

Some Adsorption Characterizations of the Egyptian Glauconite Ore

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MANY industrials use different adsorbent agents in purification of the liquid products or the wastewater. The use of natural materials as low cost sorbents for the removal of cations, especially heavy metals from aqueous solutions has recently received increasing attention. The present study was conducted to evaluate the some adsorption properties of the Egyptian glauconite ore. Batch equilibrium experiments were conducted using Cu (II), Fe (II) or Ca (II) with glauconite. The results reveal that the thermodynamic equilibrium of Cu (II), Fe (II) or Ca (II) with glauconite is attained at 20 min the curves obtained are single and smooth indicating the formation of monolayer of cations on the adsorbent surface of glauconite. The pH is one of the most important factors that affect the adsorption process. The maximum removals of Cu (II), Fe (II) or Ca (II) with glauconite are at pH 6, 5.5 and 8, respectively. The process of different cations uptake follows both Langmuir and Freundlich isotherm models and the first-order kinetics. The values of Langmuir constants for glauconite Q_{max} and b were found to be equal 3.76, 4.61 and 6.32 mg/g and 0.215, 0.046 and 0.062 l/mg, for Cu (II), Fe (II) and Ca (II) respectively. Based on the results of the present study, glauconite can be used as low cost cations adsorbent agent. Especially it has a big advantage that is the sandy loam textural makes the glauconite use also as a sand filter.

Keywords: Adsorption, Glauconite, Heavy metals, Copper, Iron, Calcium, Langmuir and Freundlich isotherm models, First-order kinetics.

The naturally occurring iron-potassium silicate "greensand" is also called glauconite (K, Na) (Al, Fe, Mg)₂ (Al, Si)₄ O₁₀ (OH)₂. A widely held concept is that glauconitization starts from a K-poor glauconitic smectite, which progressively evolves toward an end member constituted by a K-rich glauconitic mica (Odin and Matter, 1981). On the basis of their potassium content, glauconitic grains are ranked as nascent ($K_2O < 4\%$), slightly evolved (4–6%), evolved (6–8%), and highly evolved (>8%). According to Odin and Létolle (1980) and Odin and Matter (1981), the term *glauconite* should be restricted to a dark green, mica-type glauconitic mineral, whereas the general term *glaucony* should be used to designate green pellets enriched in glauconitic minerals, especially when precise mineralogical connotation is lacking. (Amorosi *et al.*, 2007).

In Egypt, Glauconites occur in the Western Desert associated with phosphorites and iron ores (Baioumy, 2007). According to El-Aref *et al.* (1999) the thickness of the overlying glauconitic sediments varies in thickness from up to 25 m in the Western and Eastern Wadis areas to less than 1 m in the high central area, El-Gideda mine of the Bahria Oasis.

Glauconite used as a substrate for growing agricultural plants (Pishmanov and Petkova, 2002), as soil amendment (Heckman and Tedrow, 2004), source of potassium for growing crops (Rao, and Rao, 1999 and Eid, 2012) or as low cost sorbent agent. Smith *et al.* (1996), evaluated adsorption equilibria for Cd(II), Pb(II) and Zn(II) on a sample of pass 200 mesh Lithuanian glauconite. They recorded that the relative adsorption capacity for the metals was in the order Pb > Cd > Zn. Although glauconite is a complex clay mineral, a single site approach provided a satisfactory description for many of the cases studied. Henriksen *et al.* (1998) investigated the possibility of segregating liquid manure so that all the N is transferred into a thick slurry phase by different natural and synthetic adsorbents, in order to allow the remaining liquid to be spread onto fields without causing environmental problems. They recorded that the clinoptilolite showed the highest capacity for ion exchange but glauconite was preferred due to its faster reaction time. Petkova *et al.* (2000) tested a zeolite - glauconite - phosphorite mixtures for decrease of lead uptake by plants. They found that the input of zeolite-glaucuite and zeolite-phosphorite compositions did not result in soil acidity neutralization but resulted in decreasing lead content of radishes by approximately two times compared to radishes grown on contaminated soil, which is due to their high ion exchange capacity and hemosorption. Lukyanik *et al.* (2001) used a natural sorbents for decalcination of purified juice. They suggested that the natural adsorbent separated glauconite should be used for softening of sugarbeet juices. Ringqvist *et al.* (2002) determine metal adsorption from wastewaters onto sphagnum and carex (peat samples) and compare it to the adsorption onto peat granules, clinoptilolite, glauconite and a flue dust from steel production. They reported that the combination of glauconite or clinoptilolite with the peat samples in column experiments gave a minor improvement in metal removal. The responses of calves receiving feed contaminated with radionuclides and heavy metals to the addition of glauconite to the basic ration as enterosorbents was studied by Fomichev *et al.* (2005). They concluded that the use of glauconite as enterosorbents of radionuclides and heavy metals for raising calves in Russia is effective.

The present study was conducted to evaluate the adsorption properties of Egyptian glauconite ore as a natural low cost sorbent agent.

Material and methods

Characterization of the adsorbent

The glauconite ore was obtained from the Central Metallurgical Research and Development Institute, Helwan, Cairo, Egypt, that was taken from Bahariya

Oasis, Egypt. Characterizations of some physical and chemical properties of glauconite ore were determined with the standard methods according to Page *et al.* (1982) and the results were listed in Table 1.

TABLE 1. Some physical and chemical properties of glauconite ore.

Characteristic	Units	Glaconite
Sand	%	69.3
Silt	%	17.2
Clay	%	13.5
Textural class		Sandy Loam
Saturation	%	70
EC	dS/m	7.03
Soil saturation extract		
pH		3.67
Soil paste		
CaCO ₃	%	0.1
Organic matter	%	0.12
Total N	mg/kg	168
Total K	%	2.5
Cation Exchange capacity		48.5
Exchangeable Ca		8.2
Exchangeable Mg	Meq/100g	2.5
Exchangeable Na		2.7
Exchangeable K		32.6

Procedure for adsorption measurement

A stock solution (1000mg/l) of Cu (II), Fe (II) or Ca (II) was prepared by dissolving accurate weight of CuSO₄.5H₂O, FeSO₄.7H₂O or CaCl₂.2H₂O, respectively in definite volume of distilled water. Proper concentrations of the adsorbate were prepared from the stock solution through proper dilution. The pH of the adsorbate was adjusted during the dilution step using diluted hydrochloric acid or sodium hydroxide to study the effect of pH factor on the adsorption process. The pH of the solution was measured with a WPA CD720 pH meter. Adsorption measurements were made in triplicate by the batch technique at room temperature (25±2 °C). A shaker model Gerhardt LS20 was used for shaking the adsorption batches (shaking rate 200 rpm). At the end of a predetermined time interval, the adsorbent was removed by filtration. The concentrations of solutions before and after adsorption were measured by using a flame atomic absorption spectrometer, Varian AA-20. The working standards for calibration of the atomic absorption spectrophotometer were prepared by diluting a stock solution of 1000 mg/l cations. Fresh calibration was done before the analysis of each batch of samples for cation determination. The percent adsorption was estimated by employing the following expression (Milonjic *et al.*, 1992).

$$\% \text{ Removal} = (C_0 - C_e / C_0) \times 100$$

where C_0 and C_e are the initial and the equilibrium cation concentrations, respectively. The effect of contact time (2-120 min), the adsorbent dosage (0.5-6 g/50ml) and pH profile [1- 6 for Cu (II) and Fe (II) and of 1- 8 for Ca (II)]

in adsorbate concentration (100 mg/l) were investigated. Also, kinetic studies and adsorption isotherm of adsorbate concentration (50-300 mg/l) on the adsorption process, as well as to what extent the adsorption data obey Langmuir and Freundlich adsorption isotherms were studied.

Results and discussion

Effect of agitation time

Time taken for the adsorption process to attain thermodynamic equilibrium is very important in characterization and prediction of both the efficiency and the feasibility of an adsorbent for its use in water pollution control or in the other usages. The effect of agitation time was studied for 100 mg/l adsorbate concentrations. Fig. 1 shows that the percentage of Cu (II), Fe(II) or Ca(II) removal increases sharply with increasing agitation time up to 10 min and then it increases very slowly and becomes nearly constant after 20 min. The constant percent removal after 20 min means that thermodynamic equilibrium is attained at that extent. Henriksen *et al.* (1998) investigated the possibility of segregating liquid manure so that all the N is transferred into a thick slurry phase by different natural and synthetic adsorbents, in order to allow the remaining liquid to be spread onto fields without causing environmental problems. They recorded that the clinoptilolite showed the highest capacity for ion exchange but glauconite was preferred due to its faster reaction time. It is clear from Fig. 1 that the lower the adsorbate concentration, the higher the percent cations removal and this is a benefit because, as example, industrial wastewater is actually a very diluted solution of different pollutants. The curves obtained are single and smooth indicating the formation of a monolayer of cations on the adsorbent surface. Smith *et al.* (1996) found the same result. They evaluated adsorption equilibria for Cd(II), Pb(II) and Zn(II) on a glauconite and recorded that although glauconite is a complex clay mineral, a single satisfactory description for many of the cases studied. site approach provided a single site approach provided a satisfactory description for many of the cases studied.

Effect of adsorbent dose

The dependence of cations removal on adsorbent dose was investigated by varying the amount of glauconite from 0.5 to 6 g/50ml, while keeping other parameters (pH, agitation speed and time and adsorbate concentration) constant. Fig. 2 shows the dependence of cations removal extent on the adsorbent dose. It is clear from the figure that the extent of Cu (II) and Fe (II) removal increases by increasing glauconite dose. This can be explained on the bases that the higher adsorbent dose in the adsorbate solution, the greater the availability of exchangeable sites for Cu (II) and Fe (II) ions. Such effect was true for Ca (II) below 4g of adsorbent dose, where increasing glauconite dose decreased the percentage removal of Ca (II). This may be resulted from the exchangeable of H cations of solution with exchangeable Ca of glauconite (glauconite had 8.2 meq. Ca/100g, Table 1).

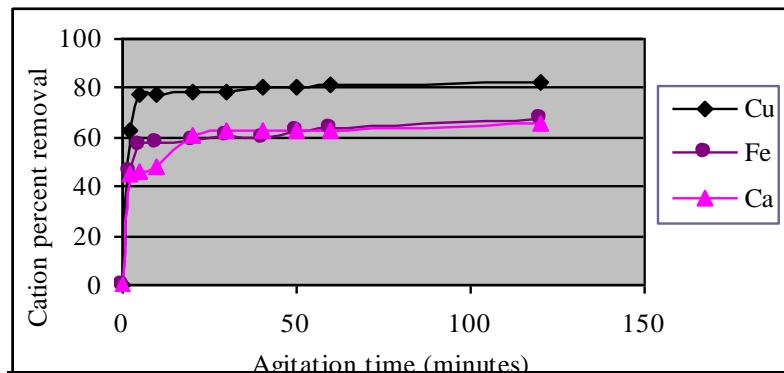


Fig.1. Effect of agitation time on cation adsorption onto glauconite. A dsorbent dose, 1 g/50ml, 100mg cation /1, pH 4.4.

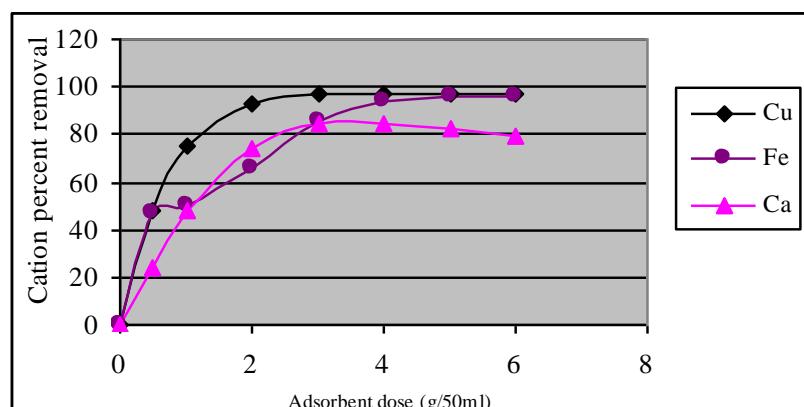


Fig. 2. Effect of adsorbent dose on cation a dsorption onto glauconite. Agitation time 20 min, 100mg cation/1, pH 4.4.

Effect of pH

Initial pH is one of the most important factors that affect the adsorption process. It affects not only the surface charge of the adsorbent, but also the ionization degree of the adsorbate. To investigate the role of pH in Cu (II) or Fe (II) removal efficiency, the initial pH of the adsorbate solution was varied in the range of 1–6. The adsorption study could not be carried out experimentally at pH values higher than 6 due to the precipitation of copper or iron hydroxide above this pH range. While the pH of Ca (II) solution increased up to 8.

Figure 3 shows the percent of cations removal as a function of pH. It can be seen that the percentage of Cu (II), Fe (II) and Ca (II) removal is maximum

(83.5%), (90.5%) and (74.2%) at pH 6, 5.5 and 8, respectively. Silber *et al.* (2012) recorded that zinc adsorption by perlite was dependent on pH, it involved a combination of specific chemical affinity to adsorption sites and an electrostatic component that is related to the surface charge and is controlled by pH. Elevating temperature significantly and systematically raised the pH and enhanced Zn adsorption. Abdel-Halim and Al-Deyab (2011) found that adsorption of Zn (II) ions onto acidified sodium alginate (ASA) was pH-dependent and maximum adsorption was obtained at pH 6. At pH values less than 4, the percent removal decreases continuously until it vanishes about 11%, 0.0% and 0.0% for Cu (II), Fe (II) and Ca (II) at pH 1, respectively.

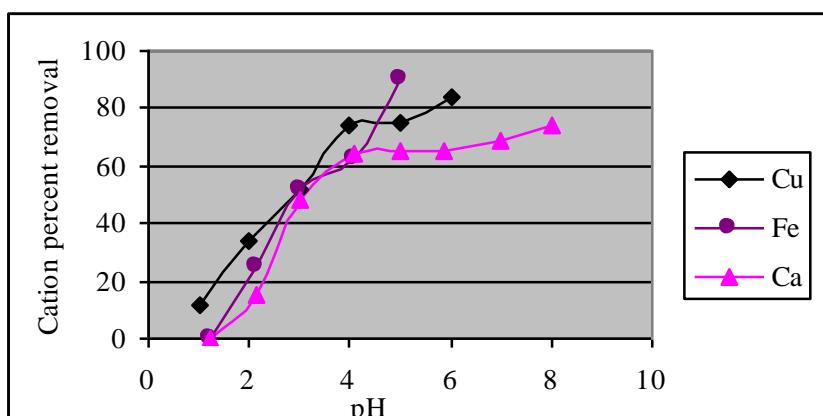


Fig. 3. Effect of pH on cation adsorption onto glauconite. (Agitation time 20 min, Adsorbent dose 1g/50ml, 100mg cation/1).

The low percent removal at $\text{pH} \leq 1$ can be attributed to both the fact that at, the high concentration of H^+ in the adsorption medium and to the protonation of ion exchange sites on glauconite surface. Under such conditions, the metal ions do not exchange and remain in the solution. As the pH value increases, the deprotonation of the glauconite surface increase and as a result, the adsorption capacity increases.

Adsorption isotherms and kinetic studies

The relation between cations equilibrium concentration and its extent of removal from aqueous solutions was studied at various cation concentrations and at fixed adsorbent dose. Adsorption data for a wide range of adsorbate concentrations is the most commonly described by adsorption isotherms, such as Langmuir or Freundlich isotherms, which relate adsorption density, q_e (adsorbate uptake per unit weight of the adsorbent) to equilibrium adsorbate concentration in the bulk fluid phase (C_e).

According to Langmuir (1918), maximum adsorption corresponds to a saturated monolayer of adsorbate molecules on the adsorbent surface. The linear form of Langmuir isotherm is given by the following equation:

$$\frac{C_e}{q_e} = \left(\frac{1}{Q_{\max} \times b} \right) + \left(\frac{C_e}{Q_{\max}} \right)$$

where C_e is the equilibrium concentration of adsorbate (mg/l), q_e is the amount of metal adsorbed at equilibrium (mg/g) and Q_{\max} (mg/g) and b (l/mg) are the Langmuir constants related to the adsorption capacity and energy, respectively.

The adsorption process was found to obey Langmuir adsorption isotherm, thus on plotting C_e versus C_e/q_e (Fig. 4, 5 and 6) a straight line with correlation coefficient (R^2) of 0.9977, 0.9887 and 0.9987 for Cu (II), Fe (II) and Ca (II), respectively were obtained. The values of Langmuir constants for glauconite Q_{\max} and b were calculated and found to be equal 3.76, 4.61 and 6.32 mg/g and 0.215, 0.046 and 0.062 l/mg, for Cu (II), Fe (II) and Ca (II), respectively.

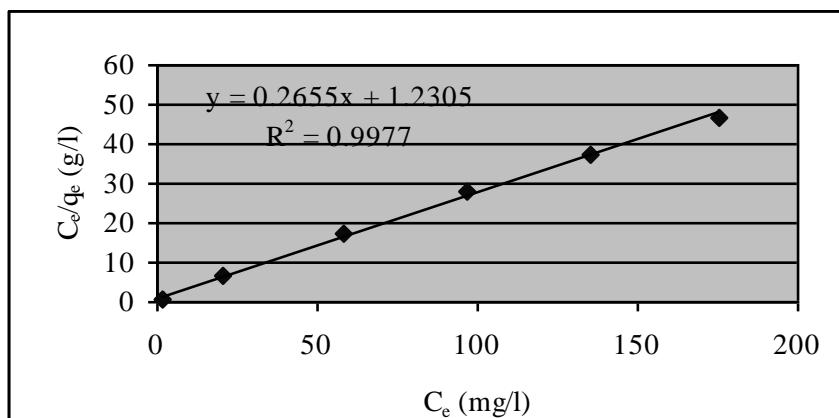


Fig. 4. Langmuir adsorption isotherm of Cu (II) onto glauconite.

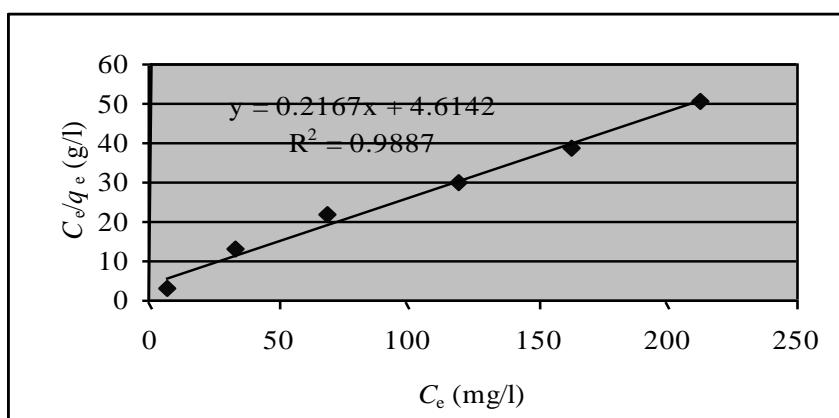


Fig. 5. Langmuir adsorption isotherm of Fe (II) onto glauconite.

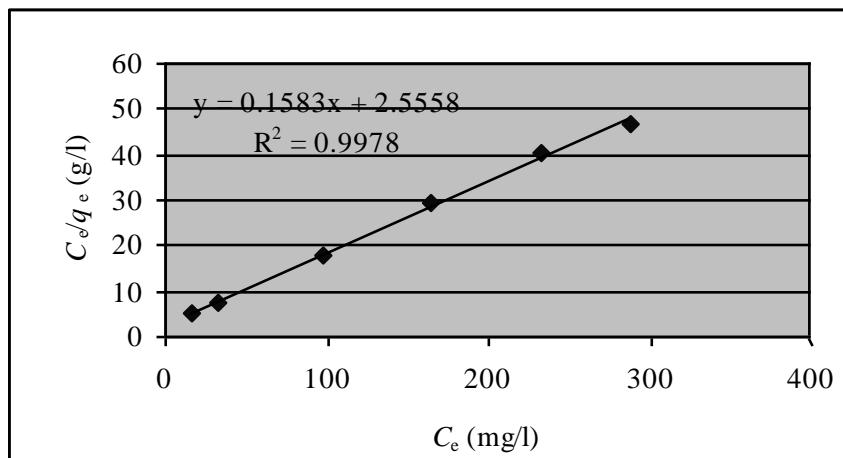


Fig. 6. Langmuir adsorption isotherm of Ca (II) onto glauconite.

The essential characteristics of Langmuir can be expressed in terms of a dimensionless equilibrium parameter, RL, which describes the type of isotherm (Hall *et al.*, 1966) and is defined by:

$$R_L = \frac{1}{1 + b \times C_0}$$

where b (l/mg) is the Langmuir constant and C_0 (mg/l) is the initial concentration of cation solution. The parameter RL shows the shape of isotherm according to Table 2. The values of RL for different Cu (II), Fe (II) and Ca (II) initial concentrations are listed in Table 3. It is clear from Table 2, all RL values range between 0 and 1, indicating the favorable adsorption of Cu (II), Fe (II) and Ca (II) onto glauconite.

Table 2. Effect of separation factor, RL on isotherm shape.

RL value	Type of isotherm
$RL > 1$	Unfavorable
$RL = 1$	Linear
$0 < RL < 1$	Favorable
$RL = 0$	Irreversible

Table 3. RL values based on Langmuir equation for Zn (II) adsorbed on ASA.

Cation concentration (mg/l)	Cu (II) RL value	Fe (II) RL value	Ca (II) RL value
50	0.085	0.303	0.243
100	0.044	0.178	0.139
150	0.030	0.127	0.097
200	0.022	0.098	0.075
250	0.018	0.080	0.061
300	0.010	0.068	0.051

Moreover, the adsorption process was also found to obey Freundlich adsorption isotherm (Freudlich, 1907), thus on plotting $\log q_e$ against $\log C_e$ (Fig. 7, 8 and 9) a straight line with a correlation coefficient (R^2) of 0.9704, 0.9978 and 0.9947 were obtained for Cu (II), Fe (II) and Ca (II), respectively. Freundlich isotherm is represented by the equation:

$$\log q_e = \log K_F + (1/n)\log C_e$$

where q_e is the amount of adsorbate adsorbed per unit weight of adsorbent (mg/g), C_e is the equilibrium adsorbate concentration (mg/l), and K_F and n are Freundlich constants, related to capacity of adsorbent and favorability of the adsorption, respectively. The values of Freundlich constants for glauconite, K_F were calculated and found to equal 1.945, 1.073 and 1.913 for Cu (II), Fe (II) and Ca (II), respectively. As shown from the results, the values of n are 7.662, 1.353 and 1.267 for Cu (II), Fe (II) and Ca (II), respectively, (*i.e.*, $0 < n < 10$) showing that the adsorption of this cations onto glauconite is favorable.

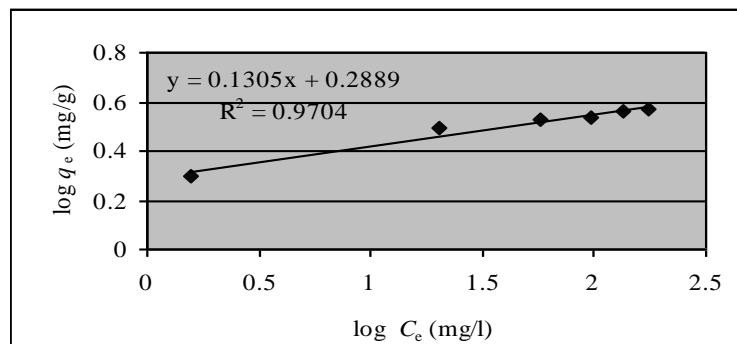


Fig. 7. Freundlich adsorption isotherm of Cu (II) onto glauconite.

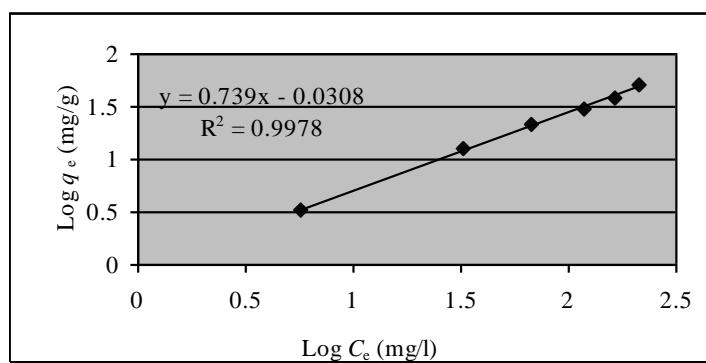


Fig. 8. Freundlich adsorption isotherm of Fe (II) onto glauconite.

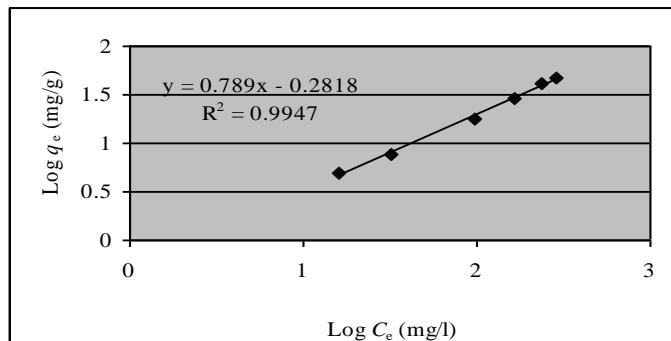


Fig. 9. Freundlich adsorption isotherm of Ca (II) onto glauconite.

The rate constants of adsorption (K_{ad}) for cation removal from solution by glauconite were determined by using the following first-order rate expression by Lagergren (Singh *et al.*, 1996 a and 1996 b).

$$\text{Log}(q_e - q) = \text{log } q_e - (K_{ad}/2.303) \times t$$

where q_e and q (both in mg g⁻¹) are the amount of Cu (II), Fe (II) or Ca (II) adsorbed at equilibrium and any time, t , respectively.

The linearity of the plot $\text{Log}(q_e - q)$ versus t (Fig. 10) indicates the applicability of the first-order rate expression. A straight line with a correlation coefficient (R^2) of 0.7949, 0.9318 and 0.7575 for Cu (II), Fe (II) and Ca (II), respectively were obtained. The K_{ad} values, calculated from the slope of the lines in Fig. 10 are 34.8×10^{-3} , 20.7×10^{-3} and $36.4 \times 10^{-3} \text{ min}^{-1}$ for Cu (II), Fe (II) or Ca (II), respectively. Tahir and Rauf (2004) reported that the bentonite clay has been used for the adsorption of Fe(II) from aqueous solutions over a concentration range of 80–200 mg/l, shaking time of 1–60 min, adsorbent dosage from 0.02 to 2 g and pH of 3. The process of uptake follows both the Langmuir and Freundlich isotherm models and also the first-order kinetics and the K_{ad} of Fe (II) on bentonite was $19.4 \times 10^{-3} \text{ min}^{-1}$.

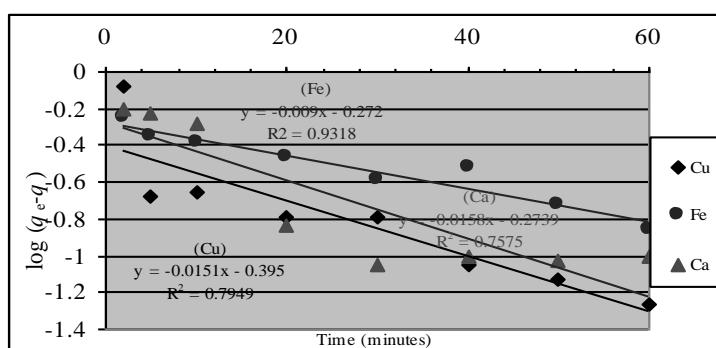


Fig. 10. Lagergren plot for the adsorption of different cations on glauconite (adsorbent dose, 1 g/50ml, 100mg cation/l, pH 4.4).

It could be concluded that the adsorption of Cu, Fe and Ca cations on glauconite is dependent on the pH, time and the dose of the adsorbent. The adsorption process was also found to obey Langmuir and Freundlich isotherm models. The values of Langmuir constants for glauconite Q_{max} and b were found to be equal 3.76, 4.61 and 6.32 mg/g and 0.215, 0.046 and 0.062 l/mg, for Cu (II), Fe (II) and Ca (II), respectively. The values of Freundlich constant for glauconite, K_F were found to equal 1.945, 1.073 and 1.913 for Cu (II), Fe (II) and Ca (II), respectively. It clear that glauconite is efficient as an adsorbent agent for a heavy metals.

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بعض الخصائص الامتصاصية لخام الجلوكونيت المصري

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عديد من الصناعات تستخدم مواد إدمصاص مختلفة في تنقية المنتجات السائلة أو مياه الصرف مما أدى إلى زيادة الإهتمام حديثاً باستخدام مواد إدمصاص طبيعية رخيصة لإزالة الكاتيونات و خصوصا العناصر الثقيلة من السوائل. و إقيمت هذه الدراسة لتقييم بعض خصائص الإدمصاص لخام الجلوكونيت المصري حيث أجريت مجموعات من تجارب الإلتران بين محاليل من $\text{Cu}^{(II)}$ أو $\text{Fe}^{(II)}$ أو $\text{Ca}^{(II)}$ مع الجلوكونيت. و أشارت النتائج إلى أن الكمية المدصصة من $\text{Cu}^{(II)}$ أو $\text{Fe}^{(II)}$ أو $\text{Ca}^{(II)}$ على الجلوكونيت وصلت إلى الإلتران بعد 20 دقيقة مع تكون طبقة إدمصاص واحدة على سطح الجلوكونيت. و تعتبر درجة حموضة محلول (pH) أهم عامل مؤثر على عملية الإدمصاص حيث وصلت أقصى كمية إدمصاص للنحاس والحديد والكالسيوم على الجلوكونيت عند $\text{pH} = 6$ و 5,5 و 8 على الترتيب. و أن عملية إدمصاص الكاتيونات تتبع كل من معادلتي لأنجمير و فروينديش للا ADMAS و أيضا سرعة تفاعل الدرجة الأولى. و كانت قيم ثوابت معادلة لأنجمير (Q_{max}) و (b) لـ $\text{Ca}^{(II)}$ و $\text{Fe}^{(II)}$ و $\text{Cu}^{(II)}$ لـ Q_{max} على الجلوكونيت 3,67 و 4,61 و 6,32 مليجرام/جرام و 0,215 و 0,046 و 0,062 لتر/مليجرام على الترتيب. و النتائج السابقة تدل على أن خام الجلوكونيت المصري يمكن استخدامه كمادة إدمصاص رخيصة و خصوصاً أن له ميزة كبيرة و هي قوامه الرملية الطينية الذي يسهل استخدامه كمرشح رملي أيضاً.