

Removal of Hg⁺⁺ from aqueous solution using TiO₂ nanoparticles

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Abstract

Nanometer-sized titanium dioxide prepared in the Lab chemically modified with 8-hydroxyquinolin and used for selective solid phase extraction processes, separation and preconcentration process of aluminum (III) from aqueous solutions prior to its determination by Inductively Coupled Plasma optical emission-Mass spectrometry(ICP-MS) and Graphite furnace atomic Absorption Spectrometry(GFAAS). The optimal conditions for the proposed solid phase extraction (SPE: 0,1g of TiO₂- modified oxine, 6h shaking time, pH 6.5). The experimental results was fitted well to Langmuir isotherm equation to determine the maximum adsorption capacity. The static maximum adsorption capacity was 69.013mg/g. The lowest concentration of aluminum (Al⁺³) was (30.0µg/L) and the highest concentration was (14585µg/L). The method was successfully applied to the determination of trace of aluminum in areas near to the factories in east of Tripoli.

Keywords

Adsorption • Nanop Particles • Aqueous Solution • Hg

Introduction

The environmental contamination has given rise to concern about the accumulation of heavy metal species in the human body, in order to access exposure to and absorption of trace elements, biological samples such as blood are often be analyzed. A deficiency in trace element such as the deviation of zinc from its normal levels in the blood is used for the early diagnosis of certain illnesses [1]. Cancer is a complicated multi-factorial, multifaceted, and multi-mechanistic illness and is the second leading cause of death in many developing countries. It has thus become a major public health problem in the world. The association of serum trace of elements and high cancer risk has been found in many studies [2]. In recent years, there has been an increased concern over the concentration of mercury in drinking and natural water due to its high toxicity and pollution to the environment and especially the aquatic system, mercury is leached from rocks and soil into the water system by natural processes, some of which are accelerated by human activities. The World Health Organization (WHO) recommends a limit of 1 mg/L of mercury in drinking water, which requires a very accurate, selective and sensitive method of measurement. Because of its simplicity, high sensitivity and relative freedom from interference, cold vapor atomic absorption spectrometry (CVAAS) has generally been used for the determination of mercury [3]. But on account of extremely low

concentration of mercury (mg/L) in water samples, the high salinity of seawater, and the growing awareness of environmental Mercury pollution, a separation and preconcentration step is required .

The most frequent used method for pre-concentration of mercury from natural water is co-precipitation, solvent extraction, electrochemical precipitation and solid phase extraction. Solid phase extraction of the immobilized organic compounds is attracting great interest because of its high enrichment capability and operation simplicity. Immobilization of organic ligands on the surface of an inorganic or organic solid support is usually aimed to modify the surface with certain target functional groups that can be exploited for further analytical uses [4]. Chelating sorbents can be obtained in a very simple manner by impregnation of inorganic or organic solid support with organic ligands . Several methods for pre-concentration of mercury utilizing such an approach have been devised ; among them are, dithizone immobilized on different base, sulfonic acid dithizone on anion exchanger; dithiocarbamate resin, thiomichelers keton, N-(2-pyridyl methyl) chitison (PMC), 2,3-dimercapto propane-1-sulfonate (DMPS), dithiocarbamate, solium diethyl dithiocarbamate and dithizone derivation [5] .

The use of nanoparticles for separation and preconcentration to analytical chemistry opens a new methodology that is quicker, simpler, and more precise than those old methods . The biggest advantage of this method consists of the fact that the desired

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Received: 02 December, 2021; Accepted: 16 December, 2021; Published: 23 December, 2021

materials are separated from solutions by a simple and compact process while fewer secondary wastes are produced. Other advantages are a big active surface area for amass given by particles; the ability to process solution that contains suspended solid; avoidance of channeling effects, which are common in packed beds; and the capacity to immobilize particles in a specific position of the column by application of external field gradients [6,7,8].

The Objectives of the study:

1. Preparation of TiO₂ Nanoparticles using Sol-Gel method.
2. Characterization of TiO₂ Nanoparticles using SEM, XRD and FTIR Techinques .
3. Study the effect of different parameters such as contact time, pH concentration of Hg (II), interfering metal ions on adsorption of Hg(II) on TiO₂ nanoparticles.
4. Removal of Hg(II) from contaminated water samples .
5. Fitting the experimental data to Langmuir mathematical Isotherm
6. Determination of Hg (II) in real water samples .
7. Study the effect of binary mixtures such as (Hg (II),Cd(II)) , (Hg(II),Zn(II)) on the adsorption capacity of Hg(II) on TiO₂ nanoparticles..

Material and Method

Chemicals: All Chemical used in this study were of high purity(Mercury nitrate99 % Merck, Germany, Titanium tetrachloride99 % PARK chemical LTD Pool England, Sodium dodecyl sulfate(SDS)99.5 % PARK Northo m pton, UK, Titanium sulfate 99.5 % BDH. Chemicals LTD, Hydrochloric acid 37% Sigma –Aldrich,bsA, Soduim hydroide Merk,Germany, Nitrac acid 65% Milano-italy)

Instruments: Shaker(Edmund Buhllr,k1-2 Germany), Digital pH meter(WTW525,Germany), Mercury analyzer(AA-6800,shimadzu,Japan), Furnace(Mettler Toledo AB 104-s-Germany), Analytical balance(Memner 600-Germany- (0.00001/g), Furnace(Qallenhamb,Hotspot temperature (0-1000C)Germany, Ultrasonic bath(Model Isolab, Germany).

Preparation of TiO₂ nanoparticles

Preparation of Titanium dioxide (TiO₂) nanoparticls from

Titanium Tetrachloride: The Titanium nanoparticles were synthesized by drop wise by addition of Titanium Tetrachloride (TiCl₄) in ethanol. The reaction was performed at room temperature while stirring under a fume hood due to the large amount of chlorine (Cl₂) and hydrochloride acid HCl gases evolved in this reaction. The resulting yellow solution was sonicated in Ultrasonic bath for about three hours. The suspensions obtained were dried in an oven for several hours at 500C until amorphous, drid and TiO₂ particles were obtained. The obtained powder samples were calcined for one hour in a box furnace at temperature ranging at 500 0C in an ambient atmosphere [8] .

Preparation of titanium dioxide (TiO₂) nanoparticles from titanium sulfate: Nanoparticles titanium dioxide TiO₂ (TiO₂ NPs) was synthesized from titanium sulfate according to a previously reported method [19]. Titanium sulfate (120.01g) was carefully hydrolysed for 10min at 100°C in 0.5L distilled deionized water

(D.D.W) 120.12g of carbamide and 2.45g of sodium dodecyl sodium sulphonate were added to the solution. The solution was stirred for 3h at 80°C by a magnetic stirr and sonicated in Ultrasonic bath for about three hours. The precipitate was filtered, washed with(D.D.W) to neutral, and dried at 100°C for 8h. The powder obtained was ground in an agate mortar and calcined for 2h at 500°C. The producer was then extracted and stored.

Effect of Contact time: 0.1g of TiO₂ of nanoparticles was added to 20 ml of 10 ppm Hg+2 solutions and shaken for 30 min, 1h, 2h, 3h, 4h, 5h and 6h. The mixture was then centrifuged at 350 rpm . The suspension was filtered.

The concentration of Hg+2 in solution was directly determined by Mercury analyzer .

Effect of pH

The effect of pH on the adsorption of Hg (II) on TiO₂ nanoparticles was studied.

0.1g of TiO₂ nanoparticles was added to 20ml of 10 ppm Hg (II) at different Values of pH 2, 3, 4, 5, 6 and7 and putted on shaker for about 2h. The mixture was then centrifuged at 350 rpm .

The suspension was filtered the solution was directly determined by mercury analyzer.

Effect of Mercury (Hg+2) Concentration

A series of standard solution of Mercury (Hg+2) with concentrations 1,2,4,6,8 and 10ppm were transferred in to 100ml erlemeyeh flask and 0.1 of TiO₂ nanoparticles was added at pH =3. The mixture was shaken vigorously for 2h to facilitate adsorption and Hg+2 ions onto the nanoparticles .

After, that the solution was centrifuged for 2h, the concentration of Hg+2 in the solution was directly determined by Mercury analyzer.

Determination of Mercury Hg (II) in contaminated water samples

Hg+2 concentration was determined in contaminated water samples. Samples were taken from three different places including Souk Althulatha Square (Tripoli), Libyan Fish Company (Alkhmus) and car wash place (Tarhuna). Each sample was devided into portions and the first portion was taken for analysis without any treatment and it was the base of comparison. The second portion was treated as following :

- 100ml of the water sample was placed in 250 ml beaker.
- 2ml of 0.1 M HNO₃ and 5ml of 0.1 M HCl were added.
- The mixture was heated at 900C untill the volume reduced to about 50 ml .
- The mixture was cooled at room temperature finally, the samples were filtered.
- (D.I.W) was added to the filterate to reach 100ml total volume.
- Samples were transferred to conical flasks contain 0.1g of TiO₂ nanoparticles was added to each.
- Treated water samples were placed in the shaker for 2hrs . After that treated samples were filterated using filter papers (N. 42) .

- Determination of Hg+2 concentration in filtered samples using Mercury analyzer.

Effect of mixtures (Hg(II) Cd(II)), (Hg(II), Zn(II)) on adsorption Capacites of Hg(II)

By applying the following conclusions of different pH values and different time of adsorption as well as the concentration of the mixtures contents .

Effect of Contact time: 0.1g of TiO₂ of nanoparticles were added to 20 ml of 10 ppm (Hg(II),Zn(II)) and (Hg(II),Cd(II)) and shaken for 30 min, 1h, 2h, 3h, 4h, 5h and 6h. The mixture was then centrifuged at 350 rpm . The suspension was filtered

The concentration of Hg+2 in solution was directly determined by Mercury analyzer .

Effect of pH: The effect of pH on the adsorption of Hg (II) in the mixtures (Hg(II),Zn(II)) and (Hg(II),Cd(II)) on TiO₂ nanoparticles was studied.

0.1g of TiO₂ nanoparticles was added to 20ml of 10 ppm (Hg(II),Zn(II)) and (Hg(II),Cd(II)) at different Values of pH 2,3,4,5,6 and 7 and putted on shaker for about 2h . The mixture was then centrifuged at 350 rpm .

The suspension was filtered . The solution was directly determined by Mercury Analyzer

Result

Characterization of TiO₂ nanoparticles

TiO₂ nanoparticles will be characterized by X-ray Diffraction, Scanning Electron Microscopy, Fourier Transform Infrared Techniques and Transmission Electron Microscopy techniques.

X-ray Diffraction(XRD): XRD pattern was recorded on prepared and calcined samples using instrument as described in section (1-5).the 2θ scans were recorded at several resolution using CuKα radiation of wave length 1.54Å 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 scherrers formula.

$$T = \frac{0.9\lambda}{B \cos\theta}$$

Where λ is x-ray wave length,B is peak width and θ the Bragg's angle K.

Fig (4-1) shows the (XRD) pattern of TiO₂ nanoparticles synthesized via method (1). Prepared nanoparticles showed as anatase has tetragonal symmetry (simple tetragonal) and belongs to D_{4h}19

-14/and space group with lattice constants of a=0.348nm,C=0.952nm and c/a=0.73nm.The crystal structure of the obtained powder was indicated in figure (4-1).All the diffraction peaks can be indexed as the pure (anatase) phase for TiO₂ nanoparticles.The obtained powder has high purity of anatase phase.No charities peak impurity of impurities such as Na₂TiO₃ was

detected.The average crystalline size of nanoparticles were determine to be (27 nm) utilizing sherrer formula ($T = \frac{0.9\lambda}{B \cos\theta}$),where T is the mean crystallize size of the powder,is the wavelength of CuKα,J 1.5405Å,B is the full width at half maximum (FWHM) intensity of the peak in radian. θ 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.

$$T = \frac{0.9\lambda}{B \cos\theta} = \frac{0.9 \times 1.54056}{0.6469 \times 7.93 \times 10^{-3}} = 270 \text{ \AA} = 27 \text{ nm}$$

Ba measured breath of balk material about maximum intensity B0 measured breath of observed material in half maxima.

$$[B = (B_2 - B_1) / 2]$$

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

2-The phases formed are pure.

tragonal symmetry (simple tetragonal) and belongs to D_{4h}19

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

Sample No	λ	2θ	B1/2	Dp	Lattice strain
Sample 1	1.54056.	24	0.2369	35.28nm	0.0048
Sample 2	1.54056	24	0.1986	43.5nm	0.004
Sample 3	1.54056	24	0.3149	27.30nm	6
Sample 4	1.54056	24	0.1968	42.39nm	0.0087

Table1: Relation between wave length for X-ray and crystal size.

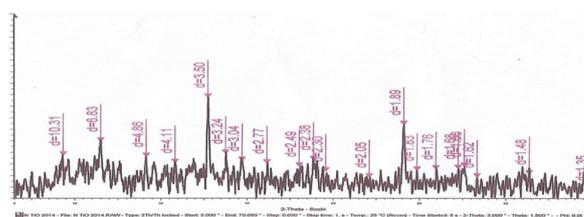


Figure 1: X-ray diffraction patterns of TiO₂ nanoparticles prepared From TiSO₄.

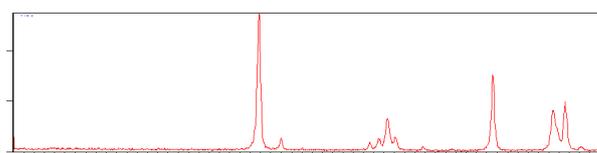


Figure 2: X-ray diffraction pattern of TiO₂ nanoparticles prepared from TiCl₄.

Fourier transform Infrared technique (FT-IR)

FT-IR Spectra of TiO₂ nanoparticles is shown in figure (3) and .According to FT-IR spectra of TiO₂ nanoparticles.

$$Q_e = \frac{C_0 - C_e \times V_m}{Wt(g) \times 1000}$$

Table (5) shows the relation between concentration of Hg (II) (ppm) and adsorption capacity Qe (mg/g). Table (6) shows the mathematical Langmuir adsorption isotherm for adsorption of Hg +2(II) onto TiO2 nanoparticles. It was found that the maximum adsorption capacity of TiO2nanoparticles for Hg+2 (II) up to.

Slop=1.136,intercept=0.0145.

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

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

$$b = \frac{\text{intercept}}{\text{Slope}}, \quad b = \frac{0.0145}{1.136} = 0.01276$$

$$0.01449Q_0 = 1, \quad Q_0 = 69.013 \text{ mg \ g}$$

$$Q_e = \frac{C_0 - C_e}{1 + \frac{C_0}{bQ_0C_e}} \times 1000$$

$$Q_e = 1 - 0.28 \times 20 / 1 \times 1000, \quad Q_e = 0.138 \text{ mg/g}$$

C (ppm)	Ce (ppm)	(C0-Ce)x 20	Qe (mg/g)
1	0.28	13.8	0.138
2	0.52	31.4	0.314
4	0.56	69.4	0.694
6	0.3	108.8	1.088
8	0.42	147.8	1.516

Table 2: The relation between concentration of Mercury Hg+2ppm and the adsorption capacity (Qe) (mg/g).

1/C	1/Q
1	7.25
0.5	3.185
0.25	1.441
0.17	0.919
0.13	0.659

Table 3: The Langmuir adsorption data.

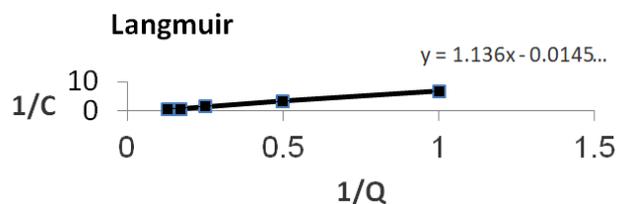


Figure 10: Langmuir adsorption isotherm for adsorption of Hg(II) onto TiO2 nanoparticles, W=0.1g, pH=4 time=2h, Conc=8ppm,250C.

Determination of Mercury (II) in contaminated water samples

Mercury concentration was determined in contaminated water samples. The samples were collected from three different places, including Souk Althulatha square (Tripoli), Libyan Fish company(Alkhums) and Car Wash Place(Tarhuna) .The results of samples before and after treatment with TiO2 nanoparticles are shown in table (4)

Removal%	ppm of Hg +2	ppm of Hg +2	Location	Source	Samples	
	After tretm ent	Before tre atment				
0.58%	87	0.3701±0.070	0.979 ±0.052	Tripoli	Souk Althulatha Square	S1
0.7295	40	0.2442±0.040	0.1004 ±0.072	Alkhums	Libyan Fish Company	S2
0.8837	30	0.2874±0.030	0.7201 ±0.026	Tarhuna	Car Wash place	S3

W=0.1, n=3, tim=2, Conc=8, pH =3, Tem=25 0C

Table 4: Mercury concentration in contaminated water samples as well as percentage of Hg+2 removals.

Effect of contact time on the preconcentration of Hg+2 in the mixtures Hg(II),Zn(II)) and (Hg(II),Cd(II))

The effect of contact time on the preconcentration of Hg+2 in the mixtures Hg(II),Zn(II)) and (Hg(II),Cd(II)) were studied using different times. Figures (11) and (12) showed the relation between contact time and the concentration of treated Hg+2(ppm). The best time for Hg(II) adsorption on TiO2 nanoparticles was 120 min. The results are shown in figure (11) and (12) .The effect of contact time on

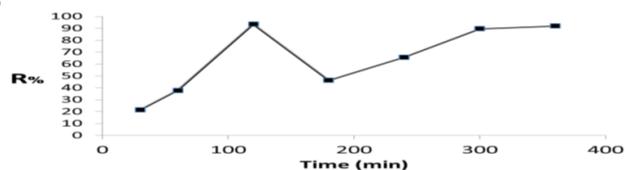


Figure 11: Effect of contact time on adsorption of mixture (Hg+2, Zn+2) ontoTiO2 nanoparticles. W=0.1g, pH=4 time=2h, Conc=8ppm, 250C.

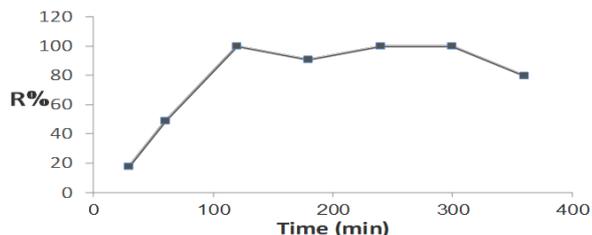


Figure 12: Effect of contact time on adsorption of mixture (Hg+2 , Cd+2) onto TiO2 nanoparticles, W=0.1g, pH=4 time=2h, Conc=8ppm, 250C.

Effect of pH of Hg(II) in (Hg(II),Zn(II)) and (Hg(II),Cd(II))

The effect of pH on adsorption of 10ppm of (Hg(II),Zn(II)) and (Hg(II),Cd(II)) on TiO2 nanoparticles were studied and the results are shown in Figures(13) and (14). It was found that pH=4 has the optimum conditions for pre-concentration of Hg+2 from aqueous solutions. pH=4 was chosen for further examination with different Hg +2 concentration .

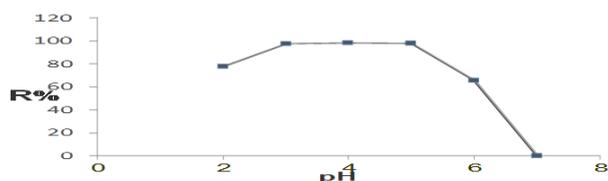


Figure 13: Effect of pH on adsorption of mixture (Hg+2, Cd+2) ontoTiO2 nanoparticles. W=0.1g, pH =4, Conc=8ppm time=2h, 250.

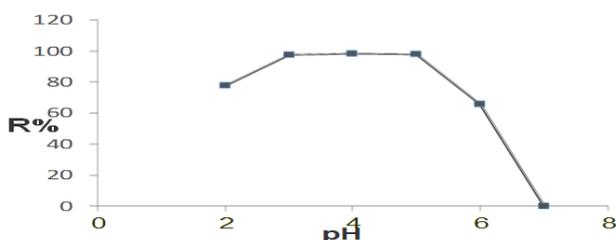


Figure 14: Effect of pH on adsorption of mixture (Hg+2, Zn+2) ontoTiO2 nanoparticles, W=0.1g, pH =4, Conc=8ppm time=2h,250C.

Effect of concentration of Hg +2 in the mixtures (Hg(II),Zn(II)) and (Hg(II),Cd(II))

The adsorption of Hg +2 in the mixtures (Hg(II),Zn(II)) and (Hg(II),Cd(II)) on the surface of TiO2 NPs were examined by using different concentration of Hg+2: 1,2,4,6 and 8 ppm in contact with 0.1g of TiO2 nanoparticles figure(15)and(16) The figures shows increasing for Hg+2 Ions. Concentration leads to increasing of the percentage of removal of Hg+2. This may can be explained because of the obesite intraction between positive Hg+2 ions

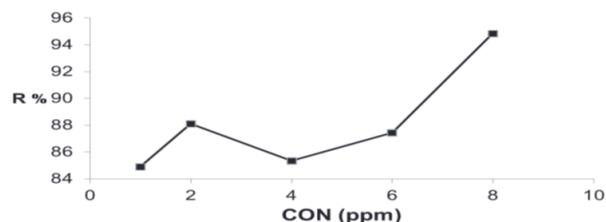


Figure 15: Effect of concentration on adsorption of mixture (Hg +2 , Cd+2) ontoTiO2 nanoparticles, W=0.1, pH =4, Conc=8 time=2h, 250C.

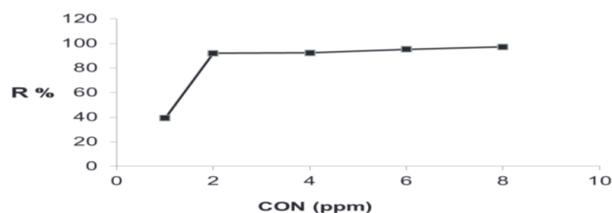


Figure 16: Effect of concentration on adsorption of mixture (Hg+2 , Zn+2) onto TiO2 nanoparticles. W=0.1g, pH =4, Conc=8ppm, time=2h, 250C.

Adsorption capacity of Hg(II) in the mixture Hg(II),Zn(II)

It was found that the maximum adsorption capacity of Hg (II) in mixture Hg(II),Zn(II) on TiO2 nanoparticles 38.43mg/g.

Slop =1.3839, intercept =0.026

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

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

$$b = \frac{\text{intercept}}{\text{Slope}}, b = \frac{0.0132}{1.0825} = 0.01219$$

$$1.0825 = \frac{1}{bQ_0}, 1.3839 = \frac{1}{0.01219Q_0}$$

$$Q_e = \frac{Q_0 - Q_0 \cdot (C_0 - C_e)}{C_0 - C_e} \times 1000$$

$$Q_e = 1 - 0.37 \times 20 / 1 \times 1000, Q_e = 0.126 \text{mg/g.}$$

1/C	1/Qe
1	7.94
0.5	3.18
0.25	1.401
0.17	0.88
0.13	0.649

Table 5: Concentration of Hg+2 (mg/L) and adsorption capacity (Qe) (mg/g).

C0 (ppm)	Ce (ppm)	(C0-Ce)x20	$Q_e = \frac{C_0 - C_e}{0.1 \times 10.0} \times 20$
1	0.37	12.6	0.126
2	0.52	36.4	0.364
4	0.43	71.4	0.714
6	0.32	113.6	1.136
8	0.29	154.2	1.542

Table 6: Langmuir adsorption of mercury in mixture Hg(II),Zn(II).

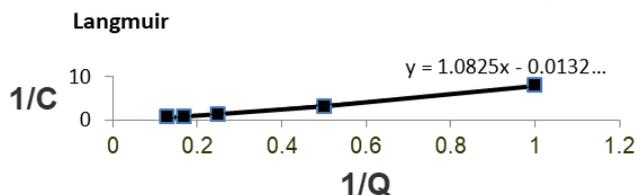


Figure 17: Langmuir adsorption isotherm for adsorption of mercury (Hg+2) in the mixture (Hg(II),Zn(II)) onto TiO2 nanoparticles W=0.1g, pH=4, Conc=8ppm, time=2h,250C.

Adsorption capacity of Hg(II) in the mixture Hg(II),Cd(II)

It was found that the maximum adsorption capacity of Hg(II) in mixture Hg(II),Zn(II) on TiO2 nanoparticles 29.585mg/g.

Slop =1.163, intercept =0.0139

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

$$\frac{1}{bQ_e}$$

$$b = \frac{\text{intercept}}{\text{Slope}}, \quad b = \frac{0.0139}{1.163} = 0.01195$$

$$1.163 = \frac{1}{bQ_0}, \quad 1.163 = \frac{1}{0.01195Q_0}$$

$$0.0139Q_0 = 1, \quad Q_0 = 71.94\text{mg/g}$$

$$Q_e = 1 - 0.13 \times 20 / 1 \times 1000, \quad Q_e = 0.174\text{mg/g.}$$

1/C	1/Qe
1	5.75
0.5	2.841
0.25	1.48
0.17	0.96
0.13	0.661

Table7: Concentration of Hg+2 (mg/L) and adsorption capacity (Qe) (mg/g).

C0(ppm)	Ce (ppm)	(C0-Ce)x 20	$Q_e = \frac{C_0 - C_e}{0.1 \times 10.0} \times 20$
1	0.13	17.4	0.174
2	0.24	35.2	0.352
4	0.62	67.6	0.676
6	0.79	104.2	1.042
8	0.44	151.2	1.512

Table 8: Langmuir adsorption of Hg(II) in the mixture Hg(II),Cd(II).

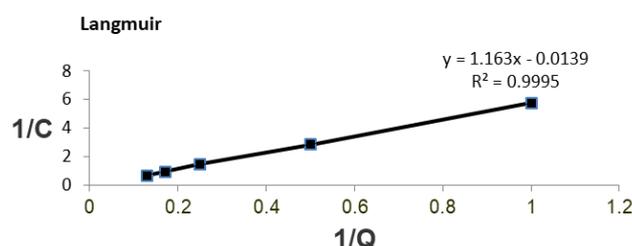


Figure18: Langmuir adsorption isotherm for adsorption of mercury (Hg+2) in the mixture (Hg(II),Cd(II)) onto TiO2 nanoparticles.

Conclusion

The results show that TiO2 nanoparticle is a good adsorbent for removing Hg (I) and Hg (II) ions from aqueous solutions. The optimum conditions for the adsorption of Hg (I) ion onto TiO2 nanoparticles are as the following: contact time=75 min; adsorbent dosage=0.05 g; initial concentration of Hg (I) ion=50 mg/L; temperature=22°C, and pH=9. In contrast, the optimum conditions for the adsorption of Hg (II) ion onto TiO2 nanoparticles are: contact time=45 min; adsorbent dosage=0.05 g; initial concentration of Hg (II) ions=40 mg/L; temperature=22°C and pH=7. Maximum adsorption of Hg (I) and Hg (II) ions onto TiO2 nanoparticles under the optimum conditions are 97.5% and 98.6%, respectively. Comparison of experimental results with adsorption isotherms of Langmuir, Freundlich and Temkin shows that adsorption of Hg (I) and Hg (II) ions by titanium dioxide nanoparticles corresponds fairly to the Langmuir adsorption isotherm model. The kinetics study of the adsorption process demonstrate that the model of kinetics for adsorption of Hg (I) and Hg (II) ions from aqueous solutions onto TiO2 nanoparticles corresponds to a pseudo-second order model.

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How to cite this article: Etorki AM, Aboushloa EM. "Removal of Hg⁺ from aqueous solution using TiO₂ nano-particles." *Chem Sci J*12 (2021) : 265