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# REMOVAL OF AS (III) ION FROM AQUEOUS SOLUTION USING IRON PARTICLES PREPARED FROM LEMON JUICE

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## ABSTRACT

The present study reports a benign approach to prepare citrate capped iron particles using juice of a lemon fruit. Resultant particles were subjected to detailed adsorption studies for removal of arsenite aqueous solution of defined concentration. The planer reflection of XRD analysis suggested that iron particles were crystalline in nature. FT-IR study suggested that functional groups like -$NH_2$, -$NO_2$, -CO, and –CS were involved in arsenite ion sorption. The composite removal efficiency for As (III) was found to be 88.58 %, at pH 4.5, with contact time of 30 min and temperature at 30°C. Data of adsorption were analyzed using various parameter like isotherm models, thermodynamic and kinetic models. Chemisorptions resulted in to formation of monolayer arsenite ions over the surface of prepared iron particles with maximum adsorption capacity; 55.53 mg/gm. Adsorbent hold good promises to remove arsenic from contaminated water.

***Keywords:*** Arsenite, Adsorption, Iron particles, *Citrus limon* L., Kinetics

## INTRODUCTION

Today, chemical precipitation, electrochemical method, ion exchange, membrane filtration, and adsorption methods are used to the remove the metals and metalloids from water. On considering the other techniques it has been found that the adsorption method is the best choice because adsorption is appreciable removal capacity, offers less energy consumption with favorable hassle free operation conditions (Lakshmanraj *et al.*, 2009). Common adsorbents that are used for the adsorption are of low-cost materials usually such as carbonaceous materials and modified materials. These adsorbents are of wide ranges, variety and sources that it can be selected according to the local conditions by different countries and regions (Mohan and Pittman 2006)considered a priority pollutant. Industrial sources of Cr(VI. Recently, the method of adsorption by nanoparticles has been proposed as an innovative treatment for the purification of water from. It has been witnessed for the past decades that the increasing applications of nanomaterials have achieve a notable achievement in the environmental protection field (Zhang 2003). Generally nanoparticles have external dimensions are in the nanoscale (usually 1–100 nm) or those who have a nanoscale internal structure/surface. Under the nanoscale, nanomaterials often exhibit some special properties, such as a surface effect, small size effect, quantum effect, and macro quantum tunnel effect (Nurmi *et al.*, 2005). These properties pay greater contribution to their extraordinary adsorption capacity and reactivity, both of which are favorable for the removal of heavy metal ions.

Arsenic contamination of subsurface water and its subsequent consumption have been seen as a serious public health related issue (Bhowmick *et al.*, 2018). Developing countries e.g. eastern part of India and Bangladesh, Nepal, Inner Mongolia, Cambodia, Thailand, Argentina, Chile including developed nation like USA, have witnessed geogenic arsenic contamination and disease linked to arsenic exposure (Singh et al., 2015). In and around India like East Bengal and West Bengal, some area of North Bihar (Gangatic plain) where over 40 million people are being exposed to more than 50 ppb of arsenic whereas according to EPA, the limit is set to 10 ppb. Out of 4 million, 1.2 million tube wells of Bangladesh is found containing arsenic more than 50 ppb and 7600 peoples are afflicted with chronic arsenicosis (Mukherjee *et al.*, 2006). More than 10 ppb of arsenic is still being consumed for lack of alternate access (Polizzotto *et al.*, 2008). Findings have suggested an association of high levels of arsenic in drinking water with significant increase in the risk of cancers for affected population (Bates *et al.*, 1992). Arsenite ($AsO_3^{3-}$) and arsenate ($AsO_4^{3-}$), referred to $As^{+3}$ and $As^{+5}$ respectively are forms of arsenic are commonly present in natural water i.e. (Prasad *et al.*, 2011). Pentavalent species is predominant in surface water and shallow ground water while trivalent arsenite is found in deep-seated groundwater (Nickson *et al.*, 1998). In the environment, iron plays an important role in contaminant mobility, sorption and breakdown due to its role as an electron donor (Bendell-Young and Harvey

1992). Zero valent iron nanoparticle (nZVI) are important candidate for remediation of contaminated sites as iron is inexpensive, non-toxic and environmentally compatible (Li *et al.,* 2006). Iron particle can be prepared by several methods e.g. sodium borohydride ($NaBH_4$) (Song and Carraway 2005)pentachloroethane (PCA ethylene glycol (Raveendran *et al*., 2003) and corbothermal synthesis (Allabaksh et al., 2010). Iron particles tend to rapidly agglomerate to form larger aggregates due to Vander Waals and magnetic forces, rendering them undeliverable to the targeted contaminant locations.(Kim *et al*., 2008). The stabilizer molecules are used for repulsions to outweigh the attractive Vander Waals and magnetic forces. Biodegradable polymers or surfactants to cap these nanoparticles that prevents them from aggregation. Thus nZVI is surface-modified (SM) with polymers or surfactants to increase its migration and therefore proximity to the pollutant materials (Saleh *et al.,* 2008).

Iron particles have been prepared using different biological agents e.g. extract of plant leaves and tea extract (poly phenols) was chosen as a reducing agent and stabilizing agent for nZVI preparation because poly phenols are biodegradable (nontoxic) and water soluble (Nadagouda et al., 2010)or MTS, (3-(4,5-dimethylthiazol-2-yl. Polyphenol, caffeine, and/or a natural solvent or surfactant yielded zerovalent nano scale iron has been employed to remove chromium from contaminated soil (Chrysochoou *et al*., 2012)which rebounded to 4.5 after the equivalent of 45 days and remained stable for the next equivalent 3 years. Metals concentrations in the effluent (Pb, Cr and Fe. Nanoscale zero-valent iron (NZVI) have been tested

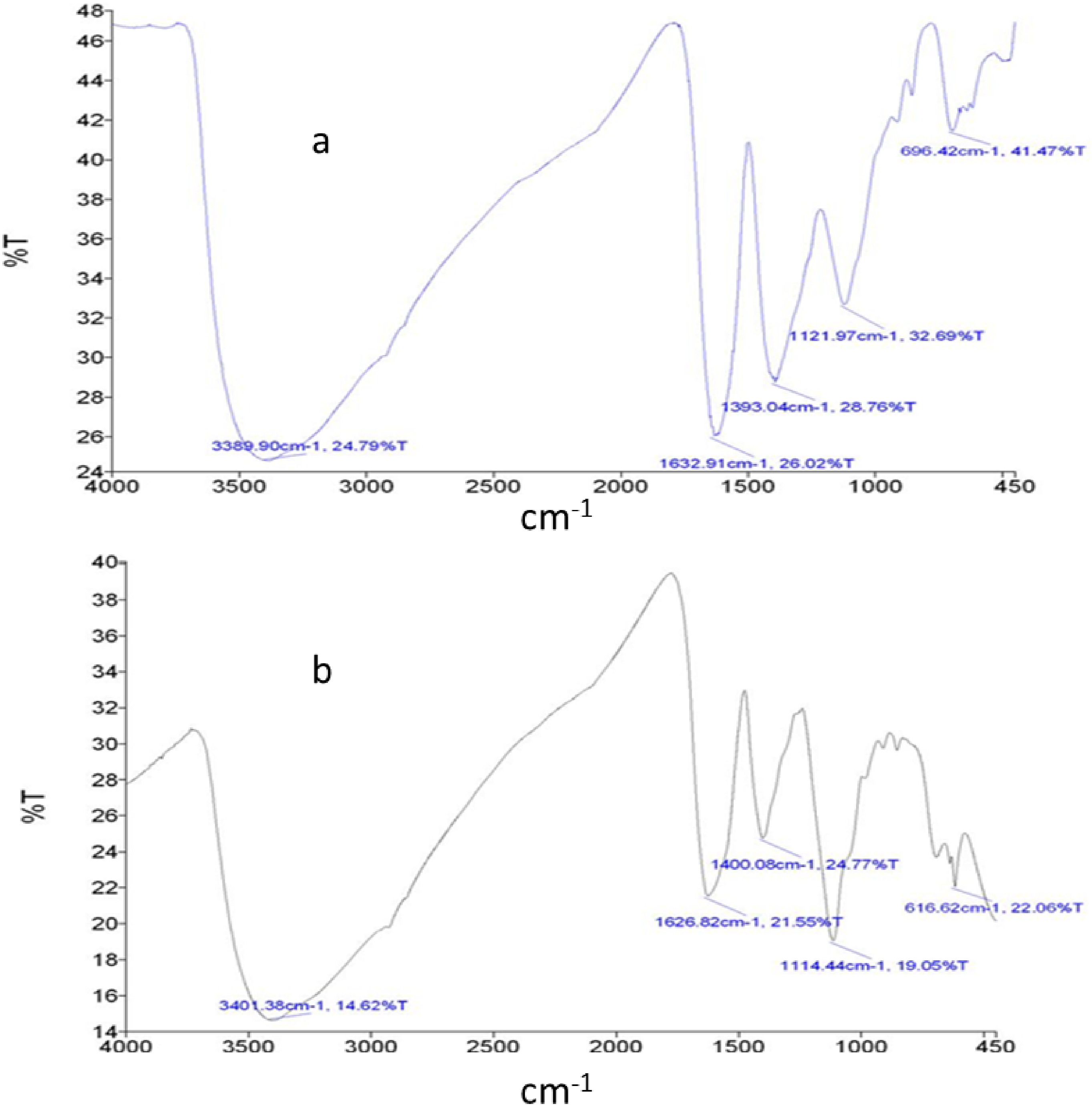

**Figure. 1** FT-IR spectra of fruit juice capped iron particles: a, after adsorption of As(III) ions: b.

for the removal of As(III), which is a highly toxic, mobile, and predominant arsenic species in anoxic groundwater (Kanel *et al*., 2005). Several biosorbents have been tested for removal of arsenic from aqueous solution e.g orange juice residue (Ghimire *et al.,* 2002), Zr (IV) loaded orange waste (Biswas *et al*., 2008)produced during juicing has been loaded with zirconium(IV, Zr-loaded diacetic acid chelating agent (Balaji et al., 2005)Nalpha diacetic acid functional groups. Arsenate ions strongly adsorbed in the pH range from 2 to 5, while arsenite was adsorbed between pH 7 and 10.5. The sorption mechanism is an additional complexation between arsenate or arsenite and Zr complex of LDA. Adsorption isotherm data could be well interpreted by Langmuir equation for As(V, iron-oxide coated polymeric material (Katsoyiannis and Zouboulis 2002) etc. The present study reports preparation of citrate capped iron particles using a green approach and its further uses as effective adsorbent for removal of As(III) and As(V) from aqueous solution.

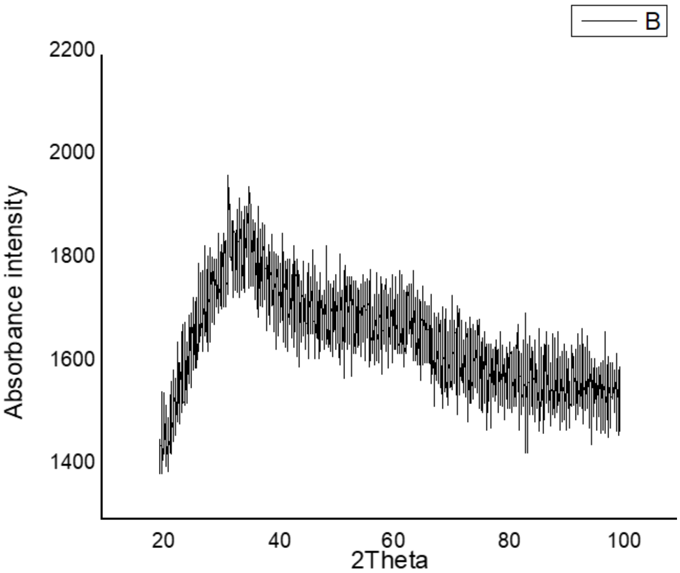

**Figure. 2.** XRD of citrate capped iron particles.

## MATERIAL AND METHODS

### Synthesis of citrate capped iron nanoparticles

Lemon (*Citrous limon*) fruit juice was used as capping and nucleating agent for the synthesis of Iron nanoparticles. In a typical synthesis of iron nanoparticles, requisite amount of ferric nitrate was added in to 20 ml of MiliQ water for final concentration to 10 mM. Fruit juice (5 ml) was added drop wise in to the solution of $Fe(NO_3)$. The content was shaken using rotatory orbital shaker operating at 200 rpm for 72 hrs. The incubation of the mixture was carried out at 35°C in dark condition. The reduction of iron ions was monitored by measurement of the UV-vis spectra using spectrophotometer. A spectral scanning analysis was carried out by measuring optical density of the content

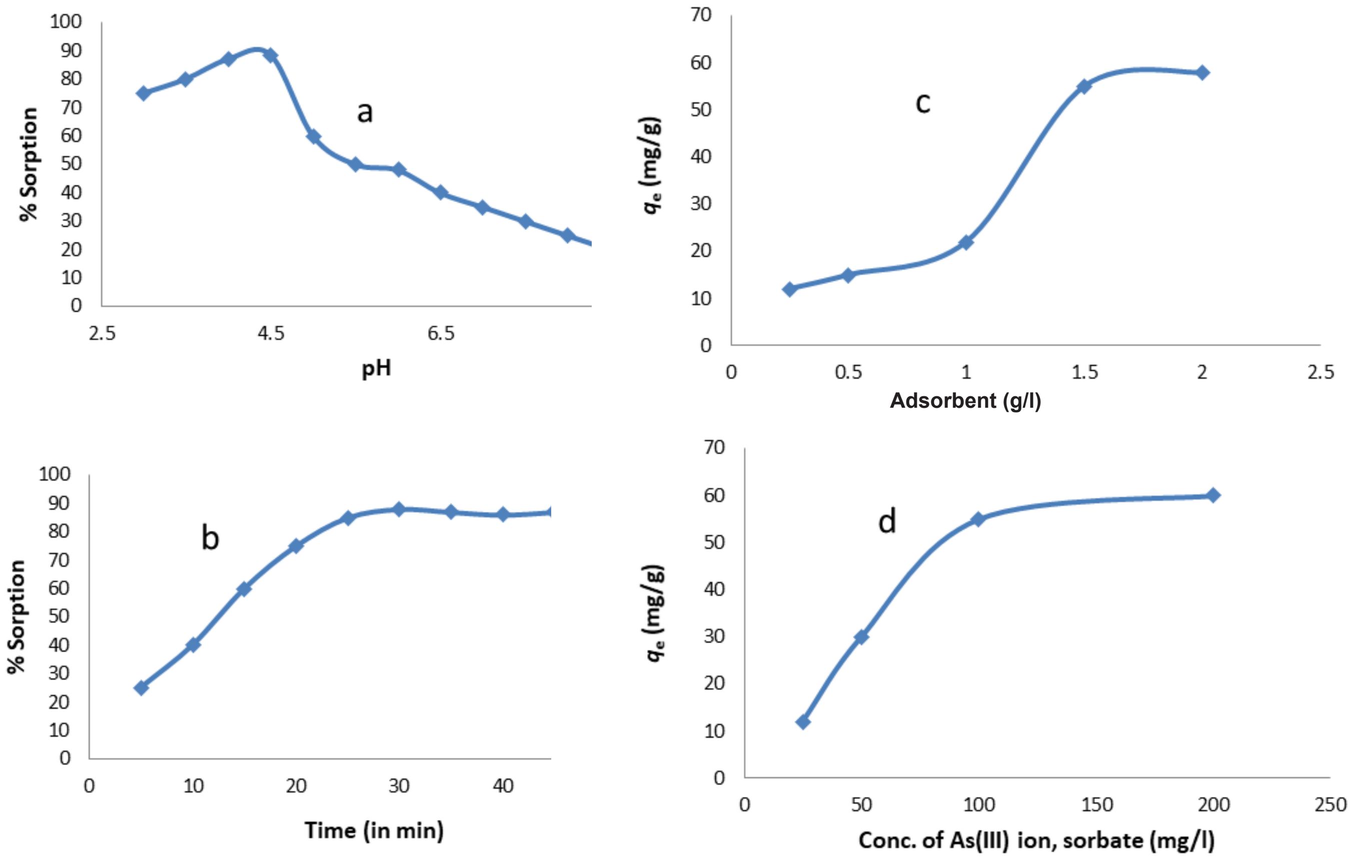

**Figure. 3** Effect of pH, where optimum pH was found to be 4.5 (a), Effect of contact time on sorption of arsenite onto sorbent (As(III): 100 mg/L, sorbate concentration: 2g/L at 30°C) (b). Effect of sorbent, (c) and effect of sorbate concentration (d) on over all sorption processes at 30°C.

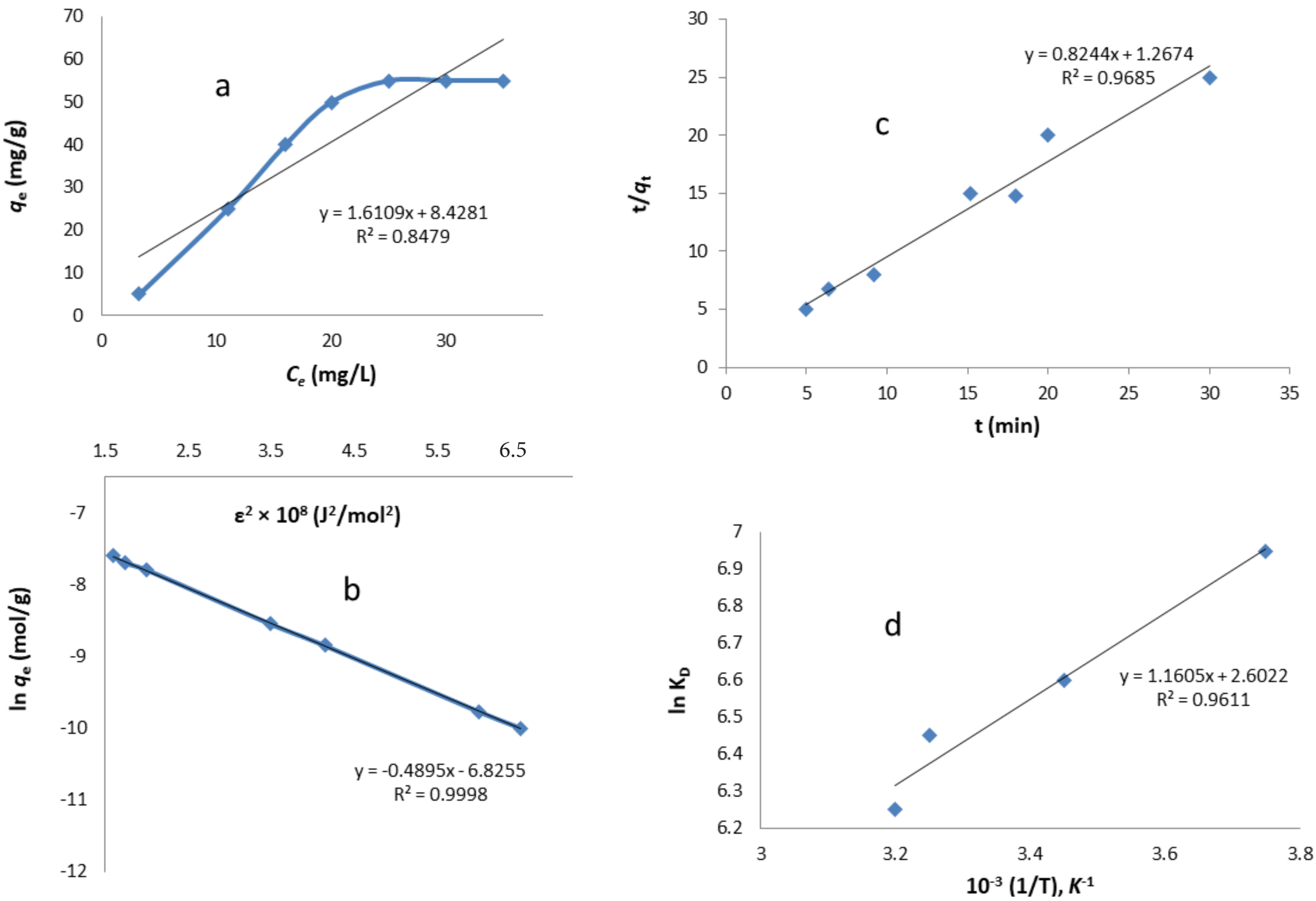

**Figure. 4** Langmiur isotherm plots for sorption of arsenite onto sorbent (a). D-R isotherm plots for sorption of arsenite (b) Second order sorption kinetic models for metals uptake (c) Plot of ln $K_D$ Vs. 1/T for the estimation of thermodynamic parameters for sorption of arsenite (d).

from wavelength, 350 to 700 nm. All the chemicals used in this experiment were procured from Sigma Chemicals Co. (St. Louis, USA) unless mentioned otherwise.

### Sample preparation XRD and FT-IR analysis

The citrus juice broth containing iron nanoparticles were subjected to centrifugation at 13000 rpm for 10 min. Impurities were removed by washing of pellet using deionized water. Pellet was dried using hot air oven and grounded in powder using mortar and pestle. The powder of synthesized nanoparticles obtained in this manner was subjected to XRD analysis. XRD data were collected using a Phillips PW1710 diffractometer with Co-Kα radiation operating at 40 KV, 35 mA in step scan mode, between 5 and 75° and with a counting time of 8 s per step. Disc of 100 mg KBr containing 1% powder was subjected to FT-IR analysis. A spectral scan analysis was carried out at wave number ranging from 400-4000 $cm^{-1}$ by using a FT-IR spectrometer (Perkin Elmer, Spectrum GX) with resolution of 0.15 $cm^{-1}$ to evaluate functional groups that might be involved in particles formation process.

### Adsorption optimization and arsenic estimation

The adsorption experiments were carried out at batch scale by adding different amount of sorbent in to 50 ml of either As(III) solution. *F*lasks containing mixture of sorbate and sorbent were placed over magnetic stirrer till reaching of equilibrium time. Aliquot of sample was collected over studied period of time of time and filtered using 0.22 µm filter paper. This sample was persevered at 4°C until quantification of arsenic was performed. The optimization for maximum sorption of arsenite and arsenate was carried out for contact time 5, 10, 15, 30, 45 and 60 min, sorbent concentration 0.25, 0.5, 1.0, 1.5, 2 g/L and sorbate concentration 25, 50, 100, 150, 200 mg/L, respectively. The percentage sorption of As(III) and ion was calculated as follows equation:

$$\text{Sorption }\% = \frac{C_i - C_f}{C_i} \times 100 \tag{1}$$

The sorption capacity, expressed as the amount of As(III) ions sorbed per gram of sorbent (mg/g) was calculated as follows:

$$q_e\,\frac{(mg)}{g} = \frac{(C_i - C_f)\,V}{M} \tag{2}$$

Where $C_i$ and $C_f$ are the initial and final concentrations of As(III) ions in the aqueous solution (mg/L), respectively. V is the volume (L) of test solution and M is the mass of sorbent (g) used.

Estimation of arsenic was performed with Atomic Absorption Spectrophotometer-Hydride Vapour Generation [AAS-HVG] (Shimadzu, Japan). Hydride generation (arsine) from total arsenic occurred after reduction of sample with HCl and $NaBH_4$ ($pH < 1$). Concentration of $NaBH_4$ and of NaOH was 6.0 g/L, 2.0 g/L, respectively (Prasad *et al.,* 2014).

## RESULT AND DISCUSSION

### FT-IR and XRD study

FT-IR analysis was carried out to identify the interaction among functional group of synthesized sorbent and As(III) ions. The fig. 1a shows the spectrum of the adsorbent before adsorption while, fig. 1b is the spectrum of sample containing As (III) ions. The peak at 3359 $cm^{-1}$ in control was shifted at 3401 after As(III) ion sorption, that indicated the involvement of primary aliphatic amine –NH stretch of biofunctionalised iron particles. Peak positioned at 1632 was found to be shifted at 1626. The prominent shift as well as decrease in intensity of peak indicated the involvement of primary amine –NH group after As(III) sorption. Similarly, peak at 1393 was shifted to 1400 $cm^{-1}$ indicated the involvement of aromatic nitro $–NO_2$ group. Peak of 1121 was found to be shifted at 1114 $cm^{-1}$, indicated the involvement of C-O stretch. Peak at 696 $cm^{-1}$ is shifted to 616 $cm^{-1}$, can be attributed to involvement of disulfide stretch (C-S) of citrate capped iron particle after As(III) adsorption.

The XRD diffractogram contained two prominent peaks and can be perfectly indexed to iron peaks with 2θ values of 36.0 and 53.5 and 62.26, corresponded to the crystal planes of 311, 400, and 511 of synthesized iron particles (fig. 2). Although inclusion of organic molecules suppresses the peak intensity, but diffraction pattern of prepared iron particles shows the shows the presence of inorganic material in the adsorbent. The observed planes in synthesized iron particles were matched with JCPDS (file no. 19-0629) confirming the presence of iron in synthesized particles.

### Effect of pH, contact time and adsorbent doses on adsorption

The effect of pH for sorption of As(III) ion is studied for determination of maximum removal of sorbate at different pH. The maximum adsorption was found to be at pH 4.5 (fig. 3a). At low pH arsenite ions are positively charged and attracted towards negatively charged citrate anchored over iron particles. Effect of contact time on sorption of arsenite on to citrate capped iron particle was studied using sorbent doses 2.0 g/L and sorbate 100 mg/L at room temperature (fig 3b). Sorption of arsenite (88.58%) was observed after mixing sorbate and sorbent for a period of 30 min. Figure 3c, shows the pattern for absorption of arsenite for different dose of sorbent. Maximum sorption occurred when 2 g/L of citrate capped iron particles were subjected to sorption studies. An increase in sorbate concentration to more than 200 mg/L, when sorbent doses was 2g/L, did not enhance over all sorption in batch sorption model (fig. 3d). Iron nanoparticles prepared using polyvinyl alcohol solution, removed 96% As(III) (Yang and Yan 2012). Iron nanoparticles coated over pottery granules removed 98% of total arsenic(Dong *et al*., 2009). Iron-doped activated carbons prepared by ferric chloride removed 94% of total arsenic (Fierro *et al*., 2009).

### Adsorption isotherms

The sorption isotherms were investigated using three equilibrium models namely Langmuir, Freundlich and Dubinin-Radushkevich (D-R) isotherm models. The Langmuir model hypothesizes a monolayer adsorption process, without any interaction between sorbed ions. Langmuir isotherm can be defined according to the following formula

$$q_e = \frac{q_m K_L C_e}{1+K_L C_e} \tag{3}$$

where $q_e$ is the equilibrium metal ion concentration on the adsorbent (mg/g), $C_e$ is the equilibrium metal ion concentration in the solution (mg/L), $q_m$ is the monolayer biosorption capacity of the adsorbent (mg/g) and $K_L$ is the Langmuir biosorption constant (L/mg), relating the free energy of sorption. A non-linear relationship of As(III) ions were sorbed per unit sorbent. The coefficients of determination ($R^2$) was found to be 0.847 for As(III) sorption (fig. 4a). The maximum sorption capacity ($q_m$) of the sorbent was found to be 55.53 mg/g while $K_L$ value was calculated as 0.0195 L/mg for As(III) ions. The Freundlich model assumes a heterogeneous adsorption surface and active sites with different energy. This isotherm can be explained by following formula:

$$qe = K_f C_e^{1/n} \tag{4}$$

Where $K_f$ is a constant relating the sorption capacity and $1/n$ is an empirical parameter relating to sorption intensity which varies with the heterogeneity of the material. A Freundlich isotherm was obtained by plotting $q_e$ Vs. $C_e$ values, which showed a nonlinear relationship between two. The $R^2$ value, 0.825suggested that the Freundlich model was not able to describe the relationship between the amounts of sorbed As(III) ions adequately to their equilibrium concentration in the solution. The Langmuir isotherm model best fitted the equilibrium with higher $R^2$ value than Freundlich model. Physical or chemical nature of sorption processes were examined by analyzing the equilibrium data using D-R isotherm model. The linear form of D-R isotherm model is presented by following equation:

$$\ln q_e A = \ln q_m - \beta\varepsilon^2 \tag{5}$$

where β is the activity coefficient related to mean sorption energy ($mol^2/J^2$) and ε is the Polanyi potential $\varepsilon = RT \ln (1 + 1/C_e)$. The D-R isotherm model well fitted the equilibrium data since the $R^2$ value was found to be 0.999 for As(III) sorption (fig. 4b). The sorption mean free energy (E; kJ/mol) gives physical or chemical nature of adsorption. The mean sorption energy (E; kJ/mol) is expressed as follows:

$$E = \frac{1}{\sqrt{2\beta}} \tag{6}$$

The adsorption process is regarded as a chemical in nature when values lie between 8 and 16 kJ/mol, similarly adsorption is physical in nature when E value is less than 8 kJ/mol. The mean sorption energy was calculated as 12.82 for As(III) ions suggested chemisorptions type of sorption.

## Adsorption kinetic models

Pseudo-first order and Pseudo-second order kinetic models were used to analyze the sorption rate of As(III) ions on to citrate capped ion particles. The pseudo-first order rate equation is given as;

$$\log(q_e - q_t) = \frac{\log q_e - k_t t}{2.303} \tag{7}$$

where $q_e$ (mg/g) is the amount of metal ions sorbed at equilibrium and $q_t$ is the amount of metal sorbed at any time (mg/g) and $k_1$ is the rate constant of the equation ($min^{-1}$). The biosorption rate constant $k_1$can be can be determined experimentally by plotting, log ($q_e$-$q_t$) versus t. Experimental data were also tested by pseudo-second-order equation

$$\frac{t}{q_t} = \frac{1}{k_2 q^2} \quad \left(\frac{1}{q_e}\right)t \tag{8}$$

Where $k_2$ is the equilibrium rate constant (g/mg/min). The values of correlation coefficient of pseudo-second order model was found to be 0.968 for As(III), higher than pseudo-first-order model i.e. 0.875 (fig. 4c). The pseudo-second order model can explain the biosorption kinetic behaviour of As(III) onto sorbent satisfactorily with a good correlation coefficient.

## Biosorption thermodynamics

Thermodynamic parameters including the change in free energy (ΔG°), enthalpy (ΔH°) and entropy (ΔS°) have been studied. The change in free energy (ΔG°) was calculated from following equations:

$$G^\circ = - RT\ln K_D \tag{9}$$

where, R is the universal gas constant (8.314 J/mol K), T is temperature (K) and $K_D$ ($q_e/C_e$) is the distribution coefficient .The enthalpy (ΔH°) and entropy (ΔS°) parameters were estimated from the following equation:

$$\text{In } K_D = \left(\frac{\Delta S^o}{T}\right) - \left(\frac{\Delta H^o}{RT}\right) \tag{10}$$

The slope and intercept of the plot of ln $K_D$ versus $1/T$ gives value of ΔH° and ΔS° Fig. 7d. Gibbs free energy change (ΔG°) was found to be -17.93, -17.89, -16.88 and −16.49 kJ/mol for As(III) adsorption at 20, 30, 40 and 50°C, respectively (fig.4d). The negative ΔG° values indicated thermodynamically feasible and spontaneous nature of the sorption. The decrease in ΔG° values with increase in temperature suggested a lesser feasibility of sorption at high temperatures. The enthalpy of biosorption ΔH° parameter was found to be −24.26 As(III) sorption. The negative ΔH° indicates the exothermic nature of sorption at 20 to 50°C. The enthalpy or the heat of sorption ranging from 2.1 to 20.9 KJ/mol corresponds to physical sorption where as ranging from 20.9 to 418 KJ/mol is regarded as chemical sorption. Therefore the ΔH° value suggested that the sorption process of As(III) occurred due to chemisorptions. The ΔS° parameter was found to be −17.34J/mol K for As(III) sorption, suggested a decrease in the randomness at the solid/solution interface during the sorption process.

## CONCLUSIONS

Citrate capped colloidal iron particles were synthesized and used as an adsorbent for removal of As(III) ions from aqueous solution. XRD analysis revealed the particle nature of synthesized material. In batch sorption studies maximum sorption capacity of prepared sorbent was found to be 55.53 mg/g for As(III) ions at optimal experimental conditions. FT-IR analysis indicated the involvement of functional groups ($-NH_2$, $-NO_2$, -CO, and –CS) in arsenite sorption processes. The sorption pattern fitted well with the Langmiur model and followed pseudo-second-order kinetics. The mean free energy values derived from the D-R isotherm model indicated that the sorption primarily occurred due to chemisorptions. This low citrate capped iron particle can be used for removal of arsenic from contaminated water.

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