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Treatment of arsenic-contaminated groundwater by a low cost activated alumina adsorbent prepared by partial thermal dehydration

Mou Sen, Parimal Pal*

Environment and Membrane Technology Laboratory, Department of Chemical Engineering
National Institute of Technology, Durgapur, India-713209
Fax. +91343-2547375; email: parimalpal2000@yahoo.com

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ABSTRACT

Experimental investigations were carried out to remove arsenic from contaminated groundwater by low cost activated alumina based adsorbent prepared by partial thermal dehydration. Activated alumina based adsorbent with high surface area was prepared following partial thermal dehydration of gibbsite precursor and an attempt was made to study the effects of dehydration temperature, residence time, rate of increase of temperature and particle size on development of active surface area of the adsorbent. The operating parameters were found to have significant effect on active surface area development. BET Surface area (by nitrogen adsorption) and ignition losses were determined for all the samples. It was found that an adsorbent of surface area of around 335–340 m^2/g could be developed when dehydrated at 500°C for a residence time of 30 min in a rapid heating system (rate of increase of temperature 200°C/min) with particle size of 200 mesh (85%). The arsenic adsorption capacity of this adsorbent was determined both in batch and column studies. The adsorbent was found to be very effective in removing arsenic. The adsorbent placed in column could successfully remove arsenic from water up to a level below 10 $\mu g/L$ for more than 6000 bed volume water.

Keywords: Activated alumina; Arsenic adsorption; Adsorption surface; Thermal dehydration

1. Introduction

Contamination of groundwater by arsenic leachate is now a worldwide problem affecting millions of people from South-West U.S.A. to India, Bangladesh, Taiwan, Vietnam, and Nepal [1]. Through extensive studies [2–5] carried out over the last few decades on removal of arsenic from drinking water, adsorption, chemical coagulation-precipitation, ion exchange and membrane separation have been established as the broad technology options of purification. Pal et al. [6] have shown that for large scale treatment, physico-chemical separation technique is possibly the best for the developing South-East Asian countries, for small scale treatment facilities like the community water filters, however, activated alumina-based adsorption technique could be a favourable option provided it is produced at low cost as adsorbent needs periodic replacement. Clifford [7] Fox and Sorg [8], Hathway and Rubel [9], Chen et al. [10], Lin et al. [11] have all shown that activated alumina based adsorption could be a very effective point-of-use water treatment device by virtue of its good mechanical properties, stability under most reaction conditions and high surface area [12]. Different studies for state of the art technologies were discussed by many researchers [13–18]. The moot question that needs to be solved is how to the produce activated alumina at low cost. There are different methods for manufacture of active alumina adsorbents. It is produced mainly by gel precipitation of gibbsite powder with sodium hydroxide and sulfuric acid. This method gives more chemically pure activated alumina

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*Corresponding author.

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with well-defined physical parameters, which is used for different catalyst preparations. Cost of this active alumina is very high and, therefore, use of this adsorbent for water treatment is a costly affair in the developing countries like Bangladesh and India where vast rural areas have been affected due to contamination of groundwater by arsenic. The alternate route for production of active alumina is by partial thermal dehydration of gibbsite powder. This process comprises a thermal treatment, at a suitable temperature for a suitable time which removes 28–31% of water of crystallization from the gibbsite, leaving a loss on ignition of 4–7 wt%. This gives rise to high active surface area. Though investigations on effects of independent parameters like dehydration temperature, rate of increase of temperature [19], residence time and particle size [20] on the generation of surface area have been carried out, there has hardly been any study on the combined effect of these parameters on the development of surface area. Thus the present studies aim at better understanding the critical preparation parameters and their combined effects on production of activated alumina in an environmentally benign way through a low cost approach and to study the effectiveness of the newly formed activated alumina for removal of arsenic from water. So, an adsorbent was prepared by partial thermal dehydration rather than by conventional gel precipitation method and to find out the effectiveness of activated alumina powder (0.5–1.0 mm) in removing arsenic from contaminated groundwater, column study was carried out.

2. Materials and methods

2.1. Materials

The raw material used for the thermal dehydroxylation is known mineralogically as gibbsite and chemically as aluminium trihydroxide, Al $(OH)_3$. Commercial grade Gibbsite powder of specification as given in Table 1 was procured from Indian Aluminium Company (INDAL), Muri Works, India. All chemicals used were of reagent grade and were used without further purification. Arsenic standards and supra pure HCl were procured from E. Merck, Germany. Potassium permanganate was procured from Ranbaxy, India.

2.2. Equipment

Muffle Furnace (Amalgamated Engg. Works., India) of temperature range 1200°C ± 10°C and an Electric Oven (0–700°C, Amalgamated Engineering Works., India) was used for thermal dyhydroxylation of the powder for slow heating and fast heating respectively. A Standard Pot Mill (CHEM MACO, India) of capacity 1.5 kg and made of Stainless Steel (dia-10″, length-12″) with 55 rpm was used for mechanical disintegration of raw gibbsite powder.

Table 1
Physicochemical specification of gibbsite powder.

Chemical analysis	
Fe_2O_3, % by wt	0.008
SiO_2, % by wt	0.005
Na_2O, % by wt	0.280
Al_2O_3, % by wt	65.00
H_2O, % by wt	Rest
Mesh size	
+50 mesh	30
+100 mesh fraction	80
+200 mesh fraction	100
Loss on ignition, %	34–36
Free moisture content	4%
Surface area, m^2/g	5

2.3. Instrument

Surface area of the sample was measured in a surface area analyzer (SMARTSORB-90/91) of Smart Instruments Co. Ltd Mumbai, India. The instrument works on the theory first proposed by Brunauer–Emmet–Teller known as BET theory. According to this theory, at known partial pressure, amount of N2 adsorbed by the sample at Liquid N2 temperature can be expressed as:

$$(P/P_0).V.(1 - P/P_0) = 1/VmC + (C - 1)\ (P/P_0)/VmC \quad (1)$$

where, P/P_0= relative pressure of adsorbate, V = Volume adsorbed at P/P_0 at 0°C
Vm = Monolayer volume, C = Constant

2.4. Experimental procedures

2.4.1. Preparation of high surface area active alumina by partial thermal dehydration

10 g of dry gibbsite powder was taken in a small stainless steel bowl having diameter of 50 mm and height 30 mm, heated in muffle furnace at temperatures starting from 200°C to 700°C with a temperature interval of 50°C. The rate of increase of temperature was kept as 10°C per minute and dehydration was done for 30 min. The bowl was immediately placed in desiccators to avoid moisture adsorption. The preparation scheme is shown in Fig. 1.

To determine the effect of time of stay at the dehydration temperature, same quantity of raw gibbsite powder was taken and dehydrated at 400°C, 450°C and 500°C (as the maximum surface area was obtained in this temperature zone) with time of dehydration 15 min, 30 min and 45 min at the three temperatures.

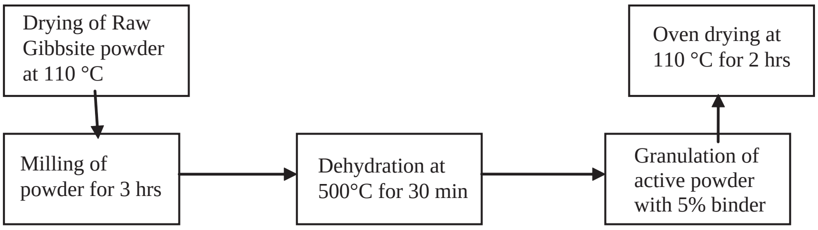

Fig. 1. Schematic diagram of preparation of activated alumina granules of size 0.5–1.0 mm.

Study was carried out to determine the effect of rate of increasing temperature on development of high surface area. The sample was heated to 350°C, 400°C, 450°C, 500°C and 550°C in an electric oven with rate of increase of temperature as 200°C per minute. To observe the combined effect of residence time and rate of heating, samples were kept for a residence time of 15 min, 30 min, 45 min, 60 min, 75 min, 90 min, 105 min and 120 min in the oven for each temperature.

To determine the effect of particle size for development of surface area 1.5 kg of gibbsite powder was first dried for 6–8 h in a tray dryer at 110°C to remove the free moisture. This powder was then milled for 1 h, 2 h, and 3 h respectively in a standard Pot Mill. Sieve analysis (Table 2) was done for the milled powder. This milled powder was then dehydrated at 450, 500, 5500C in the electric oven. The residence time were kept at 15 min, 30 min, 45 min in each case.

The surface area and ignition loss was measured for all the samples. Thus the combined effect of dehydration temperature, residence time, rate of heating and particle size on development of surface area was studied. The activated alumina powder thus formed is granulated in a granulator with the help of spraying 5% acetic acid binder. Granules are then air dried and oven dried to remove surface moisture. The method of preparation of activated alumina granules is as shown in Fig. 1. Granules of size range 0.50 mm to 0.9 mm were used for determination of arsenic removal capacity. The physicochemical specification of newly developed activated alumina granules is as shown in Table 3.

2.4.2. *Determination of arsenic adsorption capacity of the active alumina thus formed*

For each experiment 100 mL of 200 ppb As (III) and As (V) solution was taken in a 150 mL bottle, and agitated at 180 rpm for 16 h at 30°C. To study the effect of adsorbent dose on arsenic (V) and arsenic (III) removal, adsorbent dose was varied from 5–50 gm/L. The pH of the solution was maintained at 7 ± 0.1 by checking the pH value after each 2 h and adding N/10 HNO_3 drop wise.

To study the effect of arsenic concentration of the raw water on the removal efficiency, synthetic water sample containing 100–1000 ppb of arsenic (As-III and As-V separately) was added to 2.5 gm (optimum dose) of adsorbent in 100 mL water. The sample was agitated for 16 h in a shaker at 30°C with 180 rpm. The pH of the solution was measured after each 2 h and adjusted to 7 ± 0.1 by addition of N/10 HNO_3.

Table 2
Sieve analysis of the milled gibbsite powder and surface area generated.

Milling time (h)	Sieve analysis at 500°C	Surface area (m^2/g)
0	+50–30 +100–80 +200–100 +325–Nil	269
1	+50–Nil +100–65 +200–100 +325–Nil	316
2	+50–Nil +100–Nil +200–60 +325–100	327
3	+50–Nil +100–Nil + 200–1% +325–85%	336

Table 3
Physicochemical specification of activated alumina granules produced by partial thermal dehydration.

Physical characteristics	
Surface area, m^2/g	335
Pore volume, cm^3/g	0.5
Bulk density, g/cm^3	0.8 ± 0.05
Chemical composition, %	
Al_2O_3	92.0
SiO_2	0.3
Fe_2O_3	0.05

2.4.3. Column study

The schematic diagram of the experimental apparatus employed for the column study is shown in Fig. 2. Virgin activated alumina granules of size 0.5–0.9 mm were packed (approx. 53 gm) in a glass column having length of packed bed 20 cm and bed porosity of 0.44. ID of the tube was 20 mm.

Ground water spiked with 1 ppm Arsenic V was used to find out the column performance of the newly formed activated alumina. The raw water having arsenic (V) concentration of 1000 ppb was charged in down flow mode from the top of the column at a flow rate of 4 mL/min. Initially all arsenic ions were being adsorbed by the media resulting in zero effluent concentration. Most of the solute was removed initially by a narrow band of activated alumina granules at the top of the column (adsorption zone). As operation continued the adsorption zone proceeded towards lower layers of activated alumina. Ultimately when the adsorption zone reached the bottom of the column, arsenic concentration in the outlet began to increase. A plot of solute concentration in the effluent (mg/L) versus bed volume referred as 'break through curve' was obtained by plotting arsenic concentration in the effluent against the bed volume.

2.5. Analytical procedures

Analysis of the samples for arsenic concentration was done following flame-fias technique in a Perkin-Elmer brand (AAS-100) atomic adsorption spectrophotometer. In the flame-fias technique arsenic was analyzed after its conversion to volatile hydride which was formed through the following reactions:

$$NaBH_4 + 3H_2O + HCl = H_3BO_3 + NaCl + 8H$$

$$As_3 + H \text{ (excess)} \qquad AsH_3 + H_2 \text{ (excess)}$$

Volatile hydride was then transported to the quartz cell of the atomic adsorption spectrophotometer where it was converted to gaseous arsenic metal atoms at 900°C in air acetylene flame and analysis was done at 193.7 nm wavelength using a hollow cathode lamp. Samples to be analyzed for arsenic was pre-reduced using a reducing solution 5% (W/V) potassium iodide (KI) and 5% (W/V) ascorbic acid. The reduced sample was allowed to stand at room temp for 30 min. In hydride generation, 0.2% sodium borohydrate ($NaBH_4$) in 0.05% NaOH was used. 10% (v/v) HCl was used as carrier solution Hydride generator was purged using 99.99% pure nitrogen. pH of the oxidation unit and the filtered water was measured by a pH-ion meter (Micro-2, EDT). Percentage removal of arsenic was calculated using the initial value (C_{As0}) and the residual value (C_{As}) of arsenic concentration in feed water and treated water (permeate) respectively as below.

$$\% \text{ removal of arsenic} = (1 - C_{As}/C_{As0}) \qquad (2)$$

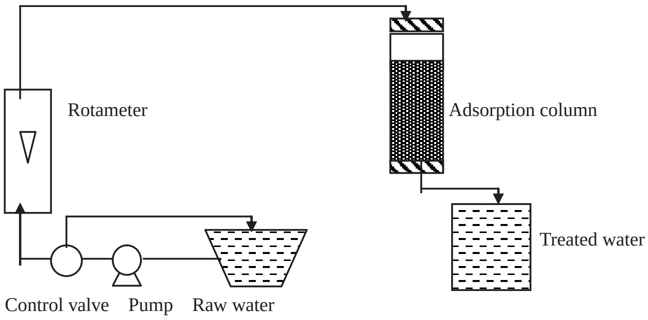

Fig. 2. Schematic diagram of the experimental adsorption column.

3. Results and discussion

3.1. Effect of temperature on surface area development

The surface area of raw gibbsite powder was approx 5 m^2/g, which did not show any significant increase in the surface area up to a temperature of 200°C but thereafter rapid increase in surface area with rapid release of structural water and a sharp peak, was observed in the temperature range of 400–500°C when heated for 30 min in a slow heating system (rate of increase of temperature was 10°C/min). Fig. 3. shows the variation in surface area when heated at various temperatures and a maximum surface area was achieved at 450°C, which was approximately 280 m^2/g. It indicates that surface area increases with the rise in temperature as gibbsite converted to bohemite during heating. It is due to the fact that gibbsite remains in crystalline form at normal temperature but it converts to amorphous alumina due to heating up to a range of 450–500°C, which is having more surface area than crystalline alumina.

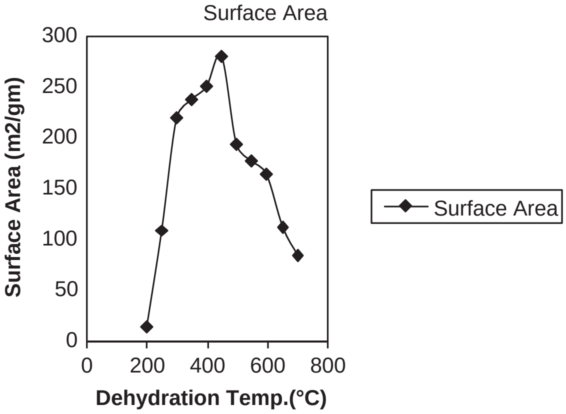

Fig. 3. Effect of change in the dehydration temperature on surface area.

3.2. Effect of residence time on surface area development

Fig. 4. exhibits the effect of residence time on generation of surface area and loss on ignition. The residence time at a particular dehydration temperature was also a varied for determination of optimum condition of thermal dehydration to get a high surface area active material directly from partial dehydration of gibbsite powder. In our studies it was found that a residence time of 15 min at a temperature of 450°C is the most suitable residence time for development of high surface area when heating was done in a slow heating system. From the figures it was observed that the surface area development at a particular temperature depends on heating period. The surface area thus achieved was 298 m^2/g with ignition loss 8.7%. Thus, by reducing the time of stay from 30 min to 15 min a better surface area has been achieved. Surface area formation depends on the presence of 0.5 H_2O as shown by Blanchin [21] in the alumina. It is a matter of controls on the heating system, how the water should be released at a particular temperature. Staying for less or more time will have an effect on release of water molecule from the alumina powder and will have effect on surface area development. Blanchin showed in his research that maximum surface area achieved when 0.5 molecules is attached with alumina molecule. Staying at a particular temperature reduces the presence of ½ molecule of H_2O, thereby reducing the surface area.

3.3. Effect of rapid dehydration

Earlier studies show that rapid dehydration route produce activated alumina with higher surface area [22–23]. However, there is hardly any data available regarding the effect of rate of increase of temperature on the development of higher surface area.

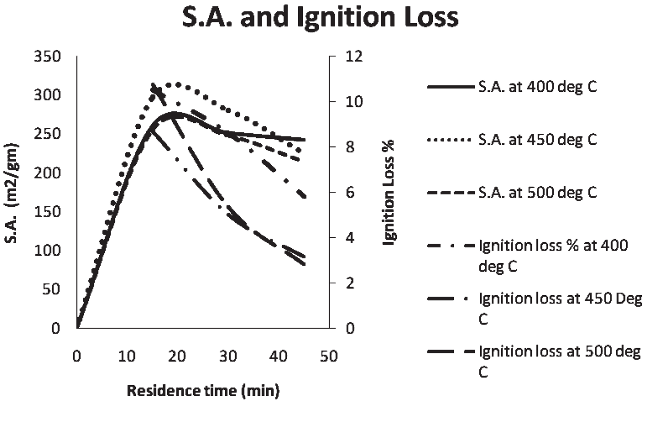

Fig. 4. Effect of residence time (min) on surface area (m^2/g) and loss on ignition (%) of gibbsite powder at 400°C, 450°C, 500°C in a slow heating system.

It was observed that the optimum dehydration temperature shifted from 450°C to 500°C when same test was carried out under very rapid dehydration. The surface area increased to a value of 310 m^2/g at 500°C and with a residence time of 30 min (Fig. 5). The reason behind the high surface area was probably the development of amorphous ρ, χ, η, and γ Alumina, which were obtained by rapid heating and rapid removal of water vapour as reported by B. Whittington [24].

3.4. Effect of particle size

Attempt has been made to increase the surface area of the thermally dehydrated gibbsite by applying mechanical grinding for reduction of particle size to increase the surface area further. The results are as given in Fig. 6. It is observed that no significant increase in the surface area is achieved after 1 h milling but after 2 h and 3 h milling surface area increased by about 5% and 8.5% respectively over the unground material dehydrated under the same operating conditions (Table 2). The particle size of the raw material has an effect on the surface area of an activated alumina powder when derived from partial dehydration of gibbsite. The sieve analysis shows the size range of the particle. This finely ground particle when partially dehydrated at 500°C for 30 min gives a maximum surface area of 336 m^2/g. This is due to the fact that in case of smaller particle size the water vapour

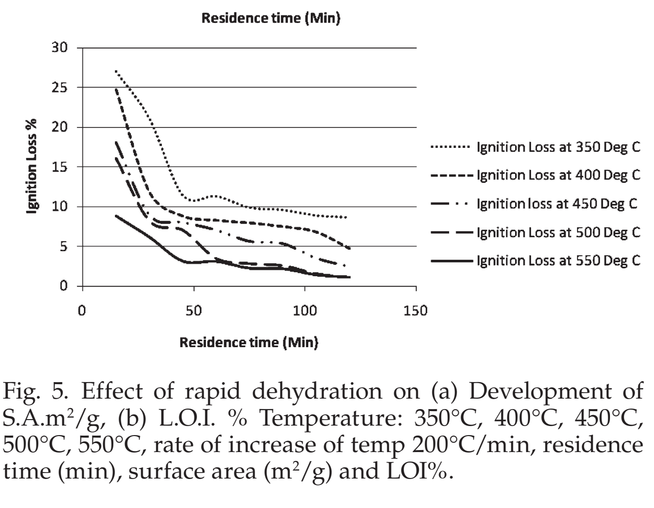

Fig. 5. Effect of rapid dehydration on (a) Development of S.A.m^2/g, (b) L.O.I. % Temperature: 350°C, 400°C, 450°C, 500°C, 550°C, rate of increase of temp 200°C/min, residence time (min), surface area (m^2/g) and LOI%.

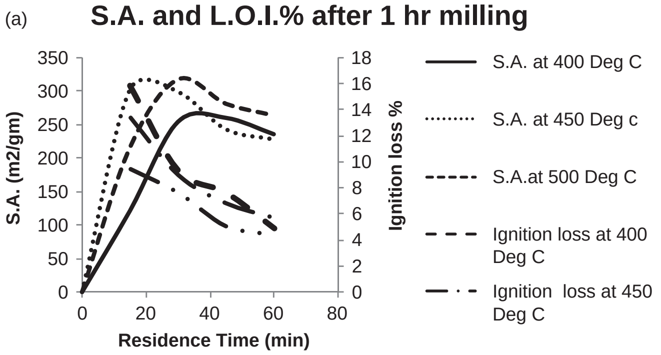

Fig. 6(a). Effect of particle size on development of surface area oven dried material milled for 1 h dehydrated at 400°C, 450°C and 500°C.

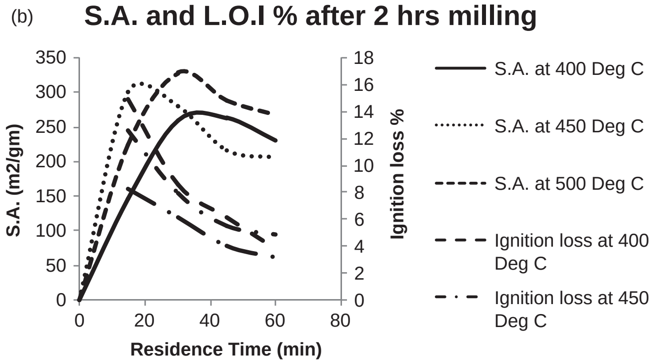

Fig. 6(b). Effect of particle size on development of surface area oven dried material milled for 2 h dehydrated at 400°C, 450°C and 500°C.

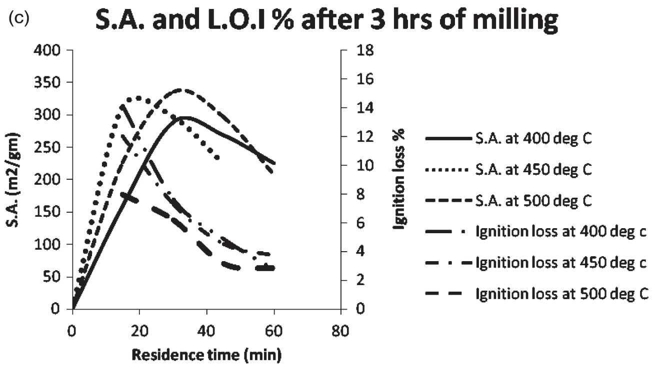

Fig. 6(c). Effect of particle size on development of surface area oven dried material milled for 3 h dehydrated at 400°C, 450°C and 500°C.

formed can rapidly diffuse out of the trihydroxide particle. The smaller particle sizes thus resists building up of vapour pressure inside the particle and thereby prohibit formation of boehmite which has a less surface area. Literature shows that relatively less boehmite is formed due to small size of the crystal [25,26]. Thus the best product obtained when dehydrated in a fast heating system at 500°C for 30 min after milling for 3 h. The surface area thus formed was 336 m^2/g.

3.5. Efficiency in removing arsenic from water

Fig. 7. shows the effect of adsorbent dose on % removal of arsenic. % removal was found to increase with adsorbent dose initially till it reached around 25 g/L. Beyond this value, however, no further increase in % removal accompanied the increase in dose. Thus the optimum adsorbent dose for contaminated water with 200–400 μg/L As concentration was found to be around 25 g/L. In the batch test, the adsorbent could remove as high as 90% As (V) and 42% of As (III) when feed concentration was up to 400 ppb. This is because the anionic As (V) is more attracted by activated alumina than neutral As (III). Above 400 ppb the removal percentage decreased gradually resulting a lowest adsorption of 72% and 35% for As (V) and As (III) respectively (Fig. 8) when arsenic concentration in raw water was 1000 ppb.

The breakthrough curve against an initial As (V) concentration of 1 ppm was plotted in Fig. 9. The point on the breakthrough curve where the arsenic concentration

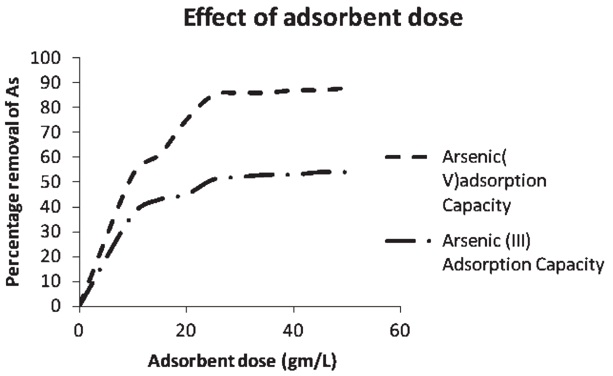

Fig. 7. Effect of adsorbent dose in removal of arsenic (V) and arsenic (III).

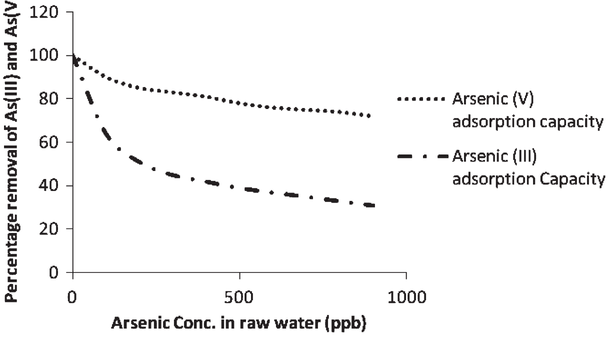

Fig. 8. Effect of concentration of arsenic (V) and arsenic (III) in raw water.

in the effluent becomes 0.01 mg/L is the breakthrough point. The point where arsenic concentration reaches 90% of the influent (900 ppb) is the point of exhaustion. The accumulation and subsequently the removal of arsenic were mainly dependent on the quantity of the adsorbent available in the bed.

Column study had shown the break through bed volume as 6000 up to which the effluent arsenic concentration was below 10 µg/L. The exhaustion point corresponded to a bed volume of 13,000, when the effluent arsenic concentration was found to be 90% of the influent arsenic concentration. Thus the activated alumina formed by partial thermal dehydration of gibbsite powder could efficiently remove arsenic from drinking water.

It was observed that residence time of 15 min gives better result than the 30 min when heated at 450°C in a slow heating system. Dehydration temperature shifted from 450°C to 500°C when heated in a rapid dehydration system instead of a slow heating system. The surface area improved by about 9% when particle size changes from 100% +200 mesh to 1% +200 mesh. The maximum surface area thus achieved was 336–340 m^2/g when dehydrated at 500°C in a rapid dehydration system for 30 min and after milling for over 3 h. The ignition loss (LOI) was determined in all the cases as it gives an idea of the amount of structural water remaining in the active alumina powder. This is because the literature shows that with the increase of dehydration temperature the ignition loss decreases keeping very less amount of water in it and thereby reduce the surface area. The adsorbent thus prepared by this active alumina powder showed As (V) removal over 90% whereas As (III) removal only 42% for raw water As conc. 400 ppb. This is because of the neutral characteristics of As (III) and anionic characteristics of As (V), respectively. However, the column performance in removing arsenic V was found to be satisfactory where arsenic level in water could be reduced to well below 10 ppb for more than 6000 bed volumes. The exhaustion bed volume was found to be more than 13,000. As concentration of arsenic in the groundwater of the Delta basin of Ganga is found to be in the 300–400 µg/L range, the cheap adsorbent is expected to be suitable for application in the concerned region.

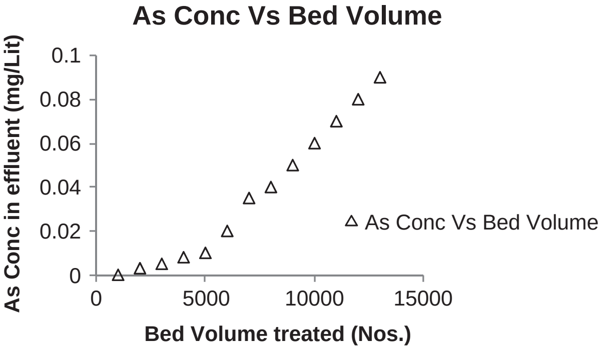

Fig. 9. Arsenic removal in a laboratory-based adsorption column.

3.6. Comparison of costs

The cost factors associated with production of activated alumina by gel precipitation method and by partial thermal dehydration of gibbsite powder are presented in Table 4. Cost estimates are based on Indian market prices of raw materials, utilities, labor and overhead charges. These estimates do not give absolute costs but indicate relative costs associated with the conventional method and the suggested present method. The data were collected from Oxide (India) Catalysts Pvt. Ltd. From the Table it is seen that the cost of manufacturing alumina-based adsorbent using the present technique is approximately half of the cost associated with gel precipitation method. The adsorbent thus produced is less complicated, much cheaper than that produced by gel precipitation and it involves no use of alkali or acid thus the production route is environmentally benign also.

Table 4
Cost comparison of activated alumina produced by gel precipitation process and thermal dehydration process for 1 MT material.

Components	Gel precipitation method		Thermal dehydration method	
Raw material		Cost ($)		Cost ($)
	Aluminium sulphate 1560 kg @ $0.42/kg	650	Gibbsite Powder 1500 kg @ $0.42/kg	625
	Gibbsite powder 1120 kg @ $0.42/kg	467		
	Caustic soda 560 kg @ $0.62/kg	350	Binder	62
	Binder	30		
Utilities	Electricity and fuel	210	Electricity and fuel	270
Over head and labour		220		145
Total		1927		1102

4. Conclusion

For removal of arsenic from contaminated groundwater a low cost adsorbent based on activated alumina was prepared by partial thermal dehydration of gibbsite. The adsorbent successfully reduced arsenic level in contaminated water well below WHO-prescribed level (i.e. 10 μg/L). This simple scheme involving fewer steps, less energy and little harsh chemicals was not only cheaper than conventional gel precipitation method but was environmentally benign also. As most of the arsenic-affected people live in the rural backwaters of South-East Asian countries where community water filters fail because of the need for frequent replacement of costly adsorbent material, the proposed manufacturing scheme is likely to be a highly promising one.

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