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# Adsorptive Removal of Zn(II),Co(II) and Their Radioactive Isotopes $^{65}$Zn, $^{60}$Co on The Surface of Sodium Nano Bentonite Coated with Oleyl-Amine

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**Abstract**: A novel adsorbent material was designed through coating the surface of sodium nano bentonite with oleyl-amine to produce an efficient nanosorbent **Na.Bent-O-Amine** for the adsorptive removal of Zn (II), Co (II) ions from tap water and $^{65}$Zn, $^{60}$Co radioactive isotopes from radioactive wastewater. The nonabsorbent was characterized using the Fourier-transform-infrared (FT-IR), X-ray diffraction (XRD), High-resolution transmission electron microscopy (HR-TEM). The particle size of **Na.Bent-O-Amine** nonabsorbent was in the range (13.59-16.31) nm according to the HR-TEM. Different experimental parameters included, initial pH, contact time, nonabsorbent dosage, initial metal ions concentration were investigated and optimized. The results indicated that the optimum values were obtained by using 5.0 mg of **Na.Bent-O-Amine** nanosorbent at pH 7.0 and reaction time 50, 60 min for Zn (II), Co (II), respectively. The maximum adsorption capacity values were 225.68, 195.54 mg/g for Zn (II) and Co (II), respectively. Also, the multistage microcolumn system was implemented to remove Zn (II) and Co (II) from tap water and their radioisotopes $^{65}$Zn and $^{60}$Co from radioactive wastewater. The results of this study confirmed an excellent recovery and the percentage of removal were (91.5-94 %) for Zn (II) / $^{65}$Zn and (93-95%) for Co (II) /$^{60}$Co.

**Keywords:** Natural Radioactivity, Specific Activity, Soil, Radiation hazards, NaI(Tl) Detector.

## 1 Introduction

Nanoscience is a modern science focus on the synthesis of different materials in the range of 1-100 nm, within this scale the properties of material is different from its bulk size. The improvement of nanomaterials properties are attributed to their small particle size which consequently increases its specific surface area and increases their activity. Nano-materials is implemented in a lot of applications, like medicine, solar cells, industry and water treatment from the heavy metals as well as the radioactive isotopes **[**1,2**].** Radioactive isotopes and heavy metals resultant from industrial and nuclear activities are hazard materials. Wastewaters treatment is required to remove these unhealthy materials to protect the environment from its risk effects **[**3-4**].** Several treatment techniques like chemical precipitation, solvent extraction, and adsorption were implemented for the removal of heavy metals, radio nuclides from industrial waste water and radioactive wastewaters **[**5-6**].** Adsorption is a suitable technique due to its simplicity and low-cost. Many kinds of materials were applied to adsorb heavy metals and radioactive isotopes as nano metal oxides, organic polymers, surfactants and different kinds of clays **[**7-10**].** Clay minerals as, bentonite, sericite, kaolinite, montmorillonite and vermiculite were established and investigated as a novel adsorbents due to their advantages as, high quantity, very cheap materials, its large surface area, mechanical stability and high cation exchange capacity **[**8-12**].**

Bentonite is aluminosilicate mineral, has the structure of one alumina octahedral layer sandwiched between two

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silica tetrahedral layers. The high ability of bentonite to remove heavy metals and radioactive isotopes was attributed to its adsorption sites for metal ions on bentonite clay include silanol "SiOH" and aluminol "AlOH", hydroxyl groups on the mineral edges as well as the permanently charged sites, " O- " on the basal surfaces [8]. However, the adsorption properties of bentonite can be increased using different types of surface modifications [11-18]. Activated bentonite with sulfuric acid to increase its active sites was implemented for the adsorptive removal of Cu (II) and Pb(II) [8]. A comparative study between activated bentonite with acid and natural bentonite was held for the adsorptive removal of Pb (II), Cd (II) and Cu (II), which was shown a high removal efficiency for activated bentonite compared to natural bentonite [9]. Removal of Zn (II) and Co(II) from water and wastewater was a target for a number of researchers. For example, activated carbon, bentonite and iron powder were used to prepare $Fe_2O_3$–ceramisite by sintering at 800°C as a low-cost adsorbent for the adsorptive removal of Zn (II) and Pb (II) [10]. A comparative study between raw bentonite, alkaline Ca-bentonite for the adsorptive removal of Zn (II) was investigated [11]. Tricalcium aluminate as a selective agent for the adsorptive removal of Zn (II) was reported [12]. A lot of researchers have established the adsorptive removal of Co (II) and $^{60}$Co radioactive isotope from waste water using different kinds of materials. Application of magnetic composite of chitosan mixed with polyvinyl alcohol to uptake $^{60}$Co was reported [13]. Removal of Co (II) from wastewater using activated Saudi bentonite was reported [14]. Removal of Co (II) through polymer ultrafiltration using three kinds of water sulfonated soluble polymers based on polyvinyl alcohol was reported [15]. $Fe_3O_4$ nanoparticles for the adsorptive removal of Co (II) was reported [16]. Silicotitanate and four different iron oxides (ferrihydrite, goethite, hematite, and magnetite), were prepared for the adsorptive removal of Co(II) and strontium metal ions from nuclear wastewater through using of adsorption and microfiltration system [17]. The adsorption of radioactive Eu (III), Cs (I), and Co (II) using nanocomposite of $TiO_2$ mixed with Polyacrylamide styrene sodium sulfonate was investigated [18]. A comparative study between bentonite and bentonite modified with formaldehyde for the adsorptive removal of the radioactive isotope $^{60}$Co from radioactive waste was investigated [19]. Also, other papers were investigated at this field [20-23]. In this study, nano bentonite was activated with sulfuric acid to increase its active sites then was treated with sodium hydroxide to obtain sodium nano bentonite which increases the number of hydroxyl groups on the surface of nano bentonite and consequently increase its adsorption capacity. The coating process of nano bentonite with oleyl-amine as a surfactant using dispersion coating technique via aVander Vall's forces to improve the adsorption capacity of nanosorbent through the amine group ($NH_2$) of oleyl-amine was established in this study. The results indicated that the coating process increases the values of adsorption capacities.

## 2 Experimental Section

### 2.1 Materials and Chemicals

All chemicals and reagents used in this work were of analytical grade and used as received without any purification. Nano bentonite was provided from the Sigma-Aldrich chemical company, USA. Oleyl amine extra pure ($C_{18}H_{35}NH_2$, FW= 267.5 g/mole and assay 99.9%) was provided by Cross, USA. Sodium hydroxide (NaOH, FW = 40 g/mol and assay 98%), hydrochloric acid (HCl, FW = 36.46 g/mol and 37 %) and zinc chloride ($ZnCl_2$, FW = 136.29 g/mol and assay 98%) were purchased from SDFCL (SD fine Chem limited), India. Cobalt chloride ($CoCl_2$ FW = 129.84 g/mol and assay 97%) purchased from BDH. EDTA, ethylenediaminetetraaceticacid disodium salt, ($C_{10}H_{14}N_2O_8Na_2$ FW = 372.24 g/mol and assay 98%), sodium acetate anhydrous ($CH_3COONa$ FW = 82 g/mol and assay 98%), were purchased from El-Nasser pharmaceutical chemicals company, Egypt.

### 2.2 Instrumentations

The nano-adsorbent, **Na.Bent-O-Amine** was characterized using (FT-IR), (XRD) and (TEM).The FT-IR spectra of **Na.Bent-O-Amine** was recorded in the range of 400–4000 $cm^{-1}$ using a BRUKER Tensor 37 Fourier transform infrared spectrophotometer. HR-TEM, (JEOL-JEM2100F) was used to examine the surface morphology and determine the particle size of nanosorbent using scanning image observation device to give bright and dark-field TEM images at 200 kV. EDTA titration and Perkin Elmer flame atomic absorption spectrophotometer model 2380, were used to determine the concentration of heavy metal ions, Zn (II) and Co (II). NaI scintillator detector was used to determine the values of radioactivity for $^{65}$Zn, $^{60}$Co isotopes in the radioactive waste water.

### 2.3 Preparation of (Na.Bent-O-Amine)

Activation of nano Bentonite was established using 5 g of nano bentonite, 500 ml of 1M $H_2SO_4$ was added under vigorous stirring at 80$^0$C for 4 hours. Activation process of nano bentonite increases from its adsorption capacity, this is attributed to increasing the number of active sites, via increase its surface area and remove the traces of heavy metals, then the sample was washed with deionized water for several times to remove the excess of $H_2SO_4$, finally the sample was dried in an oven at 120°C for 4 hours. Activated sodium nano bentonite was treated using 1M of NaOH at 80°C for 2 hours to obtain the nano bentonite in its sodium form, the excess of NaOH was removed by washing with water for several times then the sample was dried in an oven at 120°C for 4 hours. About of 5g of the dried sodium nano bentonite was mixed with 2.5 mL of oleyl amine dispersed in 100 mL of water then the mixture was heated for 90 minutes at 80°C with vigorous stirring. The produced **Na.Bent-O-Amine** nanosorbent was transferred to an oven and dried at 80°C for 4-6 hours.

### *2.4 Adsorptive Removal of Zn (II) And Co(II) Ions Using The Batch Technique.*

The different experimental controlling parameters as pH, contact time, nanosorbent dosage and initial metal ion concentration on the adsorptive removal of Zn(II) and Co(II) were investigated using batch equilibrium technique according to the following procedures.

### *2.4.1 Effect of pH On The Metallic Adsorption Capacity*

The effect of pH was studied as the following, 25.0±1 mg of **Na.Bent-O-Amine** was placed in a 50.0 mL measuring flask, 9.0 mL of the acidic buffer solution from pH 1.0 to 7.0 was added to the flask, then1.0 mL of 0.1 mol $L^{-1}$ of the selected metal ion Zn(II) or Co(II) solution was added to the previous reaction mixture and shaken for 30 min at room temperature using automatic shaker, finally a filtration process was performed using suitable filter paper and the free metal ion in the filtrate was mixed with the appropriate buffer solution and indicator to titrate against 0.01 mol $L^{-1}$ EDTA. The procedure was repeated for three times and the average value was calculated to determine the metal capacity value according to **Eq. (1).**

$$\mathbf{q} = \frac{(\mathbf{C}_{\circ} - \mathbf{C})\mathbf{V}}{\mathbf{m}} \qquad \mathbf{(1)}$$

Where, Co and C are the initial and residual metal ion concentration in mol $L^{-1}$, respectively, V is the aqueous volume of the adsorption reaction (L), m is the mass of adsorbent (g) and q is the adsorption metal capacity in $mmolg^{-1}$, that represents the amount of metal ion mmol, adsorbed per gram of dry adsorbent.

### *2.4.2 Effect of Contact Tme On The Metallic Adsorption Capacity*

Investigation the equilibrium for this reaction was occurred by taking 25.0±1 mg of **Na.Bent-O-Amine** in a 50.0 mL measuring flask, 9.0 mL of the optimum value of buffer solution for each metal ion was added to 1.0 mL of 0.1 mol $L^{-1}$ from the selected metal ion Zn (II) or Co(II) and shaken for different periods of time like "1, 5, 10, 20, 30, 40, 50 and 60 min". The procedure was repeated three times, completed as described at 2.4.1 step and the metal capacity values were calculated using **Eq. (1).**

### *2.4.3 Effect of Nanosorbent Dosage on The Metallic Adsorption Capacity*

Nano adsorbent dosage effect on the adsorptive removal of Zn(II) or Co(II) was studied using different masses (5.0, 10, 15, 20, 25, 30, 40, 50, 60, 70, 80, 90 and 100.0±1 mg) of **Na.Bent-O-Amine**, mixed with 9.0 mL of the optimum buffer solution and 1.0 mL of 0.1 mol $L^{-1}$ solution of selected metal ion Zn(II) or Co(II) in a 50.0 mL measuring flask, this mixture was shaken under the previous optimum conditions of pH and contact time using automatic shaker. The procedure was repeated three times and the metal capacity values were calculated using **Eq. (1).**

### *2.4.4 Effect of Different Ionic Concentration On The Metallic Adsorption Capacity*

A sample of **Na.Bent-O-Amine** 25.0±1 mg was placed in a 50.0 mL measuring flask, 9.0 mL of the optimum value of buffer solution for each metal ion was added in the presence of different metal ion concentrations ranged from 0.1 to 2.0 ml of 0.1 mol $L^{-1}$ from the selected metal ion Zn(II) or Co(II). The mixture was shaken for the optimum time of each metal ion as indicated in section 2.4.2. The procedure was repeated three times and the metal capacity values were calculated using **Eq. (1).**

### *2.4.5 Adsorptive Removal of Zn(II) and Co(II) Metal Ions Using Column Technique*

100.0 ml of tap water sample was spiked to obtain concentration 1.0 ppm of the target metal ion and passed through column system packed with 20 ±1 mg of **Na.Bent-O-Amine** for Zn (II) or Co (II), using a flow rate of 2 mL $min^{-1}$ under air pressure. The initial concentration and the effluent solution concentration were determined using atomic absorption spectroscopy to calculate the percentage of removal values according to **Eq. (2)**.

$$\mathbf{Removal\%} = \frac{\mathbf{(C_O - C_F)}}{\mathbf{C_O}} \mathbf{X\,100} \qquad \mathbf{(2)}$$

Where, $C_O$ and $C_F$ (mg $L^{-1}$) were the initial and final metal ion concentration, respectively. The application procedure was repeated three times and the average percentage removal values were calculated.

### *2.4.6 Adsorptive Removal Of $^{65}$Zn, $^{60}$Co Radioisotopes From Radioactive Wastewater*

Irradiation of Zn (II) and Co(II) was achieved in Egypt Second Research Reactor to obtain simulated radioactive waste water contaminated with $^{65}$Zn and $^{60}$Co. Removal of $^{65}$Zn and $^{60}$Co radioisotopes from this radioactive waste water was performed using 100 mL with concentration 1.0 ppm of the radioactive sample was passed over a micro-column system packed with the same amount of **Na.Bent-O-Amine** at step 2.4.5 at a constant flow rate of 2 ml/min under air pressure. Both of the initial and effluent activities of the produced isotopes due to (n, γ) reaction were determined using NaI detector.

## 3 Results and Discussion

### *3.1 Characterization of Nanoadsorbent*

The acquired FT-IR spectra of **Na.Bent-O-Amine** was illustrated in Fig.1 in the frequency range of 400 - 4000 cm$^{-1}$. **Na.Bent-O-Amine** was characterized by the presence of a characteristic peak at 1038.59 cm$^{-1}$ which was attributed to the Si-O in the plane, the presence of the Si-O band was strong evident for the silicate structure of the clay. Also the bands at 466.95, 524.10 cm$^{-1}$ were corresponded to Si-O-Al octahedral and Si-O-Si bending vibrations, the two peaks at 625.11, 841.32 cm$^{-1}$ were assigned to O–Si–O asymmetric stretching, the presence of the two peaks at 1646.77, 3438.31 cm$^{-1}$, were referred to the bending and stretching vibrations of the–OH groups respectively for the water molecule adsorbed on the surface of clay, a band at 3622.98 cm$^{-1}$ represented the stretching vibrations of the OH groups coordinated with the octahedral Al$^{3+}$ cation **[**8,12,15,21**].**

The oleyl amine was observed through the following the two peaks at 2855.19 and 2927.99 cm$^{-1}$were corresponded to the symmetric and asymmetric stretching vibration of the CH$_2$, respectively. The peak at 722.2 cm$^{-1}$ is due to the rocking vibration and the peak at 787 cm$^{-1}$ can be attributed to a NH$_2$ wagging vibration, which was the characteristic peak for oleyl amine **[**29**].**

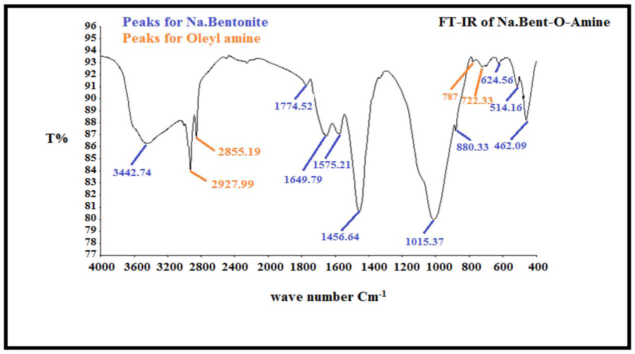

**Fig 1.**FT-IR spectra of **Na.Bent-O-Amine** nanosorbent

XRD diagram of **Na.Bent-O-Amine** was shown in Fig.2 as the following series of peaks at 2θ = 19, 36, 63 were characterized due to the presence of aluminum silicate structure of bentonite, with the consideration that the oleyl amine is not detected through X-ray diffraction analysis **[**8,12,15,21**].**

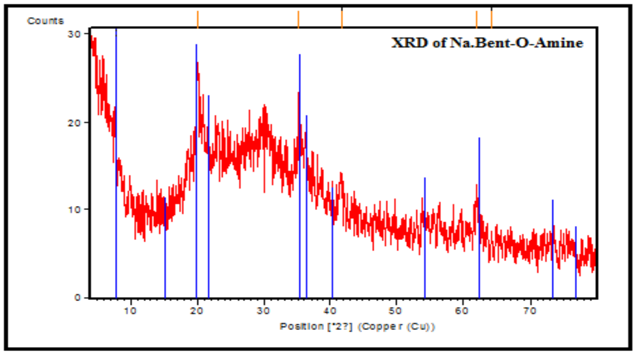

**Fig 2.** XRD pattern of **Na.Bent-O-Amine** nanosorbent

The high-resolution transmission electron microscopic (HR-TEM) imaging of **Na.Bent-O-Amine** was acquired using 200 k, as represented in Fig.3 its particle size was found in the range from (13.59-16.31) nm.

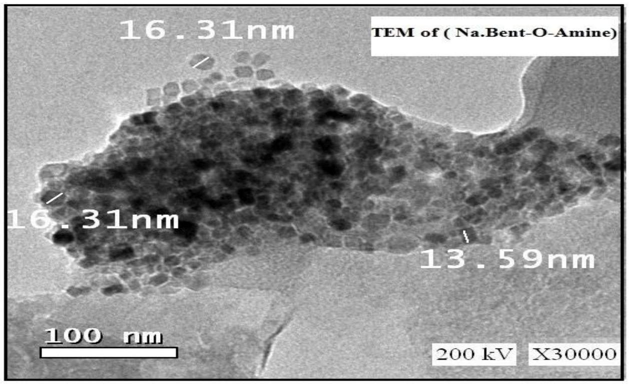

**Fig 3.** HR-TEM of **Na. Bent-O-Amine** nanosorbent

### *3.2 Adsorption Removal of Zn (II) And Co(II) Using Na. Bent-O-Amine*

#### *3.2.1 Effect of pH On The Adsorption Capacity Values*

The pH factor is the most important factor to study the adsorptive removal of Zn (II) and Co(II) using **Na.Bent-O-Amine** nanosorbent. The results of this study were shown in Fig.4.a the metal capacity values were expressed with mmol g$^{-1}$ versus the pH values for metal ion solution. Nano bentonite has a lot of advantages as its high surface area which attributed to the small particle size, large active sites result from the activation process with $H_2SO_4$, also NaOH treatment was established to increase its adsorption capacity **[**8, 9**].** The structure of activated nano bentonite has sheets of silicate layers these layers are loaded with –OH groups which result from the combination of Si-O with the hydrogen atom in the aqueous medium. Coating the surface of nano bentonite with oleyl amine increase the metal uptake in this case through the functionalization the surface of bentonite with the amine group (-$NH_2$) which is responsible for the metal adsorption removal through binding via ion exchange or the formation of a metal complex **[**24**].**The reactivity of surface loaded with (-$NH_2$) groups was dependent on the pH value of solution, at low pH values less than 3.0 there was a strong competition between the target metal ion Zn (II) or Co (II) and the high concentrations of hydrogen ions which protonate the surface of active binding sites which lead to increase the positive charge density on the surface of nano-adsorbent, this process leads to increase in the repulsive forces between positively charged protonated surface functional groups and positively charged metal ions **[**25**].**

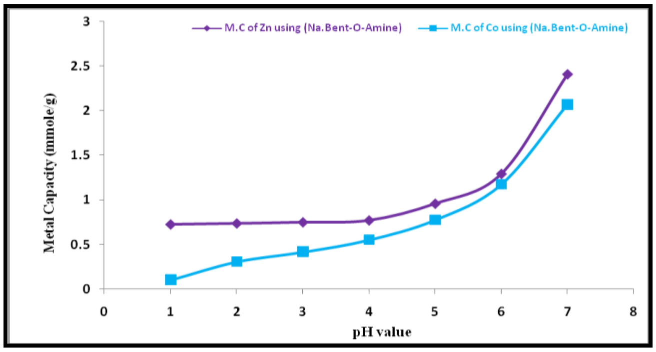

**Fig 4 a** Effect of different Values of pH on the metal capacity for Zn (II) / Co(II)

In Fg.4.a for pH 1.0-3.0, the metal capacity values of Zn(II) increased slightly within the range 0.728-0.752 mmolg$^{-1}$. The same trend was observed for Co (II) but with obvious increasing in the adsorptive removal, within the range 0.1 - 0.415 mmolg$^{-1}$. At high pH values 6.0-7.0,

surface protonation decreased, this lead to the (-$NH_2$) on surface of activated bentonite had a strong binding affinity with the target metal ions for Zn(II) and less stronger for Co(II), the metal capacity was 2.409 mmolg$^{-1}$ for Zn(II) , 2.071 mmolg$^{-1}$ for Co(II) at pH 7.

### *3.2.2 Effect of Contact Time On The Adsorption Capacity Values*

The contact time for the adsorptive removal of Zn(II) or Co(II) using **Na.Bent-O-Amine** was investigated at different time (1, 5.0, 10, 15, 20, 30, 40, 50 and 60 min) at the optimum reaction pH 7 for Zn(II)/Co(II) in the presence of initial metal ion concentration was 0.1 mol L$^{-1}$.
Fig.4.b represented the metal adsorption capacity values with (mmolg$^{-1}$) for Zn(II)/Co(II) versus the contact reaction time in (min). A gradually increasing in the metal capacity with increasing the time from 1 to 50 min for Zn(II), at 50 min the equilibrium was obtained, the metal capacity was 2.393 mmolg$^{-1}$, for Co (II) the equilibrium was obtained at 60 min with the metal capacity 2.333 mmolg$^{-1}$

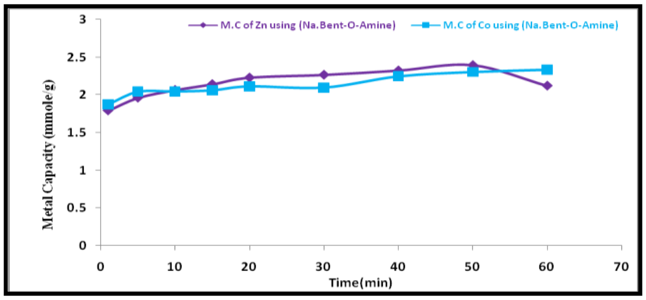

**Fig 4 b** Effect of shaking time on the metal capacity for Zn (II)/Co (II)

### *3.2.3 Effect of Nanosorbent Dosage On The Adsorption Capacity Values.*

The nanosorbent dosage was achieved in the presence of optimum pH and contact time conditions for Zn (II) and Co (II), with different masses of nanosorbent as (5.0, 10.0, 15.0, 20.0, 30.0, 40.0 to 100.00 mg) to determine the variation in the metal adsorption capacity values.

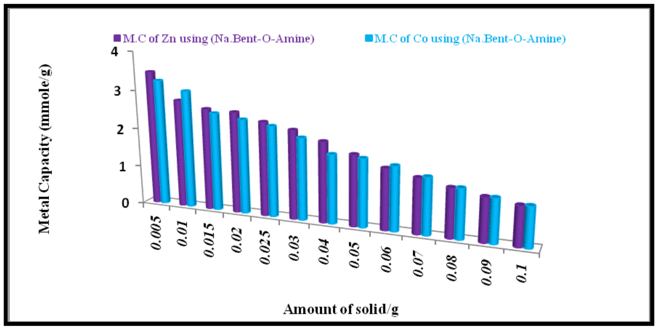

**Fig 4 c.** Effect of nanosorbent dosage on the metal capacity for Zn (II)/Co (II)

Fig.4.C showed the effect of the amount of nanosorbent on the removal processes. The obtained results indicated that the metal capacity values decrease with increasing the mass of nanosorbent from 5 to 100 mg. The highest metal capacity for Zn (II) was obtained with 5.0 mg as 3.472 mmolg$^{-1}$, the lowest was obtained with 100.00 mg as 0.995 mmolg$^{-1}$. The same behaviors were observed for Co(II) ions, the highest metal capacity value was obtained as 3.259 mmolg$^{-1}$ using 5.0 mg and the lowest value was obtained with 100.00 mg as 1.01 mmolg$^{-1}$ using 100 mg. The observed behavior in low nanosorbent dosage was mainly attributed to the greater availability of metal ions compared to the active surface functional groups on the nanosorbent. On the other hand, the low metal adsorption

capacity values at high mass were mainly due to the less availability of the functional groups compared to a number of available metal ions in aqueous solution **[**27**].**

### *3.2.4 Effect of Initial Metal Ion Concentration On The Adsorption Capacity Values.*

Different values of initial metal ion concentrations from 0.1 to 2.0 ml of 0.1mol$L^{-1}$ were used to investigate the change in the adsorption capacity. Fig.4.d showed the results of the metal capacity values for Zn (II)/Co(II) using **Na.Bent-O-Amine**. The obtained results indicated that by increasing the initial metal ion concentration, this led to an increase in the metal adsorption capacity values of Zn (II) / Co (II). The adsorption capacity values of both Zn (II) / Co(II) were found to gradually increase with increasing the metal ion concentration from 0.1 to 2.0 ml of 0.1 mol$L^{-1}$. The highest metal capacity values were obtained at concentration 2.0 ml of 0.1 mol$L^{-1}$ as 2.926, 3.26 mmol$g^{-1}$ for Zn(II) and Co(II), respectively using **Na.Bent-O-Amine** nanosorbent. These results were argued to the generated driving force by increasing the initial metal ion concentration this affected the mass transfer barrier between the nanosorbent and the metal ion in the contact solution **[**28**].**

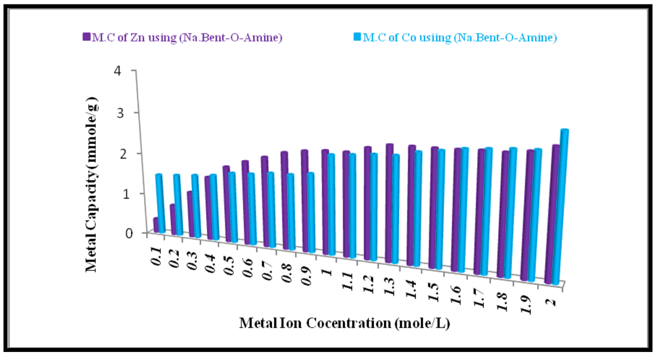

**Fig 4 d.** Effect of metal ion concentration on the metal capacity for Zn (II)/Co (II)

### *3.3 Applications of Na.Bent-O-Amine In The Removal of Zn (II), Co (II) And Their Radio-Active Isotopes $^{65}$Zn, $^{60}$Co From Water Samples*

The application of **Na.Bent-O-Amine** to remove 1.0 mg $L^{-1}$ from Zn (II), Co(II) from tap water and $^{65}$Zn, $^{60}$Cofrom radioactive wastewater were investigated using multi-stage micro-column system packed with 20 ±1 mg of **Na.Bent-O-Amine** nano-adsorbent. Table.1 showed a good obtained results of this study which indicated a good correlation values between removal of Zn(II), Co(II) metals from tap water and $^{65}$Zn and $^{60}$Co from their radioactive waste water, the removal process was achieved through three runs, the first run removal process was in the range 20-25% for Zn(II), $^{65}$Zn , 24.5- 30 % for Co(II), $^{60}$Co. The second run was in the range 60-65% for Zn (II), $^{65}$Zn, 67-70 % for Co (II), $^{60}$Co. The third run was in the range 90-91.5 % for Zn (II), $^{65}$Zn, 90-93.5% for Co (II),$^{60}$Co.

**Table 1.**Removal of Zn (II), Co(II), $^{65}$Zn and $^{60}$Co from tap water and radioactive wastewater.

| Sample | Metal | Percentage Extraction (%) |
|---|---|---|
| Tap water | Zn(II) | 90% |
| | Co(II) | 90% |
| Radiowastewater | $^{65}$Zn Radioisotope | 91.5% |
| | $^{60}$Co Radioisotope | 93.5% |

## 4 Conclusion

The **Na.Bent-O-Amine** nanosorbent was characterized using FT-IR, XRD, and HR-TEM. The application of this nanosorbent for the adsorptive removal of Zn (II)/Co(II) from aqueous solutions was investigated in the different pH conditions whereas the optimum value for the extraction of the target metal ions was identified as pH 7. The maximum adsorptive capacity values were found to correspond to 225.68, 195.54 mg/g for Zn (II) and Co(II), respectively using 5.0 mg of **Na.Bent-O-Amine** nano-adsorbent. The efficiency of **Na.Bent-O-Amine** nanosorbent was evaluated with respect to recovery of Zn(II) and its $^{65}$Zn radioisotopes as well as Co(II) and its $^{60}$Co radioisotope from tap water and radioactive wastewater samples and the collected results were referred to excellent percentage recovery values in the range 90-91.5 % for Zn (II), $^{65}$Zn and 90- 93.5 % for Co (II) $^{60}$Co.

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