Preparation and characterization of ferrihydrite: application in arsenic removalfrom aqueous solutions

ABSTRACT

Arsenic pollution is a public health hazard in Burkina Faso due to its impact on human health and water resources. To mitigate this pollution, ferrihydrite material has been synthetized and characterized to be used as adsorbent for arsenic removal in aqueous solutions. This study aims to contribute to the improvement of access to clean drinking water by removing arsenic from water using ferrihydrite. Arsenic removal experiments were carried out using batch mode and aqueous solutions. The characterization of ferrihydriteusing Scanning Electron Microscopy (SEM) coupled with Energy Dispersive Spectroscopy (EDX), X-ray Diffraction (XRD), Infrared (IR), and Brunauer Emmett Teller (BET) showedthat an amorphous and microporous ferrihydrite«2»line. The total specific surface area and pH of the charge were 184.518 m²/g and 9.41, respectively. The optimal adsorbent doses were 4g/L forAs(V) and 8g/L withAs(III). The optimum pH range for the adsorption of As (V) and As (III) was between 2 and 10. The adsorption capacity ranged from 2.0 to 15.07 mg/g for As (V) and from 2.0 to 13.01 mg/g for As (III) with increasing concentration between 2 and16 mg/L. Equilibrium time for As (V) and As (III) on ferrihydrite was found to be 720 min and 960 minwith anadsorption capacities of 623.75 and 618.75 µg/g, respectively. The adsorption of As(V) and As(III) was consistent with the Langmuir monolayer model on ferrihydrite. A very favourable spontaneous chemical reaction was observed.Arsenic removal followed the pseudo-second order kinetic model and occurred on a monolayer.

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Comment [RC2]: “To improve”

Comment [RC3]: Please indicate from here on the use of As(III) and As(V) solutions for the removal experiments.

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Comment [RC5]: Do the authors mean “point of zero charge” instead fo “pH of the charge”? Please revise and clarify the nomenclature of this parameter.

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Comment [RC7]: It would be better to report only the maximum adsorption capacicities for As(V) and As(III).

Comment [RC8]: Please remove the adsorption capacities obtained from kinetic experiments.

Keywords*:adsorption, arsenic, characterization,ferrihydrite, removal.*

1. INTRODUCTION

In different forms, arsenic compounds are known for their high toxicity on living organisms, mainly humans and animals [1, 2]. Due to the human activities, the increase in global mining and mineral processing processes has contributed to the accumulation of arsenic in waste rock and mine drainage water [2,3,4]. The transfer of arsenic with other heavy metals in soil and water resources has been observed in many regions of the world [5,6,7]. Arsenic (III) is less common in water but more toxic than arsenic (V) [2,5]. In addition to its role as a trace element in life, arsenic exposure through daily consumption of arsenic water is responsible for many diseases (cancers, respiratory problems, etc.)

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worldwide [8, 9, 10]. In Burkina Faso, arsenic contamination has been noted in several regions due to the consumption of water with concentrations higher than the standard of 10 µg/L according to the World Health Organization (WHO) [10,11]. Boreholes and water sources built in rural areas by the state and partners have been closed for contamination reasons linked to arsenic [10,11]. Arsenic removal from water at a rate of less than 10 µg/L (WHO standard) is becoming a global necessity for the supply of drinking water in developing countries [2,9].To reduce the harmful effects of arsenic compounds, treatment techniques such as filtration, coagulation-precipitation, reverse osmosis, electrodialysis, adsorption, and coupled techniques have been developed [12,13,14]. Among these techniques, adsorption has been studied in recent years owing to its effectiveness, economy, low cost, and environmental friendliness [14,15]. In the implementation of adsorption technique, the adsorbent material must be effective, selective, and have a high specific surface area to accumulate different forms of arsenic [15,16]. Adsorbents such as activated carbon, clays, laterites, and metal oxy-hydroxides have been tested for the removal of As (III) and As (V) in water[15,16,17]. Literature indicated that adsorbents based on iron oxides and hydroxides are effective for the adsorption of different forms of arsenic because of their amphoteric character [18,19,20].

In this work, ferrihydrite has been used as adsorbent for arsenic removal. Specifically, ferrihydrite has been prepared and characterized using analytical methods. In addition, arsenic removal capacity of percentage and removal mechanism have been investigated under various operating conditions.

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2. MATERIALS AND METHODS

2.1 Preparation of ferrihydrite

To prepare theferrihydrite granular, the method described by Scwertmann*et al.*[21]were used:100 mL of a 0.25M ferric nitrate monohydrate solution (purity ≥ 98%) (Sigma-Aldrich) has been prepared with distilled water at initial pH of 2.08. The granular ferrihydrite was stored in an enclosure at a temperature between 19 and 21 °C.

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2.2 Physico-chemical characterization of ferrihydrite powder

2.2.1 Physical characterization

The moisture content (TH) of the ferrihydrite was determined rightafter centrifugation of the paste to evaluate the quantity of free water it contained before drying. 50 g (mass m_1) of moisture ferrihydrite was placed in an oven at 110°C for 72h to constant mass denoted m_2.The moisture content (TH) of the ferrihydrite has been calculatedaccording to the following relationship [19]:

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$$\boldsymbol{TH}(\%) = \left(\frac{\boldsymbol{m_1 - m_2}}{\boldsymbol{m_1}}\right) \times \mathbf{100} \qquad (\mathbf{1})$$

The bulk density (d) of the ferrihydrite was determined using a method described elsewhere [16]. Equation 2 was used to determine the bulk density of ferrihydrite:

$$\boldsymbol{d} = \frac{(\boldsymbol{m_1 - m_0})}{\boldsymbol{V}} \qquad (\mathbf{2})$$

The pH at the point of zero charge (pH_{PZC}) has been studied to understand the neutral surface charge of ferrihydrite [22]. The pH_{PZC} value was determined from the curve (initial pH – final pH) as a function of the initial pH intercepting the x-axis of pHi – pHf = 0 [23]. The surface area and the pore dimensions of the ferrihydrite were analyzed using Micromeritic (TriStar II plus version 3.02) controlled with MicroActivesoftware. Before nitrogen (N2) adsorption at 77.350 K, the ferrihydrite was dried and dispersed in a glass quantrachrome cell at 105 °C for 24 h to remove the adsorbed water and gas. The surface area was determined using the Brunauer, Emmett, and Teller (BET) method and the one of Langmuir[22,24]. The nitrogen adsorption and desorption isotherms obtained by Barrett, Joyner, and Halenda (BJH) methodwere used to determine the volume and radius of pore.

2.2.2 Chemical characterization

Elemental chemical analysis of the ferrihydrite was carried out using a Microwave Plasma Atomic Emission Spectrometer (MP-AES, Agilent 4200) controlled by Spectra software. The powder was mineralized by the «eau regale method» according to the following protocol: 10 g of the dry powder was weighed into a 100 ml gauge flask. Subsequently, 15 mL of hydrochloric acid (37%, Honeywell) and 5 mL of nitric acid (68%, Flucka) of analytical grade (AR) were added and heated to a temperature of 200±5 °C on a hot plate for one hour. After cooling, the flask was completed up to the gauge mark, homogenized, and used for the determination of chemical elements by MP-AES previously calibrated with standard solution [24]. The Fourier Transform -infrared (FT-IR) spectrum was recorded in the range of 500 to 4000 cm^{-1} with a resolution of 4 cm^{-1} using an OUAFO device (controlled by OPUS software) to evaluate the surface chemical functions. The mineral phase of the ferrihydrite was studied using a Shimadzu diffractometer equipped with a copper tube and graphite monochromator. The diffractogram was collected in an angular range of 2Ɵ from 2° to 90° at a speed of 2° min^{-1} under a voltage of 40 kV and intensity of 30 mA [23]. The morphology and surface composition of the ferrihydrite have been observed using a Microspec-WDX 600/OXFORD brand device coupled to energy dispersive spectroscopy (EDX) [19,25].

Comment [RC18]: Is it a SEM device? Please indicate it on the manuscript.

2.3 Preparation of arsenic solutions

A stock solution of As (III) (1000 ppm) was prepared by dissolving a sodium arsenite salt ($NaAsO_2$) in a solution of 20% NaOH (Flucka) [20]. The As (V) solutions of were prepared from a stock solution of 1000 ppm ($Na_2HAsO_4·7H_2O$, Merck) by dilution with ultrapure distilled water. Reagents such as NaOH (Flucka) and HNO_3 (Sigma-Aldrich) were of analytical grade (AR) and were used to adjust the pH of the matrix solutions. The pH of As(III) and As (V) solutions was adjusted to Be between 7 and 8 with a branded portable pH meter (HANNA, waterproof HI98318).

2.4. Arsenic removal experiments

Batch experiments were conducted to remove As(III) and As(V) from aqueous solutions. 25 mL solution of As (III) or As (V) ata concentration of 5 mg/L was added to 1.0 g of ferrihydrite in test tubes and initial pH was adjusted between 2 and 12. Absorbent dose was varied over a range of 4 g/L to 14 g/L using initial arsenic concentration of 5 mg/L. The effect of initial arsenic concentration was then evaluated using concentrationranging from 1 to 16 mg/L.Similar experiments were carried out to assess adsorption kinetics by testing from 01 h to 24 h with a 10g/L dose of adsorbent at an arsenic concentration of 5 mg/L. All experiments were running for 24 h at room temperature in the laboratory 24 ± 0.15°C. Residual arsenic concentration has been analyzed with a Microwave Plasma Atomic Emission Spectrometer (MP-AES Agilent 4200) after filtration. The evaluation of the adsorption efficiency using ferrihydrite was carried out using the percentage of removal denotedAs (%) and the adsorption capacity denoted Qe (mg/g). The arsenic removal was calculated using the following relation:

$$As(\%) = \frac{C_i - C_e}{C_i} \times 100 \qquad (4)$$

The adsorption capacity of ferrihydrite was determined by the following formula:

$$Q_e(mg/g) = \frac{(C_i - C_e) \times V}{m} \qquad (5)$$

Where Ci and Ce (mg/L) represent the initial and equilibrium concentrations of As (III)or As (V), respectively,m (g) and V (L) are the mass of the ferrihydrite and the volume of solution, respectively.

3. RESULTS AND DISCUSSION

3.1. Characteristics of ferrihydrite

Figure 1 shows a moist red-brown chocolate cake batter (fig. 1A) and brown-colored ferrihydrite powder (fig. 1B). The mesh size of the ferrihydrite grains used in this work is less than or equal to 75 µm. The appearance and texture of the ferrihydrite powder obtained were similar to those reported by Otgon *etal.*[18]at pH (3-4) .

Figure 1: Images of the prepared ferrihydrite

Physicochemical characteristics of the ferrihydrite are listed in Table 1.In this Table, we noticed that ferrihydrite has a large positive surface area relative to its basic pH at zero point (pH_{PZC}). In addition, this material contains a very high moisture content.Ferrihydrite's positively charged surface is therefore capable of adsorbing anions such as arsenic species[23]. The bulk density, which is almost half the density of pure iron (d = 7.874 g/cm^3), indicates the predominance of iron in the ferrihydrite [19]. The specific surface area of the ferrihydrite powder (184.518 m^2/g) is lower than the value reported by Milton *et al.* [26]. The higher Langmuir surface area than the other surfaces recorded in

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Table 1 indicates that monolayer adsorption may control the removal of As (V) or As (III) from ferrihydrite [15,19]. The quantitative analysis of ferrihydrite showed the high content of iron (1257 mg/L) confirming its nature of iron oxyhydroxide.

Table 1 : Physicochemical characteristics of ferrihydrite powder

Physical parameter	**Quantitative value**
Particle size (µm)	≤ 75
Moisture content TH (%)	69.00
Density (d)	4.07
Zero charge pH pHCN	9.41
BET specific surface area (m^2/g)	59.986
Langmuir specific surface area (m^2/g)	73.315
External specific surface area (m^2/g)	51.216
Total specific surface area (SS) (m^2/g)	184.518
Total pore volume (cm^3/g)	0.052
Quantitative analysis of ferrihydrite	
Fe(mg/L)	1257.010
Al(mg/L)	*
Cu(mg/L)	*
Pb(mg/L)	*
Si(mg/L)	*
Ti(mg/L)	0.019
Na(mg/L)	2.310
Zn(mg/L)	0.007

*Not determined

The IR spectrum in Figure 2 shows the characteristic bands of the ferrihydrite. The different vibration bands were assigned according to the literature [19, 26, 27]. The bands around 3400 cm^{-1} correspond to the stretching vibrations of the O-H bonds in the surface hydroxyl groups and water molecules. The wave numbers around 1630, 1400, and 900 cm^{-1} would correspond to adsorbed water molecules, deformation of Fe-O bonds, and deformation of Fe-OH, respectively. Milton*et al.*[26] showed that the characteristic bands of ferrihydrite are mainly due to the hydroxyl O-H groups, the Fe-O and Fe-OH function of the structure. The relatively weak intensity of the stretching bands of the O-H bonds in the hydroxyl groups can be explained by the metastable nature of ferrihydrite [28].

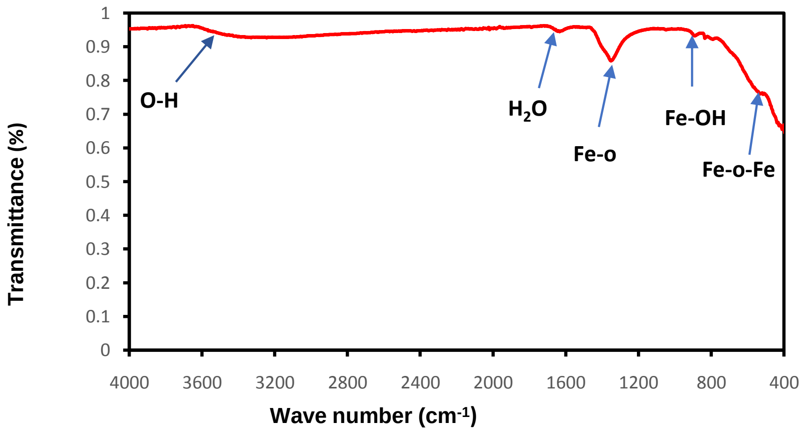
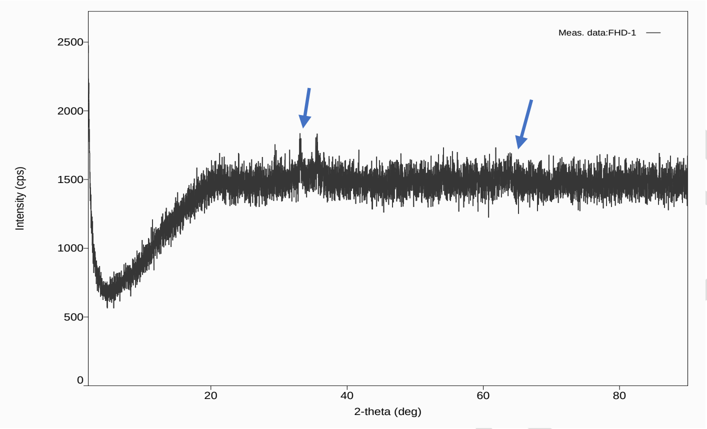
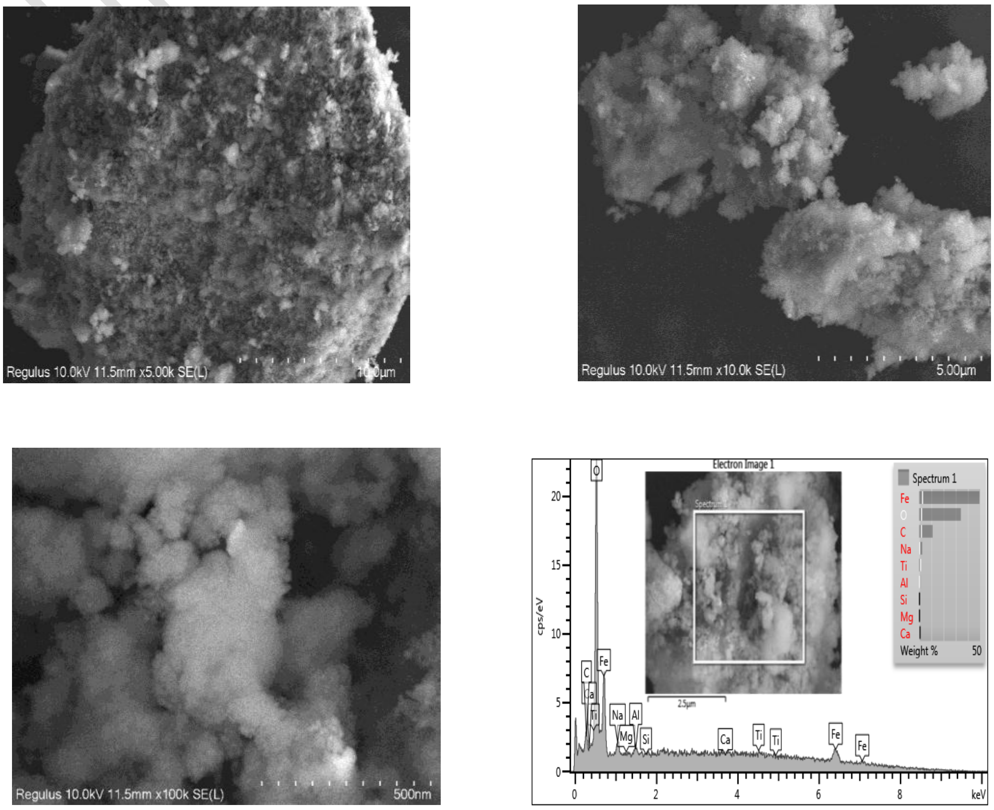


Figure 2: FT - IR spectrum of ferrihydrite powder

The X-Ray diffraction (XRD) pattern of the ferrihydrite is shown in Figure 3.It highlighted the presence of two broad lines at 2θ values close to 35° and 67°. According to the literature[18,28], this diffraction pattern can be attributed to "2-line" ferrihydrite rather than "6-line" ferrihydrite, whose diffraction pattern shows 6 lines.Background noise with relatively low peak intensities is observed in the diffraction pattern, indicating that the synthesized ferrihydrite is amorphous format. Otgon *et al.*[18] showed that the intensity of the peaks depended on the morphology and crystallinity of ferrihydrite. The ferrihydrite prepared could therefore be an amorphous 2-line ferrihydrite.



The images in Figure 4 show the surface morphology of ferrihydrite powder particles at different magnification scales. Magnification A (5X) shows a cluster of aggregates formed by small particles. Whereas magnification C (100 X) shows whitish irregular spherical shapes with relatively close pores indicating a larger specific surface area with small pore volumes [21,26]. These irregularly distributed whitish particles indicate the very amorphous character of the ferrihydrite powder revealed by the diffractogram. In their studies on the removal of As (V) by iron-doped activated carbon, Sanou *et al.* [19] showed that the whitish particles were due to the presence of iron and its magnetic nature. Spot chemical analysis of the surface (figure 4D: spectrum EDX) revealed that the ferrihydrite wasmainly composed of iron (49.54%) and oxygen (34.18%). The other trace chemical elements are carbon (11.08%), sodium (2.50%), titanium (1.53%) and aluminium (1.05%).



3.2. Influence of operating parameters on arsenic removal

3.2.1. Effect of initial pH

Figure 5 shows the adsorption of As(III) and As(V) over the pH range of 2–12. As(III) and As(V) was 100% removed until pH $_{ZPC}$= 9.41. The elimination rate decreased slightly in the pH range 10–12 for As (III) and As (V). The efficiency of the adsorption of different ionic and neutral forms of arsenic, such as $H_3A_SO_4$, $H_2A_SO_4$-, $HA_SO_4^{2-}$ and $H_3A_SO_3$ on the surfaces of ferrihydrite at a pH lower than 9.41 could be explained by a process of attraction due to the positive charge of the ferrihydrite surface. However, beyond pH $_{PZC}$ = 9.41, the reduction in elimination could be explained by the repulsion of the ionic forms of arsenic (H_3AsO_3, $H_2AsO_3^-$, AsO_3^{3-}, AsO_4^{3-}) by the negatively charged surface [23,24]. The adsorption of neutral forms (H_3AsO_4 and H_3AsO_3) in the pH range ≤ pH_{PZC} would be an ionization of the arsenate and arsenite forms on ferrihydrite followed by a ligand exchange mechanism [23]. The average equilibrium pH of the final pH value was 7.29. Thus, the mechanism for eliminating the forms of arsenic on the ferrihydrite surface would be bidentate or monodentate [18,27]. The optimal pH range is 2–10, which corresponds to that of Qi *et al.* [29].

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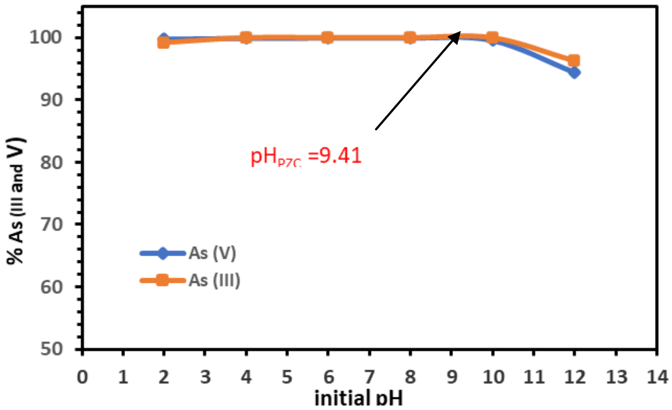

3.2.2. Effect of Adsorbent amount

The influence of the mass of ferrihydrite was evaluated for adsorbent doses ranging from 4 to 40 g/L with an initial concentration of 5 mg/L of As (V) and As (III) at pH 7.45 for 24 h. Figure 6shows an increase in the arsenic removal from 99.64 to 100% for As (V), and from 87 to 100% for As (III). The optimal adsorbent doses under these conditions were 4 g/L for As (V) and 8 g/L for As (III) on the ferrihydrite. The adsorbent dose for As (III) was double that for As (V) because of the oxidation of As (III) to As (V) before its fixation on the surface of ferrihydrite [25,29]. The increase in the removal rate was due to the increase in the number of active sites on ferrihydrite. The adsorption capacity of ferrihydrite depends on the number of active sites and specific surface area [29].

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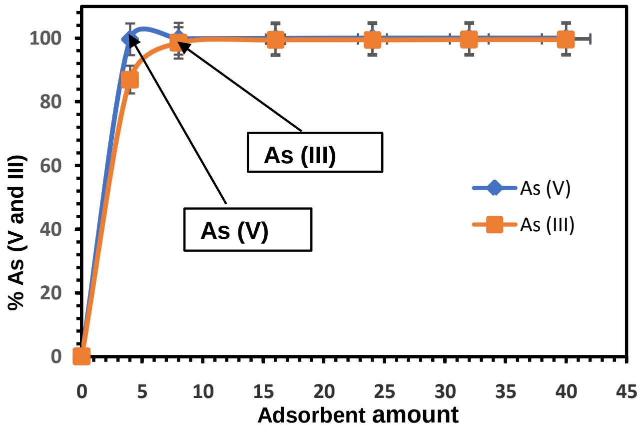

3.2.3. Effect of initial arsenic concentration

The behavior of the adsorption of As (V) and As (III) onto ferrihydrite was studied over a concentration range of 2–16 mg/L at pH (6.89) with a mass of1.0 gduring 24 h. Figure 7shows that the removal percentages of As(V) and As(III) on the ferrihydrite surface decreased with the increase of initial arsenic concentration. For As (V), the removal percentage decreased from100 to 93.95% while by

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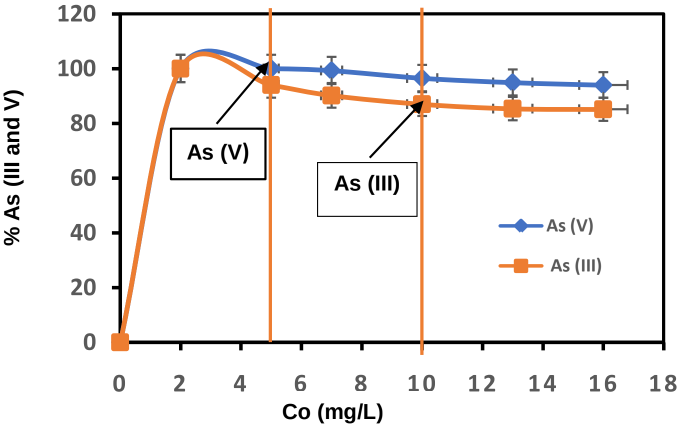
removing As (III), itdecreasedfrom 100 to 85.10%. The adsorption capacity of ferrihydrite increases from 2.0 to 15.07 mg/g for As (V) and from 2.0 to 13.01 mg/g for As (III). This increase in adsorption capacity could be explained by the increase in initial As (V) and As (III) ions fixed on the active sites of the ferrihydrite [20,30]. However, the decrease in arsenic removal percentage could be due to the limited number of arsenic ions adsorbed on the ferrihydrite at low concentrations [30,31].The optimal concentration of ferrihydrite was 5 mg/L for As (V) and 10 mg/L for As (III).

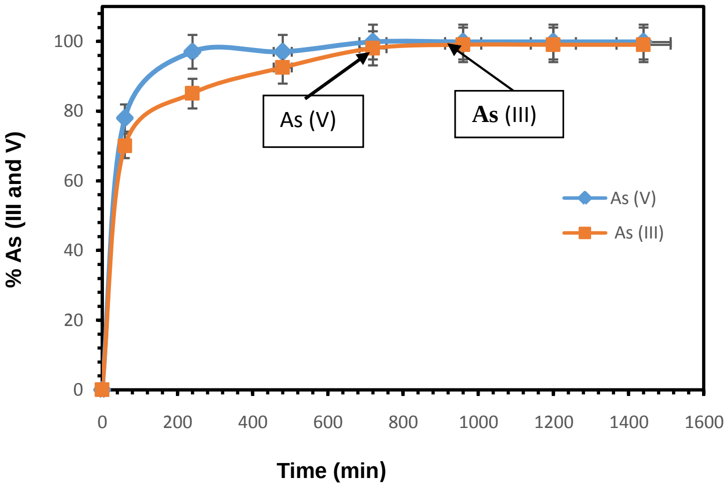


3.2.4. Effect of contact time

The effect of the contact time was studied from 1h to 24 h (60-1440 min) with a concentration of 5 mg/L and a mass of 10 mg as shown in Figure 8. The removal percentage as a function of the contact time showed an increase according two steps:

First step indicated an increase of arsenic (V) removal from 78 to 99.80% when the contact time was increased between 60 and 720 min.By removing As(III), it increased from 70 to 99.00% with an increase of contact time between 60 and 960 min.The second step indicated that arsenic removal remained constant after 720 min and 960 min for As(V) and As(III), respectively.

The equilibrium time for optimum removal of As (V) and As (III) on ferrihydrite was 720 min (12h) and 960 min (16h) corresponding to adsorption capacity of 623.75 and 618.75 µg/g respectively for As(V) and As(III). Adsorption of As (V) and As (III) onto ferrihydrite could be chemical reactions of the internal and external complexes on the functional groups of the adsorbent, as shown by the IR spectrum [20,27].



3.2.5. Modelling of isotherms

The adsorption behavior of As (V) and As (III) on ferrihydrite as a function of the initial concentration was modeled using the LangmuirandFreundlichequilibrium isotherms [15,31,32]. Langmuir isotherm model describes the monolayer adsorption with homogenous sites of adsorbent, while multilayer adsorption and heterogenous sites were described by Freundlich isotherm model.

The linearized form of the Langmuir isotherm model is given by the relation:

$$\frac{C_e}{Q_e} = \frac{1}{Q_m}(C_e) + \frac{1}{K_L Q_m} \quad (8)$$

The equation of the Freundlich model is given by the following relation:

$$LnQ_e = lnK_f + \frac{1}{n} lnC_e \qquad (9)$$

Figure 9 shows the plots Ce/Qe = f(Ce) and LnCe= f(Ce) asfunctions of the equilibrium concentration Ce for the Langmuir and Freundlich models. The slopes and intercept of the curves were used to calculate the different constants listed in Table 2.

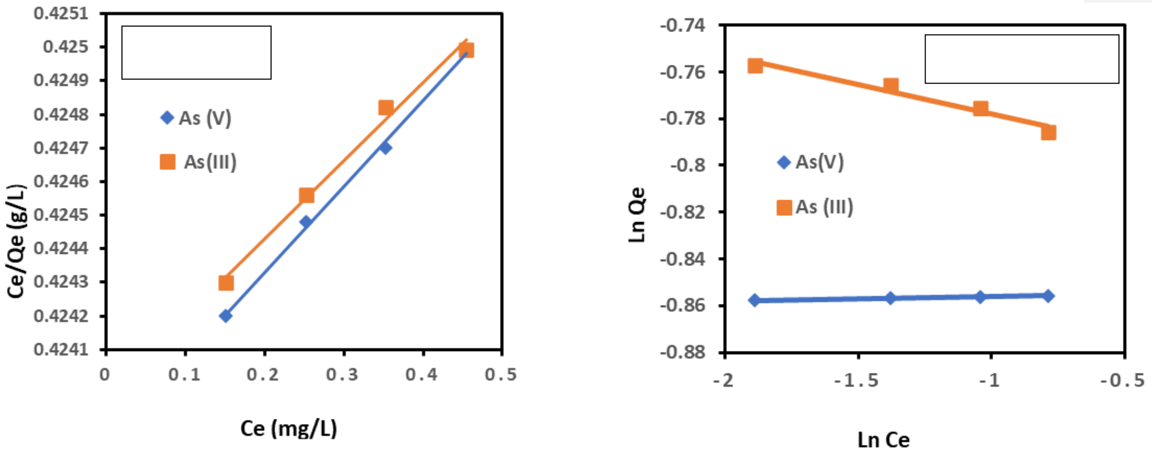


Figure 9: Representation of Langmuir and Freundlich isotherms for As (V) and As (III)

Table 2: Langmuir and Freundlich constants

Ferrihydrite	**Langmuir**			**Freundlich**	
	Q_m (mg/g)	K_L (L/mg)	R^2	K_f (mg/g)	nR^2
As (V)	12.01	1.73	0.98	10.31	30.97
As (III)	11.36	3.28	0.99	10.96	40.98

The values of the Langmuir and Freundlich constants R^2 between 0.98 and 0.99 show a good correlation with the experimental data. The adsorption capacity values for As (V) and As (III) were 12.01 and 11.36 mg/g respectively according to the Langmuir model and 10.31 and 10.96 mg/g respectively according to the Freundlich model (table 2). The removal of As (V) and As (III) better follows the monolayer chemisorption process of homogeneous active sites. The Freundlich affinity constants (n) of As (V) and As (III) are 2.78 and 3.86 respectively, and are all greater than 1, indicating very favourable spontaneous chemical reactions [19,32].

The equilibrium parameter R_L and the Gibbs free energy (ΔG) have been evaluated from the following relations:

ΔG_0 = -RT. Ln K_L(10)

$$R_L = \frac{1}{1 + K_L C_O} \qquad (11)$$

The calculated R_L values were between 0 and 1, indicating a favorable adsorption process for As (V) and As (III). The positive Gibbs free energies are 2.70 and 1.24 kJ/mole respectively for As(V) and As(III)indicated that the reactions on ferrihydrite are spontaneous process [19].

3.2.6. Kinetic modeling

To study the kinetic of arsenic adsorption onto ferrihydrite, the pseudo-first-order and pseudo-second-order model described by Lagergren and the one given by Ho and Mckay, respectively have been widelystudied[19,33].

The integration of the Lagergren'sequation (12) and the one of Ho and McKay (13) are given in the following formulas:

$\mathrm{Ln}\ (Q_e - Q_t) = \ln Qe - k_1 t$ (12)

$\frac{t}{Q_t} = \frac{1}{Q_e} t + \frac{1}{K_2 Qe^2}$ (13)

The representation of these equations is given in Figure 10.

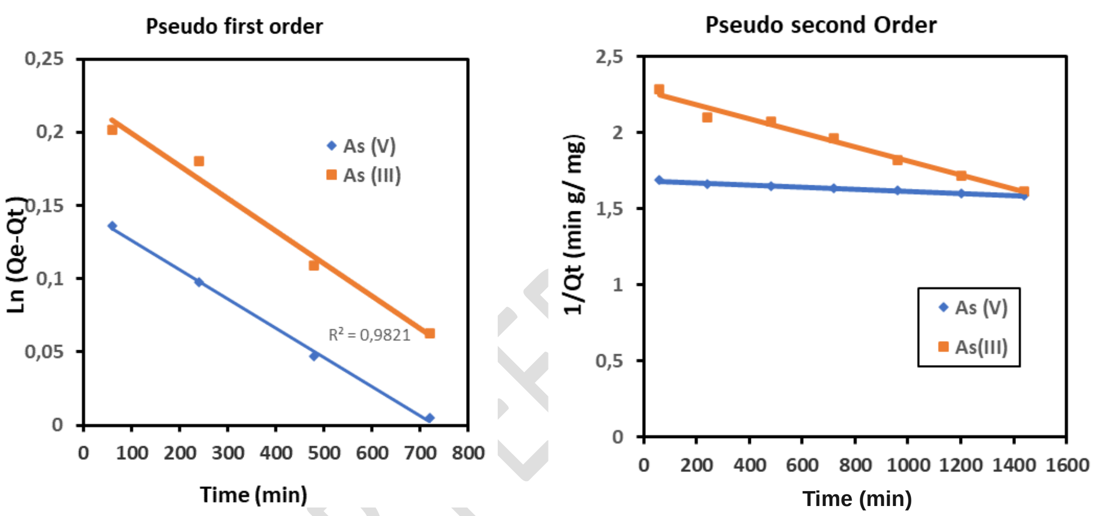


Figure 10: Representation of the kinetic models for the removal of As (V) and As (III) on ferrihydrite

Table 3: Kinetic constants in the removal of As (V) and As (III) on ferrihydrite

Ferrihydrite	**Pseudo-first-order**				**Pseudo-second-order**			
	Qe_{exp}	Qe_{theo}	K_1	R^2	Qe_{exp}	Qe_{theo}	K_2	R^2
	mg/g	**mg/g**	**min^{-1}**		**mg/g**	**mg/g**	**min^{-1}**	
As (V)	1.07	0.31	0.012	0.99	1.00	0.99	3.14	0.98
As (III)	0.99	0.29	0.010	0.98	0.98	1.01	3.78	0.97

The correlation coefficients (R^2) ranging from 0.97 to 0.99 for both models indicate that data are perfectly correlated between the variables. Using the pseudo-second kinetic model, the theoretical values of the adsorption capacity (Qetheo) are closeto the experimental values (Qe exp) indicating that arsenic removal followed the pseudo-second-order model. Consequently, chemisorption would controlthe adsorption process of As (V) and As (III) on the surface of ferrihydrite through an inner-sphere reaction [19,33].

Comment [RC23]: Is tehre any difference between As(V) ansd As(III) adsorption kinetics? Wich one si faster according to the kinetic constants?

4. CONCLUSION

This study enabled the successful preparation and characterization of ferrihydrite as adsorbent in arsenic removal. Characterization of the powder ferrihydriteindicatedanamorphous 2-line ferrihydrite with hydroxide and surface hydroxyl functionalities to give butter results. The efficiency of ferrihydrite in arsenic removal, mainly As(V) or As(III) species depended on the initial pH, adsorbent dose, initial concentration, and contact time. The mechanism of the removal of arsenic on ferrihydrite could be described by monolayer adsorption occurred following a spontaneous process. The removal of As (III) or As (V) has been occurred according the pseudo-second-order kinetic and chemisorption process.

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