Removal efficiency of heavy metals from aqueous solutions by Iraqi natural clays

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ABSTRACT

This study investigated the removal process of (Cr, Mn, Fe, Co and Ni) ions from aqueous solutions using the adsorption process onto Iraqi clays. The main goal was given to investigate the adsorption process as a function of pH and time onto Iraqi clays (Natural bentonite and Kaolin) and results were compared with Italian Kaolin behavior. The batch method has been employed using initial metal concentrations solution 25 mgL-1 at pH 4.0, 6.7 and 9.0. Experiments are performed, to study the behavior of different kind of clays toward single metals and for metal in competition with each other. The percentage adsorption was determined for the adsorption system as a function of adsorbate concentration. The results indicates that sorption of metals is competitive, and each clay has better metal removal efficiency when only one metal is present in the solution compared to multiple metals under constant conditions. Samples were analyzed for aqueousmetal concentration using Acetylene-Air Flame/Atomic Absorption (phoenix 986), with single-element hollow cathode lamps. It is shown that the studied clays were sensitive to pH changes, and the amounts of metal adsorbed increase with pH increase.Batch studies showed that Fe and Cr were removed efficiently from neutral as well as from acidic and alkaline solutions on Iraqi bentonite and Iraqi Kaolin clays, whereas the uptake of , Co, and Ni, increased at alkaline solutions pH (9). pH change does not improve the Mn uptake and it was the lower absorbed cation at different pH media , and on all type of studied clays. Using Italian Kaoline as Sorbent for the cation under investigation showed that Fe was removed efficiently from neutral as well as from alkaline solution, While Cr uptake was in its maxima from neutral solution, The retention efficiency of clay samples for test metals follows the order of Iraqi bentonite> Iraqi Kaolin > Italian Kaolin. The results also shows that the equilibrium time for the sorption of metals by the clay was less than 1h (30 minutes) for all metals.

I. Introduction

The presence of heavy metals in aquatic environment because of their toxic nature and other adverse effects on receiving eco systems has been of great concern to scientific community [1] .The release of heavy metals into terrestrial and aquatic ecosystems can be mainly attributed to human activities [2] .Many toxic heavy metals have been discharged into the environment as industrial wastes, causing serious soil and water pollution [3] . These metals tend to accumulate in organisms, causing numerous diseases and disorders[4].The treatment of rich heavy metal aqueous solutions is realizable by mean of many methods including: ion exchange, chemical precipitation, ultrafiltration, electrochemical deposition, among which, adsorption on appropriate adsorbent (low cost and abundance of the latter) is one of the most efficient in terms of simplicity and feasibility of operation and low consumption of energy [5], [6].

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The use of alternative low-cost materials as potential sorbents for the removal of heavy metals has been emphasized recently. The use of natural clays has been the favored method of reducing or eliminating such hazardous contaminants[7].

The wide usefulness of clay is a result of their high specific area, high chemical and mechanical stability, and variety of surface and structural properties ,The natural clays are readily available ,cheap and environmental friendly[8]. The clay mineral properties, high cation exchange capacity, swelling property and surface areas make clay such as favorable materials to be widely used as rheology control reagents, sorbents in pollution prevention and environmental remediation.[9]. In addition to these properties, the clay materials have already been shown to have high adsorption capacity, which may even exceed that of activated carbon under the same conditions of pH and temperature, because of their high specific surface area, high cation exchange capacity, Brönsted and Lewis acidities and positive or negative surface change against pH [10].

Extensive researchs have been reported on the use of clays for metal ions removal from aqueous solutions: Zn(II) with natural bentonite [11], Zn(II), Cd(II), Pb(II), with raw smectite [12], Pb(II), Cd(II) and Ni(II) with montmorillonite and kaolinite [13], Pb(II), Ni(II), Cr(VI) and Cu(II) with palygorskite [14],Cu(II) and Cd(II) with bentonite [15], Mn(II) from aqueous solution with natural clay from Bikougou (Gabon) [16], Co (II) on the surfaces of Attapulgite, bentonite, and kaolin, provided locally in Iraq [17], Zn(II) and Pb(II) on clays (especially bentonite, zeolite and quartz sand) [18], also various forms of modified clays have been used [19][20].

The current study focused on 1- the performance of a naturally available and inexpensive, untreated Iraqi clays {(bentonite and Kaolin) from west of Iraq} to remove divalent cations individually and in competition with each other from artificially prepared metal aqueous solution of constant metal concentration in a single batch system. 2- to determine the effect of, pH and time on adsorption

2. Materials and Methods

2.1. Chemicals

All chemicals used were of reagent grade from fluka and BDH . The synthetic solutions were prepared from Cr $(NO_3)_2$. 9H₂O, MnCl₂.4H₂O , Iron metal , Co $(NO_3)_2$, Ni $(NO_3)_2$. 6H₂O, in doubly distilled water .Studied metals concentration in the equilibrium solution was determined using Acetylene-Air Flame/Atomic Absorption spectrophotometer (phoenix 986), with single-element hollow cathode lamps. The pH measurement were performed with a pH meter (Mettler -Toledo)

2.2. Preparation of sorbent:

Iraqi kaolinite clay and bentonite was supplied from Doukhla site in the west part of Iraq (Ramadi governance) with the chemical composition of Al2Si2O5(OH)4. ,while Italian Kaoline was supplied from Ceramic factory, Ramadi, Iraq. The clay samples were ground, air - dried and pass through 106 μ m sieve. The chemical composition of the clays material was determined by fluorescence spectroscopy using a Spectro X-Lab 2000 X-ray instrument. Table (1).

Table 1	Chemical composition of Iraqi Doukhla kaolin, bentonite and Italian kaolin (wt.%).			

<u>Constituent</u> <u>Weight %</u>	<u>Iraqi</u> kaolin	<u>Italian</u> kaolin	<u>Iraqi</u>
SiO ₂	50.4	44.2	56.7
Al ₂ O ₃	28.9	11.9	15.9
Fe ₂ O ₃	4.5	4.7	5.29
CaO	1.8	10.6	4.5
MgO	0.3	4.7	3.4
Na2O	0.3	0.9	1.1
K2O	0.3	2.3	3.58
L.O.I (wt.%)	13.5	20.7	12.51

L.O.I = loss on ignition

2.3. Adsorption procedure. Effects of time :

The sorption of the metals ions on these clays was studied in the time range from 10 minutes to one hours and it was found that the initial 1 hour (30 minutes) was sufficient to exchange most of the metal ions.

Effects of Competing Cations.

A (500 mg) of clay was equilibrated with 50 mL of 25 mgL-1 concentration of the prepared metal (Cr, Mn, Fe, Co and Ni) aqueous solution in a 100 mL stopped propylene flask, at room temperature at different interval of time and pH values (4.0, 6.7 and 9.0) in a single batch system. After the equilibration using a constant stirring speed for 30 minutes , the suspension was filtered out and analyzed for metal concentration, using atomic absorption spectrometer (phoenix 986) supplied with air acetylene flam.

The effect of several parameters such as type of clay, pH, contact-time, on the adsorption was studied . The same procedure was repeated for metals competition effect using artificially prepared metals aqueous solution (25 mgL-1 for each metal ion , Cr, Mn , Fe ,Co and Ni) in a single batch system. All experiments were carried out in triplicate with appropriate blanks. For clarity, only the mean values are presented in the graphs. The percent adsorption (% ads) was calculated according to the equation below:

 $(\% \text{ ads}) = (C_0 - C_t) \setminus C_0 \times 100$

. where C_0 = initial Metal (II) concentration and C_t = Metal (II) concentration at any time

Effects of pH :

The effect of solution pH on heavy metal adsorption was also studied in the pH range from 4.0 to 9.0, the pH was adjusted to values of 4.0, 6.7, and

9.0 by adding 0.1 and 0.01 M NaOH under constant stirring. After the desired pH was reached a sample of 10 mL was taken for desired study. The study involve the effect of pH on metal adsorption with time change

3. Results

The adsorption of (Cr, Mn, Fe, Co and Ni) ions onto three type of clays was studied at different pH values and time, while keeping all others parameters such as particle size, temperature, metal concentration, and adsorbent to adsorbate ratio constant.

Due to previous research. a constant concentration of metal ions (25 mgl-1) was used in this study, it was observed that the adsorption capacity of bentonite clay has linearity increased with metal ion concentration up to 50 mg\l then adsorption didn't change with metal ion concentration , and this indicate that the pores of clay minerals filled after a certain period. It was observed that medium temperature has a negative effect on adsorption of metal, the metal adsorption percent followed the order $20 \ {}^{0}C > 50 \ {}^{0}C > 75 \ {}^{0}C > 90 \ {}^{0}C.[8]$. A constant room temperature 30 °C was chosen to performed this research. Batch studies showed that the equilibrium time for the sorption of metals by clay was 30 minutes for all metals.

The batch method experiments are performed, to study the behavior of different type of clays toward single metals solution and mixed metal ions solution. The results showed that Cr was removed efficiently (100%, 98%,96%) onto Iraqi bentonite, Iraqi kaolinite and Italian Kaolinite respectively as single ion . whereas the uptake of Cr was decreased to be (78%, 95%, 65%) when it was in competition with other metals on Iraqi bentonite, Iraqi kaolinite and Italian Kaolinite respectively. The results showed that Mn removal efficiency was (97%,92%,91%) onto Iraqi bentonite, Iraqi kaolinite and Italian Kaolinite respectively as single ion. Whereas the removal efficiency was decreased to be (67%, 41%, 41%) when Mn was in competition with other metals on Iraqi bentonite, Iraqi kaolinite and Italian Kaolinite respectively.

The results showed that Fe was removed more efficiently(100%) on the three type of clays as single ion and in competition with other metals , whereas the uptake of Fe was decreased 8% and 34% when it was in competition with other metals on Iraqi kaolinite and Italian Kaolinite respectively. The decrease in the removal efficiency of Co and Ni was the same (about 10%) when they were in competition with other metals on Iraqi bentonite and Iraqi kaolinite. The sorption Onto Italian Kaolinite of Co and Ni was decreased 30% and 42% respectively when they were in competition with other metals comparing to single ions batch test.

In general Cr and Fe uptake was the strongest and Mn was the weakest. While Co and Ni was moderately high adsorbed. Although the Italian Kaolinite did not perform as well as the Iraqi kaolinite and Iraqi bentonite . Sorption of studied metals to Italian Kaolinite exhibited competitive sorption ,While the Iraqi bentonite and Iraqi kaolinite performed good sorption for metals, has little competitive sorption, and appears to be an effective sorbent for heavy metals , and these sorbent be well suited for metals removal from aqueous solutions. . Fig (1-3)

The batch method experiments are performed, to study the effect of solution pH on heavy metal adsorption at different type of clay's. The effect of solution pH was studied in the pH range from 4.0 to 9.0; the results are shown in Fig. (5-13). The process of cation exchange between solid surface and the solution containing the heavy metal cations is highly dependent on the pH value of the medium.

The results showed that Fe was removed efficiently from neutral as well as from acidic and

alkaline solutions on Iraqi bentonites, whereas the uptake of Cr, Co, and Ni, increased at alkaline solutions pH (9). pH change does not improve the Mn uptake and it was the lower absorbed cation at different pH media (it's minimum percentage at acidic solution), and on all type clays.

Fe and Cr were removed efficiently from neutral as well as from acidic and alkaline solutions on Iraqi Kaolin clays, whereas the uptake of Co, Ni, increased at alkaline solutions pH (9). Using Italian Kaoline as Sorbent for the cation under investigation showed that Fe was removed efficiently from neutral as well as from alkaline solution, While Cr uptake was in its maxima from neutral solution. The order of affinity for the adsorbate is Fe2+ > Cr3+ > C02+ > Ni2+ > Mn2+ , and this is associated with the size of the ion and the pore development in the clays. Fig. (15-24) shows contact time effect.

The contact time effect on the clays removal of 25 mg L–1 of the studied metals with pH change was studied. The metal Removal increases with time and reaches a maximum after 30 min of agitation.

4. Discusion

The heavy metal sorption is attributed to different mechanisms of ion-exchange processes as well as to the adsorption process. During the possible ion exchange process, firstly metal ions may move through either the pores of the bentonite mass or through channels of the crystal lattice. Secondly, they may replace exchangeable cations, mainly such as sodium, magnesium and calcium. Diffusion may also occur by slowly or fastly passing the metal ions through the adsorbent pores, but diffusion might be retarded at which the metal ions or hydrated metal ions the couldn't pass through smaller diameter channels.[8]. In this case, the metal ion sorption could mainly be attributed to ion-exchange reactions in the microporous minerals of the bentonite samples.

There are two main factors that have made clays very suitable to be developed as sorbent in many areas .The first is that clays have very large surface area, usually between 10 and 700 m2 g and the second is clay mineral's expansive surface often has an electrical charge that result in accumulation of inorganic and organic cations[21]. Smectite group clays such as bentonite possess a net negative structural charge resulting from isomorphic substitution of cations in crystal lattice. This negative charge is neutralized by the adsorption of positively charged species, giving clay the ability to attract and hold cations such as toxic heavy metals. [22][23]. In the same way, the surface area of the adsorbent can influence in the adsorption process. Bentonite exhibits an enormous surface area when it is hydrated in water. It consists of numerous microscopic platelets, each with negative charges on the flat surfaces and positive charges on the edges. Hydration causes these platelets to separate into a porous structure containing both positive and negative charges. It is well known that the ionic charge and the porous structure give bentonite the ability to adsorb the toxins [11]. The high Si/Al mole ratio of bentonite results in a low anionic field that gives rise to a good selectivity. These results show that bentonitic clay holds great potential to remove the relevant heavy metal cations from industrial wastewater. Each clay will likely have better metal removal efficiency when only one metal is present in the solution compared to multiple metals (assuming everything else is equal). This indicates that the sorption processes of metals were affected by the type of clay and competition of metals with each other (Fig 4), and it was decreased descending in the order:

Iraqi Bentonit > Iraqi kaolinite > Italian Kaolinite From the experimental results, the selectivity

order can be given as

This explained that, Mn2+ was the least adsorbed metal ion, while Fe2+ , Cr 3+ were the most easily adsorbed metal ion on all the type of the studied clays. Since the clays are used is a micro porous adsorbent [24]. The metals penetrate easily into these pores when the ionic size becomes small. Similar results have been reported in literature [25]. It can be seen that metal ions are easily adsorbed when the hydrated ionic size decreases.[26].

It is evident that the adsorption phenomena depend on the surface charge density of adsorbent and hydrated ion diameter depending upon the solution Owing adsorption pH. to their capacity, aluminosilicate and mineral oxides are capable of removing many metals over a wide pH range representing an efficient way for the remediation of contaminated soils. Numerous researches have been conducted on natural bentonite to examine its potential for the removal of heavy metal ions. Extensive the research has assessed metal adsorption characteristics of natural clays and has demonstrated that pH variation influences metal adsorption. [27-29]. The increase in adsorption is probably due to cation hydrolysis, since hydroxyl complexes are adsorbed preferentially over uncomplexed cations or to the increase in the negative surface charge of the adsorbent related to the increase in the concentration of hydroxyl ions, leading to an increase in attraction for cation in solution. [19].

The results showed that adsorption of the heavy metal cations on clays are very sensitive to the pH value of the medium. Adsorption takes place slightly at lower pH values (pH= 4.0), above this value adsorption becomes significant and maximized at pH= 9 for most ions (Fig 14). This trend may be explained in the following three ways. Firstly, it is related to the change of surface charge of bentonite particles, that increases negatively with increasing solution pH,

Fe2+=Cr3+>Co2+>Ni2+>Mn2+

which results in the increase of Kd values[30] [31]. That is, the higher negative surface charge allows the relevant metal ions to have easier electrostatic reaction with the bentonite particles. Secondly, it is due to a competitive ion-exchange reaction at low pH. The metal ions compete with H+ ions for the available sorption sites, giving a reduction of their Kd values. The contribution of ion competition, however, will decrease with increasing pH. Thirdly, a sharp increase in Kd at pH 9.0 may be due to precipitation or formation of complexation products of metal ions by hydrolysis. At higher pH the metal cations begin to hydrolyze and precipitate and hence become not available for adsorption. Thus the cation which is most readily hydrolyzed in solution also has the highest affinity to be adsorbed. [32]. As a matter of fact, increasing pH was reported to increase the adsorption of metal ions from kaolinite suspensions [30]. Studying the adsorption behaviour of Sr, Cs and Co by Camontmorillonite showed that Co adsorption increase above the pH of precipitation of Co(OH)2. [33]. In another study [34], concluded on the adsorption mechanism of Co2+ ions on natural sepiolite, that the clay is sensitive to pH changes, so that the amounts of heavy metal cations adsorbed increase as pH increase in adsorbent-adsorbate system. In general, results indicated that the adsorption is highly pH dependent. Similar results have been reported in literature. [35].

5- Conclusion

Under experimental conditions, it can be concluded that removal of heavy metals from aqueous solution was possible using the studied clays at the studied concentrations. The sorption processes of metals were affected by the type of clay and competition of metals with each other, and it was decreased descending in the order: Iraqi Bentonit > Iraqi kaolinite > Italian Kaolinite, and the selectivity order can be given as Fe2+ = Cr3+> Co2+> Ni2+.> Mn2+.It was seen that adsorption took place for these metals within 30 minutes for the concentration levels studied at the pH 9.0 that selected for an optimal rate of adsorption for all ions investigated.

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Fig (1) % adsorbed metals on Italian Kaoline as single ion and mixed solution



Fig (2) % adsorbed metals on Iraqi Kaoline as single ion and mixed solution



Fig (3) % adsorbed metals on Iraqi bentonite as single ion and mixed solution



K.lt: Italian Kaoline K.Irq.:Iraqi Kaolinite B.Irq.: Iraqi bentonite

Fig (4) Normalized Clay type effect on the average of metal adsorbtion %



Fig (5) % adsorbed metals on Italian Kaoline at Neutral pH



Fig (6) % adsorbed metal on Italian Kaoline at acidic pH



Fig (7) % adsorbed metal on Italian Kaoline at alkaline pH



Fig (8) % adsorbed metal on Iraqi Kaoline at neutral pH



Fig (9) % adsorbed metal on Iraqi Kaoline at acidic pH



Fig (10) % adsorbed metal on Iraqi Kaoline at alkaline pH



Fig (11) % adsorbe metal on Iraqi bentonite at neutral pH



Fig (12) % adsorbe metal on Iraqi bentonite at acidic $$\rm pH$$



Fig (13) % adsorbe metal on Iraqi bentonite at alkaline pH



Fig (14) Normalized pH effect on the average of metal adsorbtion %



Fig (15) % adsorbed metals variation with time on Italian Kaoline at Neutral pH



Fig (16) % adsorbed metals variation with time on Italian Kaoline at acidic pH



Fig (17) % adsorbed metals variation with time on Italian Kaoline at alkaline pH



Fig (18) % adsorbed metals variation with time on Iraqi Kaoline at Neutral pH



Fig (19) % adsorbed metals variation with time on Iraqi Kaoline at acidic pH



Fig (20) % adsorbed metals variation with time on Iraqi Kaoline at alkaline pH



Fig (21) % adsorbed metals variation with time on Iraqi bentonite at Neutral pH



Fig (22) % adsorbed metals variation with time on Iraqi bentonite at acidic pH



Fig (23) % adsorbed metals variation with time on Iraqi bentonite at alkaline pH



Fig (24) Normalized Time effect on the average of metal adsorbtion %

كفاءة إزالة العناصر الثقيلة من المحاليل المائية باستخدام الأطيان الطبيعية العراقية

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الخلاصة:

تضمنت هذه الدراسة عملية إزالة أيونات (الكروم، المنغنيز ، الحديد، الكوبالت والنيكل) من المحاليل المائية بالاعتماد على عملية الامتزاز على الأطيان العراقية . كان الهدف الرئيسي للدراسة متابعة عملية الامتزاز كدالة تغير لدرجة الحموضة والوقت على الطين العراقي (البنتونايت الطبيعي والكاؤلين) ومقارنة النتائج مع الكاولين الإيطالي. استخدمت طريقة (الوجبة) الدفعة باستعمال تراكيز أولية للعناصر المدروسة (25 ملغم لتر – 1) عند رقم هيدروجيني 4.0 فر 6.7 و 7.0 و 9.0 . اجريت التجارب، لدراسة سلوك أنواع مختلفة من الطين تجاه عنصر منفرد في المحلول وتجاه العناصر مجتمعة في محلول متنافسة مع بعضها البعض. استخدمت تقنية الامتصاص الذري اللهبي (لهب هواء – استيلين) لتعيين تركيز العناصر المدروسة في المحاليل المائية محلول متنافسة مع بعضها البعض. استخدمت تقنية الامتصاص الذري اللهبي (لهب هواء – استيلين) لتعيين تركيز العناصر المدروسة في المحاليل المائية بعد امتزازها على سطوح الأطيان قيد الدرس. تم تقدير النسبة الملوية لامتزاز العناصر كدالة لتركيز العنصر الممتز، وقد بينت النتائج تنافس العناصر في عملية الامتزاز على سطوح الأطيان وتزداد كفاءة إزالة الأطيان للعناصر من محاليلها المائية في حاله محلول الممتر، وقد بينت النتائج تنافس العناصر الذي يحتوي كل العناصر المدروسة تحت نفس الظروف. أظهرت الدراسة تأثير تغير حامضية محاليل العناصر على كفاءة الأطيان المدروسة، وتبين الذي يحتوي كل العناصر المدروسة تحت نفس الظروف. أظهرت الدراسة تأثير تغير حامضية محاليل العالي لعناصر على كفاءة الأطيان المدروسة، وتبين الذي يحتوي كل العناصر المدروسة تحت نفس الظروف. أظهرت الدراسة تأثير تغير حامضية محاليل العراقي في ازالة ايوني الحروم من المحالي الذي يحتوي كل العناصر المدروسة تحت نفس الظروف. أظهرت الدراسة تأثير تغير حامضية محالين العراقي في ازالة ايوني في الراسة المائيني من محلولة المائي مع ارتفاع قيم الـ 10. الذي يحتوي كل معادي والقاعدية، بينما كانت الزادة لأيوني العراقي في الولولي في ازالة أليون العراقي في ازالة المرين في المائيني في ازالة الميني في المنغنيز من محلولة المائي مع تغير درجة الحوضة وكانت كفاءة الازالة لأيوني المنيكل في الوسط القاعدي، وتبين مكاءة الأطيان في ازالة المن من من محلولة المائي مع تغير درجة الحوضة وكانت كفاءة الازالة الوراقية في ازالة أيون المنييا في المنغنيز