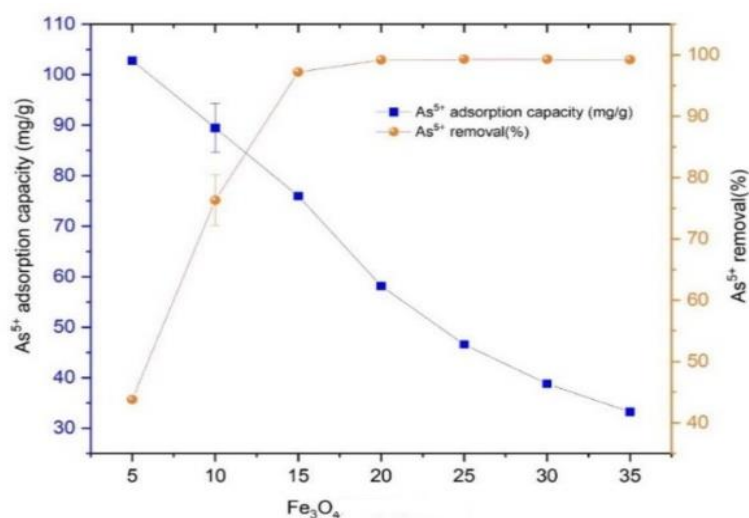


Figure: 1.3. The effect of adsorption dose

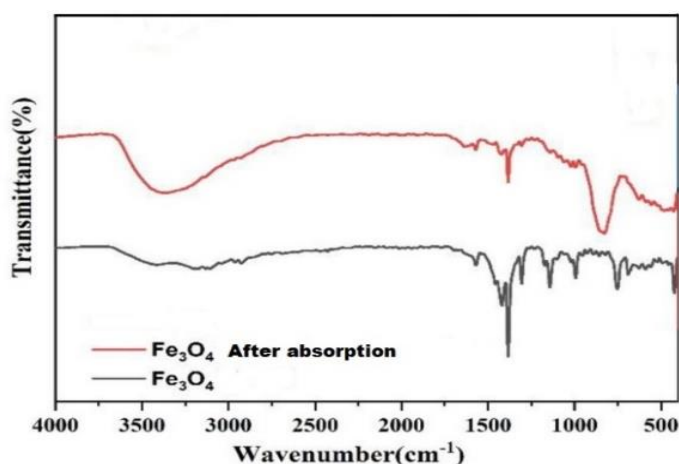


Figure 1.4. FTIR patterns of synthesised Fe₃O₄ before and after adsorption

However, in the current experiment, the removal extent remained nearly constant with additional increases in adsorbent dose, showing the saturation of adsorption sites. The saturation of the active sites may also be due to the overlapping of active sites at higher dosage as well as the decrease in the effective surface area resulting in the accumulation of exchanger particles. So, 13 g L⁻¹ is considered as the optimum dose and is used for further study. Percentage removal of arsenic at different pH (1–12) was studied using 13 g L⁻¹ of adsorbent dose at room temperature (26 °C), and contact time of 60 min for initial arsenic (III) concentration of 10 mg L⁻¹, 50 mg L⁻¹, and 100 mg L⁻¹. The results are presented in figure 4.3. After the adsorbent is added, the pH of the solution does not change; it stays at pH 7. The graph clearly shows that at pH 5, the removal is maximum at "97.62% for 50 mg L⁻¹, 95.81% for 100 mg L⁻¹, and 93.99% for 150 mg L⁻¹, respectively." The following factors might be to blame for the reduction in arsenic removal with a rise in pH. (1) When the concentration of As ions is more in water solution, then the magnitude of positive charge over amino group is more and (2) competition for active sites by excessive amount of hydroxyl ions present in the water in alkaline pH, the above attained pH indicates a good agreement with zero-point surface charge (pH_{zpc}) study [37].

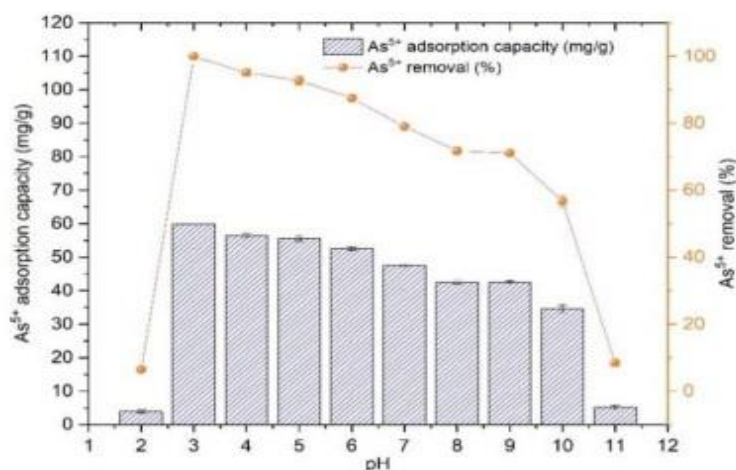


Figure: 1.5. Adsorption effect of pH

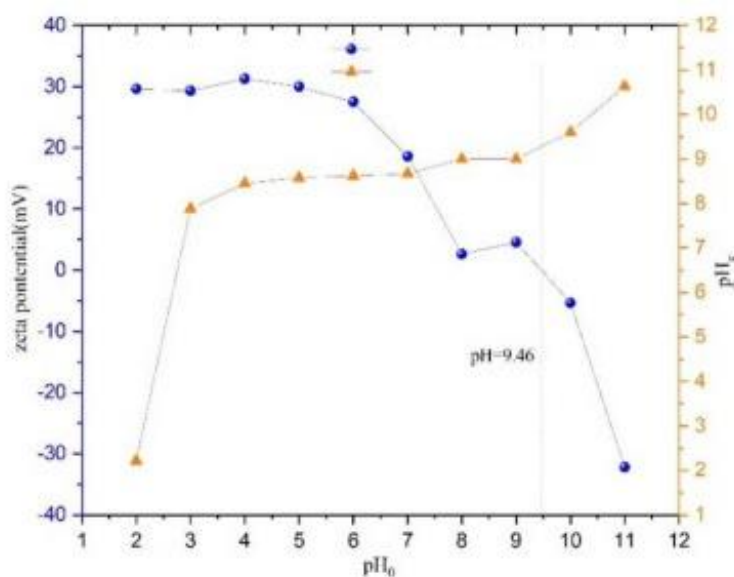


Figure 1.6. Isoelectric effect of PANI/Fe₃O₄

Contact time and adsorption kinetic studies

The effect of contact time on the removal of arsenic (III) was studied in the range of 10 min to 140 min by using 13 g L⁻¹ of adsorbent at room temperature (26 °C), at pH 5. From the Figure 4.8, it is understood that the effective removal achieved within 120 min, which is “97.62%, 95.81% and 93.99%” for initial concentration of 50 mg L⁻¹, 100 mg L⁻¹ and 150 mg L⁻¹ respectively. Since there is no change in the removal % after 120 minutes, 120 minutes was taken into consideration as the actual adsorption equilibrium period. Because there are more open adsorption sites in the adsorbent at first and there is a larger gradient of solute concentration, the rate of removal is initially higher. Several kinetic models, including the Lagergren first order rate equation and second order rate equation, are used to test the experimental data using initial concentrations of 50 mg L⁻¹, 100 mg L⁻¹, and 150 mg L⁻¹ in order to look at the controlling mechanisms of the adsorption process, such as mass transfer and chemical reaction. Below is a representation of the integrated linear pseudo-first order rate equation and second order rate equation [38, 39, 40].

Table 1.1. Kinetic parameters of As (III) adsorption at 25 °C.

Polyaniline magnetic nanocomposite Fe ₃ O ₄	Pseudo first order equation			Pseudo second order equation		
	K ₁	R ²	q _e	K ₂	q _e	R ²
50 mg L ⁻¹	0.03	0.98	1.99	0.99	1.99	1.33
100 mg L ⁻¹	0.04	0.95	9.11	0.10	9.11	1.20
150 mg L ⁻¹	0.05	0.94	17.34	0.06	17.34	1.32

$$\text{Log}(q - q_t) = \text{Log} q - K_1 \{t/2.203\} \dots \dots \dots (2)$$

$$t/q_t = [1/(K_2 q_c^2) + 1/q_c]t \dots \dots \dots (3)$$

Where q_e and q_t (both in mg g^{-1}) are the amount of arsenic (III) adsorbed at equilibrium and at time “ t ” respectively, K_1 is adsorption rate constant of pseudo-first order adsorption, K_2 is the rate constant of second order rate equation ($\text{g mg}^{-1} \text{min}^{-1}$), and q_c is the maximum adsorption capacity (mg g^{-1}) for the second order adsorption. The values obtained from the plots were represented in Table 1.1.

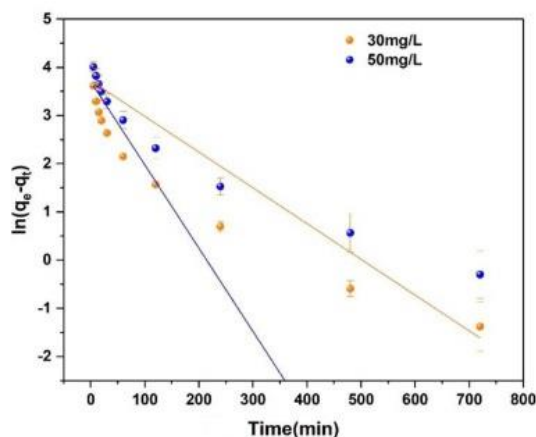
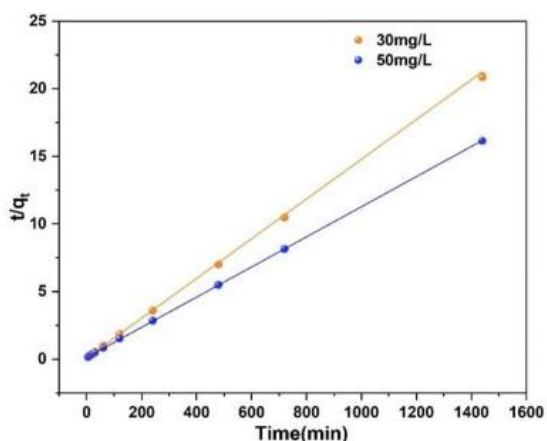


Figure 1.7. Pseudo-First order model Figure 1.8. Pseudo-Second order model

CONCLUSION

The arsenic contamination in all ecosystem has become a major environmental problem which results in adverse environmental impacts as well as cause serious threat to plants, animals and human beings. Accordingly, there is need to understand the chemistry, toxicology, contamination and to research on removal technologies of arsenic from different mediums.

According to literature review the arsenic is a naturally occurring element of the earth's crust and is found in large quantities in the air, water, and land. It is exceedingly toxic in its inorganic form. People are exposed to large amounts of inorganic arsenic by smoking cigarettes, eating contaminated food, drinking polluted water, and utilising contaminated water for crop irrigation and food preparation. Long-term exposure to inorganic arsenic, often through food and water consumption, can cause chronic arsenic poisoning. Skin cancer and skin lesions are the most recognisable outcomes. Individuals, population groupings, and geographical regions all experience long-term high inorganic arsenic exposure differently in terms of symptoms and indications. Thus, the sickness brought on by arsenic lacks a common definition. This makes it more difficult to determine how harmful arsenic is to human health.

The analysis examined the six main remediation procedures currently in use to remove arsenic from water: oxidation; coagulation; precipitation; and filtration. Adsorption,

membrane technology, bio-remediation, and ion exchange round out the list. Some techniques and technology for clean-up combined these kinds.

In this research work extensive study on arsenic problems and analysis of removal of arsenic from saline water using polyaniline magnetic nanocomposite/Fe₃O₄ has been carried out.

The polyaniline magnetic nanocomposite/Fe₃O₄ were created. Briefly, deionized water was used as the solvent to dissolve ferrous chloride tetrahydrate (FeCl₂.4H₂O) and ferric chloride hexahydrate (FeCl₃.6H₂O). With a molar ratio of Fe³⁺/ Fe²⁺=2, the solution of FeCl₃ and FeCl₂ was combined. Co-precipitation happened after adding ammonium hydroxide (NH₄OH) as a precipitating agent to the solution while it was being vigorously stirred.

Polyaniline magnetic nanocomposite/Fe₃O₄ can be potential adsorbents for the removal of Arsenic from water due to their great capacity for adsorption and simplicity in separating from water in the presence of a weak external magnetic field. Comparing the magnetic properties of Fe₃O₄ and polyaniline doped Fe₃O₄ nanoparticles was found to be more effective.

The second-order kinetic equation and the Langmuir isotherm model both provide excellent explanations of the mechanism of arsenic adsorption on the Fe₃O₄. The largest amount of arsenic that could be adsorb by the Fe₃O₄ at pH 3 and 298 K was 115.113 mg/g. The following reasons could explain why the elimination of arsenic declines when pH rises. (1) When the concentration of hydroxyl ions in a solution of water increases, the magnitude of the positive charge over the amino group increases, and (2) An excess of hydroxyl ions present in the water at an alkaline pH competes for active sites; the aforementioned pH indicates a good agreement with the zero-point surface charge (pHzpc) study. It has been demonstrated through the investigation of thermodynamic parameters that the process of arsenic adsorption by the polyaniline magnetic nanocomposite/Fe₃O₄ is a spontaneous endothermic reaction. Because of its excellent adsorption capability, good water stability, and ease of preparation, the polyaniline magnetic nanocomposite/Fe₃O₄ has a wide range of potential applications for eliminating As (III) from waters.

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