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Activated carbon from bamboo waste modified with iron and its application in the study of the adsorption of arsenite and arsenate

Research Article

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Abstract: Activated carbon obtained from bamboo waste was synthesised and modified with iron (BAC-Fe) and used for the removal of arsenic from aqueous solutions. Two different adsorption models were used for analysing the data. The adsorption capacities were determined for BAC-arsenite, BAC-Fe-arsenite, BAC-arsenate and BAC-Fe-arsenate, with a qmax (μg g^{-1}) of 14.89, 19.19, 22.32 and 27.32 respectively. Adsorption capacity varied as a function of pH and modifications to the sorbent. Adsorption isotherms from an aqueous solution of arsenite and arsenates on activated carbons were determined. These adsorption isotherms were consistent with the Langmuir and Freundlich adsorption models. Adsorption kinetics followed a pseudo-first order rate equation, as did the kinetics for BAC-Fe-arsenite and BAC-Fe-arsenate adsorption.

Keywords: *Activated carbon • Bamboo waste • Isotherms • Freundlich • Langmuir*

1. Introduction

Bamboo is used for several purposes for both domestic and industrial uses. It is used for making handicrafts and furniture, building, decorating, paper making and as firewood in many parts of the developing world. However, its potential as an adsorbent has not received much attention. Environmental pollution problems caused by heavy metals cannot be overemphasised. Heavy metals are very toxic and pose a threat to humans and the environment. The United States Environmental Protection Agency has classified some heavy metals such as lead, cadmium and mercury as priority pollutants because of their toxicity [1,2]. The amount of these heavy metals in our environment has increased as a result of industrialisation. The anthropogenic sources of heavy metals include waste from the electroplating and metal finishing industries, metallurgical industries, tannery operations, chemical manufacturing, mine drainage, battery manufacturing, leachates from landfills and contaminated ground water from hazardous waste sites [3,4]. As a result of these, the need to remove and/or recover these heavy metals has increased. The removal and recovery of toxic and/or valuable metals from aqueous effluents have received much attention in recent years.

Arsenic is one of the most dangerous and harmful to ecosystems pollutants. This element is found in rivers and agricultural land. Contact and ingestion of this element is very harmful to human health, in different oxidation states and particularly in its oxidation states II and V. Arsenic is an element that attacks all sanguine system, and if ingested it generates very stable chemical complexes that are difficult to break putting at risk the lives of living beings and, therefore, the ecosystem in general [5-19].

Arsenic is additionally associated with other pollutants such as organic compounds generating a highly toxic liquor. In industrialized countries the permissible quantity of arsenic is controlled rigorously, but in developing

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countries it is not always the case. Thus, development of new technologies to remove these contaminants at low cost is very important [20,21].

The main sources of arsenic are contamination herbicides and fungicides used indiscriminately to protect their crops. Different solutions have been proposed for removing arsenic from industry waste water have provided to have a good future. However, the complexity of their applicability and high costs have prevented the widespread application. Furthermore, it had been shown that obtaining porous solids from waste materials is a promising methodology for the removal of metallic ions. Besides, agricultural waste can be used for the synthesis of porous solids, such as activated carbons. Additionally, these materials have been tested for the retention of complex and bulky dye molecules in textile industry. All of those reported findings assure that future research will use and modify this kind of materials for the adsorption of specific pollutants.

This study aimed to prepare activated carbons from bamboo waste furniture waste and to investigate their properties and adsorption capacities for arsenic in water treatment. A chemical activation process using NaOH as the activating agent prepared bamboo-derived activated carbon. Carbonisation was carried out at 950ºC under a nitrogen atmosphere. A comparative study of the adsorption capacity of As (III) and As (V) on activated carbon modified with iron has been performed in this study. Results are analyzed and compared with results obtained in the literature. The outcome of this research will analyze the possibility of applying the results to pilot plant level. In the case of Colombia, this applicability is a novel contribution of this research and provides a great social contribution.

2. Experimental procedure

2.1. Preparation of activated carbon

Bamboo waste discarded from production by the Colombian bamboo furniture industry was cut into pieces approximately 1 square inch in size. The pieces were washed with distilled water and dried at 105ºC for 12 h. These bamboo precursors were immersed in a sodium hydroxide (NaOH) solution (25% by volume). The weight ratio of bamboo precursors to NaOH was fixed at 2. Next, the mixture was dehydrated in an oven overnight at 105ºC. The dried bamboo waste/NaOH mixture then was put on metal mesh inside a stainless steel chamber placed and in an electrical furnace (Carbolite™) Nitrogen gas (150 mL min^{-1}) was fed into the chamber and heating was initiated to start the carbonisation process. The heating rate was 5ºC min^{-1} and continued until the final temperature of 950ºC was reached. Carbonisation was then maintained at 950ºC for 6 h. The activated carbon product was cooled to room temperature under a nitrogen atmosphere. To remove the remaining impurities such as ash, the synthesised activated carbon was washed with a hydrochloric acid (HCl) aqueous solution (10% by weight), followed by washing with distilled water several times until the pH of the washing solution was neutral. The HCl-treated activated carbon was then dried at 105ºC for 24 h and ground into 130-180 µm particles prior to characterisation and the adsorption study. Here, we designated activated carbon carbonised at 950ºC as BAC.

2.1.1 Fe modification

Twenty grams of adsorbent were placed in a 125 mL Nalgene bottle with 100 mL of 0.5 M $Fe(NO_3)_2$ solution prepared with N_2 purged distilled water (to remove oxygen present in the water, thus preventing the oxidation of ferrous sulphate to ferric sulphate). Here, we designated this activated carbon treated with an iron solution as BAC-Fe.

2.2. Characterisation of activated carbon

Proximate and ultimate analyses of the bamboo precursor were carried out using a thermogravimetric analyser (TGA, ASTM D5142-90) and a CHNO analyser (ASTM D5373-93), respectively. The heating value was determined using a bomb calorimeter. The physical properties (BET surface area and pore volume) of BAC and BAC-Fe were determined by nitrogen adsorption at its normal boiling point (-196ºC). The specific area (S_{BET}) was calculated from the isotherm data using the Brunauer, Emmet and Teller (BET) model. Total pore volume (V_{total}) micro pore volume ($V_{micropore}$), volume of pores less than 1 nm (V<1 nm) and mean pore width (L) were calculated using the density functional theory approach (DFT) [23]. For FT-IR measurements, activated carbon synthesised from bamboo waste samples was blended with KBr to form pellets; the spectra were obtained on a Nicolet 7500 FT-IR spectrophotometer (Thermo Nicolet Corporation, USA) in the range of 4000–400 cm^{-1} at 4 cm^{-1} resolution. For SEM observations, an FEI Quanta 200F scanning electron microscope (FEI Company, Holland) was utilised. The accelerating voltage was 20.00 kV. Activated carbon obtained from bamboo waste powder was placed on an iron slice. All samples were observed directly under low vacuum without being sputtered with gold.

2.3. pH study

Measurements of the effects of variable pH on adsorbent performance used 0.1000 g of each adsorbent (iron-

treated activated carbon) added to 50.00 mL of 50 μg L^{-1} arsenate or arsenite solutions. The variable pH solutions were prepared by adding dilute NaOH or HNO_3 dropwise to achieve pH values of 2.0 through 12.0. Bottles were shaken at 125 rpm for 48 h at a constant temperature (25.0 ± 1ºC).

2.4. Adsorption equilibrium isotherm

The batch sorption experiments conducted for the isotherm study were performed using different weights of adsorbents (0.0500, 0.100, 0.150, 0.300 or 0.500 g) to which a constant volume (100 mL) of solution (100 μg L^{-1} arsenate or arsenite) was applied in 100 mL Nalgene bottles; all chemicals were of reagent grade and solutions were prepared by Milli-Q water (Q-H_2O, Millipore Corp.) with resistivity of 19.4 MΩ-cm. Sodium arsenate (Na_2HAsO_4•$7H_2O$, 99%) and sodium arsenite ($NaAsO_2$, 99%), were obtained from Sigma to prepare solutions (100 μg L^{-1}); these were placed in closed 250 mL amber bottles. Solutions were stirred at 200 rpm for periods between 5 and 240 min at 25±1ºC. Activated carbons were removed by filtration and arsenic concentrations were analysed by a Spectra AA Model AAS-8010 with a Graphite Furnace Atomic Adsorption spectrometer (GFAAS) (Young Lind Instrument Co. Ltd., Korea) with a Young Lind Graphite Tube Analyser (GTA) 110. Calibration curves were completed before each sample run with 0, 5, 10, 25 and 50 μg L^{-1} solutions and a %RSD <5 for triplicate sample sets. Blank solutions were also prepared and analysed. The solution pH changed as the metal ion concentration changed during adsorption process. A previous survey was done to determine the solution pH associated with maximum adsorption.

2.5. Adsorption kinetics

We followed the procedure proposed by Le [24]. Arsenic adsorption kinetics were evaluated at 25ºC and pH 10.5. The initial As (arsenite or arsenate) solution concentration was 50 mg As per L (corresponding to 50 mg As per g adsorbent). The kinetic experiments were conducted in a 1000 mL two-neck distilling flask. The flask was magnetically stirred and partially immersed in water in a plastic tray that was connected to a circulating water bath. With this experimental setup, the temperature of the solution inside the flask was maintained at 20 ± 0.5ºC. A combination pH/electrode was inserted into the flask for on-line pH measurement. Two burettes filled separately with 0.1 N HNO_3 and 0.1 N NaOH were set on top of the flask for pH adjustment. Before the start of each kinetic run, 450 mL of distilled water and 500 mg of the adsorbent (BAC-Fe; this adsorbent was chosen because it showed better results in terms of its adsorption capacity) were added to the flask, stirred for 1 h, and pre-adjusted to pH 10.5. Then, 50 mL of arsenate or arsenite stock solution containing 500 mg As per L were added to the flask while the pH was maintained at around 10.5. The reaction solution (5–10 mL) was sampled with an air displacement pipette at different times up to 180 min. The sampled solution was immediately filtered through a 0.45 μm membrane filter, and the filtrate as analysed for arsenite and arsenate.

3. Results and discussion

3.1. Characteristics of the precursor and activated carbon

The SEM images showing the surface morphology are shown in Fig. 1. The surface of both the bamboo charcoal and BAC-Fe were highly porous, which provided a large surface area for iron loading and arsenic adsorption. The SEM image of the modified activated carbon obtained from bamboo shows large pores

The properties of bamboo waste are presented in Table 1. The results show that bamboo waste is a highly volatile matter and with a high carbon content. The latter is a favourable property for a starting carbonaceous material to be used in the production of activated carbon. The carbon content of our bamboo is in agreement with other results reported for bamboo (43.8 and 47.6%) [22,23].

A useful indication of the pore structure of a solid can be obtained by visual inspection of the shape of the isotherm. Impregnation with iron seemed to have a considerable effect on the shape of the N_2 adsorption isotherm. The isotherms and pore size distribution of carbons prepared from bamboo waste with and without iron treatment are shown in Fig. 2. The isotherm of BAC without impregnation with iron increased sharply at a low relative pressure and reached a plateau. This is typical behaviour for a type I material using the Brunauer, Deming, Deming and Teller (BDDT) classification and is characteristic of microporous adsorbents.

Fig. 2 shows the nitrogen adsorption isotherms obtained at 77 K. Furthermore, Table 2 shows the apparent area BET, the micropore volume and mesopore volume. Isotherms show that the BAC is fundamentally microporous carbon and presents a type I isotherm according to the IUPAC classification, which has a high adsorption level at low pressure and then quickly reaches a plateau.

The carbon modified with iron (BAC-Fe) generates a hysteresis loop typical for active carbons with mesopores; thus, shown as the iron fix it modifies the

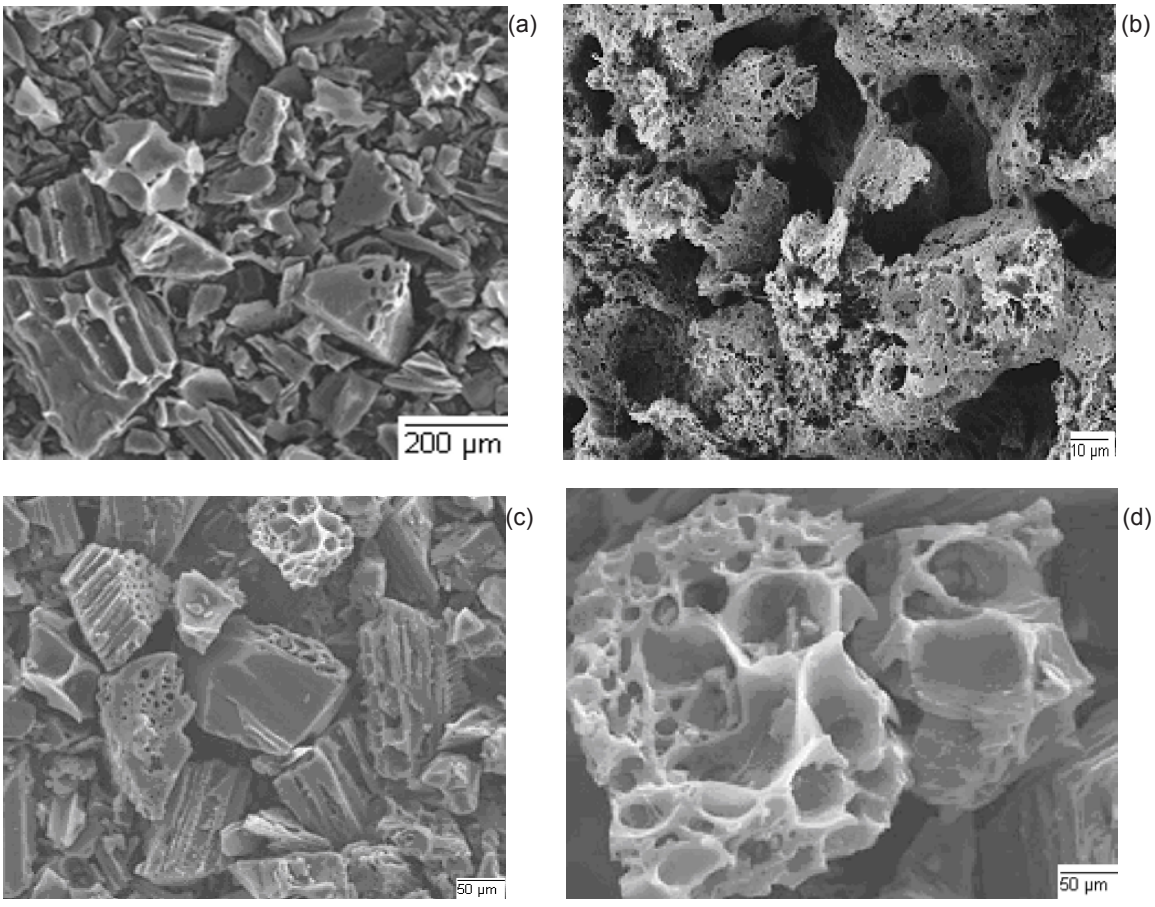

Figure 1. Scanning electron microscope (SEM) micrograph: a, b bamboo charcoal (BAC), and c, d modified bamboo charcoal with iron (BAC-Fe).

Table1. Properties of bamboo used in this research.

Analysis last	
Carbon	47.56
Hydrogen	5.32
Nitrogen	0.87
Oxygen, sulphur and others*	46.25

** Obtained by difference*

textural characteristics of the starting coal. This type of phenomenon has been reported in the scientific literature [22-25].

Results reported in Table 2 show that the samples have a significant micropore volumes which is interesting while analyzing the probability that these materials can be used as adsorbents effective in aqueous solution.

It should be noted that the BET area result is a result of the apparent area. When considering the mechanism for the mesopores and micropores filling it is important to analyze apparent area, because those pores can be filled before generation of the adsorption monolayer, which results in an overestimated measure of the area with no physical meaning.

In the case of activated carbons from bamboo this type of analysis must be considered.

FTIR spectra are employed to understand the formation of composites. Fig. 4 shows the FTIR spectra of bamboo waste, BAC and BAC-Fe. The FTIR spectrum (Fig. 4) shows a slight change between the original sample (waste bamboo), the activated carbon obtained (BAC) and the modified BAC (BAC-Fe).The most interesting changes in the corresponding band were found at 485 cm^{-1} that can be assigned to the O-Fe vibration.

3.2. pH study

The influence of pH on the adsorption of As(III) and As(V) ions is presented on the arsenic speciation diagram (Fig. 5). It shows the presence of each one of the species as a function of pH. This diagram clearly shows that stable species studied in this research may be removed between pH 7-10 [22-24].

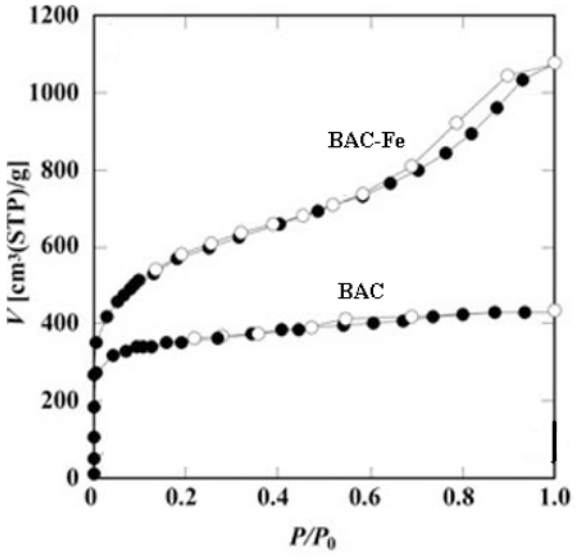

Figure 2. Isotherms of the adsorption of nitrogen at -196ºC for the synthesised sorbents.

Table 2. Pore characteristics of the activated carbon samples.

Samples	BAC	BAC-Fe
S_{BET} (m^2/g)	985	1357
V_{total} (cm^3/g)	0.42	1.3
$V_{micropore}$ (cm^3/g)	0.28	0.47
V< 1nm	0.18	0.15
L (nm)	0.6	1.1

Fig. 6 shows that activated carbon obtained and subsequently modified by iron adsorption has an adsorption of arsenate in the pH range of 7.0 - 11.0, approaching 100% removal at pH ~11.0. The other activated carbon (BAC) removes about 50% in the pH range 5.0-7.0.

Adsorption of arsenite with the BAC-Fe is most effective in pH range of 6.0-9.0 and reaches about 60% removal. These results are shown in Fig. 7.

These results clearly shows the dependence of adsorption capacity from the pH function of As(III) and As(V) ions in porous materials synthesized in this work.

The influence of the pH on the adsorption has been widely explored in the literature [26].

However, in this paper we will focus on explaining the influence of pH on arsenic retention capacity due to the texture and chemical properties developed in porous materials obtained from waste of bamboo. By modifying activated carbon with iron, the chemistry superficial of carbon changes due the modification of the functional groups.

With pH changes the concentrations of hydroxyl and hydronium ions change too, generating generate various adsorption capacities with respect to arsenic.

3.3. Adsorption isotherms from aqueous solution

Judgment of which model best represents arsenate or arsenite adsorption on the iron-treated adsorbents was based on the calculated respective correlation coefficients (r^2). There are several models in thermodynamics to interpret and analyze the type of adsorbate-adsorbent interactions. The most used are the Langmuir and Freundlich models that were applied in this investigation.

The Langmuir equation in its linear form can be written as presented below (Eq. 1):

$$\frac{c_{eq}}{q} = \frac{1}{q_{max} b} + \frac{c_{eq}}{q_{max}} \quad (1)$$

Linear plots of c_{eq}/q *vs.* c_{eq} (not shown) were used to evaluate characteristic parameters of the equation (see Table 3). q_{max} and b were obtained from the slope and intercept of the plots, respectively. The most important parameter in the Langmuir equation is the R_L – an equilibrium parameter, which is defined according to Eq. 2:

$$R_L = \frac{1}{(1 + b c_o)} \quad (2)$$

where b is the Langmuir constant and co is the initial concentration of the metal ions. When R_L is greater than 1 [27] the adsorbate-adsorbent interaction is unfavorable, whereas the value in the range of 0 to 1 is considered to be favorable for the adsorbate-adsorbent interaction. In this research determined values for R_L varied from 0.003 to 0.405 for an initial concentration of arsenite and arsenate of 100 um L^{-1}. Therefore, the adsorption process was favourable for arsenate and arsenite (for the activated carbons modified with iron, BAC-Fe) and was adjusted using the Langmuir model due to the R_L values.

The Freundlich isotherm was chosen to estimate the adsorption intensity of the adsorbent towards the adsorbate. The Freundlich equation is represented by Eq. 3:

$$q = K_F c_{eq}^{\frac{1}{n}} \quad (3)$$

where C_{eq} is the equilibrium concentration of the ion in aqueous solution, q is the amount of ions adsorbed and n and K_F are constants incorporating all parameters affecting the adsorption process between the adsorbate and the adorbente.

To assess this parameter, the equation is linearized:

$$\ln q = \ln K_F + \frac{1}{n} \ln c_{eq} \quad (4)$$

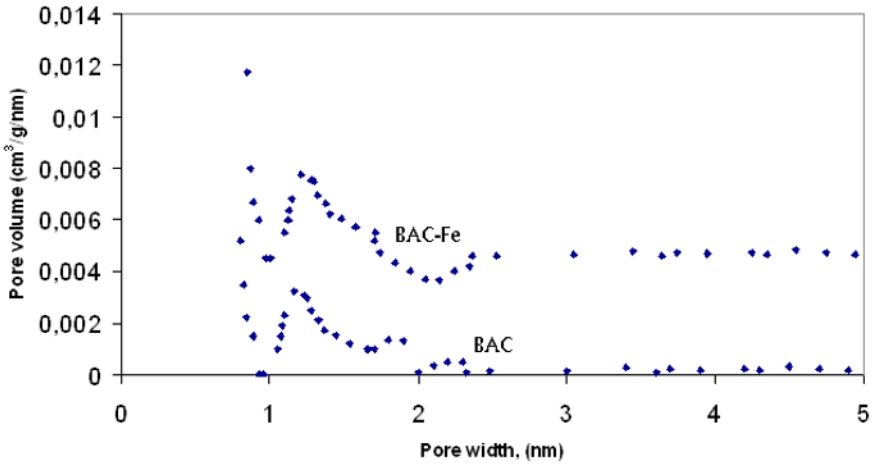

Figure 3. Pore size distribution for BAC and BAC-Fe carbons.

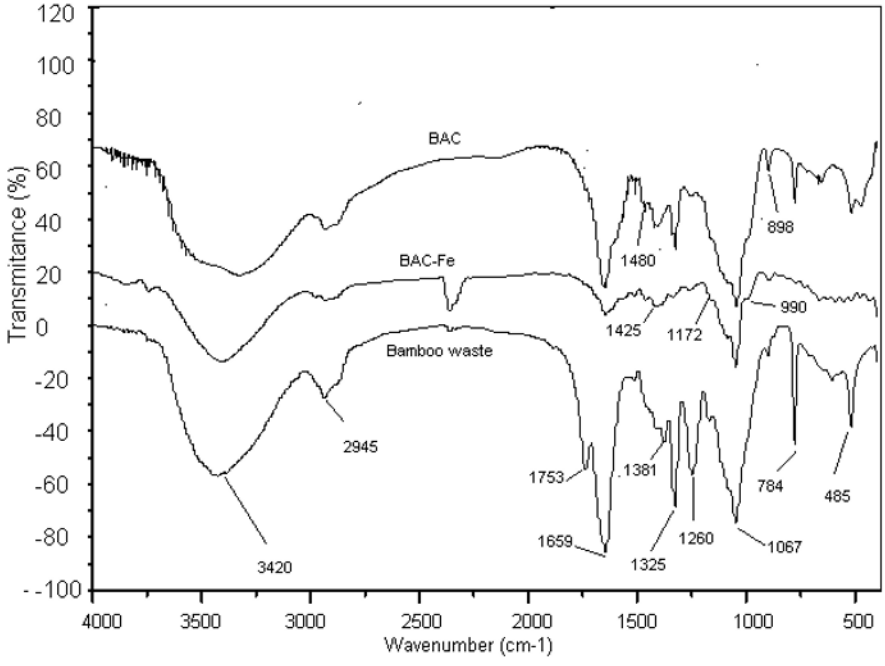

Figure 4. FT-IR spectra of synthesised activated carbons.

The results are also shown in Table 3. Parameter 1/n shows whether or not the adsorption is favorable. When the value is between 0.1 and 1 adsorption is considered to be favorable. The Freundlich model is usually one of the most applied in the phenomenology of adsorption as it is a simple model and can be applied to not only homogeneous surfaces but also has its reach in heterogeneous surfaces [26-28]. The magnitude of K_F and n show that the arsenite and arsenate data were well fit to the Freundlich model. Also, an increased K_F value indicates a greater adsorption intensity. Therefore, the K_F values were higher for arsenate. On the other hand, a relatively high R^2 value indicates that this model is fitted more confidently; this parameter is shown in Table 3. According to the obtained values, the Langmuir model provides the best fit to the experimental data in the present study.

The results of this work are interesting, because under our experimental conditions, they show that the adsorption capacity of arsenic depends on using BAC or the BAC-Fe, which shows that this is an important factor that dominates the adsorption and retention of arsenic, as shown in Fig. 8.

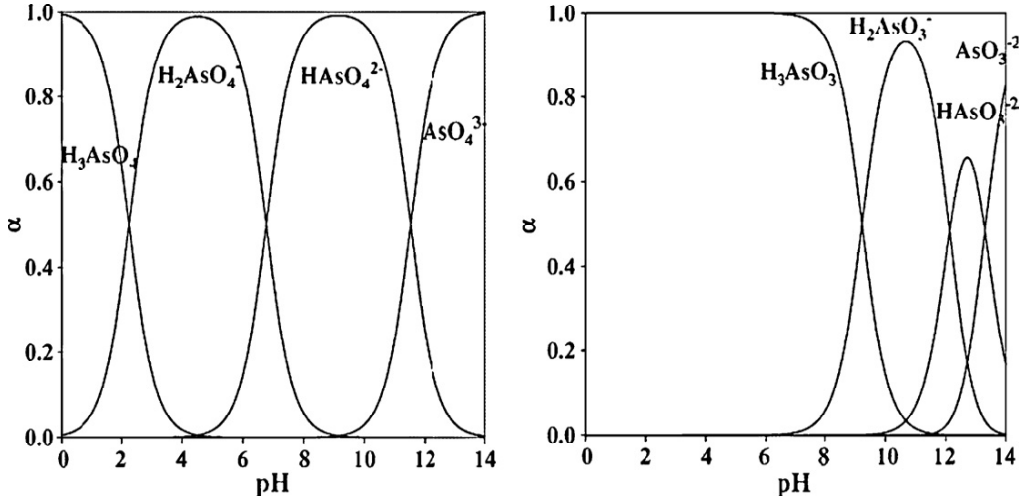

Figure 5. Distribution of arsenate and arsenite as a function of pH (after [28]).

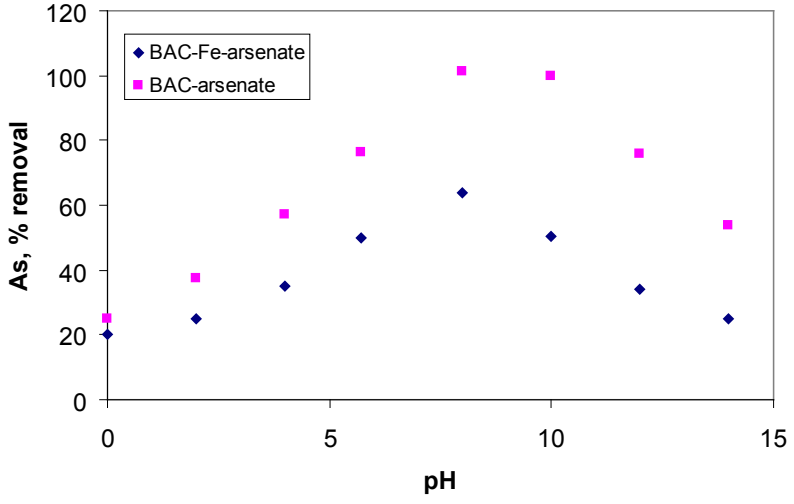

Figure 6. Study of the adsorption of arsenate on sorbents as a function of pH. Conditions: 100 μg L^{-1} arsenate, pH 9.0.

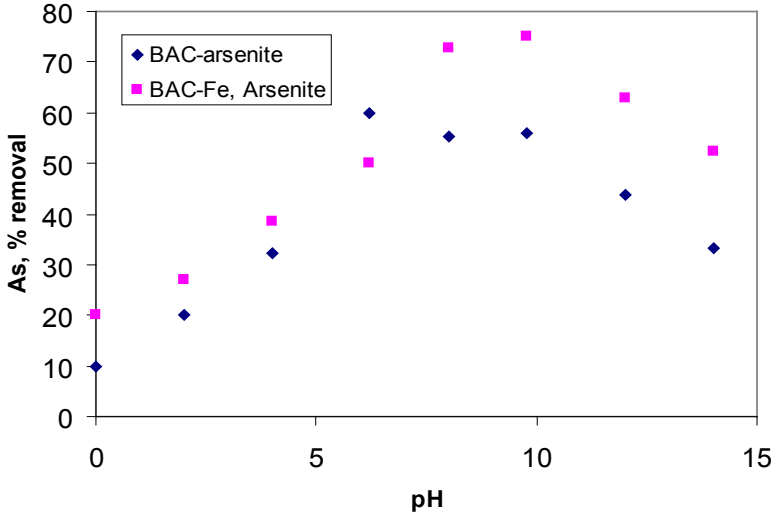

Figure 7. Study of arsenite adsorption by the BAC-Fe and BAC-Fe sorbents as a function of pH conditions: 100 μg L^{-1} arsenite, pH 9.0.

Table 3. Isotherm parameters of arsenite and arsenate adsorption on activated carbon from bamboo and activated carbon modified with iron.

	Freundlich model				Langmuir model			
	Linear K_D (L g^{-1})	K_F	1/n	R^2	q_{max} (μg g^{-1})	B (L g^{-1})	R_L	R^2
BAC-arsenite	8.03	12.365	0.540	0.9993	14.89	1.09	0.307	0.9734
BAC-Fe-arsenite	9.45	15.967	0.548	0.9710	19.19	1.05	0.004	0.9988
BAC-arsenate	12.33	20.786	0.678	0.9996	22.32	1.12	0.405	0.9799
BAC-Fe-arsenate	12.33	23.678	0.798	0.98215	27,32	1.22	0.003	0.9999

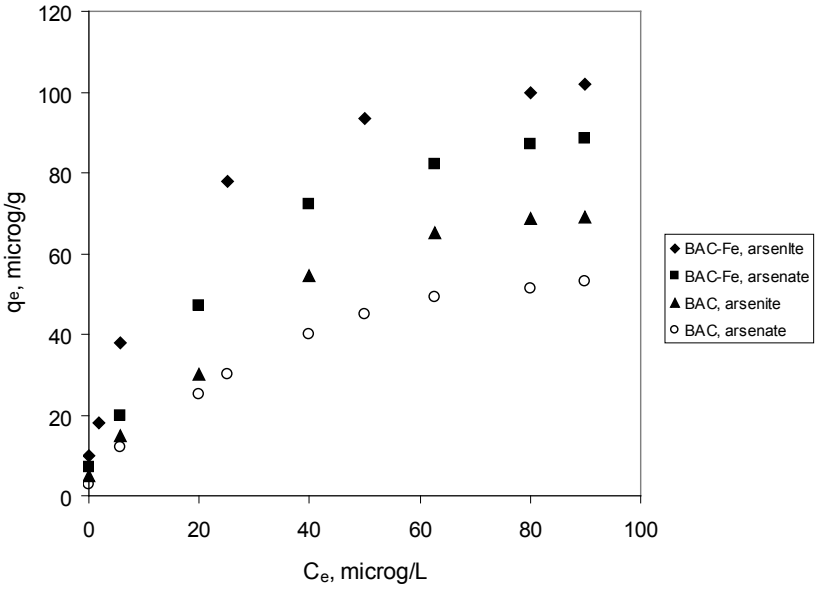

Figure 8. Adsorption isotherms of arsenic and arsenate ions by BAC-Fe and BAC. Experimental conditions: 100 μg L^{-1} arsenite and arsenate, pH 9.0.

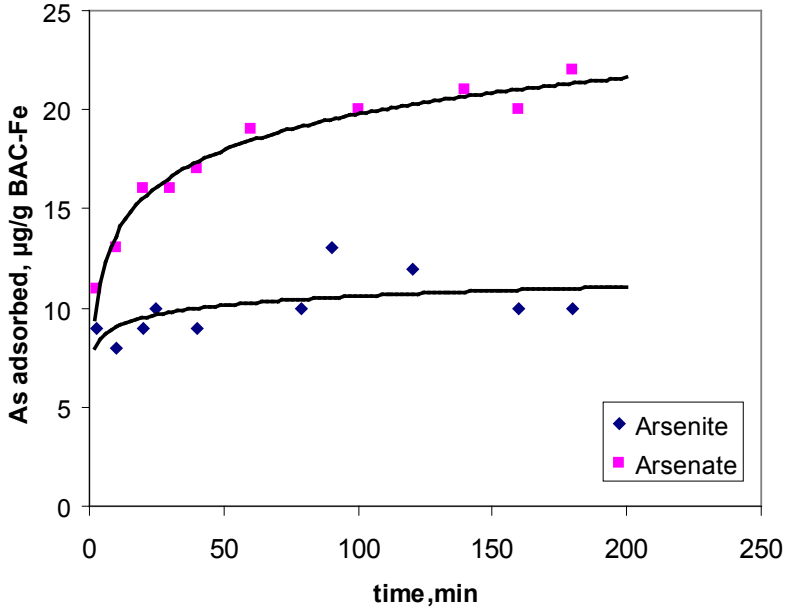

Figure 9. Kinetics of adsorption of arsenate and arsenite. Experimental conditions: 100 μg L^{-1} arsenite and arsenate, pH: 9.0.

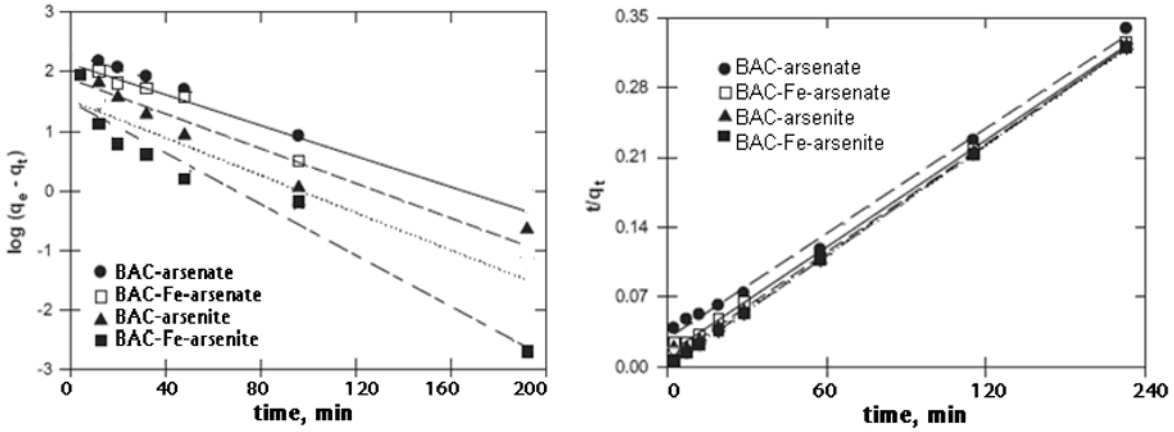

Figure 10. Pseudo-first order kinetic and pseudo-second kinetic models of arsenite and arsenate on BAC and BAC-Fe.

3.4. Adsorption kinetics

The kinetic results presented in Fig. 9 were obtained using the BAC-Fe at pH 9.0. Both ions adsorbed very quickly and after about 20 minutes both achieved a balance in the adsorption kinetics. The initial charge of arsenic was 50 mg As per 1 g adsorbent, with a higher adsorption capacity of the arsenite (45.1%), while the capacity of present arsenate was 27.6%. These results are in agreement with the data obtained during the study of pH influence.

Application methods from chemical reactions [26-28] could be used to expand our knowledge of kinetic behavior of above mentioned materials.

Simplified equation of arsenate sorption at iron sites:

$$S(BAC) - FeOH + H_3AsO_4 \rightarrow \rightarrow S(BAC) - Fe - H_2AsO_4 + H_2O \quad (5)$$

Simplified equation of arsenite sorption at iron sites:

$$S(BAC) - FeOH + H_3AsO_3 \rightarrow \rightarrow S(BAC) - Fe - H_2AsO_3 + H_2O \quad (6)$$

Here, S(BAC) represents the surface of the activated carbon [26].

We believe that the more rapid adsorption of arsenite was a function of the size of the arsenate ion, which allows further distribution into the interior of the porous structure. This is in agreement with the results reported by other authors [50,51], *i.e.*, using ferrihydrite, more rapid adsorption of arsenite was a function of the size of the arsenate ions, which allows further distribution into the interior of the porous structure.

Ion size is important because according to the diffusion processes occurring within the porous network it affects the rate of adsorption within the modified activated carbon. So, normally, bulky ions tend to have a slower adsorption rate than the smaller ones, because the latter can easily enter the network for meso- and micorporos carbon.

It is therefore necessary to establish a design appropriate for the adsorption treatment process, and it is also important to understand the rate at which arsenic is removed. Therefore, the study of adsorption kinetics is crucial, as it describes the solute uptake rate, which in turn controls the residence time of adsorbate uptake at the solid–solution interface. Fig. 9 demonstrates the time profile of arsenite and arsenate onto activated carbon obtained from bamboo treated with iron (BAC-Fe). It was found that most of the uptake of arsenite took place rapidly in the first 50 min, followed by a relatively slow process.

Adsorption equilibrium could be obtained within 100 min, which is longer than that of the adsorption of inorganic arsenate. The lower adsorption rate of arsenate onto the sorbent might be due to the properties of BAC-Fe. If adsorption mainly occurred due to electrostatic interaction, the rate of adsorption would be very fast, on the order of minutes [26].

The uptake of arsenite on activated carbon modified by iron was on the order of minutes, which implies that the specific adsorption that occurs between arsenite and BAC-Fe may be by a diffusion process. To better understand the adsorption kinetics of arsenite, pseudo-first order and pseudo-second order models were used to simulate the adsorption process. The models are expressed as follows:

$$\frac{dq}{dt} = k_1\left(q_{e1} - q_t\right) \quad (7)$$

$$\frac{dq}{dt} = k_2\left(q_{e2} - q_t\right)^2 \quad (8)$$

where k_1 and k_2 are the rate constants for the pseudo-first-order (1/h) and the pseudo-second-order models [g $mmol^{-1}$ h^{-1})], respectively, while q_t and q_e are the amounts of arsenite and arsenate adsorbed (mmol g^{-1}) at time t and at equilibrium onto BAC-Fe, respectively. From Fig. 9, it can be found that the adsorption kinetics data were well described by both pseudo-first order and pseudo-second order rate models; however, the pseudo-first order rate model fit better with a correlation coefficient r^2 above 0.99.

In the arsenate adsorption kinetics study, the kinetics were also analysed and similar results were found. This research shows that the kinetics fit well to a pseudo-first order model (see Figs. 10a and 10b).

4. Conclusions

The activated carbon synthesised from bamboo waste and modified by iron showed promise as an efficient low-cost adsorbent for arsenate and arsenite removal. Arsenite and arsenate were effectively removed by BAC-Fe via adsorption; the difference in the adsorption capacity for arsenite and arsenate was very significant. The extent of arsenic adsorption depended on surface loading and pH. The adsorption isotherm data obtained for BAC-arsenite and BAC-arsenate fit the Freundlich model well and the adsorption kinetics followed a pseudo-first order rate equation, as did BAC-Fe-arsenite and BAC-Fe-arsenate adsorption. The adsorption isotherm data obtained for BAC-Fe-arsenite and BAC-Fe-arsenate fit well to the Langmuir model. Both adsorption and desorption proceeded very rapidly, and within 50 minutes, the amount of arsenic adsorption or desorption reached up to 60-80% of levels detected under equilibrium conditions.

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