

SYNTHESIS, CHARACTERIZATION OF ANALCIME AND ITS APPLICATION IN WATER TREATMENT FROM HEAVY METAL

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ABSTRACT

In this study analcime was successively synthesized from kaolinite as a raw material using the fusion with NaOH method. The conditions of hydrothermal crystallization (zeolitization) were found to be at temperature of 170 C°, and time span between 36 h and 72 h for kaoline. The synthetic materials have been characterized by X-ray diffraction (XRD), scanning electron microscope (SEM), Fourier transform infrared spectroscopy (FT-IR) and thermo gravimetric (DTA/TGA) analysis. The results indicate that the crystallization of analcime not affected by the hydrothermal reaction time. The synthetic analcime experimented to up taking some of heavy metals from prepared slandered solution or natural contaminated ground water. The results showed that the synthetic analcime has good efficiency in removal of heavy metals ions (Fe and Mn) with concentrations up to 50 ppm, and Cd and Pb ions to about 10 ppm. The percent adsorption (%) was evaluated with changes in the parameters such as, dosage of adsorptent, PH and time for different heavy metals ions. The order of removal for the different ions is as follows: $Pb^{2+} > Cd^{2+} > Mn^{2+} > Fe^{2+}$. The Langmuir constants model for Fe^{2+} ions sorption on the adsorption isotherms is fitted well. The R_L value in the present investigation at concentration more than 5 ppm was equal or less than one, and equal one for Mn^{2+} , indicating that the adsorption of the metal ion by analcime is favorable.

Keywords: *Analcime, Kaoline, Hydrothermal Reactions, Zeolite, Clay ground water, aqueous solution, characterization, element contents, water treatment.*

INTRODUCTION:

Many toxic heavy metals have been discharged into the environment as industrial wastes, causing serious soil and water pollution (Lin, 2002). Pb^{2+} , Cd^{2+} , Fe^{2+} , and Mn^{2+} are especially common metals that tend to accumulate in organisms, causing numerous diseases and disorders (Inglezakis, 2003). They are also common

groundwater contaminants at industrial and military installations. Numerous processes exist for removing dissolved heavy metals, including ion exchange, precipitation, phytoextraction, ultrafiltration, reverse osmosis, and electro-dialysis (Thyfault and Mackayla, 2016). The use of alternative low-cost materials as potential sorbents for the removal of heavy metals has been

emphasized recently. Various treatment processes are available, among which sorption is considered to be cost-effective if low-cost sorbents such as zeolites are used. (Raeis, et al., 2014). Zeolites are naturally occurring hydrated aluminosilicate minerals. They belong to the class of minerals known as "tectosilicates. Most common natural zeolites are formed by alteration of glass-rich volcanic rocks (tuff) and alkaline rocks. Analcime is a hydrated sodium aluminum silicate which exists in cubic crystals. The chemical formula of analcime is $\text{NaAlSi}_2\text{O}_6 \cdot \text{H}_2\text{O}$. (Gatta et al., 2004), with small amount of potassium and calcium. The structure and chemical properties are more similar to the feldspathoids, even though they are classified to the zeolite mineral. Natural analcime can be found in the analcime basalt and other alkaline igneous rocks. Analcime can be used as an ion-sieve (Fabrizio et al., 2008). One of the analcime characteristics is exchange ions at room temperature, and increasing temperature make the ion-exchange more easily. This is due to the presence of smaller pores in analcime structure (Azizi et al., 2013). Generally, zeolite minerals are rare in all of the world, therefore over 200 synthetic zeolites have been synthesized either using chemicals or natural materials. The main objective of this study focused on utilization of the low cost and abundance materials such as clay (kaolinite) for synthetic analcime, and used it to remove heavy metal ions such as Fe^{2+} , Mn^{2+} , Pb^{2+} and Cd^{2+} from prepared standard solutions and natural ground water from Wadi Qena area.

1. HYDROGEOLOGICAL SETTING OF WADI QENA

Wadi Qena is located in the north part of the Eastern Desert. It lies between latitudes $26^{\circ}10'$ and $28^{\circ}15'$ N, and longitudes $32^{\circ}31'$ and $32^{\circ}45'$ E Fig. (1). Most of the hydrogeological studies that have been carried out in wadi Qena were focused mainly on the downstream of the wadi. Among these studies; El-Ramly 1972, Gomaa 1992; Thorweihe et al. (1993); Aggour 1997; Assiut University 2001; Yehia A. 2001; Abdel Moneim 2005; Seleem 2014; Abdel Moneim et al. (2015). The review of the previous publications indicated that groundwater in Wadi Qena and its surroundings exist with different potential in six aquifers under different hydrogeological conditions. These units are; quaternary Alluvium aquifer, Pliocene Sandstone aquifer, Lower Maestrichtian phosphate aquifer, Companion Marl aquifer, Turonian-Santonian Sandstone aquifer, Precambrian Basement Complex aquifer. Also, the groundwater in the study area can be classified into shallow and deep. Shallow groundwater aquifers occur mainly within the Quaternary alluvial sediments. The main recharge is from the local rainfall and flash floods that occur due to short-period heavy storms falling on the mountainous zones and sedimentary hills. Recharge from the deep aquifers through subsurface joints and fractures, as well as from the Nile aquifer, could be expected. The deeper aquifer is in Nubian sandstone and is potentially good for agriculture. Its thickness ranges from 300m to 800 m. At relatively shallow depths (less than 500 m), the groundwater is brackish (total dissolved

solid; TDS more than 4876 ppm). However, at greater depths (more than 500 m), the groundwater is fresh (TDS of 580; Rashed et al., 2006).The study of hydrochemical

characteristics in Wadi Qena revealed on the high concentration of Fe and Mn in some studied wells relative to (WHO, 2004) and (A.R.E, 2007).

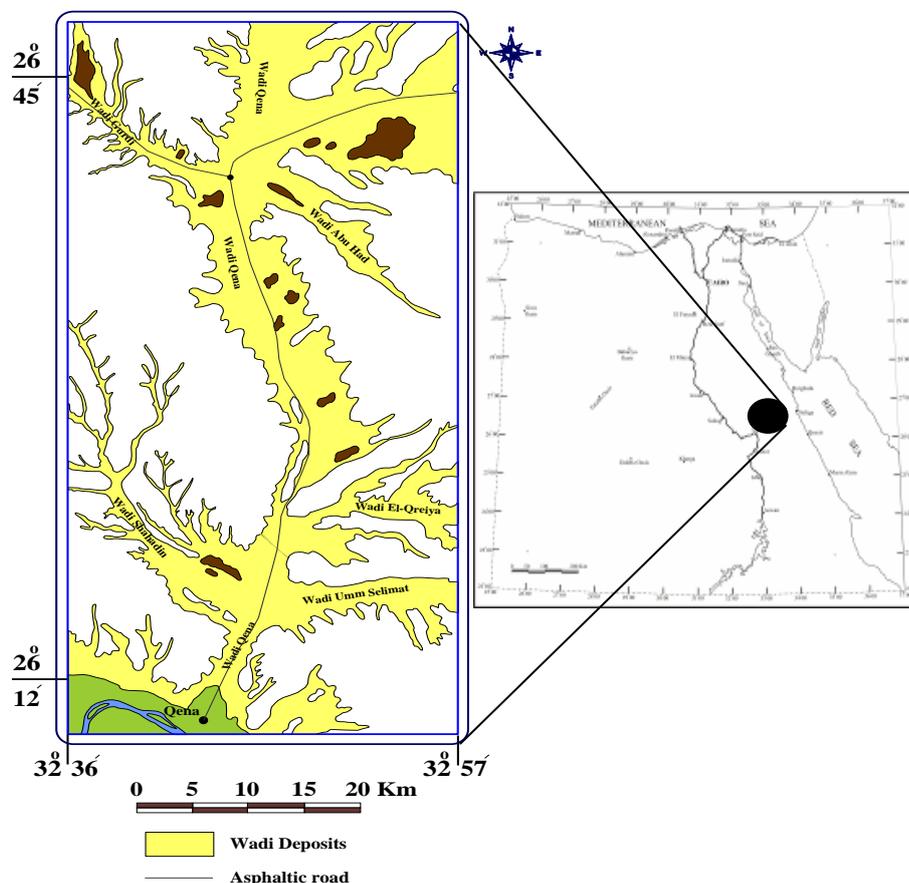


Fig. (1): Location map of the Wadi Qena

3. METHODOLOGY

Two method were used to synthetic of analcime using kaolinite raw material as

a source of Al and Si. The chemical composition of the raw material kaolinite are given in Table (1)

Table (1).Chemical composition of kaolinite

Composition	SiO ₂	TiO ₂	Al ₂ O ₃	Fe ₂ O ₃	MnO	MgO	CaO	Na ₂ O	K ₂ O	P ₂ O ₅	SO ₃
Content (wt. %)	49.2	2.82	32.97	6.82	0.24	2.2	9.43	0.8	0.05	0.13	0.26

1. By using alkaline fusion (after Rios et al, 2008)to synthetic analcime using kaolinite NaOH pellets was mixed with the calcinated Kaoline by ratio 1.2: 1 and the mixture was fused at 600 C° for 1 h . The alkaline reagent added to the starting material acts as an activator agent during fusion. The alkali-fused products were then dissolved in water (H₂O/alkali fused starting material

ratio)=(1:4.9), generally under stirring conditions until the reaction gels were homogenized aged for 24 h under static conditions. The mixtures were transferred into polytetrafluoroethylene (PTFE) bottles of 200 ml and a stainless steel autoclave, then crystallized under static conditions at 170 C° for 5, 3, 1.5, days .After removal from the oven they were quenched in cold water

and the product recovered by vacuum filtration, washed well with distilled water until the pH reach up to 11 and dried at 80 C° overnight.

2. Analcime was synthesized through a hydrothermal method using the following reaction condition:

$6Na_2O: 0.75Al_2O_3:30SiO_2. 780H_2O$. At first, 4.75g of sodium hydroxide were dissolved in 10 g water. To this solution about 3.575 of kaolinite were added. The mixture was stirred (200-300 rpm), thereafter 161.25 g of water were added and stirred. The gel was then transferred to polytetrafluoroethylene (PTFE) bottles (200 ml) and a stainless steel autoclave, then put in oven at 170 C° for 36h. After removal from the oven, quenched in cold water, and the product recovered by vacuum filtration, washed with distilled water and dried at 80 C° overnight. The synthetic zeolite was identified and characterize by the next measurements. The X-ray diffract graphs of the raw material and synthetic zeolite were obtained by using X-ray diffraction pattern, recorded on a Philips Expert (30 mA, 40 kV) with CuK α radiation, The morphologies of the raw material and synthetic zeolite were examined by scanning electron microscopy (SEM using a Jeol JSM-5400 LV instrument. SEM sample was prepared on a

copper holder by placing a smooth part of the Zeolites powder and then covered with gold-palladium alloy. SEM images were taken using a Penta Z Z-50P Camera with Ilford film at an accelerating voltage of 15 kV using a low-dose technique. FT-IR spectra were recorded on IR-470, Infrared spectrophotometer, Shimadzu by using the KBr pellet technique. And thermo gravimetric analysis (TGA) and differential thermal gravimetric (DTG) were carried out in air with Shimadzu DTG-60 at heating rate of 10°C/min.

4. RESULTS AND DISCUSSION

4.1. CHARACTERIZATION

1. X-RAY DIFFRACTION

The identification and characterizations of the raw material and synthetic zeolite shown in fig.(2). a 2θ range of 5-55°. Kaoline is the predominant mineral phase in the raw material which can be identified by its characteristic XRD peaks at 12.34° and 24.64° 2θ Fig.(2a). However, minor mineral impurities, such as quartz, illite, muscovite and halloysite, also occur. The X-ray diffractograms of the synthesized give reflection peaks at 15.76°, 25.93° and 30.48° As shown in the XRD patterns Fig. (2b), which is consistent with the analcime (see Treacy and Higgins, 2001; Zhao et al., 2004).

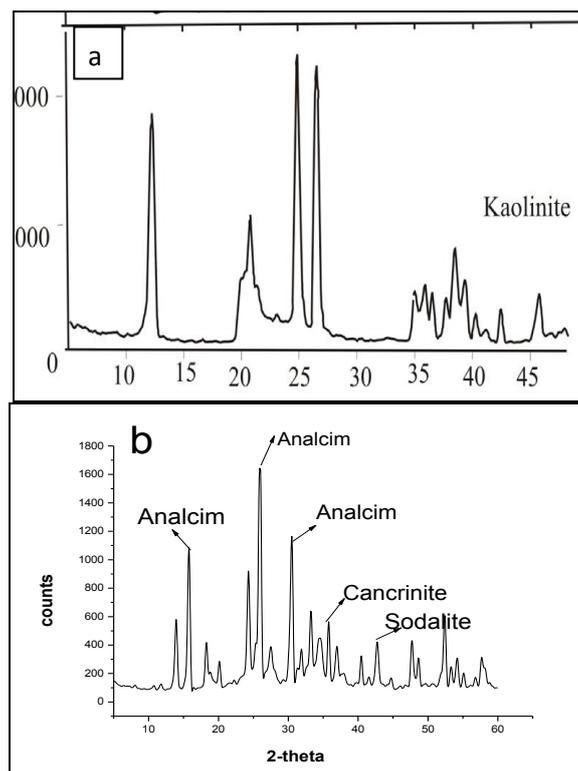


Fig.(2): XRD patterns of a) Raw material kaolinite b)Synthetic analcime

2. SCANNING ELECTRON MICROSCOPE (SEM)

The morphology of the raw material and synthetic zeolite were examined shown in

Fig. (3 a, b). The main morphological features observed in the synthesized analcime from met kaolinite are, spherical and cubical Fig.(3 a, b).

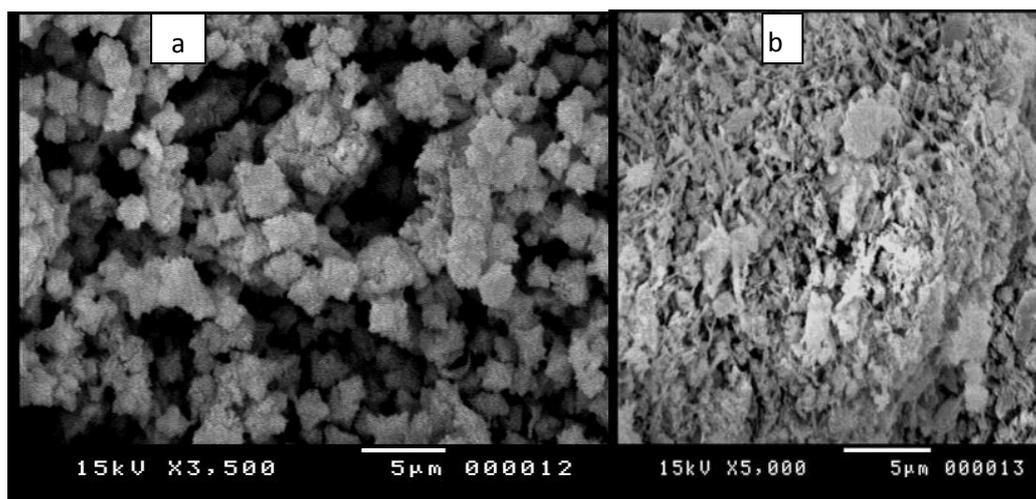


Fig. (3). SEM micrographs of (a) Raw material kaolinite ;(b) Synthetic analcime

FOURIER TRANSFORM INFRARED (FT-IR) SPECTROSCOPY

The characterization of raw material (kaolinite) and synthetic analcime with transmission Fourier transform infrared spectroscopy (FT-IR) is described in Table (2), Fig. (4 a, b). The characteristic OH-stretching vibrations of kaolinite band at 3460.71 cm^{-1} . The symmetric stretch ($753\text{-}71\text{ cm}^{-1}$), double ring vibration ($63\text{-}44\text{ cm}^{-1}$), T-O bending modes (44.04 cm^{-1}), or the internal linkage vibrations of TO4 (T=Si or Al) tetrahedral and to symmetrical stretching respectively. The bands at (1677.26 cm^{-1}) is assigned to the water in the channels of kaoline. FT-IR spectroscopy is used to probe the structure of the synthesized Analcime and monitor reactions in zeolite pores. Specifically, structural information can be obtained from the vibrational frequencies of the zeolite lattice observed in the range between 200 and 1500 cm^{-1} , (Li., 2005). In general, each zeolite has characteristic infrared pattern. However, some common features are observed which, include the asymmetric ($950\text{ - }1250\text{ cm}^{-1}$) and symmetric stretch ($660\text{ - }770\text{ cm}^{-1}$), double ring vibration ($500\text{ - }650\text{ cm}^{-1}$), T-O bending modes ($420\text{ - }500\text{ cm}^{-1}$), and possibly opening modes ($400\text{ - }420\text{ cm}^{-1}$). The FT-IR-spectral data of the synthesized analcime is presented in (Fig.4b). The double rings (D4R and D6R) in the framework structures of the zeolitic ($500\text{ - }650\text{ cm}^{-1}$), is near to 653 (s) cm^{-1} , which is characteristics Analcime, (Rios et al., 2008). The bands at 462 (s) cm^{-1} of analcime, is near the absorption bands within the range 420--

500 cm^{-1} which are related to the T-O-T bending of vibration mode (T = Al, Si) respectively. These absorption bands characterizing T-O bending vibrations may shifted to lower frequencies due to decreasing Si/Al ratio in the internal linkage due to the different length of the Al-O and Si-O bands. (Li., 2005), (Mohammed et al., 2013). The band 724 (s) cm^{-1} of analcime is near from the bands in the range $720\text{--}790\text{ cm}^{-1}$ is associated with symmetric stretching vibration of 4-membered rings. Fig. (4b). this band should be assigned to the 4-membered ring vibrations. Because these rings contain the lowest number of members of all rings occurring in the zeolite structure, therefore the bands due to these rings occur at relatively high wave numbers in the pseudo lattice band range (Wang et al., 2006). The band 1650 (w) , of analcime, is near the bands at $1647\text{ and }1648\text{ cm}^{-1}$ (Lewis sites) region is assigned to the zeolitic water in the channels of zeolite (Blanco et al., 1989). The bands at spectra $3446, 3460\text{ and }3482$ are attributed to the asymmetric stretching mode of molecular water coordinated to the edges of the zeolite channels (Zhao et al., 2006; Faghihian et al., 2009) and the band 3602 cm^{-1} of analcime is near the bands that are attributed to the asymmetric stretching mode of molecular water coordinated to the edges of the zeolite channels (Zhao et al., 2006; Faghihian et al., 2009). The bands that located at the lowest wave numbers $466\text{ and }377\text{ cm}^{-1}$, are corresponding to the characteristic bending vibrations carried out in the 4-membered rings. Table (2), (Mozgawa et al., 2005).

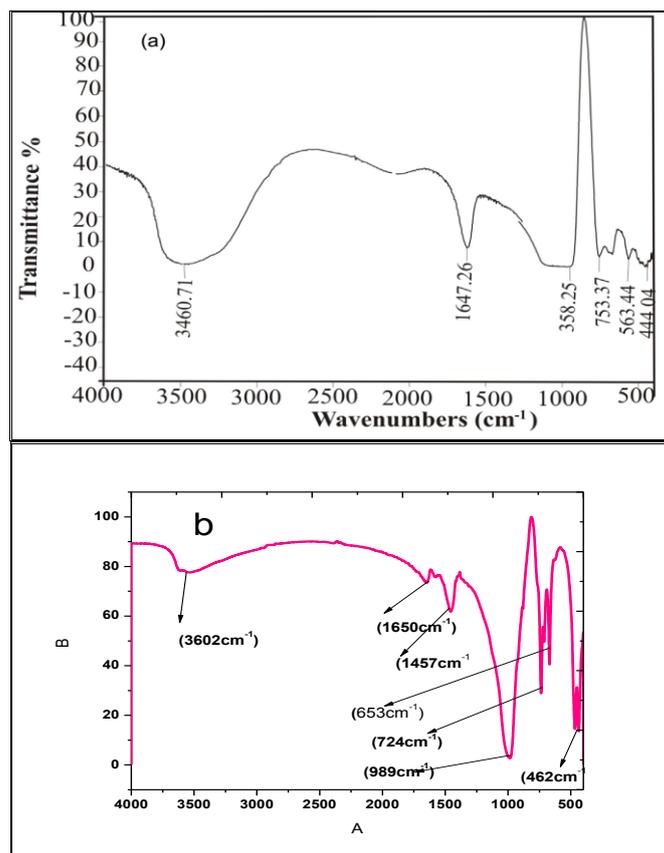


Fig. (4) Fourier transforms infrared spectroscopy (FT-IR) of. (a) Raw material kaolinite ;(b) Synthetic analcime

Table (2): Fourier Transform Infrared (FT-IR) Spectroscopy of Synthetic analcime

	Characteristic band (cm ⁻¹)	Analysis of bands
<i>Analcime</i>	3602 (b)	O—H stretching
	1650 (w)	Bending H—O—H
	1467 (m)	Na-T stretching
	939 (m)	the asymmetric Al—O stretch of sodalite
	724 (s)	4- or 6-membred double rings
	653 (s)	the symmetric Al—O stretch of sodalite
	462 (s)	Sending vibrations of Si-O and Al- O of the tetrahedral

4. THERMAL GRAVIMETRIC ANALYSIS (TGA) OF THE SYNTHESIZED ZEOLITES

The thermal behavior of synthesized analcime (TGA) and (DTG) are shown in Fig. (5). The synthesized product show up to four dehydration steps. The position of these DTG peaks and the number of dehydration steps can be attributed to the different compensating cation-water binding energies. As well as to

the different energy associated with the diffusion of the desorbed water through the porous structure of the synthesized product. Their weight loss percentages reflect the water loss from the zeolite structure, and the amount of desorbed water is related with the number of compensation cations in the framework of the zeolite. (Reyes et al., 2013).The peaks observed between 39-52 °C correspond to surface water in zeolitic materials; the peaks observed between 100 and 162 °C indicate zeolitic water, although

in some cases in this temperature range up to two peaks occur, which can be explained by the heterogeneous nature of the synthesized product. TGA curves also, showed a small weight loss in the range 0-2

% starting at 90°C until 150°C which may be attributed to loss of observed moisture and entrapped solvents. The thermographs of analcime are given in Fig. (5)

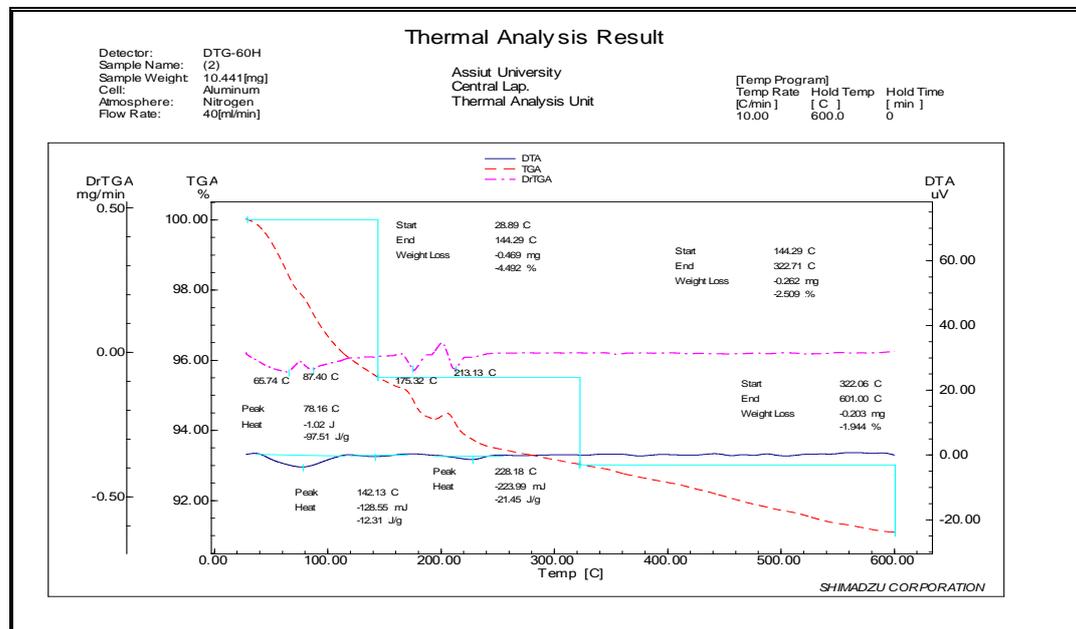


Fig.(5). Thermal analysis of the synthesized Analcime

4.2. ADSORPTIVE PROPERTIES OF THE SYNTHETIC ANALCIME

Batch adsorption studies

A batch adsorption experiment was conducted due to its simplicity In order to investigate the efficiency of the synthetic analcime in removal of some heavy metal. The experiments operated into natural ground water samples from Wadi Qena which contaminated by Fe and Mn. In addition to Fe and Mn, standard solution with concentration range from 5 to 50 ppm were prepared for Cd and Pb, by dilution Merk chemical standard solution (1000 ppm).The uptake of Cd (II), Pb (II), Fe (II) and Mn (II) onto zeolites as a function of their concentrations was studied at room

temperature, by varying the metal concentration from 5 to 50 mg/L and time while keeping all other parameters constant with respect to dose.

1. Effect of Studied analcime Dosage

The removal of Cd(II), Pb(II), Fe(II) and Mn(II) as a function of analcime dosage at the metal concentration from 5ppm, the solution pH 6-6.5, analcime dosage was varied from 0.1 to 1 g and equilibrated for 1 hrs. Increasing analcime dosage increased the percent removal of Cd(II), Pb(II), Fe(II) and Mn(II). And The results show that the optimum dosage is 0.5g, also The results clearly indicate the removal efficiency don't increase up to the optimum dosage Fig.(6).

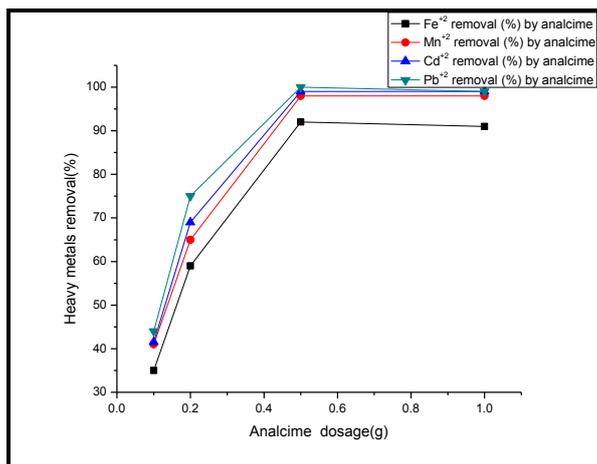


Fig.(6). Effect of Studied analcime dosage on adsorption of metal ions

2. Effect of metal ions concentration

The uptake of metal ions (Cd, Pb, Fe and Mn) onto analcime as a function of their concentrations was studied at room temperature and analcime dose equal 0.5 g, by varying the metal concentration from 5 to 50 mg/L, while keeping all other parameters constant. The results are given in Fig. (7). Percentage adsorption for Fe and

Mn decreases with increasing metal concentration in aqueous solution Fig (7). These results indicate that the order of affinity of analcime for reducing the studied heavy metal ions is as follows $Pb^{2+} > Cd^{2+} > Mn^{2+} > Fe^{2+}$. The heavy metal uptake may be attributed to different mechanisms of ion-exchange as well as the adsorption processes. (Ibrahim, et al., 2010).

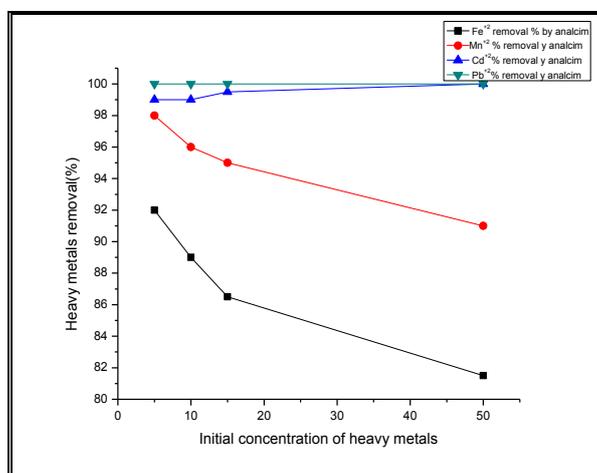


Fig. (7) Effect of initial concentration on adsorption of metal ions by studied analcime

3. Effect of contact time

The effect of contact time on the adsorption of Fe²⁺ and Mn²⁺ was examined at metal concentration 50 ppm, 0.5 g of synthetic analcime, at room temperature and pH (6-

6.5) for each ion with time varying between 5 and 60 min Fig.(8). The results show that the optimum contact time for Fe is 30 min, and 60 min for Mn Fig.(8).

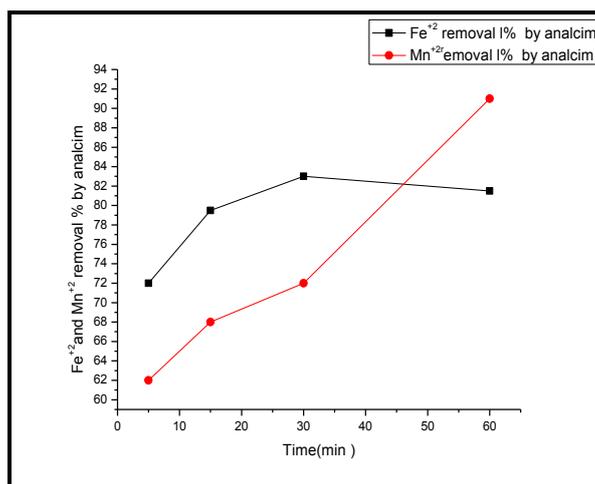


Fig. (8).Effect of contact time on adsorption of Fe²⁺, Mn²⁺ ions by analcime

4.3. REMOVAL OF (Fe²⁺, Mn²⁺) IONS FROM GROUND WATER SAMPLES AT WADI QENA

The concentration of Fe and Mn in the ground water samples from Wadi Qena were 5.5 ppm and 1 ppm respectively. The synthetic *analcime* was applied in the

removal of heavy metals (Fe²⁺, Mn²⁺) ions from ground water samples from wadi Qena. The sorption studies were carried out at different pH from 4.5- to 10.5, and time between 5 to 180 min. The optimum conditions for sorption of Fe and Mn were found at pH (6-6.5) and time at 60 min, using constant dose 0.5 g. The results are shown in Table (3).

Table (3) Heavy metal removal (%) by the studied zeolite for ground water samples from wadi Qena

Analcime	Heavy metal ion	Heavy metal removal (%)
	Fe ²⁺	88
	Mn ²⁺	64

From the above table it was noted that the synthetic zeolite analcime was found to be very effective in removing almost (Fe²⁺, Mn²⁺) ions from the studied samples. A selectivity series can be determined for analcime: Fe²⁺>Mn²⁺. It is noted that from table (3) and Fig. (8) The percentage of metal removed of (Fe²⁺, Mn²⁺) ions from the studied samples is smaller than its percentage from the standard solution at the same conditions. This is due to the effect of Effect of competitive metal

ions (Qin et al. 2006 and Sewwandi et al. 2014).

4.4. REMOVAL MECHANISM

1-The structures of zeolites consist of three-dimensional frameworks of SiO₄ and AlO₄ tetrahedral. The aluminum ion is small enough to occupy the position in the center of the tetrahedron of four oxygen atoms, and the isomorphous replacement of Si⁴⁺ by Al³⁺ produces a negative charge in the lattice. The net negative charge is balanced by the exchangeable cation (sodium,

potassium, or calcium). These cations are exchangeable with certain cations in solutions such as lead, cadmium, zinc, and manganese (Khachatryan, 2014). The fact that zeolite exchangeable ions are relatively innocuous (sodium, calcium, and potassium ions) makes them particularly suitable for removing undesirable heavy metal ions from industrial effluent waters.

• **Ion exchange:** Zeolites are capable of exchanging ions with external medium, which is the significant characteristic of zeolite. Ion exchange proceeds in an isomorphous fashion. The equilibrium ion exchange is expressed by the following equation (1)



where zA^+ and zB^+ are the valences of the respective cations, and L is defined as a portion of zeolite framework holding unit negative charge. Many zeolites contain several crystallographically distinct sets of sites that can be occupied by exchangeable ions. These sets of sites are intimately mixed with one another throughout the crystal, and each may exhibit different selectivities and ion-exchange behavior. The number of available exchange sites commonly exceeds the number of negative charges to be neutralized. Hence, the anionic charge of the framework may be neutralized when only some of the sites are occupied, and the occupancy factors may vary with the nature of the neutralizing cation. (Pabalan et al., 2001). In addition, the entering ion does not necessarily take the position of the leaving ion (Pabalan et al., 2001). (Sherry and Howard, 2003). Thus, the zeolites may exhibit a high degree of cationic disorder, both in terms of unoccupied sites and in

terms of different distributions of cations of different kinds among the site groups. Adsorption of heavy metal ions is a special characteristic of zeolites. The amount of metal adsorbed is affected by conditions like the nature and concentration of counter ions, pH, and metal solubility. (Hua et al., 2012).

• Also. The heavy metal uptake is attributed to different mechanisms of ion-exchange processes as well as to the adsorption process.

• So that according to the above expressions and the chemical composition of kaolinite (Tab.1), which considered the original material that enter in the synthesis analcime (The nature and concentration of counter ions). Analcime has affinity for heavy metals (Fe^{2+} , Mn^{2+} , Cd^{2+} and Pb^{2+}) up taking and as well as the ion-exchange process.,

2-Most Common Ions will exchange readily into most zeolites. However, the ion sieving effect is observed with the zeolites having the smallest pore openings and with the largest cations. For example Rb^+ (3.0 Å diameter) will slowly enter analcime but Cs^+ (3.4 Å diameter) will not, suggesting that analcime has an effective pore size of ~3.2 Å. So that the preference of these zeolites (analcime) for Pb^{2+} compared to the others (Tab.6). This is usually attributed to differences in metal characteristics and resultant affinity for sorption sites (Appel et al., 2002). For example, the hydrated radius of Pb^{2+} is smaller than that of Cd^{2+} , Mn^{2+} and Fe^{2+} as shown in table (3). Zeolites, in general, are weakly acidic in nature and sodium-form exchangers are selective for hydrogen (R-

Na+H2O ⇌ RH + Na+ + OH-), which leads to high pH values when the exchanger is equilibrated with relatively dilute electrolyte solutions (Wei, et al., 2006). Making metal hydroxide precipitation feasible. In natural zeolites these metals seem to reach saturation, which means that the metal had filled possible available sites and further adsorption could take place only at new surfaces. Ion-exchange capacity of heavy metal cations listed in Table (4). indicates the following selectivity sequence: Co²⁺>

Cu²⁺> Zn²⁺> Mn²⁺. The heavy metal cations are present as hexaqua complex ions with six surrounding water molecules in the solution and they passed the channel of zeolite in this form (Erdem et al., 2004). Since the adsorption phenomena depend on the charge density of cations, the diameter of hydrate cations is very important. The charges of the metal cation are the same (2+); therefore Mn²⁺ ions (the biggest diameter) have minimum adsorption, and Pb²⁺ ions (the least diameter) have maximum adsorption.

Table (4). Hydrated ionic diameters of heavy metals (adapted from Nightingale. (Abdul et al., 2011)

Metal ion	Hydrated ionic diameter (nm)
Pb ²⁺	0.401
Cd ²⁺	0.426
Mn ²⁺	0.83
Fe ²⁺	0.77

4.5. ADSORPTION ISOTHERMS OF FE²⁺, MN²⁺, CD²⁺ AND PB²⁺ IONS BY SYNTHETIC ZEOLITES

The equilibrium adsorption of the Fe²⁺, Mn²⁺, Cd²⁺ and Pb²⁺ ions were carried out by contacting 0.5g of the previous synthetic zeolites with different concentrations (5, 10, 15, 50) ppm from Fe²⁺ and Mn²⁺ ions and (5, 10, 15) ppm from Cd²⁺ and Pb²⁺ ions under room temperature, for 1 h and at pH (6-6.5) on the shaker. The mixture was filtered and the filtrate analyzed for the metal ions concentration using AAS. and the data was fitted into the following isotherms: Langmuir, Freundlich,

A. Langmuir Adsorption Isotherm

Adsorption isotherm data are quantified to describe the interactions

between the adsorbate and adsorbent and are critical in optimizing the use of adsorbent (Gupta et al., 2003). The Langmuir equation is the most popular of all the nonlinear isotherm expressions; it is a two-parameter equation (2).

$$\frac{C_e}{q_e} = \frac{1}{q_m K} + \frac{C_e}{q_m} \quad (2)$$

Where constants b and q_m relate to the energy of adsorption and adsorption capacity and their values are obtained from the slope and intercept of the plot of C_e/q_e versus C_e for room temperature. Langmuir adsorption parameters were determined by transforming the Langmuir equation (3) into linear form.

Where:

C_e = the equilibrium concentration of adsorbate (mg/L⁻¹)

q_e = the amount of metal adsorbed per gram of the adsorbent at equilibrium (mg/g).

Q_0 = maximum monolayer coverage capacity (mg/g)

K_L = Langmuir isotherm constant (L/mg).

The values of q_{max} and K_L were computed from the slope and intercept of the Langmuir plot of $1/q_e$ versus $1/C_e$ Fig. (9). (Ji, et al., 2013). Moreover the characterization of the Langmuir equation can be explained in terms of the equilibrium parameters (R_L) which is a dimensionless constant referred to as separation factor or

$$\frac{1}{q_e} = \frac{1}{Q_0} + \frac{1}{Q_0 K_L} C_e$$

equilibrium parameter equation (4).

(Pandey et al., 2010).

$$R_L = 1 / (1 + K_L C_0) \quad (4)$$

Where:

C_0 = initial concentration, K_L = the constant related to the energy of adsorption (Langmuir Constant).

R_L value indicates the adsorption nature to be either unfavorable if $R_L > 1$, linear if $R_L = 1$, favorable if $0 < R_L < 1$ and irreversible if $R_L = 0$.

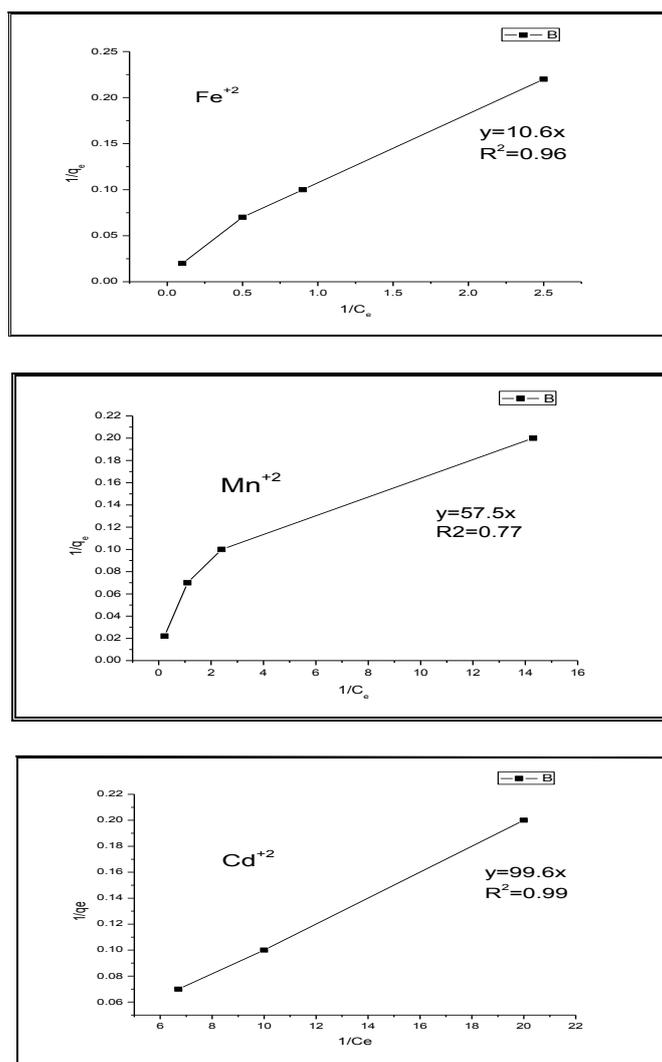


Fig. (9). Langmuir Adsorption Isotherm for adsorption of Fe^{2+} and Mn^{2+}

.From the correlation coefficient (R^2) values that are regarded as a measure of the

goodness fit of experimental data on the isotherm's model. And Table (5)

Table (5).Correlation factor for shape of isotherm

R value	Type of adsorption
$R > 1.0$	Unfavorable
$R = 1.0$	Linear
$0 < R < 1.0$	Favorable
$R = 0$	Irreversible

From the previous Table(5)The Langmuir isotherm is favorable model for Fe^{2+} , Mn^{2+} and Cd^{2+} ions sorption systems by using analcime, because of R^2 value for Fe^{2+} , Mn^{2+} and Cd^{2+} is 0.96, 0.8 and 0.99, respectively proving that the sorption data fitted well to Langmuir Isotherm modelAnd value $0 < R < 1.0$. Also, it noted that and R^2 of Cd^{2+} ion is the largest indicating that using the synthetic analcime for removal Cd^{2+} ion is morefit of experimental data on the isotherm's model. Also, the value of R_L that describes the nature of the adsorption: irreversible ($R_L = 0$); favorable ($0 < R_L < 1$); linear ($R_L = 1$); unfavorable ($R_L > 1$) (Rozada et al., 2007). From the data calculated in Table (8), in the case of Fe^{2+} , Mn^{2+} and Cd^{2+} ions are favorable ($0 < R_L <$

1); AndFrom this research work, the maximum monolayer coverage capacity (Q_0) from Langmuir Isotherm model was determined to be 10.6,57.5 and 99.6mg/g, for Fe^{2+} , Mn^{2+} and Cd^{2+} ions respectively and K_L (Langmuir isotherm constant) is 0 for the previousions.This again confirmed that the Langmuir isotherm was favorable for sorption of Fe^{2+} , Mn^{2+} and Cd^{2+} ions onto the synthetic zeolites (analcime) under the conditions used in this study (Unlu et al., 2006). (Zhanga et al., 2000). Anfortiontly from the data Tables (6), (7) it noted that although the removal (%) of Pb^{2+} ion by using the synthetic zeolites (analcime) is the highest but Pb^{2+} ion does not obeyed the Langmuir isotherm.

Table (6): Parameters for plotting Langmuir, Freundlich, Adsorption Isotherms of Fe^{+2} , Mn^{+2} and Cd^{+2} ions by the synthetic zeolites

ion/conc.		Co(ppm)	Ce(ppm)	1/Ce	Log Ce	Qe(mg/g)	1/Qe	Log Qe
Fe^{2+} /5ppm	Analcime	5	0.4	2.5	-0.4	4.6	0.22	0.7
Mn^{2+} /5ppm			0.07	14.3	-1.2	4.9	0.20	0.69
Cd^{2+}			0.05	20	-1.3	4.9	0.02	0.7
Fe^{2+} /5ppm		10	1.1	0.9	0.04	8.9	0.1	0.95
Mn^{2+} /5ppm			0.4	2.4	-0.4	9.6	0.1	0.98
Cd^{2+}			0.1	10	-1	9.9	0.1	1
Fe^{2+} /5ppm		15	2	0.5	0.3	13	0.07	1.1
Mn^{2+} /5ppm			0.9	1.1	0	13.9	0.07	1.14
Cd^{2+}			0.15	6.7	-0.8	14.9	0.07	1.2
Fe^{2+} /5ppm		50	9.3	0.1	1	40.7	0.02	1.6
Mn^{2+} 5ppm			4.5	0.22	0.7	45.5	0.022	1.65

Table (7): Linear regression equations Langmuir and Freundlich isotherm and constants for adsorption of Fe⁺², Mn⁺² and Cd⁺² ions on the synthetic zeolites

Metal ion	Analcime	Langmuir Adsorption			Langmuir equations
		Q _{max} (mg/g)	KL(L/m g)	R ²	
Fe ²⁺		10.6	0	0.96	Y=13.4x
Mn ²⁺		57.5	0	0.8	Y=57.5x
Cd ²⁺		99.6	0	0.99	Y=99.6x
		Freundlich Isotherm			Freundlich equations
		n	K _f (mg/g)	R ²	
Fe ²⁺		0.3	0	0.3	Y=0.3x
Mn ²⁺		0.01	0	-0.1	Y=0.01x
Cd ²⁺		-0.97	0	-3.1	Y=-0.97 x

Table (8) RL values for Fe⁺², Mn⁺² and Cd⁺² ions concentrations

Metal ion concentration (ppm)			Analcime	R _L value		
Fe ²⁺	Mn ²⁺	Cd ²⁺		Fe ²⁺	Mn ²⁺	Cd ²⁺
5				0.16		
10				0.09		
15				0.06		
50		-		0.02		

B. Freundlich Adsorption Isotherm

This is commonly used to describe the adsorption characteristics for the heterogeneous surface (Hutson et al., 2000). This equation predicts multilayer adsorption on heterogeneous surface, characterized by an exponential distribution of active sites. These data often fit the empirical equation (5) proposed by Freundlich:

$$Q_e = K_f C_e^{1/n} \quad (5)$$

Where

K_f = Freundlich isotherm constant (mg/g)

n = adsorption intensity;

C_e = the equilibrium concentration of adsorbate (mg/L)

Q_e = the amount of metal adsorbed per gram of the adsorbent at equilibrium (mg/g). Linearizing equation (6).

We have:

$$\log Q_e = \log K_f + 1/n \log C_e \quad (6).$$

The Freundlich coefficients k_F and n corresponding respectively to adsorption capacity and adsorption intensity have been obtained from the intercepts and slopes of linear plots log Q_e against log C_e (Fig. 10). Specifically, the linear least-squares method and the linearly transformed equations have been widely applied to correlate sorption data where 1/n is a heterogeneity parameter, the smaller 1/n, the greater the expected heterogeneity. This expression reduces to a linear adsorption isotherm when 1/n = 1. If n lies between one and ten, this indicates a favorable sorption process (Goldberg et al., 2005) and if the values of n were lower than one indicating the adsorption is less favorable

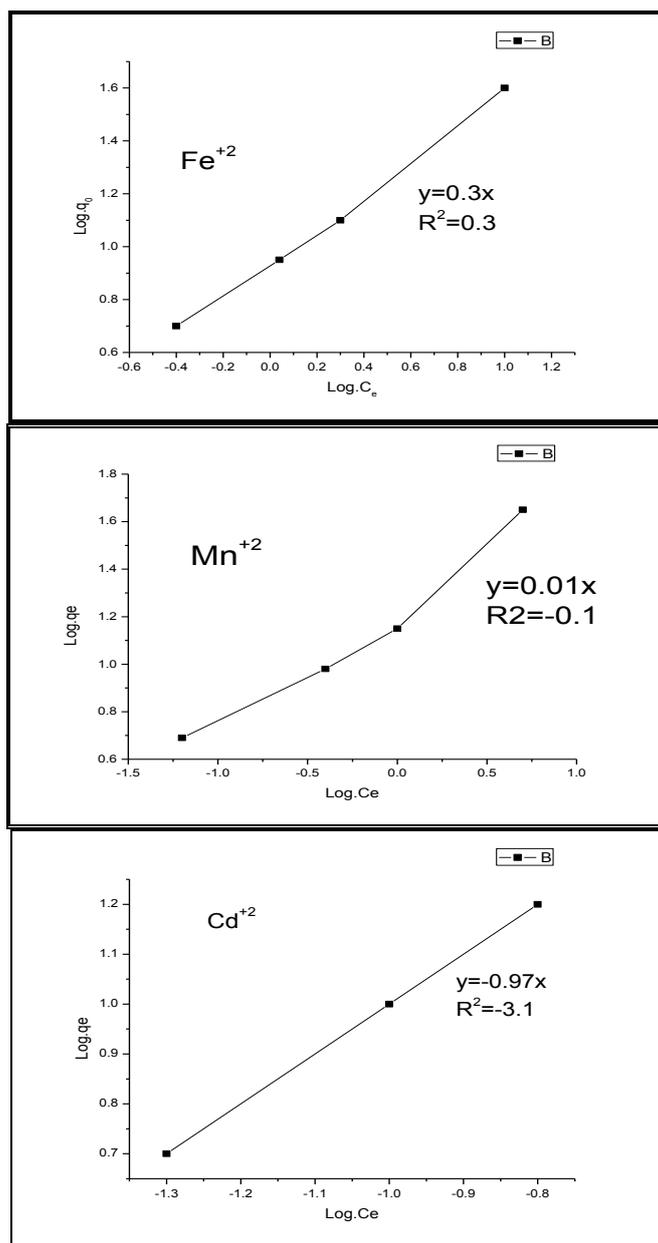


Fig. (10).Freundlich Adsorption Isothermplot for adsorption of Fe²⁺ and Mn²⁺

For the above Fig. (10).and from the calculated results of Fe²⁺, Mn²⁺ and Cd²⁺ ions it was found n were lower than one indicating the adsorption is less favorable adsorption by using synthetic zeolite (analcime). On the other hand, 1/n being above one in the case of Fe²⁺ and Mn²⁺ ions indicates cooperative adsorption and less favorable (Etale et al., 2016).but 1/n was the smaller than one in the case of Cd²⁺ ion indicating the greater the expected

heterogeneity. And favorable sorption. Also the R² value for Fe²⁺, Mn²⁺ and Cd²⁺ ions are 0.3, -0.1 and -3.1 respectively and it was demonstrated that the removal of Fe²⁺, Mn²⁺ and Cd²⁺ ions using the synthetic zeolite (analcime) did not obeyed the Freundlich isotherm (Goldberg et al., 2005). From the previous data in Table (5) it noted that although the removal (%) of Pb²⁺ ion by using the synthetic zeolites (analcime) is the highest but Pb²⁺ ion did not obeyed the

Freundlich isotherm. From the correlation coefficient (R^2) values in Tables (9) it was noted that, R^2 value of Fe^{2+} , Mn^{2+} and Cd^{2+} ions are 0.96, 0.88 and 0.99 respectively for the Langmuir isotherm adsorption model and was higher than R^2 value for the Freundlich model of Fe^{2+} , Mn^{2+} and Cd^{2+} ions that are 0.3, -0.1 and -3.1, respectively so that the experimental data for the adsorption process had better correlation coefficients values and better fits with the Langmuir isotherm model Fig. (9) than with the Freundlich model Fig. (10). Therefore, the adsorption process of Fe^{2+} , Mn^{2+} and Cd^{2+} ions onto the synthetic zeolite (analcime). Was found to follow the Langmuir isotherm model with a maximum monolayer adsorption capacity (10.6, 57.5 and 99.6) (mg/g) of Fe^{2+} , Mn^{2+} and Cd^{2+} ions, respectively. These results revealed the anchoring of Fe^{2+} , Mn^{2+} and Cd^{2+} ions to the abundant functional groups onto the synthetic zeolite (analcime) with the formation of monolayer surface coverage that was homogeneous in nature (Fernandez et al., 2003).

CONCLUSION.

Ground Water pollution with heavy metals and their purification acquires increasing importance for the development of modern industry. Higher levels of heavy metals in aquatic facilities pose a major risk to human health. It is therefore important to find ways to abate or remove such pollutants from ground water used for drinking water. In this study some heavy metals such as (Fe^{+2} , Mn^{+2} , Pb^{+2} and Cd^{+2}) was reduced from synthetic solution and natural ground water by synthetic of analcime. The use of zeolites as adsorbent

are often preferred due to their specific structure, which allows the selectivity of the processes involved (adsorption and ion exchange), and relatively simple operation with them and efficiency. Analcime was successively synthesized from kaolinite as a raw material using the fusion with NaOH method. The conditions of hydrothermal crystallization (zeolitization) were found to be at temperature of $170^\circ C$, and time span between 36 h and 72 h for kaoline. The synthetic materials have been characterized by X-ray diffraction (XRD), scanning electron microscope (SEM), Fourier transform infrared spectroscopy (FT-IR) and thermo gravimetric (DTA/TGA) analysis. Also, it was employed in solution for the removal of (Fe^{+2} , Mn^{+2} , Pb^{+2} and Cd^{+2}) ions. It was found analcime has good efficiency in removal of heavy metals ions (Fe and Mn) with concentrations up to 50 ppm, and Cd and Pb ions to about 10 ppm. The content of ions have been decreased in aqueous solutions during Batch methods of purification.

More than 92% of lead, Manganese and iron ions have been sorbed by analcime, The order of removal for the different ions is as follows: $Pb^{+2} > Cd^{+2} > Mn^{+2} > Fe^{+2}$. The experimental data for the adsorption process of Fe^{+2} , Mn^{+2} and Cd^{+2} ions onto analcime had better correlation coefficients values and better fits with the Langmuir isotherm model than with the Freundlich model.

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توليف ، توصيف الأناالسيم وتطبيقه في معالجة المياه من المعادن الثقيلة

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الملخص العربي :

يعتبر الأناالسيم من أهم معادن الزيولايت. وقد تم تخليقها بنجاح باستخدام عينات من الطين (الكاولينيت) باستخدام طريقة الانصهار مع هيدروكسيد الصوديوم ثم التفاعلات الحرارية المائية. وقد تمت عملية التبلور الحراري تحت ظروف من درجة حرارة ١٧٠ درجة مئوية، في فترة زمنية تتراوح بين ٣٦ إلى ٧٢ ساعة. وقد تمت دراسة خصائص المادة المخلفة بواسطة حيود الأشعة السينية (XRD) والميكروسكوب الإلكتروني (SEM) والتحليل الطيفي للأشعة تحت الحمراء (FT-IR) والخواص الحرارية التحليل الوزني الحراري (TGA 0)) وايضا تم استخدام هذه المادة المخلفة (الانالسيم) لإزالة بعض ايونات المعادن الثقيلة مثل الحديد، والمنجنيز، والرصاص، والكاديوم من محاليل تم تحضيرها من خلال تخفيف المحاليل القياسية لهذه المعادن (١٠٠٠ جزء في المليون). وايضا تم تطبيق (الانالسيم) في معالجة عينات من المياه الجوفية من (وادي قنا). وأظهرت النتائج أن (الانالسيم) المخلق لديه كفاءة جيدة في إزالة ايونات المعادن الثقيلة (الحديد، المنجنيز، الرصاص، الكاديوم) وقد تم تقييم نسبة الامتزاز (%) وكمية الأيونات الممتزة لكل وحدة كتلة من المواد المازة (مكافئ بالمليجرام من أيونات المعادن لكل جرام من المواد المازة) باستخدام طريقة الدفع. وايضا تم دراسة تاثير بعض العوامل مثل التركيز الابتدائي لأيونات المعادن السابق ذكرها ، والتغير في درجة الحموضة والزمن. وقد كان ترتيب إزالة الأيونات المختلفة على النحو التالي الرصاص <٢+ < الكاديوم <٢+ < المنجنيز <٢+ < الحديد. كما تم تطبيق عدة نماذج لإزالة أيونات (الحديد، المنجنيز) باستخدام (الانالسيم) المخلق ومن هذه النماذج منحنيات (Langmuir ,Freundlich isotherm's) كانت النتائج مناسبة في حالة التركيز الأكثر من ٥ جزء في المليون (<١ <RL) ، مشيرا إلى أن امتزاز هذه الملوثات على الانالسيم تتم بصورة جيدة .

كلمات البحث: الأناالسيم ، كاولين، التفاعلات الحرارية المائية، الزيوليت، الطين المياه الجوفية، محلول مائي، توصيف، محتويات العنصر، معالجة المياه.