



Adsorption of As (III) From Aqueous Solution by Groundnut Shell

KEYWORDS

Arsenic (III), groundnut shell, adsorption, isotherm

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ABSTRACT Adsorption has been demonstrated to be a useful alternative to conventional treatment systems for the removal of toxic metals from aqueous solution. In the present study the adsorption of As (III) on groundnut shell was evaluated. Batch adsorption experiments were conducted to find the effect of parameters like adsorbent dose, contact time, pH, temperature and initial metalloid concentration. The maximum adsorption efficiency was found to be 28.5 µg out of 50 µg from the As (III) bearing aqueous solution. The removal of the metalloid ions was dependent on the physico-chemical characteristics of the adsorbent and other parameters. The adsorption data were fitted to Langmuir and Freundlich isotherm models. The Langmuir model showed better representation of data, with correlation coefficient greater than 0.98.

1. Introduction

Arsenic contamination of natural water is now a worldwide crisis since the past few decades. There have been a wide spread reports of arsenic poisoning in Bangladesh (Nickson et al., 1998), West Bengal (Chatterjee et al., 1995; Kumar and Sharma, 2005), China (Guo et al., 2003), Mexico (Del Razo et al., 1990), the upper mid Western United States (Welch et al., 1998), Argentina, Chile, Taiwan and Japan. Arsenic is released to the environment through a combination of natural processes like weathering reactions, volcanic emissions, and anthropogenic activities like industrial discharges, mining activities or even agricultural use of arsenical pesticides (Min and Hering, 1998). Arsenic in trivalent and pentavalent forms are extensively found in natural aqueous system out of which As (III) is about 60 times more toxic and have high mobility than As (V). The carcinogenic and toxic effects of arsenic on living beings are quite well documented. The chronic health effects commonly include skin, lung, bladder, liver, kidney and prostate cancers (Singh et al., 2007; Flora et al., 2007; Guo et al. 2003), neurological, hematological, renal, respiratory and cardiovascular diseases (Tseng, 2007).

According to WHO and USEPA the value of arsenic in drinking water is limited to 10 µg/L (WHO, 1993; Smildley and Kinniburgh, 2002). Various physical and chemical treatment technologies were introduced for the removal of arsenic from waste water which includes coagulation or precipitation, lime softening, ion exchange, membrane filtration and adsorption method (Wilkie and Hering, 1996, Nemade et al., 2007). Most popular and widely used adsorbents were activated carbon produced from coconut shells, wood char, oat hulls, lignin, petroleum coke, bone char, peach pits, fish, fertilizer waste, waste rubber tire etc. (Pollard et al., 1992). Some activated carbon impregnated with metallic silver, zirconium and copper were also used for arsenic remediation (Manju et al., 1998; Peraniemi et al., 1994; Rajkovic 1992). The high price and regeneration cost of activated carbon limits their large scale use and has encouraged looking for low cost adsorbing materials (Mohan and Pittman, 2007). New methods like construction soil filter (CSF) (Nemade et al., 2008), low cost adsorbents like agricultural by-products, clay minerals, industrial by-products such as fly ash, zeolites, blast furnace slags, hydrotalcites, hydroxides and natural red soil disintegrated from laterite rock have so far been studied for arsenic removal from waste water (Mohan and Pittman, 2007). Hence adsorption of heavy metals using natural adsorbent including agricultural wastes is effective as well as economically viable (Sun and Shi 1998).

The present work is an attempt to remove As (III) from aqueous solution taking groundnut shell as adsorbent, and to study the effect of adsorbent dose, contact time, pH, temperature and initial metalloid ion concentration for removal of As (III) and finally to find out the optimum adsorption isotherm.

2. Materials and Method

Preparation of Adsorbents

The shells obtained from the groundnuts were initially washed in tap water thoroughly to remove the soil and clay particles. Then they were washed with distilled water and dried under sun for 2-3 days. The shells were then ground and particle sizes ≤ 1 mm were obtained by passing the milled materials through a 0.1mm steel sieve. Then, they were used for the experiments without any physical or chemical treatments.

Characterization of Adsorbent

In terms of their structure groundnut shells can be regarded as a lignocellulosic agricultural waste material containing high amount of proteins and crude fibres (Mahajan and Sud, 2011). The biomass samples were analyzed by a Shimadzu FTIR Prestige-21 spectrophotometer in KBr using DRS technique within the range of 400-4000 cm⁻¹ to identify the functional groups present in it. KBr was used as the back ground material which was mixed with very finely ground adsorbent in the ratio 96:4 (KBr:sample) and then the analysis was carried out.

Preparation of adsorbate solution

A stock solution of Arsenic was prepared by dissolving 734 gm of sodium arsenite, (NaAsO₂) in 10 ml distilled water containing 4gm NaOH and diluted to 1,000 ml with distilled water; (1 ml=1mg As). An intermediate Arsenic solution was prepared by diluting 5 ml stock solution with 500 ml distilled water i.e. 1 ml solution contained 10µg As. Then 10 ml of intermediate solution was diluted to 100 ml distilled water (1ml = 1 µg).

Analytical Method

The concentration of total arsenic was measured by Silver Diethyl Dithiocarbamate (SDDC) method (Ballinger et al., 1992). For each test 35 ml of sample was taken in a clean Gutzeit generator and to that solution concentrated HCl 5.0 ml, KI 2.0 ml (15%) and SnCl₂ 0.4 ml (40%) were added successively. Then 30 minutes time was allowed for the reduction of As (V) to As (III). In the mean time the glass wool in the scrubber was impregnated with 6-8 drops of Pb(OAc)₂

solution and 4.0 ml SDDC reagent was taken in the absorber tube. After 30 minutes of the reaction, 3.0 g zinc was added into the generator and immediately scrubber-absorber assembly was connected. The reaction was allowed to continue for another 30 minutes to ensure complete evolution of AsH₃ gas. It is also prescribed to heat the generator slightly to ensure that all arsine is released. The absorbance of the SDDC reagent was measured at 535 nm using the reagent blank as the reference.

Batch Adsorption Experiments

Batch study was conducted to find out the optimum value of adsorbent dose, contact time, pH, and temperature for maximum possible removal of the adsorbate and the best fitted adsorption isotherm model. A series of 50 ml of standard As (III) solution of 1ppm was taken. The adsorbent dose was added to sample and sufficient time was allowed for adsorption equilibrium. Then the mixtures were filtered through whatman filter paper. The residues were then digested in 250 ml conical flask with (1+1) H₂SO₄ and HNO₃. The extract so obtained after diluting up to 35 ml was transferred into a clean arsenic generator and then the sorption studies were carried out as per SDDC method. In the process all As (III) ions gets converted to AsH₃ in the generator which on passing through the scrubber is absorbed by the pyridine solution in the absorber tube forming a red coloured complex. The later was analyzed by UV-Vis spectrophotometer (ABBOTA Corporation SM 1200). The arsenic removal percentage was determined by the following equation:

$$\text{Arsenic removal \%} = C_0/C_a \times 100$$

Where, C₀ is the initial As (III) concentration in the sample and C_a is the adsorbed As (III) concentration by the groundnut shell from the sample solution.

Results and Discussion

As a pilot study four different adsorbents – rice husk, saw dust, fly ash and groundnut shell were evaluated for As (III) removal from aqueous solution. The removal efficiencies of rice husk saw dust, fly ash and groundnut shell were 5%, 28%, 21% and 30% respectively. As the ground nut shell showed higher efficiency, it was considered for further investigation.

Fourier Transform Infrared Analysis of Adsorbent

The spectra of the adsorbent showed a number of adsorption peaks which indicates its complex nature. In figure.1 the peaks observed at 3500 cm⁻¹ shows N-H stretch and peaks at 3203.76, 3157.47 and 3136.25 cm⁻¹ of O-H stretch from carboxylic acid group. The peak at 1666.5 cm⁻¹ is due to C=O stretch. Aromatic C-H bend is also present showing peak at 848.6. The peak at 1597 cm⁻¹ is associated with N-H bend of primary amine. Figure.2 shows the FTIR spectrum after adsorption. The broadening of –OH at 3331 cm⁻¹ and carbonyl group at 1666.5 cm⁻¹ was observed. These studies indicate the participation of hydroxyl and carbonyl group of the surface sites of the sorbent in the adsorbent interaction.

Effect of Adsorbent Dose on Adsorption Process

To study the optimum adsorbent dose, the experiments were conducted under the conditions of pH 8.43 and variable adsorbent dose (0.5, 0.625, 0.75, 0.875, 1, 1.125, 1.25, 1.375, 1.5, 1.625 and 1.75) in grams per 50 ml of 1 mg/l standard solution, temperature 21°C and contact time 2 hours (Fig 3). The figure reveals that uptake of As (III) increases rapidly from 0.5 gm to 1.5 gm (per 50 ml of the solution) and marginally thereafter due to more surface area with increase in adsorbent dose which confirms the finding of Gupta et al (2005). Further addition of the adsorbents did not show any considerable increase in As (III) removal. This might be due to the overlapping of the active sites at higher concentration of the adsorbent, thus reducing the surface area (Alagumuthu and Rajan, 2010). Ajmal et al. (2006) observed that by increasing the adsorbent dose, the efficiency of groundnut shell increases, while adsorption density decreases with increase

in adsorbent dose. The decrease may be due to the fact that some adsorption sites may have remained unsaturated during the adsorption sites, whereas the number of sites for adsorption increased by increasing the adsorbent doses and that resulted in the increase of removal efficiency.

Effect of Contact Time

To find out the effect of contact time all of the parameters including temperature at 21°C, adsorbent dose (1.5 g per 50 ml), pH (8.43), initial As (III) concentration except contact time were kept constant. The effect of contact time on As (III) adsorption efficiency has been shown in figure-2. The adsorption rate initially increased rapidly and removal efficiency was reached to about 55% within about 3 hours. There was no significant change in equilibrium concentration after 3 hours. The slow adsorption rate is likely due to the decrease in adsorption sites on the surface of the adsorbents (Chuming and Robert, 2008; Goldberg, 2007). Thus the contact time for 3 hours is adopted as the equilibrium time. The results of Gupta et al. (2005) show that most of the arsenic adsorption (65%) occurs in a contact time of 40 minutes but in present study the rate of this phenomenon was slower and is similar with the study of Ghanizadeh et al. (2010). These differences in optimum contact time may be related to the surface characteristics of adsorbents. The contact time of solute and adsorbents for the removal of different pollutants with adsorption process depends on the adsorbents and adsorbates characteristics and determination of minimum contact time is critical parameter in adsorption studies (Ghanizadeh et al., 2010).

Effect of pH

The pH of the aqueous solution is an important parameter that controls the adsorption process. To study the effect temperature (21°C), contact time (3 hours) and adsorbent dose (1.5 g per 50 ml solution) were kept constant with a variable pH. The pH of the solution was adjusted by using 1N HCl and 0.1 N NaOH and the arsenite removal were investigated. Figure-3 reveals that the removal is about 57% in pH range of 2-8.43 with an optimum value at pH 8. Further increase in pH resulted in the decrease of the adsorption efficiency. Hence, for all further experiments the pH is maintained at 8. Nemade et al (2007) observed 92% of As (III) adsorption on red soil surface in a pH range of 4-10 at an initial As (III) concentration of 1 mgL⁻¹. The percentage removal decreased rapidly with further increase in pH. At higher pH (> 9.8) laterite surface becomes negatively charged and arsenite adsorption becomes less due to repulsion of similar charge, as arsenite exists as anion in that pH range. The natural siderite efficiently adsorbed As (III) within a wide pH range between 3 and 10 (Guo et al., 2007). The independence of As (III) adsorption on solution pH could be explained by the amphoteric nature of Fe oxide/ oxy hydroxide (Guo et al., 2010).

Effect of Temperature

In this stage adsorption was performed with variable temperature (10°C, 15°C, 20°C, 25°C, 30°C and 35°C) keeping all other parameters constant. It was observed that 20°C is the optimum temperature. Kamsonlian et al (2010) reported that in the biosorption of Cd (II) and As (III) ions from aqueous solution by tea waste biomass the biosorption percentage increased with increase in temperature from 25 to 40°C. Further increase in temperature has no significant percentage removal of the adsorbate ions from the liquid phase. Andjelkovic et al (2011) found desorption of As (III) and As (V) at temperature higher than 200°C which may be due to dehydration of sorbents with decrease in number of –OH groups from the sorption sites.

Effect of Initial As (III) Concentration

Initial concentration is one of the effective factors to determine the adsorption isotherm. The experiments were done with variable initial arsenic concentration (1, 2, 3, 4, 5 and 6 in mg/L) and constant temperature 20°C, pH 8, contact time of 3 hours and 1.5gm/50ml. The experimental results of the

effect of initial arsenic concentration on removal efficiency were presented in the figure 7. The As (III) removal efficiency decreased with the increase in initial concentration and subsequently the fractional adsorption becomes independent of initial concentration which supports the finding of Choudhury et al. (2010). Yu et al. (2003) opined that at higher chromium concentration available sites of adsorption becomes fewer and hence percentage removal of Cr (III) decreases.

Adsorption Isotherm

Langmuir isotherm studies were conducted with varying initial As (III) concentrations (1- 6 mg/l), fixed adsorbent dose of 1.5 gm/ 50 ml and contact time of 3 hours at pH 8. The adsorption isotherms are fundamental in describing the interactive behavior between solute and adsorbent (Ofomaja and Ho, 2006). Langmuir isotherm yields a better fit to the experimental data at 20°C. The Langmuir isotherm indicates that the reaction is a reversible phenomenon and the coverage is monolayer (Altundogan et al., 2000). According to the above fact As (III) was adsorbed in the form of monolayer coverage on the surface of the adsorbent at 20°C. It has been reported that the adsorption of As (III) on hematite (Singh et al., 1988), As (III) and As (V) on activated alumina (Gupta et al., 1978) and goethite (Gimenez et al., 2007) follows Langmuir isotherm. The isotherm equations of these models are summarized below.

$$q_e = (c_0 - c_e) \frac{V}{m}$$

Where, q_e is the amount of As (III) adsorbed (mg/gm) at equilibrium contact time. C_0 is the initial As (III) concentration (mg/l). C_e is the As (III) concentration at equilibrium time in the solution (mg/l). V is the volume of the As (III) solution (L) and m is the weight of groundnut shells added into the solution (g).

The relation between the amount of adsorbate adsorbed by the adsorbent and the equilibrium concentration of the adsorbate can be expressed by the linearized Langmuir adsorption isotherm as

$$\frac{1}{q_e} = \frac{1}{Q_{max}} + \frac{1}{bQ_{max}} \times \frac{1}{C_e}$$

and the Freundlich isotherm as

$$\ln q_e = \ln k_f + \frac{1}{n} \ln C_e$$

Where, Q_{max} is the maximum adsorption intensity, b is the adsorption constant related to the enthalpy of adsorption, k_f is the constant depending upon the nature of the adsorbate and adsorbent. It represents the adsorption capacity. n , represents the adsorption intensity.

Linear Langmuir isotherm was drawn (fig. 8) by plotting $1/q_e$ versus $1/C_e$ and the linear equation obtained is and correlation factor, $R^2 = 0.9940$. The maximum adsorption capacity (Q_{max}) found was 0.024 mg/g. Freundlich isotherm for the adsorption was drawn (fig. 8) by plotting $\ln q_e$ versus $\ln C_e$ and the equation obtained is $\ln q_e = 0.085 (\ln C_e) - 3.874$ and correlation factor, $R^2 = 0.864$. It is observed from the curves and R^2 value that the adsorption follows Langmuir isotherm (Fig 9).

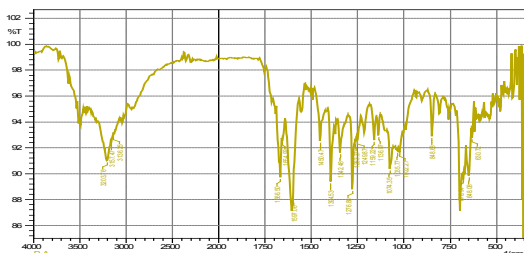


Figure.1 FTIR Spectrum before adsorption

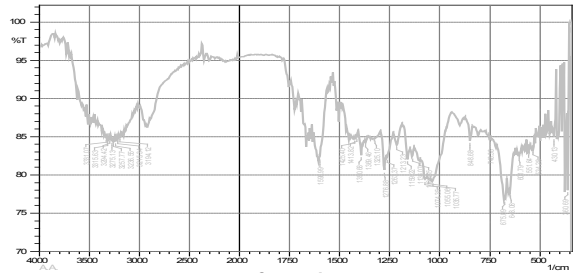


Figure.2 FTIR Spectrum after adsorption

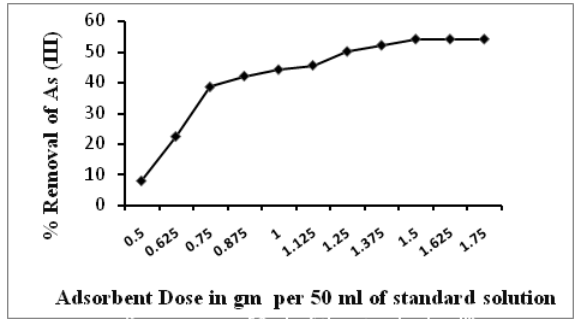


Figure.3 Effect of adsorption dose on adsorption process

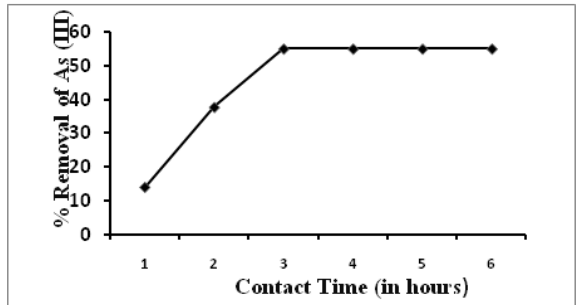


Figure.4 Effect of contact time on adsorption process

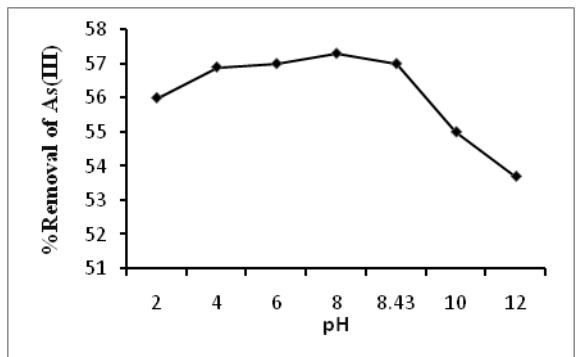


Figure.5 Effect of pH on adsorption process

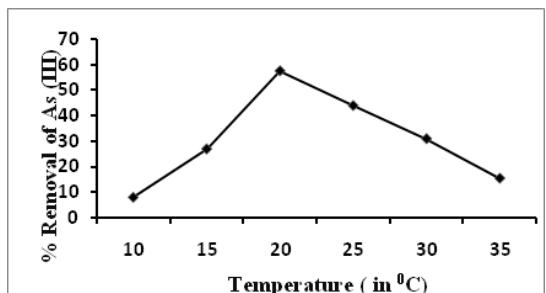


Figure.6 Effect of temperature on adsorption process

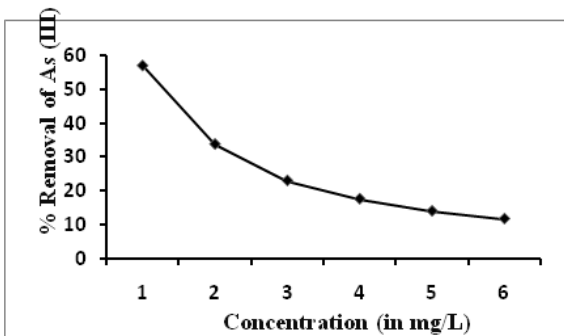


Figure.7 The effect of initial arsenic concentration on removal efficiency

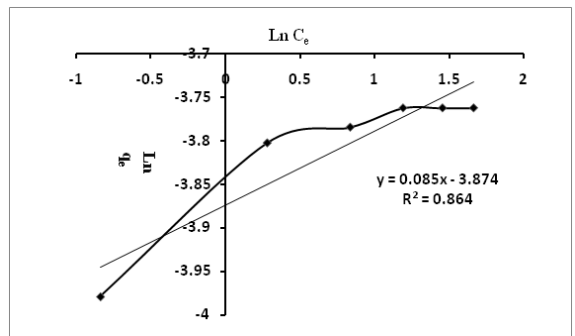


Figure.9 Langmuir isotherm curve and R^2 value

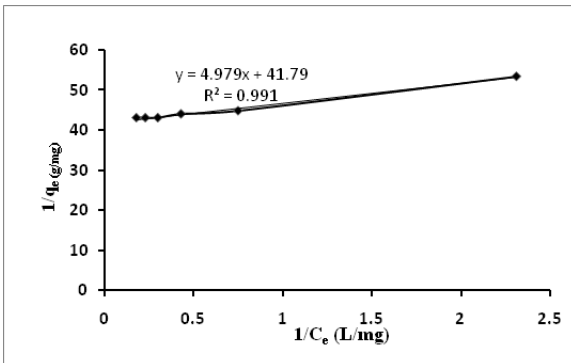


Figure.8 Linear Langmuir isotherm

4. Conclusion

In the present study, adsorption of As (III) on the groundnut shell without any chemical treatment in the arsenic bearing solution has been studied. The equilibrium studies revealed that the operating pH range should be 2 to 9. The adsorption efficiency was observed maximum at temperature 20°C within the equilibrium contact time of 3 hours. The Langmuir isotherm fits the adsorption data adequately. Since, the adsorbent is a waste biomass, it can be used as a low cost adsorbent instead of expensive adsorbents or conventional adsorbents for arsenic remediation. Moreover, India is the largest producer of groundnut (<http://www.commodities-control.com/eagrtrader/commodityknowledge/groundnut/groundnut2.htm>). So its shells would be an expedient option to be used as low cost adsorbent.

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