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Evaluation of an innovative synthesized nano zirconium tungstovanadate as cation exchange material

M.F.Elkady[1]*, E.M.EL-Sayed[1], H.A.Farag[2], A.A.Zaatout[2]

[1]Fabrication Technology Department, Advanced Technology and New Materials Research Institute (ATNMRI), City of Scientific Research and Technology Applications, Alexandria, (EGYPT)
[2]Chemical Engineering Department, Faculty of Engineering,
Alexandria University, Alexandria, (EGYPT)
E-mail: maroelkady@yahoo.com

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Abstract : The feasibility to prepare nano zirconium tungstovanadate using sol-gel technique will be studied. The prepared nano-material will be characterized using various chemical and physical evaluation techniques. It was assigned as cation exchanger as it recoded 96% lead ion sorption. The influence of the different processing parameters onto the lead ion sorption process was monitored. Equilibrium isotherm models will be examined onto the experimental data to determine the lead uptake mechanism.

Keywords : Nano zirconium tungstovanadate; Sol-gel technique; Characterization techniques; Equilibrium isotherm; Lead ion separation.

INTRODUCTION

The presence of heavy metals in the environment can be detrimental to all living organisms including humans. This pollution originates from discharge effluents from industries such as cement industries, electro plating, production of metal alloys, production of pigments for paints, mining, etc. The removal of toxic heavy metal contaminants from aqueous waste streams is currently one of the most important environmental issues being researched as their toxic nature, even at trace levels in natural waters, has been a public health problem[1]. Although this issue has been studied for many years, effective treatment options are still limited. Chemical precipitation, adsorption, reverse osmosis and ion exchange processes are the most commonly techniques utilized for removing heavy metals ions from dilute aqueous streams[2]. Lead is one of the most dangerous pollutants; even at low concentrations is extremely toxic, causing brain damage in children[3]. The ion exchange separation technique characterize over the other separation techniques by its simplicity, cheap and no energy consumable. Accordingly, the ion exchange process represents the most suitable technique for lead ion separation from polluted wastewaters. There are various types of ion exchange materials that are suitable to be utilized for the ion exchange process either from the organic or inorganic nature.

Synthetic inorganic ion exchangers have gained much attention owing to their high selectivity for certain elements, good kinetics of sorption, good selectivity and specially their greater stability to heat, ionizing radiation doses and acidic to moderately alkaline medium compared with the organic ion exchangers. Most investigations have been concerned with the use of these materials in the separation and removal of heavy toxic metal ions from industrial wastewater[4]. The heteropolyacid salts of polyvalent metals are of great interest because of their ion exchange character, as they are superior to single insoluble salts of these metals. It is possible to vary their compositions and change their selectivity to a particular ion. Thus the ion exchange behaviour, analytical application and synthesis of several zirconium based materials have been extensively studied. These inorganic ion exchangers were found to show relatively increased ion exchange capacity, selectivity and played a prominent role in water processing in the chemical and nuclear industries[5,6]. In this regard an inorganic heteropoly acid salt of zirconium based compound will be synthetized in nano-scale using the sol gel preparation technique. This investigation will be deal with optimization of the different factors that affect the lead ion sorption process onto the prepared cation exchanger. Moreover, the equilibrium lead ions sorption data will be modelled as an attempt to determine the sorption process mechanism.

EXPERIMENTAL

Materials and reagents

Zirconium oxychloride [$ZrOCl_2.8H_2O$, Germany], ammonium metavanadate [India] and Sodium tungstate [$Na_2WO_4.2H_2O$, India] have been used as the raw materials for the synthesis of zirconium(IV) tungstovanadate. Solutions of the various materials have been prepared in de-mineralized water (DMW). All these materials/reagents were mainly of analytical grade.

Synthesis of ZWV ion exchanger using Sol-gel technique

The sol–gel technique represents one of the fastest growing fields of contemporary chemistry. The attractive feature of this technology is the fact that sol–gel materials can be obtained as bulks, thin films (on various supports) and (nano) powders. Thus, nano zirconium tungstovanadate sample will be prepared by the sol-gel method. Zirconium (IV) tungstovanadate will be synthesized through addition of mixture of 0.2 M ammonium metavanadate and 0.1 M sodium tungstate into solution with 0.2 M zirconium oxy chloride in presence of 0.01M HCl gradually with continuous stirring at 25 ºC. After the addition was complete a fine yellow precipitate appeared. The reaction mixture was diluted to 1 L and allowed to settle for 24 h for complete digestion[7]. The supernatant liquid was decanted and gels were filtered by suction and the excess acid was washed thoroughly with hot water. The washed precipitate was then dried by gentle heating at 40 ºC. Then the product is grounded and immersed in 1 M nitric acid for 1 day with gentle stirring in order to transform the ion exchanger to its hydrogen form.

Chemical characterization of the prepared nano zirconium tungstovanadate ion exchangers

(a) Ion-exchange capacity (IEC)

To determine the ion-exchange capacity, which generally taken as a measure of the hydrogen ion liberation by neutral salt, The ion exchange capacities of different prepared ion exchangers were determined by acid–base titration[8]. The weighted sample of the ion exchanger in its H^+ form were soaked in 50 mL of 1 M NaCl solution for at least 12 h with shaking at ambient temperature to exchange protons with sodium ions. The ion exchanged was titrated against a standard solution of 0.1M NaOH using phenolphthalein as indicator. The ion exchange capacity (IEC) was calculated using the following equation:

$$\mathbf{IEC\ (mequiv / g) = V_{NaOH} * \frac{C\ NaOH}{Wd}} \quad (1)$$

Where; V_{NaOH}, C_{NaOH} and W_d are the volume of NaOH consumed in titration, the concentration of NaOH solution, and the weight of the dry sample, respectively.

(b) Ion exchange capacity for different metal ions

The ion exchange capacity of prepared nano zirconium tungstovanadate for alkali and alkaline earth metal ions was determined by equilibrating 0.25 g of the exchanger with 50 ml of 2 M solution of different metal salts. The liberated acid was measured by titration with standard alkali solution.

(c) Thermal effect on IEC

The effect of temperature on the ion exchange capacity of the prepared nano zirconium tungstovanadate was determined by heating a 0.5 g from the ion exchange material at various temperatures in muffle furnace for 1 h. The Na^+ ion exchange capacity after cooling the samples at room temperature was determined as mentioned in the ion exchange capacity technique[9].

(d) Chemical stability

The extent of dissolution of the material in common mineral acids (HCl, HNO_3 and H_2SO_4), bases (NaOH and KOH) and organic solvents (acetone, benzene, diethyl ether, heptane, acetic acid and alcohol) was studied. 0.5 g of ion exchanger was equilibrated with 20 ml of the solution of interest for 24 h at room temperature; the remaining amount of material was filtered and washed by DMW. After removal of excess acid or base, it was dried in oven at 40±5 ºC. The changes in color, weight and the on exchange capacity of the remaining material were observed.

Physical characterization of the differed prepared zirconium(IV) tungstovanadate ion exchangers

(a) X-ray diffraction (XRD)

X-ray diffraction pattern of the prepared ion-exchange material was recorded by X-ray diffractometer with Cu Ka radiation beam (l = 0.154060 nm) to determine the structure of the ion exchangers. Where the finely powdered samples of the ion exchanger were packed into a flat aluminium holder, and the X-ray source was a rotating anode operating at 30 kV and 30 mA with a copper target. Data were collected between 10^o and 80^o in 2θ.

(b) Thermal analysis (TGA)

The measurements of the thermal analysis of the prepared sample were carried out using thermal gravimetric analysis (TGA) with a heating rate 20 ºC/min under flow of N_2 to avoid thermal oxidation of the powder samples, starting from ambient condition up to 700 ºC.

(c) Infrared spectroscopy (FTIR)

The I.R. spectrum of the prepared ion exchange material was examined. The disc technique using KBr as a matrix was found to be suitable. In this concern, the ion exchanger was thoroughly mixed with KBr and the mixture was ground and then pressed with a special press to give a disc of standard diameter. The I.R. spectrum was scanned through a wave length range of 600–4000 cm^{-1}.

(d) Scanning electron microscope (SEM)

The grounded prepared sample was scanned to identify its structure and estimate the particle diameter at different magnifications. The mean diameter of the grains was determined from the SEM pictures by measuring at least 5 crystals for each formulation using the software Image tool.

Lead ion sorption affinity

The ion exchange efficiency of the prepared sample toward lead ions was tested. The ion exchange efficiency was determined by equilibrating 0.25 g of the selected cation exchanger sample of zirconium(IV) tungstovanadate in H^+ form with 100 ml of 50 ppm lead ion solution for 3 h with continuous shaking.

The removal efficiency is expressed as:

$$\eta = \frac{(Ci - Ce)}{Ci} * 100 \qquad (2)$$

Where, C_i is the initial metal ion concentration (ppm) and C_e is the concentration of metal ions (ppm) at equilibrium.

Batch procedure for ion exchange technique

The experiments were conducted in batch mode using synthesized wastewater which was prepared initially, where lead chloride ($PbCl_2.5H_2O$) was dissolved in distilled water to obtain the required lead concentration. These experiments were carried out to validate the effect of contact time (0-3 hr), pH (2–11), temperature (25–80 °C), mixing speed (0–400 rpm), initial concentration of the synthesis solution (5–1000 ppm), and the amount of ion exchanger (0.1 –5 g) on the ion exchange process using the most proper synthetized ion exchanger.

The experiments carried out through mixing a certain amount of ion exchanger in the H^+ form in a vial with the test solution (typically 100 ml) spiked with waste solution. The samples were mixed in shaking incubator till the solid/ solution system obtained equilibrium after 3 h. Then the different phases are separated using centrifugation at 6000 rpm for 10min. The residual lead ion concentration in the solution was analyzed using inductive coupled plasma mass spectrophotometer (ICP-AES, USA). All experiments were carried out in duplicate and mean values are presented. The lead ion measurements onto the ICP equipment were repeated three times to obtain an accurate lead ion concentration. The percentage error in the measurement of lead ion concentration is ± 0.1. The mean lead concentration values in the remaining waste solution were obtained and used for calculation the percentage ions removal onto the prepared nano-cation exchange sample as mentioned before from equation 3. The sorption capacity of the prepared material was calculated using following formula:

$$\mathbf{qe = \frac{(Co - Ce)}{m}} \qquad \mathbf{(3)}$$

where; q_e (mg/g) is the equilibrium adsorption capacity, C_o and C_e (mg/L) are the initial and equilibrium concentration of lead ions in solution, V (L) is the solution volume, and m(g) is the amount of the cation exchanger.

Equilibrium isotherm modelling for the ion exchange process

Sorption isotherms are basic requirements for designing any sorption system. The sorption isotherm is fundamental in the understanding of sorption processes since equilibrium studies the capacity of the synthesized nano zirconium(IV) tungstovanadate and indicate the affinity of the prepared material toward lead ions. This isotherm provides a relationship between the concentration of lead ion in the waste solution and the amount of ions sorbed onto the ion exchange material when the two phases were at a fixed temperature at equilibrium. The Langmuir and Freundlich equilibrium isotherms models were examined to fit the experimental data. The sorption data obtained for equilibrium conditions have been analyzed using the linear forms of these kinds of isotherms.

RESULT AND DISCUSSION

Different physical and chemical characterization methodologies were utilized to evaluate the synthesized bale yellow powdered material of zirconium tungestovanadate.

Chemical characterization

The prepared sample was chemically characterized in terms of its chemical composition, in addition to its chemical and thermal stabilities.

(a) Thermal effects on ion exchange capacity

Ion-exchange capacity of the prepared material was affected by heating. On heating at different temperatures for 1 h, the mass, physical appearance and ion-exchange capacity of the best prepared Zr(IV) tungstovanadate was changed as given in TABLE 1. One interesting property of nano-Zr (IV) tungstovanadate distinguishes by its thermal stability as the heating temperature incremented up to 700 °C.

(b) Chemical stability of cation exchanger

Results showing the extent of dissolution of the prepared material in different solutions are given in TABLE 2. It is clear that the prepared zirconium tungstovanadate exchangers is quite stable in water, organic solvents and fairly stable in acids of low concentration and bases whereas it is less stable in strong acids. This solubility experiments showed that the prepared material is highly chemically stable.

(c) Physical characterization

The X-ray diffraction pattern of the prepared material is shown in Figure 1. The X-ray diffraction spectrum shows a number of peaks at different 2θ values. The presence of some peaks suggested that the nature of the prepared nano-Zr(IV) tungstovanadate is semi-crystalline. The different intense peaks presented in the sample were compared with that in the two references of zirconium oxide phases (Card No. 01-087-1528) and (No. 01-088-0586), in order to determine the plane orientation of the cation exchange sample produced. It was clear from TABLE 3 that the produced

TABLE 1 : Effect of temperature on the ion-exchange capacity of Zr(IV) tungstovanadate on heating time for 1 h.

Heating Temperature (°C)	Appearance	IEC for Na^+ ion (mequiv.g$^{?1}$)	% loss in IEC
50	Yellow	2.5	0
100	Yellow	2.45	2
200	Yellow	2.39	4.4
400	Yellow	2.25	10
500	Dark yellow	2.15	16
600	Dark yellow	1.8	28
700	Greenish yellow	1.6	36

sample represents a mixture of zirconium tungsten oxide and zirconium vanadium oxide with cubic crystal configurations.

According to the TGA analysis given in Figure 2, the prepared zirconium tungstovanadate sample showed two main weight losses or degradation regions. The first weight loss region is maximum at around 140 ºC which is due to the free external water molecules removal[10,11]. While, the second gradual loss in the sample weight that began from temperature above 370 ºC is due to the removal of interstitial water molecules by condensation of exchangeable hydroxyl groups (-OH) from the material, which is characteristic of synthetic inorganic ion exchangers[12,13]. Above 450 ºC there is no significant degradation, which means that no structural changes can occur for the materials. This suggests that the prepared the ion exchanger is stable up to 800 ºC.

Morphological characterization of the prepared cation exchanger zirconium tungstovanadate was performed using SEM. Figure 3 indicates that the cation exchange material has average diameter equal to 28 nm. Thus, the prepared material produced in the nano-range.

TABLE 2 : Solubility of Zr(IV) tungstovanadate in various solvents (0.5 g of the material was initially taken for the treatment).

Solution	Wt. after treatment (g)	IEC for Na^+ (meq/g)
Acids:		
0.1 M HCl	0.5	2.5
0.5 M HCl	0.5	2.5
2 M HCl	0.5	2.5
5 M HCl	0.48	2.1
HNO_3	0.2	1.5
H_2SO_4	0.32	1.9
Organic solvents:		
Benzene	0.5	2.5
Heptane	0.5	2.5
Acetone	0.5	2.5
Isopropanol	0.5	2.5
Diethyl ether	0.5	2.5
Bases:		
1 M KOH	0.5	2.5
1 M NaOH	0.5	2.5

TABLE 3 : X-ray peak values for two reference phases.

Reference	2θ for strain	Plan of orientation
2θ for cubic zirconium tungsten oxide reference (card No. 01-087-1528)		
43.0067	43.1740	(3 3 1)
54.842	54.2545	(5 2 1)
66.0471	65.9729	(5 4 1)
78.8197	78.6726	(7 2 2)
2θ for cubic zirconium vanadium oxide reference (card No. 01-088-0586)		
17.4341	17.7839	(1 1 1)
51.8902	51.6046	(4 3 0)
62.351	62.9232	(1 3 5)

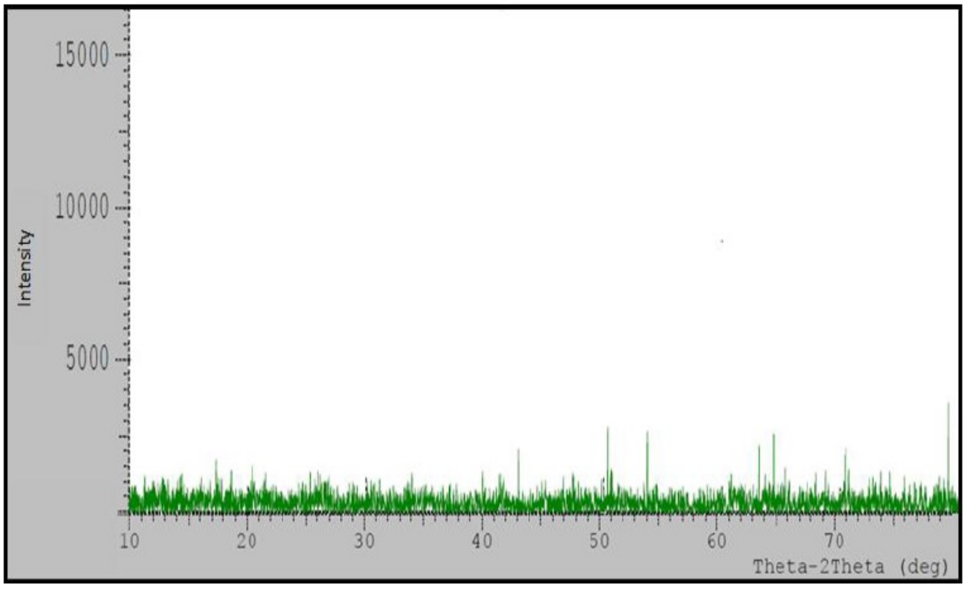

Figure 1 : X-ray curve for zirconium tungstovanadate.

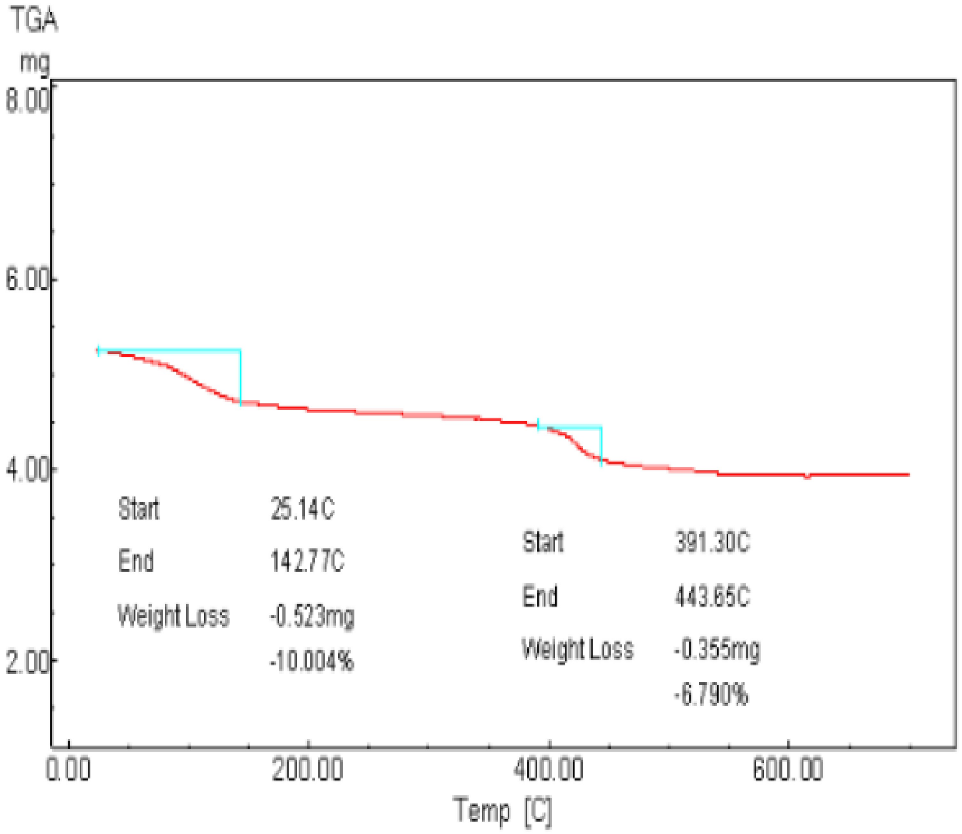

Figure 2 : TGA curve for nano zirconium tungstovanadate.

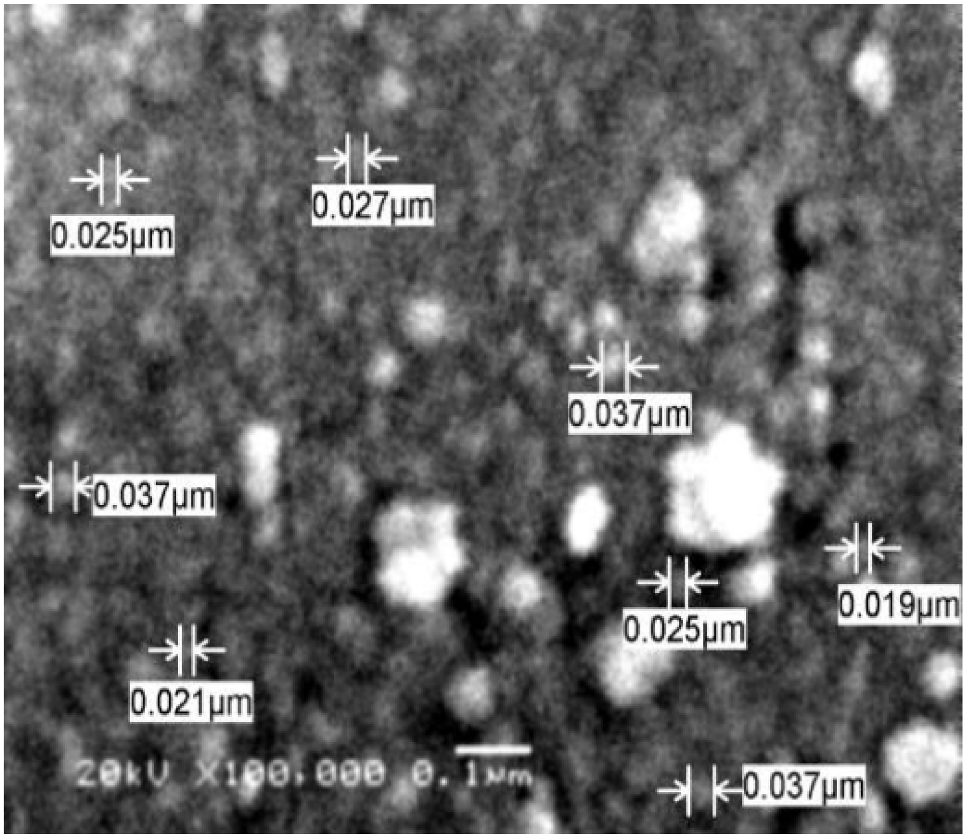

Figure 3 : SEM micrograph of prepared zirconium tungstovanadate.

The FTIR spectra of the prepared cation exchange zirconium tungstovanadate sample showed in Figure 4. The analysis of its absorption peaks investigated in TABLE 4. The appearance of the peaks has been designated accordingly[14]. This analysis assign the presence of structural H-OH that is responsible mainly for the exchangeable H^+ ion inside the prepared nano-cation exchange sample. Moreover, it was confirmed from the characteristics peaks analysis that the material composed mainly from a mixture of zirconium tungsten oxide and zirconium vanadium oxide as stated previously at XRD section.

Batch experiments for lead ion separation

(a) Effect of contact time

The effect of contact time on the lead ions sorption onto nano zirconium tungstovanadate was investigated in Figure 5. This figure elucidated that the percentage lead removal increases with contact time and attains equilibrium within 3 hr; this effect has arisen from the fact that heavy metal-zirconium tungstovanadate interaction is a dynamic process and the amount of metal uptake is a function of time[15]. Thus, 3 hr is chosen as the reaction time required for equilibrium achievement for the lead ion sorption process.

(b) Effect of ion exchanger dose

The exchanger amount is an essential parameter to obtain the quantitative uptake of metal ion. Different amount of the ion exchanger were tested in the range of (0.1 – 5 g) and equilibrated for 3 h at an initial lead concentration of 500 ppm, all experiments were carried out at the same stirring speed, 200 rpm. The lead ion removal results are given in Figure 6. It is apparent that sorption of lead ions was increased as the utilized cation exchange amount improved. This result was expected because for a fixed initial metal concentration, increasing adsorbent amount provides greater surface area and sorption sites for metal ions uptake that by its role enhance the metal ion removal[16,17]. It is readily understood that the number of available sorption sites

TABLE 4 : Details of IR spectrum of nano zirconium tungstovanadate:

Wave number (cm^{-1})	Significance
3000-3500	Hydroxostretcing vibrations (–OH groups)
1600-1630	Deformation vibrations of coordinated water (H–O–H bonding)
1400-1420	Vibrations due to metal Hydroxyl groups (M–OH)
600-750	Superposition of metal oxygen stretching vibrations (W–O, Zr–O, V–O).

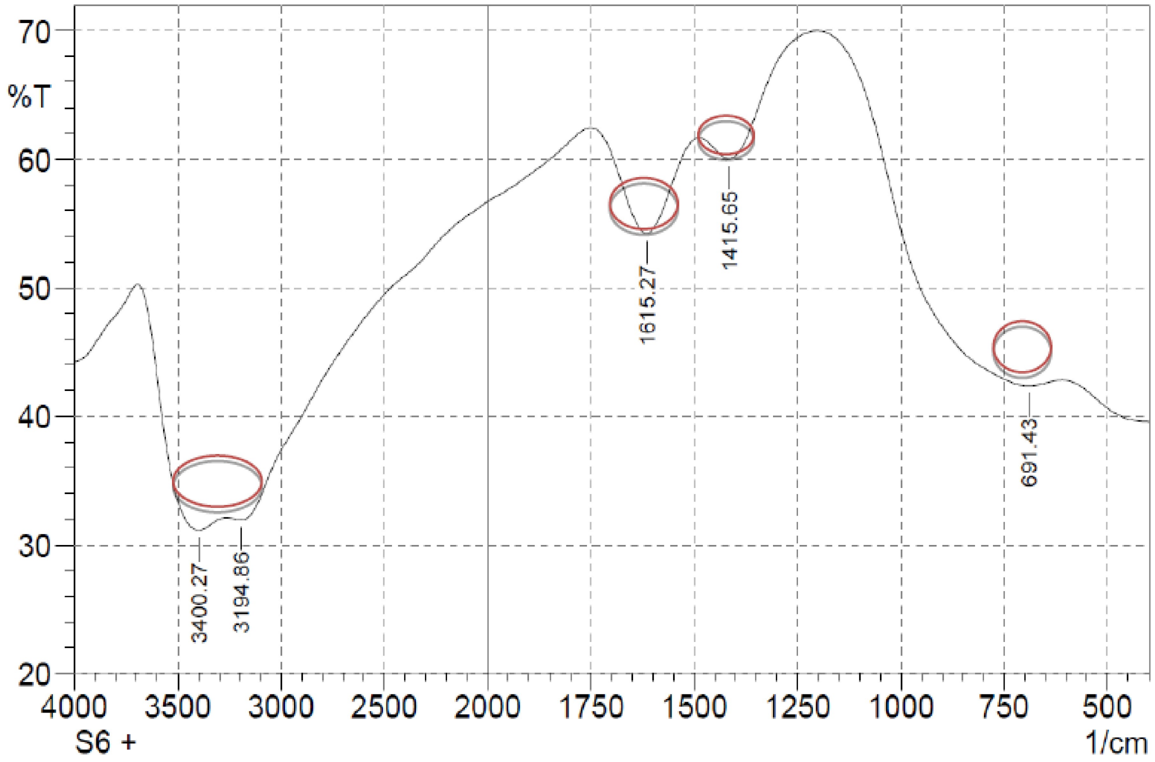

Figure 4 : FTIR spectra of Zr(IV) tungstovanadate cation exchanger.

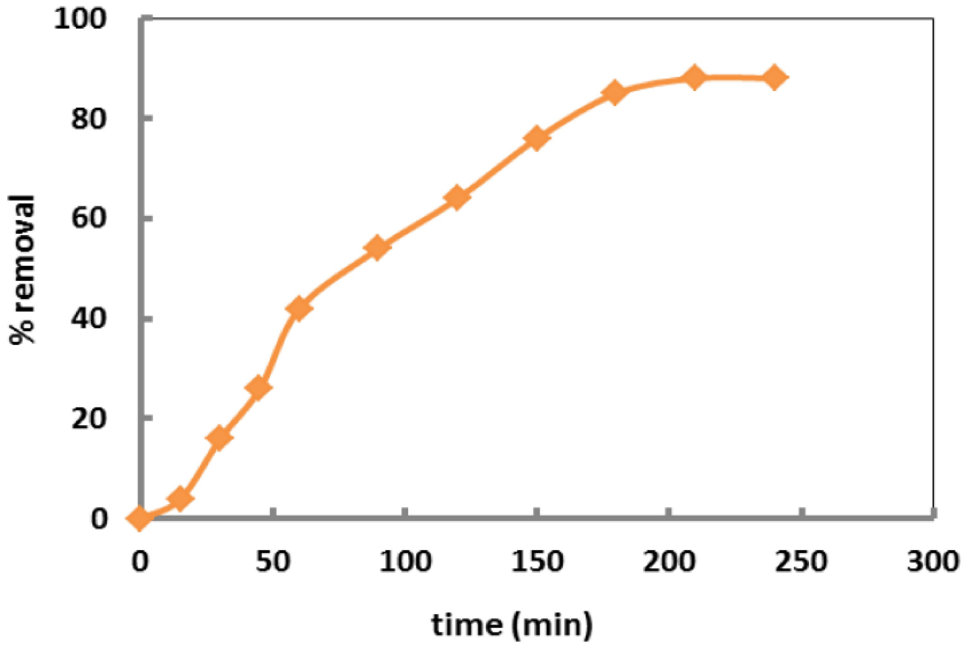

Figure 5 : Effect of contact time on the sorption of pb^{2+} by zirconium tungstovanadate (initial lead conc. = 500 ppm; resin dose = 0.25 g; solution volume, 100 ml, temperature = 25±2 °C; Agitation speed = 200 rpm; pH = 7).

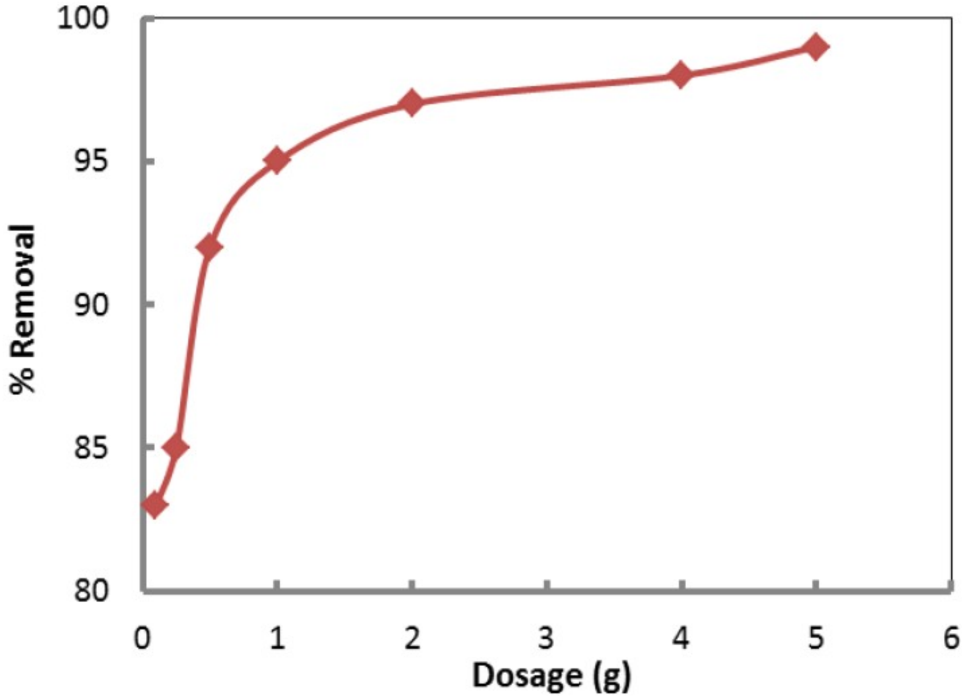

Figure 6 : Effect of cation exchange dosage on the sorption of Pb^{2+} ions (initial lead concentration = 500 ppm; solution volume = 100 ml, temperature = 25±2 °C; Agitation speed = 200 rpm; pH = 7).

increases by increasing the cation exchanger amount. From Figure 6 it was investigated that the optimum cation exchanger dosage is 5 gm that record 100 % lead ion removal.

(c) Effect of initial metal concentration

Predetermined optimal values of nano zirconium tungstovanadate dosage were used for examining effects of lead ion concentration on the lead removal process. Figure 7 illustrated that the removal percentage of lead ions decreases with increasing initial Pb^{+2} concentrations. The decrease in the rate of Pb^{2+} removal with increasing initial concentration may be attributed to that the increase in the repulsion forces between the same charged species with increasing the metal ion concentration. Thus this will hinder the mobility of metal ion toward the exchangeable sites[18,19].

(d) Effect of pH

Hydronium ion concentration is an important parameter affecting the ion-exchange process. This is partly because hydrogen ions themselves are strongly competing adsorbate. In order to investigate the effect of pH on lead removal using the prepared nano zirconium, experiments were performed in the pH range (1–11). As seen from Figure 8 optimal uptake

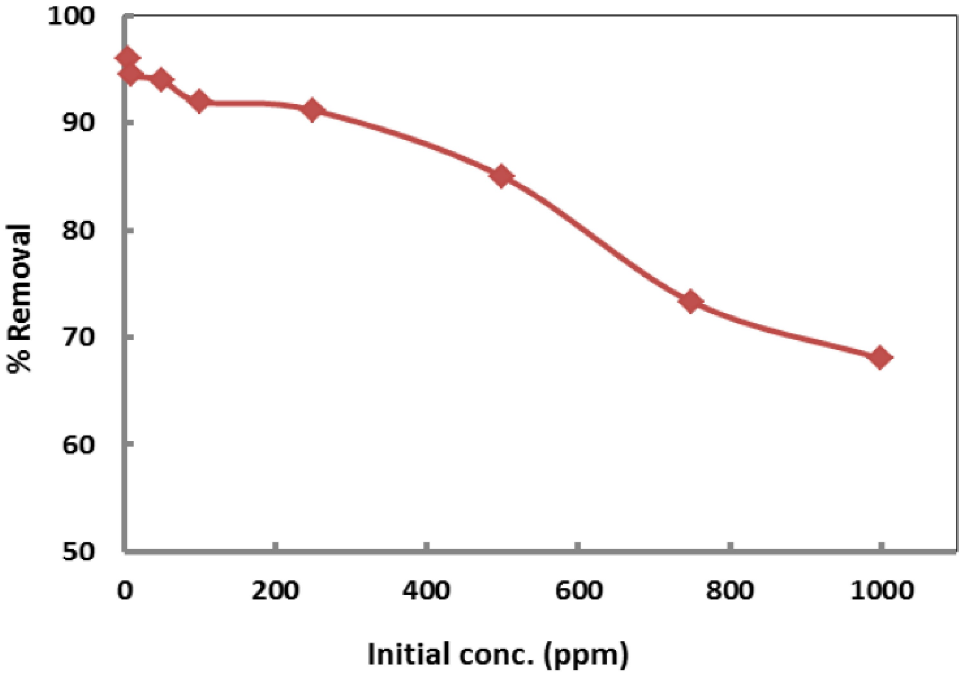

Figure 7 : Effect of initial metal concentration on the ion exchange of Pb^{2+} ions using the prepared cation exchanger (exchanger dosage = 0.25 g; volume of solution = 100 ml, temperature = 25±2 °C; Agitation speed = 200 rpm; pH = 7).

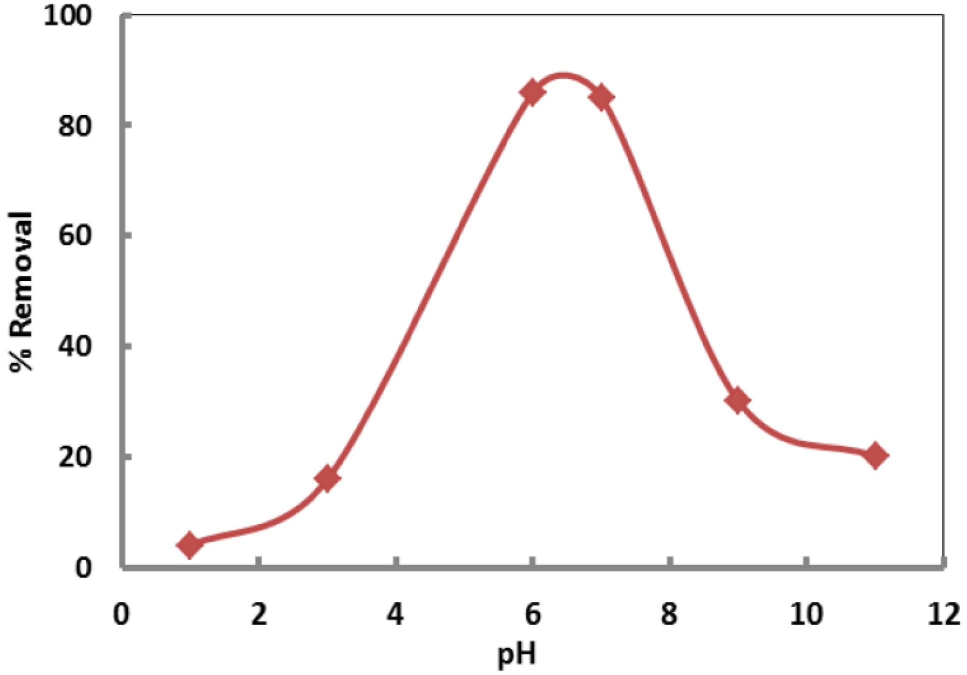

Figure 8 : Effect of initial solution pH on the ion exchange of Pb^{2+} ions (exchanger dosage = 0.25 g; solution volume =100 ml, temperature = 25±2 °C; Agitation speed = 200 rpm; initial lead conc. = 500 ppm).

of Pb^{2+} was occurred at pH range of 6-7. At high pH values, decrease in removal efficiency achieved by nano zirconium tungstovanadate can be described with formation of $Pb(OH)_2$ during reaction of Pb2+ ions with OH–. In this state, hydrolysis accompanied by precipitation of metal hydroxides may occur[16]. However, at low pH values, the decrease in lead ion removal may be returned to the competition of H^+ ions with Pb^{2+} in the ion exchange process onto the prepared cation exchanger.

(e) Effect of solution temperature

The effect of temperature on the ion exchange process is important not only because it affects the rate and extent of sorption but also due to the fact that temperature dependence of sorption provides information about possible sorbate–sorbent interaction[20]. In the temperature range of 25–80 °C, the removal capacity of the prepared inorganic ion exchanger for the lead ions was determined. From Figure 9 it is clear that lead ions removal from the aqueous solution increase with raising temperature. It may be returned to that higher temperatures activate the lead ions for enhancing sorption at the coordinating sites of nano-cation exchanger. Also, it is mentioned that cations move faster when temperature increases. Potential explanations for this are that specific or electrostatic interactions become weaker and the ions become smaller, since solvation is reduced[21]. This result indicates that the ion exchange process is an endothermic process onto the prepared nano zirconium tungstovanadate.

Adsorption isotherms

Results obtained for leas ions sorption using the prepared nano zirconium tungstovanadate were analyzed with well-known adsorption models, Langmuir and Freundlich.

(a) Langmuir model

The Langmuir isotherm is a commonly applied model for adsorption on a completely homogenous surface with negligible interaction between adsorbed molecules[22]. The model assumes uniform adsorption energies onto the surface and maximum adsorption depends on saturation level of monolayer. Langmuir model can be represented with the following linear equation:

$$\frac{Ce}{qe} = \frac{1}{kqm} + \frac{Ce}{qm} \qquad (4)$$

Where; q_e represents the mass of adsorbed lead per unit cation exchanger (mg/g), q_m is the monolayer capacity, k is the equilibrium constant and C_e is the equilibrium concentration of the solution (mg/L). Plot of C_e/q_e versus C_e should indicate a straight line of slope $1/q_m$ and an intercept of $1/q_mk$. Figure 10 illustrate the linear

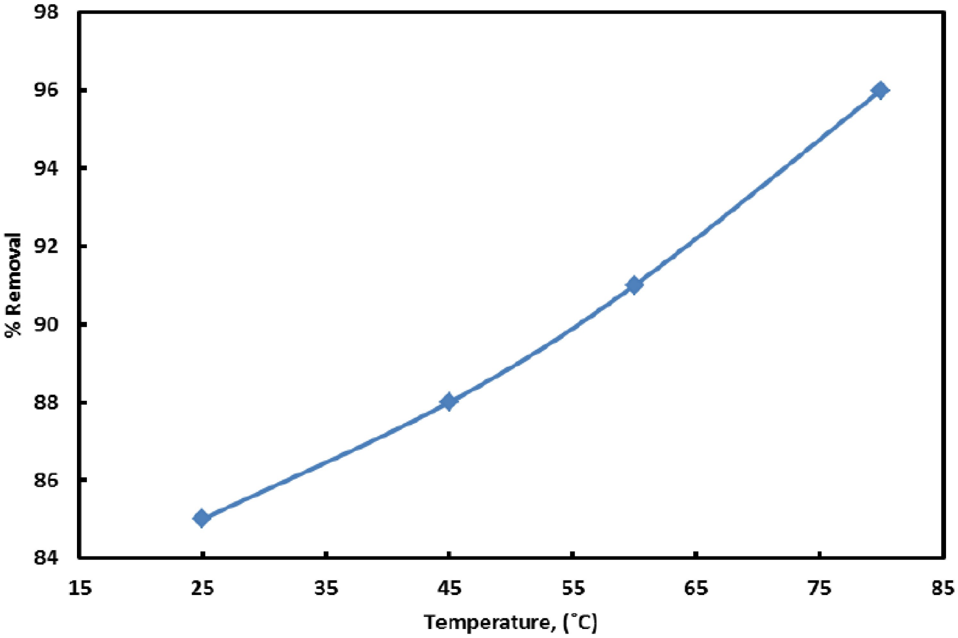

Figure 9 : Effect of solution temperature on lead removal at equilibrium using the prepared cation exchanger (initial lead conc. = 500 ppm, ion exchanger dosage = 0.25 g, agitation time = 3 h, agitation speed = 200 rpm, solution volume = 100 ml, and pH=7).

plot of Langmuir equation for lead ions removal at various initial ion concentrations onto the synthesized nano zirconium tungestovanadate ion exchanger.

From the correlation coefficient (R^2) value given in TABLE 7, which is considered as a measure of the goodness of fit of experimental data on the isotherm's model, It was clear that the Langmuir equation represents the sorption process of lead ions at the prepared nano zirconium tungstovanadate ion exchanger very well. The applicability of this model gives predication that the main mechanism for lead ions separation using the synthesized material may be takes place through the ion exchange process as mono-layer at the ion exchange material. The Langmuir parameters for lead ions re-

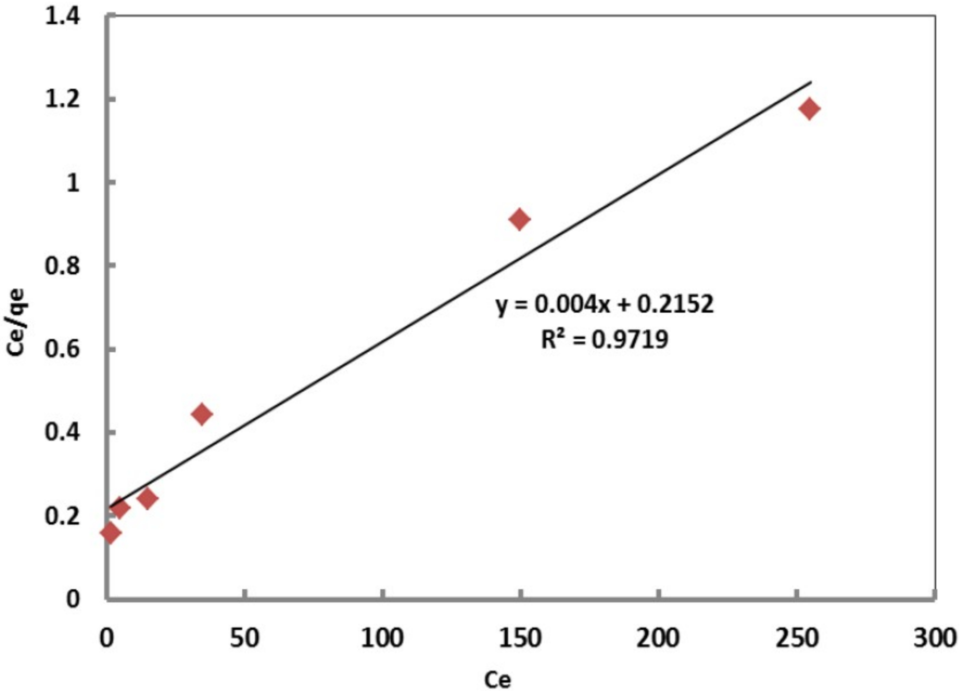

Figure 10 : Langmuir adsorption isotherm for lead sorption using nano zirconium tungstovanadate.

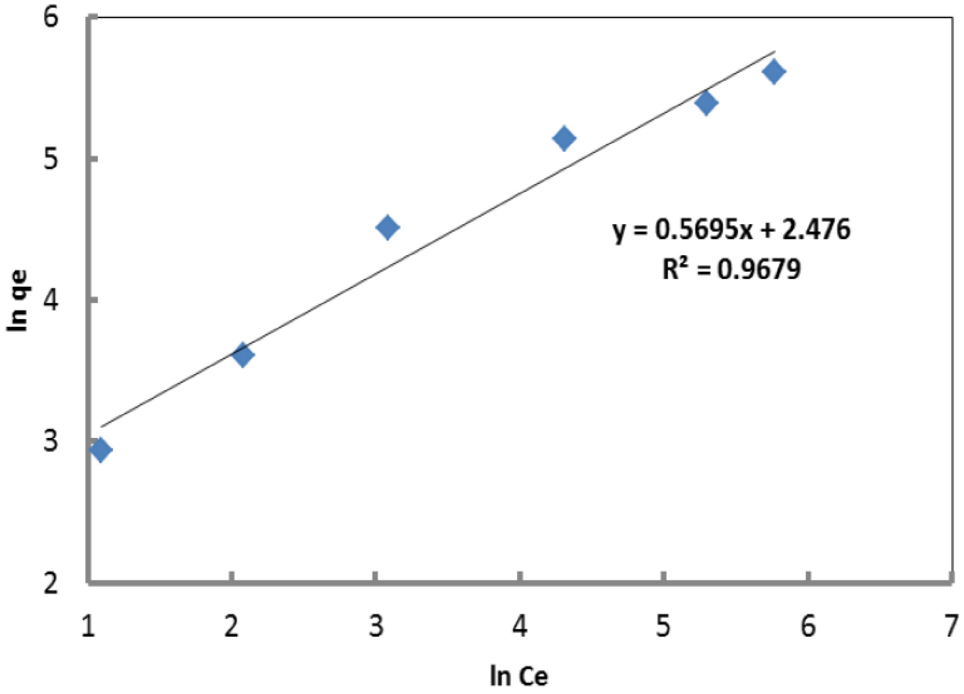

Figure 11 : Freundlich adsorption isotherm for lead ions sorption on nano zirconium tungstovanadate.

TABLE 5 : Isotherm parameters calculated for lead ions sorption using nano zirconium tungstovanadate.

Langmiur isotherm constants:		
q_m (mg/g)	**K (L/mg)**	**R^2**
250	0.018	0.9719
Freundlich isotherm constants:		
k_f (mg/g)	**n**	**R^2**
11	1.75	0.967

moval, qm and k, were calculated from the slope and intercept of these figures and tabulated in TABLE 5. The results relevant that the prepared ion exchange sample has maximum monolayer sorption capacity equals to 250 mg/g for lead ions. So, the prepared nanomaterial distinguishes by its high lead ion sorption capacity. Accordingly, it is suitable to be utilized for industrial wastewater treatment processes.

(b) Freundlich model

The Freundlich model is known as earliest empirical equation and is shown to be consistent with exponential distribution of active centers, characteristic of heterogeneous surfaces[23,24]. Freundlich equation is:

$$\text{Log } qe = \log\ kf + \frac{1}{n}\log\ Ce \qquad (5)$$

Where; K_f and n represent adsorption capacity and intensity, respectively. K_f is an important constant used as relative measure for adsorption efficiency. The magnitude of the n shows an indication of the favourability of adsorption. Values of n larger than 1 show the favorable nature of adsorption[23,24]. The plot of ln q_e against ln C_e for lead ions sorption data on the prepared nanocation exchanger is fitting well to the Freundlich isotherm (Figure 11). Where, the correlation coefficient value that equal to 0.967 is high enough for equation fitting. Accordingly, the lead ion sorption onto the prepared nano-cation exchanger may be described by both Langmuir and Freundlich isotherm models. However, the Langmuir applicability for the lead sorption process is much higher than the Freundlich applicability according to the models correlation coefficient values. This is give prediction that the adsorption phenomena have some degree of contribution beside the ion exchange mechanism in lead ions separation. The intercept of the straight line is roughly an indicator of the adsorption capacity, K_F, and the slope, n, is an indication of adsorption effectiveness. The n value (n>1) tabulated at TABLE 5 confirm the favourability nature of lead ion sorption on the prepared nano-materials.

CONCLUSION

New nano zirconium(IV) tungstovanadate ion exchanger was synthesized successfully using sol-gel technique, characterized and its analytical application was explored. The XRD spectrum of the prepared material indicated that it has semi-crystalline structure. Moreover the SEM imaging confirms that it was prepared in nanostructure. The material is fairly stable in inorganic acids, bases and organic solvents. The promising feature of the material is its affinity for Pb^{2+}ions, which is one of the most toxic metal ions present in environment.

By studying langmiur and frendlich, it was clear that the sorption of lead ions by the prepared zirconium tungstovanadate ion exchanger seemed to involve adsorption phenomenon in addition to ion exchange process.

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