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Iron Oxide Nanoparticles Assisted Arsenic, Mercury, And Cadmium Removal From Aqueous Solution

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Abstract – Iron oxide nanoparticles (IONPs) can play a significant role in the removal of heavy metal from contaminated solution .Aresnite ,As(III) highly toxic,mobile and predominant species in arsenic contaminated water IONPs have been synthesized, characterized and tested for the removal of As(III) from arsenic contaminated water. In this work , we synthesized IONPs using a chemical method involving dispersion of Fe^{3+} ions through polymer molecules of poly vinyl alcohol (PVA) in aqueous medium X-ray diffraction (XRD) analysis confirmed the formation of a single phase rhomobohrdral crystal structure, the size of nanoparticles was 50-100 nm. Transmission electron microscopic images corborate the result of IONPs (50-100 nm) . The adsorption of As(III),Hg(II) and Cd(II) On (IONPs) was studied using four parameters contact time, concentration, dose of IONPs and pH Adsorption equilibrium was achieved in 100 min and maximum removal of As(III) was obtained at pH (6-6.5) . The isotherm analysis indicated that the adsorption data better filted to the Langmuir isotherm model. The maximum adsorption capacities were 101.2, 73.10 and 60.05 mg g^{-1} for As(III), Hg(II) and Cd(II), respectively.

Keywords – Iron Oxide, Nanoparticles, Arsenic, Mercury, Cadmium.

I. INTRODUCTION

Nanoparticles are defiened as any particle with a size less than 100 nm, but like most things in particle technology, a more thorough discussion is required to achieve an unambiguous and complete response. The current agreement among the standard group is that the scale from 1-100 nm defines the size range of nanoparticles. Below 1nm may be excluded in order to avoid calling clusters of atoms a particle, but the literature contains references to particles 1 nm. Since particles have three dimensions. the ASTM standard defines two or three dimensions must be between 1-100 nm. This applies for nanotubes with a diameter of 10 nm, but a length 100 nm[1].

In nanotechnology, a particle is defined as a small object that behaves as a whole unit with respect to its transport and properties[2]. Particles are further classified according to diameter. Coarse particles cover a range between 2,500 and 10,000 nanometers. Fine particles are sized between 100 and 2,500 nanometers. Ultrafine particles[3]. or nanoparticles, are between 1and100 nanometers in size. The reason for this double name of the same object is that during the 1970-80_s, When the first thorough fundamental studies with “nanoparticles” were underway in the USA (by Grangvist and Buhrman) they were called “ultrafine particles”(UFP). Nanoparticles may or may not exhibit size-related properties that differ significantly from those observed in fine particles or bulk materials [4].

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Naturally occurring nanoparticals also include ultrafine sand grains of mineral origin(e.g oxide, carbonates). In addition to commercial produced nanopartical, many are uninfentionally created by the nocombustion of diesel fuel ultrafine particle [5].A large amount of sea salt aerosols are emitted from seas and oceane around the world [6].These aerosols are formed by water evaporation and water produced water drops are ejected into the atmosphere[7]. Their size range from 100nm to several microns[8]. An example of this phenomenon is Lake of Michigan that rests in a limestone basin ,the water containing high levels of calucium carbonate. During most of the year the calucium carbonate remains dissolved in the cold water ,but at the end of summer the water temperature increase, Iowering the solubility of calucium carbonate [9] .As result, the calucium carbonate may precipitate out of the water .Nanoparticles, can also form in bodies of water through precipitation ,as a result of temperature changes and evaporation [10].As a combustion product, tobacco is composed of nanolparticles with size ranging from around 10nm up to 700nm,with a maximum located around 150 nm[The environmental tobacco smoke has a very complex composition ,with more than 100,000 chemical components and compounds of the environmental tobacco smoke which is known to be toxic, both due to some of its gas phases as well as nanoparticals.[11]

II. MATERIAL AND METHOD

2.1-Glasswares:

All glass ware,used for this research ,were washed with (**D.I.W**) and 10‰ HNO_3 many times (Graduated,flasks, Pipettes, Beakers, Crucibles Erlemeyer flask with ground –plastic stoppers).

2.2Instruments**:**

Shaker(Edmund Buhllr,k1-2 Germany), Digital pH meter(WTW525,Germany), Graphit furnace Atomic absorption Spectrometer Mercury analysis (Gf-AAS)(AA-6800,shimadzu,Japan), Furnace(Metller Toledo AB 104-s-Germany), Analytical balance(Memner 600-Germany- (0.00001/g), Furnace(Qallenhamb,Hotspot temperature (0-1000C)Germany), High Resolution Scanning Microscope(Hitatchi Namber 5400E), TEM(Oxford systems).

2.3Chemicals

All chemical used in this study were of high purity: PVA(Poly vinyl alcohol)(BDH chemicals Ltd poole England99.9%), $Fe(NO_3)_2$ (Riedel-de-H aen98.5%),(As_2O_3)(Rieddel-de-Haen98.5%), $Hg(NO_3)_2H_2O$(Riedel-de-Haen98.5%), Soduim hydroxide (Merck,Germany Analytical grade), $Cd(NO_3)_2H_2O$(Riedel-de-Haen98.5%), Hydro chloric acid(Sigma-Aldrich USA37%),

2.4 Preparation of Fe_2O_3 nanoparticles.[sol-gel] technique

In 250ml PVA(7.5g/L), in Erlenmeyer flask, 25 ml of $Fe(NO_3)_2$ was added and mixed then few drops of sucrose was added. The mixture was heated up to the boiling , on electrical heater, for about one hour. After that the mixture was put on magnetic stirrer for an hour.The precipitate was filtered and washed with distilled water then left to dry in air for 24 hours. The dry precipitate was further dried in drying oven, at 60-80 ^oC, for an hour .The resulted granular material was darkblack in color. Finally, the powder treated in burning furnace, at 300 and 400^oC at diffemt times. The nano-material α- Fe_2O_3 is a powder and appears in different colors according to the burning heat as the following:

When the precipitate (sludge)was burned at 400 ^oC for two hours,the product was red in color,but when the precipitate (sludge)was burned at 300^oC for three hours the color was gray.

When the weight ratio of the reactants used in the preparation of sludge was changed to 100ml(PVA)+25ml($Fe(NO_3)_2$+200ml sucrose, the product (sludge) had never been formed .

2.5 The effect of Contact time:

To each of seven Erlenmeyer flasks contain 20 ml of 1ppm As(III)solutions, 0.1g Fe_2O_3 of nano-particle was added and shaken, using mechanical shaker (vibrator).Thes samples were taken at different intervals of contact time 0,20,40,60,80,120, min.The suspension was filtered .The concentration of As in solution C_t was directly determined by (GF-AAS) at the end of each time interval .The results was recorded in table (1).

Table (1) The effect of contact time on adsorption of As(III) onFe_2O_3nano-particles adsorption of wt=0.1g in c_0=1000ppb pH=6, contact time=100min.

1ppmAs	**S_1**	**S_2**	**S_3**	**S_4**	**S_5**	**S_6**	**S_7**
Time(Min)	**O**	**20**	**40**	**60**	**80**	**100**	**120**

2.6 Effete of pH:

Eight Erlenmeyer flasks each one contain 20 ml of 1ppm As(III) solutions, 0.1g Fe_2O_3 of nano-particle added and to each flask the pH was adjusted to ,2,3,,4 ,5 ,6, 7,8, 9, using HCL or NaOH. The mixture was shaken, using mechanical shaker (vibrator) for one hour. The concentration of As in solution C_t was directly determined by (GF-AAS) at different pH valces.The mean of the results was recorded in table- (2) All neplicated samples were (3) for each concentration.

Table (2)Effect of pH on adsorption of 1ppm As(III) on Fe_2O_3 nanoparticles surface adsorption of wt=0.1g in c_0=1000ppb,pH=6,contact time=100min.

No.	**pH**	**V-100ml**	**V-100ml**	**V-100ml**
1	2	2.43	2.40	2.44
2	3	3.20	3.04	3.17
3	4	3.96	4.07	4.03
4	5	5.38	5.44	4.99
5	6	6.30	6.35	5.86
6	7	7.24	7.03	7.08
7	8	8.18	8.41	8.40
8	9	8.99	9.03	8.96

Table (3) Effect of pH on adsorption of 1ppm of As on Fe_2O_3 nanoparticles for different samples adsorption of wt=0.1g in c_0=1000ppb,pH=6,contact time=100min.

No	**pH**	**V-100ml**	**V-100ml**	**V-100ml**
1	2	r_1	r_2	r_3
2	3	r_1	r_2	r_3
3	4	r_1	r_2	r_3
4	5	r_1	r_2	r_3
5	6	r_1	r_2	r_3
6	7	r_1	r_2	r_3
7	8	r_1	r_2	r_3
8	9	r_1	r_2	r_3

Table (4) Dilution sequence of 1 ppm (final volume 100 ml) adsorption of wt=0.1g in c_0=1000ppb,pH=6,contact time =100min.

NO	Ml	Concentration ppm
1	1	0.01
2	5	0.05
3	8	0.08
4	10	0.1
5	20	0.2
6	40	0.4
7	50	0.5
8	60	0.6

To each of eight Erlenmeyer flask contain 20 ml of solutions of different concentrations C_o (0.01 ,0.05,0.08,,0.2,0.4,0.5,0.6),0.1g Fe_2O_3 of nano-particle was added .the pH was adjusted to 6 .and was shaken, using mechanical vibrator for one hour. The concentration of As in solution C_t was directly determined by (GF-AAS)for each concentration.The results was recorded in table (4) All the samples were tripled to get the accurate results.

Table (5) Effect As concentration on adsorption of As on Fe_2O_3 nanoparticles surface modified by oxine adsorption of wt=0.1g in c_0=1000ppb,pH=6,contact time =100min.

Concentration	pH20 ml	pH 20 ml	pH 20 ml
0.01 ppm	6.05	6.01	6.05
0.05 ppm	6.09	6.07	6.07
0.08 ppm	5.97	6.03	6.03
0.1 Ppm	6.01	6.01	6.03
0.2 ppm	5.98	5.96	6.09
o.4 ppm	5.93	5.93	5.60
0.5 ppm	5.88	5.74	5.72
0.6 ppm	5.70	5.68	5.78

The pH 0f 6 samples (n=6) was meacred with sludge.

Table (6) Dilution sequence of 100 ppm (final volume 100 ml) adsorption of wt=0.1g in c_0=1000ppb,pH=6,contact time=100min.

No	Ml of 100ppm	Concentration of Hg
1	0.5	0.5
2	2.5	2.5
3	1	1
4	5	5
5	10	10
6	25	25

Table(7) Mixture of 10ml 0,5ppm As +10ml of Hg and pH of the solution: adsorption of wt=0.1g in c_0=1000ppb ,pH=6,contact time=100min.

No	As ppm	Hg ppm	pH	pH
1	0.5	0.5	6.23	6.34
2	0.5	2.5	6.34	5.86
3	0.5	1	6.18	6.11
4	0.5	5	6.24	6.22
5	0.5	10	6.32	6.30
6	0.5	25	6.32	5.88

1000×V=100×100

V=100×100⁄1000=100 ML

Table (8) Dilution sequence of 100 ppm (final volume 100ml): adsorption of wt=0.1g in c_0=1000ppb,pH=6,contact time=100min.

No	Ml of 100ppm	Concentration of Cd
1	0.5	0.5
2	2.5	2.5
3	1	1
4	5	5
5	10	10
6	25	25

Table (9) Mixture of 10ml o 0,5ppmf As +10ml ofCd and pH of the solution: adsorption of wt=0.1g in c_0=1000ppb ,pH=6,contact time=100min.

No	As ppm	Cd ppm	pH	pH
1	0.5	0.5	6.17	6.29
2	0.5	2.5	6.30	6.25
3	0.5	1	6.16	5.96
4	0.5	5	5.87	5.98
5	0.5	10	6.29	6.05
6	0.5	25	6.19	5.96

III. RESULT AND DISCUSSION

3.1 Charteization of prepared Fe_2O_3 nanoparticles.

XRD and TEM,SEM were used to characterize Fe_2O_3 nanoparticles for preparation of Fe_2O_3 nanoparticles that the powder was nanoscale. According to the experimental section (3-6-1) and (3-6-2).

3.1.1 XRD

XRD pattern were recorded on prepared and calcined samples using instrument as described in section (1-23).the 2$\boldsymbol{\theta}$ scans were recorded at several resolution using Cukα radiation of wave length 1.54A in the rang 20-80 with 0.05 step size. The recorded patterns were analyzed using jade soft ware to determine the peak position, width and intensity. Full width at half maxima (FWHM)data was analyzed by schemers formula.

$$\mathbf{T}=\frac{0.9\lambda}{b\cos\theta}$$

Where $\boldsymbol{\lambda}$ is x-ray wave length,B is peak width and $\boldsymbol{\theta}$ Braggs angle K.

Fig (1) shows the (XRD) pattern for Fe_2O_3 nano particles synthesized via this method .The prepared nanoparticles showed that (Fe_2O_3) has tetragonal symmetry (simple tetragonal) and belongs to D_{4h}.

-14/and space group with lattice constants of a=0.348nm,C=0.952nm and $\frac{c}{a}$=0.73nm.The crystal structure of the obtained powder was indicated in figure (4).All the diffraction peaks can be indexcd as the pure phase for Fe_2O_3 nanoparticles. The obtained powder has high purity of anatase phase. No charities peak impurity of impurities such as $Fe(NO_3)_2$ was detected. The average crystalline size of nanoparticles were determine to be (70 nm) utilizing sherrer formula (T=$\frac{k\lambda}{b\cos\theta}$),where T is the mean crystallize size of the powder,is the wavelength of CuKα,J 1.5405A,B is the full width at half maximum (FWHM) intensity of the peak in radian. $\boldsymbol{\theta}$ is Bragys diffraction anagle and K is a constant usually equal to-0.9.Thus peaks are in good agreement with rutile phase (JCPDS) card No-21-1267.

$$T=\frac{0.9x\lambda}{B\cos\theta}=\frac{0.9x1.54056}{0.6469x7.93x10-3}$$

=700A =70nm

B_a measured breath of balk material about maximum intensity B_0 measured breath of observed material in half maxima.

[$B=(B^2-B^2)^{1/2}$]

1-The XRD date peak positions are the same for all the samples.

2-The phases formed are pure.

3-All the samples show marked peak broadening confirming that the samples are indeed nanostructures, the size analysis was done

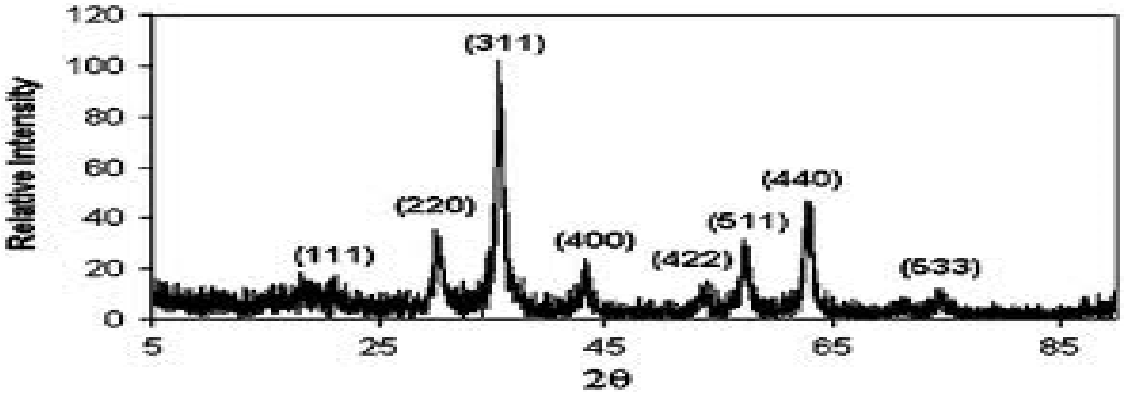


Fig (1) XRD of $\propto Fe_2O_3$ *nano particals*

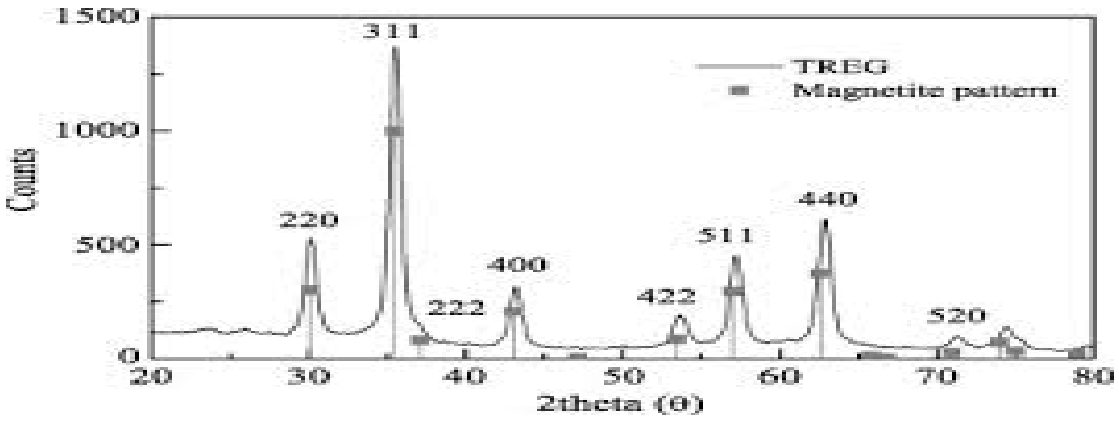


Fig (2) XRD of $\propto Fe_2O_3$ *nano particals*

3.1.2 SEM

Scanning electron microscope shows that the dimensions of the nanoparticles were between 30-70 nm Fig (3) and Fig(4) as shown in

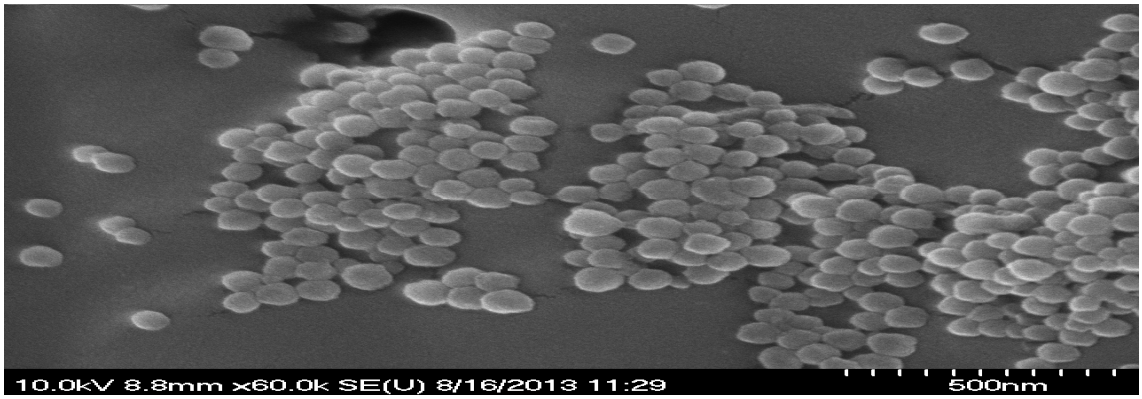


Figure (3) SEM Image of $[\propto Fe_2O_3$ *nano particals*$]$

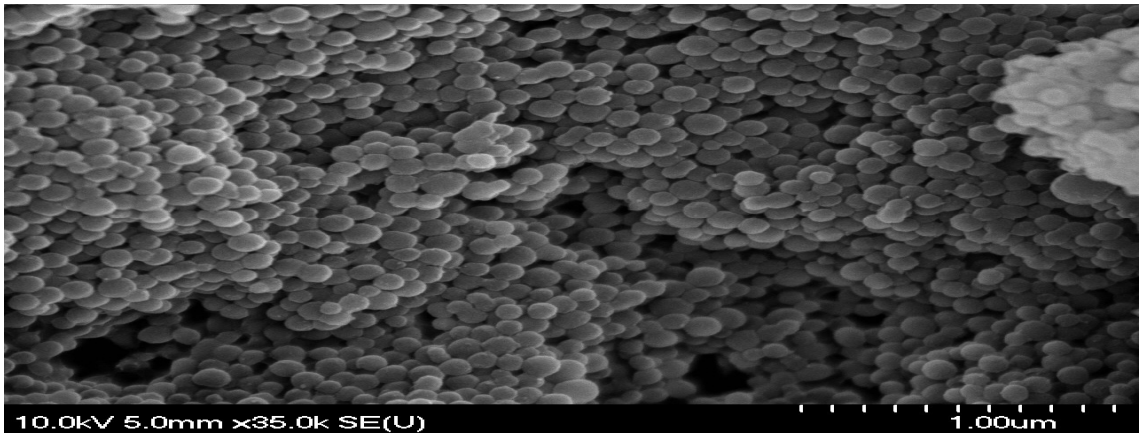


Figure (4) SEM Image of $[\propto Fe_2O_3$ *nano particals*$]$

3.1.3 TEM

TEM shows the dimensions of Fe_2O_3 nanoparticles were between 1-100 nm,as shown in figure (5).

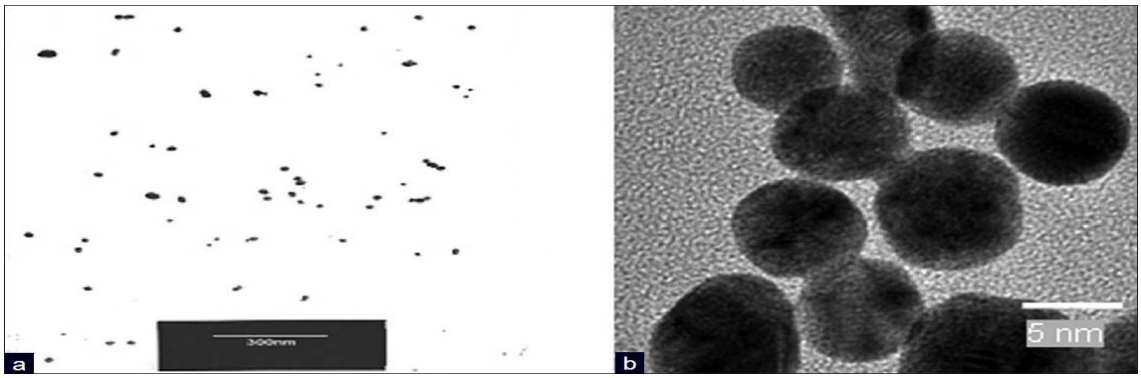


Fig (5) TEM Image of $\propto Fe_2O_3$ nanoparticles

3.2 Adsorption Process

Contaminated aqueous solution were artificially prepared from stock solution of As(III) ,Hg(II) and Cd(II).The weight of Fe_2O_3 nanoparticles (0.10 g) was meaured in erlenmeyer tubes.The contauminated aqueous solution were added to each tube.The time measuring at the moment the contaminated water is added.The adsorption behauior was studied under four different conditions of :

1-Contact time

2-Different pH Values

3-Different metal Ion concentrations

4-Different dosage of Fe_2O_3 nanoparticles.

3.2.1 The effect of contact time

The effect of conctat time was examined to find suitable contact time at which the nanoparticles are saturated and the adsorption is at equilibrium

The artificially contaminated water (20 ml) were treated with 0.1 g of Fe_2O_3 nanoparticles in room temperatare (21.5+_ 1^0C) after different periods of contact times, the times of the treatments were:20 min,40min ,60min,80min,100min,120min.It was found that 100 min was the best period of time in which maximam percent removal was reached, so it was used during the study of other behaviours.

Table (10): Mean value of final concentration, C_f, of arsenic as a function of time, wt=0.1g,contact time 100 Min , pH =6.5, C_0=1000ppb

Time	As pbb	% Removel
20	174.295	82.57%
40	271.86	85.4%
60	117.265	88.27%
80	74.30	92.57%
100	18.64	98.14%
120	10.18	98.16%

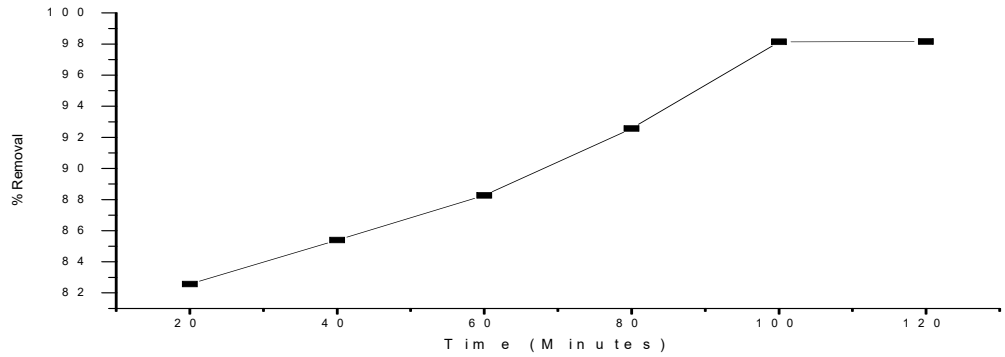


Fig (6) Adsorption of As on nano iron oxide surface

3.2.2 The effect of pH

The pH was adjusted by adding 0.1M HNO_3 and 0.1M NaOH Solutions into the ultra pure deionized water. Eight solutions of different pH (from pH 2 to pH9) were prepared. the solutions were treated for 100nm with 0.1 g of Fe_2O_3 nanoparticles in room Temperature and at pH 6.5.the concentration of metals was measured by(GFAAS) and ICP-MS before and after the treatment .the percent removal (%) of heavy metals was calculated Three triails for each PH conducted .

Table (11) mean value of final concentration ,C_f of arsenic as a function of pH, wt=0.1g, contact time 100Min, Concentration =1000ppb

Ph	Ppb of As (III)	% Removel
2	173.85	65%
3	202.51	71%
4	24.25	79%
5	5.995	97.57%
6	15.945	99.65%
7	3.545	99.40 %
8	16.125	98.38%
9	10.33	98.96%

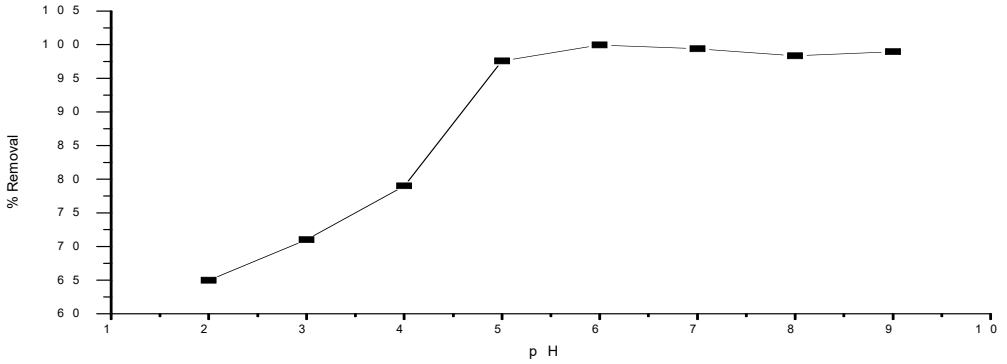


Fig (7) Adsorpion of As on nano iron oxide surface

4-2-3 The effect of concentration of target metal fon

The effect of different concentrations of metal Ions were prepared . All prepared solutions were treated for 100 min with 0.1g of Fe_2O_3 nanoparticles in room temperature and at pH 6.Three trials were made for each concentration.

Table (12)Mean value of final concentration ,C_f of arsenic as a function of C ,wt=0,1g , pH=6 , contact time=100min

C_i	C_f	Removel
10	1.8	82%
50	4.1	92%
80	1.54	98%
100	1.24	98.76%
200	1.16	99.42%
400	2.55	99.36%
500	0.62	99.98%
600	1.33	99.77%

C_i = Initial concentration

C_f = Final concentration

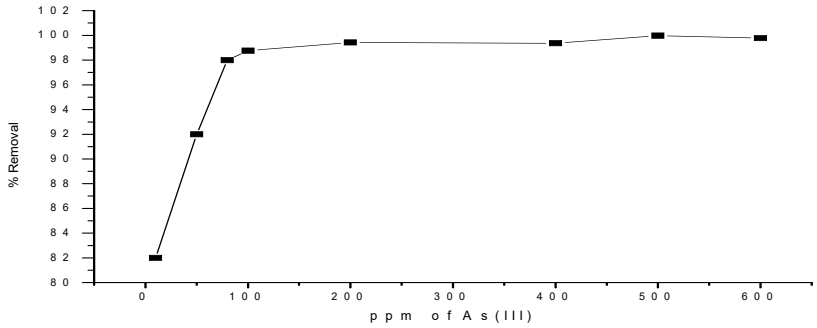


Fig (8) Adsorpion of As on nano iron oxide surface

3-2-4 The efferent of different dosage of Fe_2O_3 nanoparticles

Different mases of nanoparticles (0.1 g,0.2g,0.5 g) were used to Treat 20 ml of the metal Ion solution. Three trails for each mass were conducted.

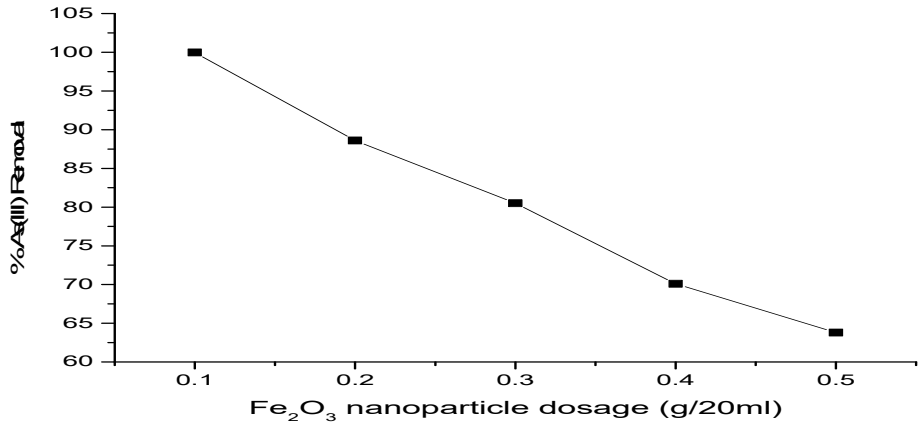


Fig (9) The effect of different dosage of Fe_2O_3 nanoparticles.

3.2.5 Effect of concentration of Hg

The results of adsorption of Hg on -Fe_2O_3 nanoparticles in the presence of 0.5 As shown in table (13) and figurer(10).

Table (13) Mean value of final concentration C_f of Hg a as a function of pH, wt= 0.1g, contact time 100 Min, Concentration =1000 ppb

C_i (Hg) ppm	C_f (Hg) ppm	% Removal
0.5	0.0650	87 %
1	0.0835	91.65 %
2.5	0.1580	93.76 %
5	0.3097	93.80 %
10	0.6350	93.65%
25	1.506	93.97 %

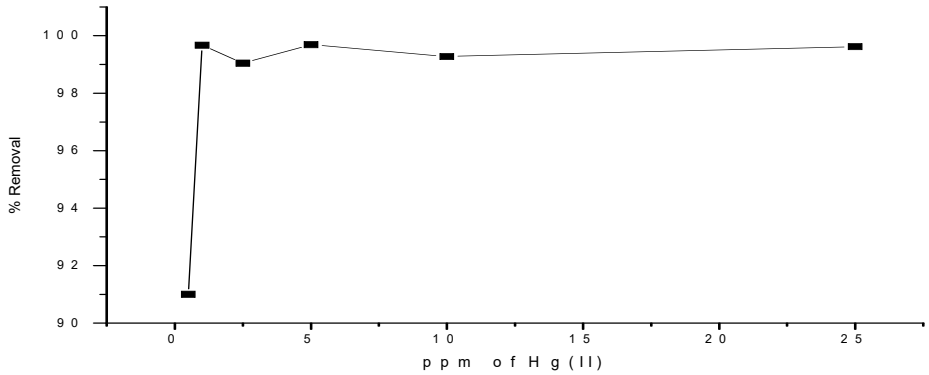


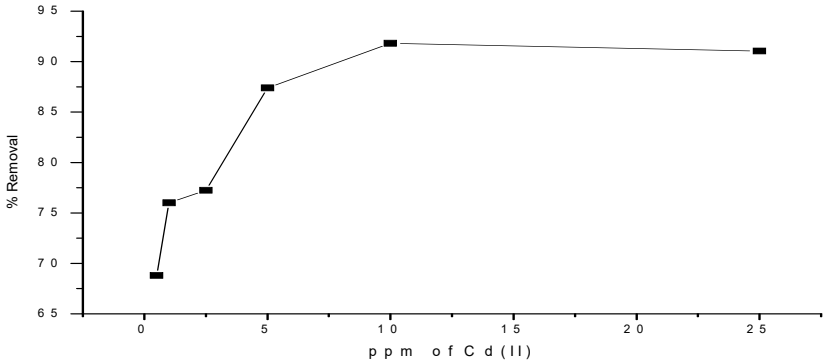
Fig (10) adsorption of Hg on-n ∝ Fe_2O_3 the presence of (As, Hg).

3.2.6 Effect of concentration of Cd

The results of effect of adsorption of Cd on n-Fe_2O_3 in the presence of 0.5 As shown in table (14) and figurer(11).

Table (14) Mean value of final concentration C_f of Cadium as a function of pH, wt=0.1g, contact time 100Min, Concentration =1000ppb

C_i	C_f	Removel
0.5	0.156	68.8 %
1	0.240	76 %
2.5	0.569	77.24 %
5	0.630	87.39 %
10	0.819	91.80 %
25	2.241	91.04 %



Fig(11) adsorption of Cd on-n ∝ Fe_2O_3 *in* the presence of(As, Cd).

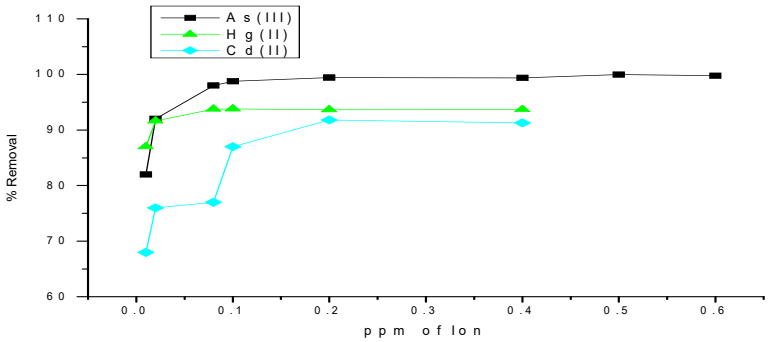


Fig (12) shows the relation between the concentration of metal ion (ppm) and **%** Removal for all the metal ions.

3.3 Adsorption capacities

The adsorption capacity is an important factor because it determines how much adsorbent is required to quantitatively concentrate the analyses a given solution. Study the adsorption capacity was mentioned in the paper recommended by Maguierira etal.

$Q_e=\frac{CO-CexVm}{Wt(g)x1000}$

Table (15) shows the relation between concentration of As(III) (ppb) and adsorption capacity Q_e(mg/g). Table (15) shows the mathematical Langmuir adsorption isotherm for adsorption of As(III) onto Fe_2O_3 nanoparticles modified oxine and figure (13).

Table (15) shows the mathematical Langmuir adsorption isotherm for adsorption of As(III) onto Fe_2O_3 nanopaticles modified oxime. It was found that the maximum adsorption capacity of Fe_2O_3 nanoparticles modified oxine for As (III) up to .

Slop=1.136,intercept=0.0145.

$$\frac{1}{Q_e} = \frac{1}{Q_0} + \frac{1}{bQ_0C_e}$$

$$\frac{1}{bQe} = \text{slop}$$

$$B = \frac{Intercept}{slop} = \frac{4.68x10-4}{0.1997} = 2.34x10^{-3}$$

$$0.199 = \frac{1}{4.68x10-4xQe}$$

Q_0 x 0.000468 =0.199

Q_0 = 427.136(mg/g)

Table (15) The relation between concentration of As^{+3}(mg/L) and adsorption capacity (Qe) (mg/g). as a function of pH, wt=0.1g, contact time 100Min, Concentration =1000ppb.

C(ppm)	Ce (ppm)	(C_O-Ce) x 20	$\frac{(C-Ce)x20}{0.1x10.0}$
0.01	0.0018	0.164	0.00164
0.05	0.004	0.920	0.0092
0.08	0.0016	1.568	0.0156
0.1	0.00124	1.975	0.0197
0.2	0.0016	3.968	0.0396
0.4	0.00256	7.948	0.0794
0.5	0.0001	9.998	0.0999
0.6	0.00138	11.972	0.1197

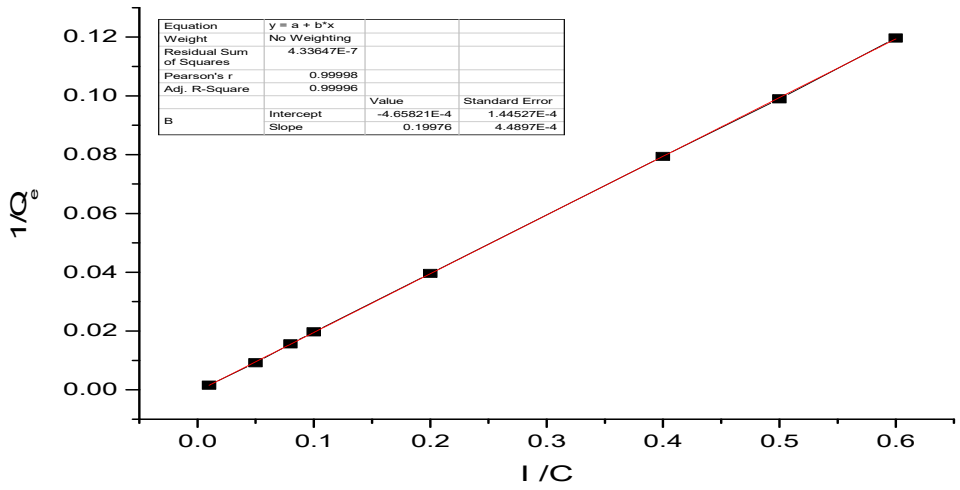


Fig (13) Langmuir adsorption isotherm for adsorption of As^{3+} onto ∝Fe_2O_3 nanoparticles.

3.4 Analysis of As(III) in real samples.

20 ml of samples was transferred into a 100ml erlemeyer flask and pH was adjusted to the desired value pH with diluted HNO_3 and NH_4OH.

Then 100mg of Fe_2O_3 nanoparticles were added and the mixture was shaken vigorously for 6h to facilitate adsorption of As(III) Ions onto its surface. After the solution were centrifuge, concentration of As(III).Ions in the solution were directly determined by ICP-MS and GFAAS using EPA3005A1992.And EPA6010C2007.Nte every samples was repeated three times (n=3) .

Table(16) the results of As in real water samples (n=2)

Sample	Concentration of As (III) mg/l	Sd(yE)
1	0.94	0.97
2	1.64	0.14
3	1.535	0.24
4	1.24	1.03
5	1.16	0.11
6	2.545	1.59
7	0.615	0.19
8	1.33	0.43

-Elution;

0.5M HNO_3 was used for sufficient complete elution. Therefore, the volume of 20 ml elute was added to filter paper after filtration the concentration the solution was measured using and GFAAS.

3.5 Stability and reproducibility tests;

To test stability and reproducibility,the new sorbent was subjected to several loading and elution bath operation.The sorption conditions are referenced according to the above experiments.The elution were carried out by shaking the sorbent with 20 ml 0f 0.5M HNO_3 together for 1h so as to ensure complete equilbartion .The sorbent showed better stability and reproducibility As(III).

IV. Conclusion

This study showed that the prepared α- Fe_2O_3 nanopaticles could be used as used an alternate to the conventional adsorbents for the removal of metal ions from wastewater with high removal efficiency within a very short time. The removal of As,Hg and Cd as a typical metal ion commonly present in wastewater ,by adsorption onto α- Fe_2O_3 nanoparticles was successfully accomplished. Adsorption was very rapid and equilibrium was achieved within As,Hg and Cd . It also showed that adsorption was highly dependent on the initial concentration of As , Hg ,Cd and initial pH value. Maximum removal efficiency was achieved at pH 5.5 at room temperature. The adsorption isotherms were also determined and were appropriately described by both Langmuir and Freundlich models ,with a better fitting to the Langmuir model than the Freundlich model. Therefore, α- Fe_2O_3 nanoparticles were recommended as fast, effective ,and inexpensive nano adsorbents for rapid removal and recovery of metal ions from contaminated water.

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