

## EFFECT OF HYDROTALCITE-LIKE COMPOUNDS ON RETENTION OF SOME HEAVY METALS FROM AQUEOUS SOLUTIONS

[54]

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### ABSTRACT

Hydrotalcite-like compounds and their calcined product which are simply referred to as hydrotalcites hereafter are a family of anionic clays. Hydrotalcites are a layered double hydroxide, with the general formula  $[M^{2+}_{1-x}M^{3+}_x(OH)_2] [A^{r-}_{x/n} \cdot zH_2O]$ . In this study, kinetics and equilibrium adsorption of Co, Mn, Ni or Zn from their aqueous solutions on Hydrotalcite-like compounds and calcined hydrotalcite have been investigated. Batch adsorption studies show that the calcined hydrotalcite mineral removed more different heavy metals than the hydrotalcite mineral at the same level of heavy metals concentrations. Calcined hydrotalcite adsorbed 71.5, 81.0, 85.8 and 86.5%, of the added amount of Co, Mn, Ni or Zn, respectively. Whereas, the hydrotalcite adsorbed, respectively, 33.3, 34.3, 52.3 and 62.5% of the same elements. The results of adsorption were fit to the simple Langmuir model. Correlation coefficients ( $R^2$ ) for the linear regression fit were  $\geq 0.82$ . The maximum sorption capacity ( $b$ ) for hydrotalcite remained highest for Ni followed by Zn, Mn and Ni, respectively; calcined hydrotalcite remained highest for Zn followed by Mn, Co and Ni, respectively. The binding energy parameter ( $k$ ) showed the highest values with calcined hydrotalcite than the hydrotalcite. Adsorption of Co, Mn, Ni and Zn increased equilibrium pH. Greater increase in pH was observed with the hydrotalcite than the calcined hydrotalcite. The percentage of extraction using DTPA solution recorded highest value with hydrotalcite and the lowest one was observed with calcined hydrotalcite. Mn showed the lowest desorption (0.94-2.17%) with calcined hydrotalcite, over all added concentrations of heavy metals. On the other hand, Zn showed the highest desorption (6.25-13.2 %) with hydrotalcite, over all added concentrations of heavy metals. It is clear that the DTPA extracting solution was not able to remove adsorbed heavy metals with hydrotalcite or calcined hydrotalcite.

**Keywords:** Hydrotalcite, Heavy metals, Adsorption, Desorption, DTPA

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## INTRODUCTION

Heavy metals are toxic pollutants that severely limit the beneficial use of water for domestic or industrial applications. Traditional methods of removal such as chemical precipitation, coagulation, sorption, ion-exchange and membrane processes are either expensive or do not allow to reduce heavy metals to the permissible levels. Among these, the ion-exchange process is probably the most attractive, especially because, instead of synthetic ion-exchange resins, it is possible to use low-cost sorbents (Kesraoui-Ouki *et al* 1994 and Bailey *et al* 1999). Hydrotalcite is an anion clay that is found in nature and can be easily synthesised in the laboratory. It is composed of mixed layers of Mg and Al,  $[Mg_{1-x}Al_x(OH)_2]^{+x}$ , with interlayer anions, most commonly carbonate, to provide overall charge neutrality (Zhu *et al* 2000). Li *et al* (1996) reported that hydrotalcite preferentially adsorbs anionic species as they are able to enter its interlayer regions and exchange with the negatively charged ions located there or to be adsorbed to the positively charged surface. With reference to the adsorption of metal ions, it is expected that this adsorption will be non-selective and depend only on the charge of the cation. Thus it is expected that the amount of adsorption of Pb and Zn will be similar and the adsorption of Cr (III) will be approximately 60% of the adsorption of these two species. The negatively charged ions located in the interlayer region of hydrotalcite may be removed by calcining the substance. In the case of hydrotalcite, prepared in this study, the interlayer carbonate anions should decompose to  $CO_2$  and  $O^{2-}$  leaving  $O^{2-}$  ions in the interlayer regions if the substance

is heated above  $400^\circ C$ . The structure of hydrotalcite is permanently lost by rising the temperature above  $900^\circ C$  (Parker *et al* 1995). The crystalline structure of hydrotalcite collapses when calcined as shown by XRD analysis; however, some short range order must remain as the substance structure is restored when it is added to water (Cavani *et al* 1991). Due to the fact that the metal cations are not likely to enter the interlayer region of hydrotalcite, it is not expected that there will be significant variation in the amount of adsorption between normal hydrotalcite and calcined hydrotalcite. Thermal decomposition of the most widely spread carbonate representatives takes place during consequent dehydration, dehydroxylation and decarbonation; those processes being partially overlapped. Until recently, it has been considered that only a reversible dehydration takes place in the low temperature region (up to  $300^\circ C$ ) while at high temperatures dehydroxylation and decarbonation proceed almost simultaneously (Miyata, 1980 and Hibino *et al* 1995). However, new studies have showed that dehydroxylation of part of the OH groups starts in the low temperature range (Rey and Fornes, 1992; MacKenzie *et al* 1993 and Stanimirova and Petrova, 1998). During the process of thermal decomposition a series of discrete metaphases with specific properties and structural features are formed (Stanimirova *et al* 1998).

The objective of this study was to examine the influence of hydrotalcite-like compounds and their calcined product on the sorption of some heavy metals such as Co, Mn, Ni or Zn from aqueous solutions. The extractability of adsorbed heavy metals was estimated.

## MATERIAL AND METHODS

### Preparation of Adsorbent

The hydrotalcite-like compounds and calcined hydrotalcite product adsorbent were synthesised using the following method:

Hydrotalcite-like compounds was prepared by co-precipitating an aqueous solution of magnesium and aluminum salts with a highly basic carbonate solution (Zhao *et al* 2003), the following steps being adopted. 1) 29.02g of  $MgCl_2 \cdot 6H_2O$  and 14.48g of  $AlCl_3 \cdot 6H_2O$  dissolved in 200mL of water under stirring. 2) 64g of NaOH and 11.44g of  $Na_2CO_3$  dissolved in 4L of water. 3) Solution from Step 1 ( $MgCl_2 \cdot 6H_2O$  and  $AlCl_3 \cdot 6H_2O$ ) is added drop wise into solution from Step 2 (NaOH and  $Na_2CO_3$ ) under severe stirring. 4) Centrifuge the mixed suspension to recover white precipitate (centrifuge at a speed of 300rpm for 20 mins). 5) Repeat steps 1-4 10 times. 6) Wash solid 3 times with distilled water. 7) Repeat step 6. 8. Place wet cake in an oven for 24 hours at 100°C.

Half of the resulting solid from hydrotalcite is calcined as follows: Solid placed in crucible in oven at 50°C. The temperature in oven is elevated to 500°C at intervals of 2°C/minute and maintained for 10 hours. Oven is cooled to 80°C at a rate of 3°C/minute. Hydrotalcite and calcined hydrotalcite are ground to a fine powder through 70  $\mu m$  sieve, homogenized, collected and stored for further use.

### Sorption of heavy metals

The heavy metals (Co, Mn, Ni or Zn) sorption experiment was conducted by both hydrotalcite-like compounds and

calcined hydrotalcite synthesis minerals. The heavy metals stock solutions were prepared by dissolving sulfate salts in distilled water. Individual aqueous solutions of different heavy metals concentrations (0.5, 1, 2, 4, 8 and 16 mmol. L<sup>-1</sup>) were prepared by diluting the stock solutions (50 mmol.L<sup>-1</sup>). The pH values of these initial stock solutions were 3.15, 6.44, 5.85 and 5.46, for Co, Mn, Ni or Zn, respectively. A fifty-mL aliquot of 0.05M  $KNO_3$  background electrolyte solution containing variable metal concentrations was equilibrated with 0.2 g from each of the two synthesized minerals. The suspensions were shaken at a room temperature for 24 hours (in the independent kinetic tests, it was found that equilibrium is reached after 12 hours). The supernatant was separated by centrifugation at 5000 rpm for 30 min and the clear supernatants were collected. Determinations of Co, Mn, Ni and Zn and pH in the supernatant were performed using atomic absorption spectrophotometer.

### Extracting of heavy metals

The residues of different heavy metals remaining in the centrifuge tubes after sorption experiments were rinsed several times with 95% ethanol to remove any entrapped heavy metals in the solution until no heavy metals were detected. Washed residues from each group were treated with 50 mL of DTPA solution and the slurries were shaken for 24 hours. Slurries were centrifuged and their supernatants were filtered using Watman filter paper No. 42 and analyzed for Co, Mn, Ni and Zn concentrations as described above for the sorption experiment.

## RESULTS AND DISCUSSION

### Sorption Isotherms

Isotherms representing sorption of Co, Mn, Ni or Zn by both hydrotalcite-like compounds and calcined hydrotalcite are shown in (Fig. 1). Nonlinear sorption isotherms were observed with all heavy metal-synthesized minerals. The isotherms are almost concave to the abscissa. Overall, the sorption behavior of different heavy metals on the two synthesized minerals were almost similar to each other, with differences in the amounts sorbed. The amount of adsorption increased with increasing heavy metals concentrations to attain a maximum value at 16 mmol.L<sup>-1</sup>. The calcined hydrotalcite mineral retained higher amounts of heavy metals as compared to hydrotalcite mineral at the same initial concentrations. For example, calcined hydrotalcite adsorbed 71.5, 81.0, 85.8 and 86.5%, of the applied 4 mmol.L<sup>-1</sup> use Co, Mn, Ni or Zn, respectively; the hydrotalcite, on the other hand, adsorbed 33.3, 34.3, 52.3 and 62.5% of these same elements at the same concentration. It is explained that, two major reasons can be mentioned for the increased adsorption values of heavy metals on the calcined hydrotalcite. First the increase in negative charge due to the decomposition of the interlayer carbonates ions (Miyata, 1980). Second, incorporation of metals ions may occur due to structure rearrangement of calcined hydrotalcite. Parker *et al* (1995) found that the interlayer carbonate anions will decompose to CO<sub>2</sub> and O<sup>2-</sup> leaving O<sup>2-</sup> ions in the interlayer regions if the hydrotalcite heated above 400°C. Due to the fact that the

metal cations are not likely to enter the interlayer region of hydrotalcite, it is not expected that there will be significant variation in the amount of adsorption between hydrotalcite and calcined hydrotalcite. Carja *et al* (2002) also reported that Layered double hydroxides, in which magnesium was partially substituted by copper or iron in the hydrotalcite-like layer, were synthesized by a coprecipitation method. This can be explained by the fact that heavy metals are associated to particles by several mechanisms: 1) adsorption, 2) co-precipitation, 3) isomorphic substitution in crystal lattice. Also, sorption of different heavy metals onto synthesized minerals varied from one metal to another in the following descending order: Zn > Ni > Mn > Co. This may be due to that heavy metals contain different forms due to a partial hydrolysis of surfaces which exhibit electrical charge properties having a strong influence on the sorption of ionic form and polar species. Also, the difference in the quantities and rates of Co, Mn, Ni and Zn adsorption to two synthesized compounds may be attributable to the different adsorption affinities of the heavy metals to surface and the type of surface complex formed. The charge on the surface of the synthesized minerals must be counterbalanced in the aqueous phase to maintain electro-neutrality. Thus it is expected that this adsorption will be non-selective and depend only on the charge of the cation (Li *et al* 1996). Therefore, Co<sup>2+</sup>, Ni<sup>2+</sup>, Mn<sup>2+</sup> and Zn<sup>2+</sup> interact as divalent or hydrolyzed monovalent ions. The adsorption of Ni<sup>2+</sup> in the form of Ni(OH)<sup>1+</sup> was described by Koppelman and Dillard (1977) in a study of Cu<sup>2+</sup> and Ni<sup>2+</sup> sorption on the clay minerals kaolinite and chlorite. The principal forms of heavy

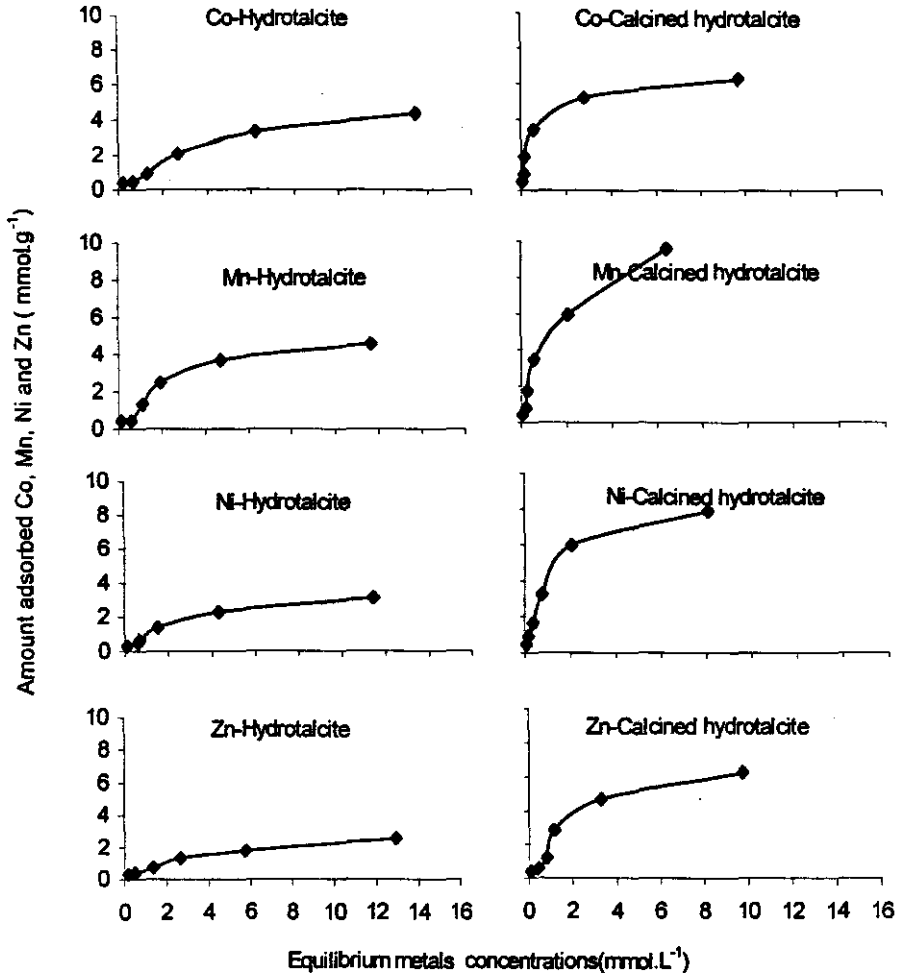


Fig. 1. Isotherms of Co, Mn, Ni and Zn adsorption on both the hydrotalcite-like compounds and calcined hydrotalcite affected by their different concentrations

metals (M) over the pH range (pH from 3.15 to 6.44) of this study were  $M^{2+}$  and  $M(OH)^+$ . Thus their adsorption was the only active process occurring in this study, while the precipitation of  $M(OH)_2$  was prevented by keeping the pH below 7 (Lindsay, 1979).

Data of heavy metals ions adsorbed on either hydrotalcite or calcined hydrotalcite were fitted to the linear form of the Langmuir adsorption isotherm. The linear form of Langmuir equation is represented by:  $(C/q = 1/kb + C/b)$  equations; C is the equilibrium concentration ( $mg.L^{-1}$ ), q is the amount of metal adsorbed onto synthesized compounds ( $mg.100g^{-1}$  mineral), b is the maximum adsorption capacity ( $mg.100g^{-1}$  mineral), and k is the affinity binding energy constant ( $L.mg^{-1}$ ). The monolayer maximum adsorption (b) from the Langmuir equation seems useful for comparing potential adsorption capacity of different soils and soil components (Sparks, 1995). Sorption parameters for synthesized compounds showed variability among the four studied metals, as reflected by their sorption maxima and Langmuir affinity constants (Table, 1). Results showed that maximum sorption capacity (b) for hydrotalcite remained highest for Ni followed by Zn, Mn and Co, respectively. Whereas, calcined hydrotalcite remained highest for Zn followed by Mn, Co and Ni, respectively. The results also indicated that the calcined hydrotalcite recorded the highest values for the maximum sorption capacity constant compared to the hydrotalcite. The binding energy parameter (k) showed the highest values with calcined hydrotalcite compared to the hydrotalcite. This can be expected, as the nature of the calcined hydrotalcite surfaces are different from those of hydrotalcite. In this respect,

Stanimirova *et al* (1998) reported that calcination of hydrotalcite up to 723-773 K yields a high surface area mixed oxide with strong Lewis basic features, which makes them suitable for various vapor-phase condensation reactions.

During the sorption experiments, pH and concentrations of Mg and Na in the equilibrium solutions were measured. After interactions of either hydrotalcite or calcined hydrotalcite with heavy metals, equilibrium pH tends to remain close to alkalinity with different heavy metals except for Zn which tends to remain close to neutrality. Adsorption of Co, Mn, Ni and Zn increased equilibrium pH (Fig. 2). Greater increase in pH was observed with the hydrotalcite than with the calcined hydrotalcite. From the pH evaluation, it is evident that, the hydrotalcite and calcined hydrotalcite have more hydroxylated surfaces. Generally, the equilibrium pH decreased with increasing the initial concentration for different heavy metals. The decrease in equilibrium pH was linearly related with the amounts of different heavy metals adsorbed for the two like minerals ( $R^2$  above 0.85). Also, the pH during interaction of aqueous metal solution with two studied minerals remained highest for Ni, followed by Mn, Co and Zn (Fig. 2). This could be attributed to the greater amounts of  $(OH)^{1-}$  and  $(CO_3)^{2-}$  which rise pH of the equilibrium solution.

#### Extraction of sorbed heavy metals

Data in (Table, 2) shows heavy metals extracted from hydrotalcite-like compounds and calcined hydrotalcite synthesized minerals as affected by DTPA extractant. The percentages of extracted heavy metals and their immobilization differed among the elements as well as

Table 1. Effect of hydrotalcite-like compound and their calcined product on Co, Mn, Ni and Zn adsorption linear correlation coefficient ( $R^2$ ), maximum sorption capacity (b) and binding energy parameter (k)

Element	$R^2$		Maximum sorption capacity (b)		binding energy (k)	
			mg.100g <sup>-1</sup>		L.mg <sup>-1</sup>	
	Hydrotalcite	Calcined hydrotalcite	Hydrotalcite	Calcined hydrotalcite	Hydrotalcite	Calcined hydrotalcite
Co	0.95	0.84	3.09	8.86	0.30	0.27
Mn	0.95	0.99	3.76	9.04	0.40	0.83
Ni	0.87	0.99	6.35	6.55	0.17	2.18
Zn	0.82	0.94	6.18	11.97	0.26	0.58

initial metals concentrations. Depending upon the amount of metal initially adsorbed, the percentage of extracted using DTPA solution recorded highest value with hydrotalcite as compared with extracted values from calcined hydrotalcite. It appears that heavy metals form complexes with calcined hydrotalcite, during the sorption process, remained more stable than hydrotalcite-heavy metals complexes. In other words, a portion of the heavy metals was sorped at a high binding energy level when using calcined hydrotalcite as adsorbate material (Table, 1). Although absolute amounts of heavy metals extracted by the DTPA solution increased with increasing amounts of adsorption onto the two studied synthesized minerals. It seems that, different heavy metals under study have similar behavior for extraction by the DTPA solution. Most of the adsorbed amounts of different heavy metals were immobilized. The maximum amount extracted from

different heavy metals didn't exceed 13.2% from amount sorbed with two studied synthesized minerals. Mn showed the lowest extraction value (0.94-2.17%) and highest immobilization (99.06 - 86.8%) with calcined hydrotalcite over all added concentrations of heavy metals; Zn showed the highest extraction value (6.25-13.2 %) and lowest immobilization (93.75 -97.83%) with hydrotalcite over all added concentrations of heavy metals. In general, the hydrotalcite or calcined hydrotalcite had a high capacity to immobilize the added heavy metals. This implies that the large amounts of different heavy metals adsorbed by hydrotalcite or calcined hydrotalcite are partially fixed and unavailable to extraction by DTPA solution. It is clear that the DTPA extracting solution didn't serve for removal of adsorbed heavy metals for either hydrotalcite or calcined hydrotalcite. This may suggest. That heavy metals under this study are associated to particles by

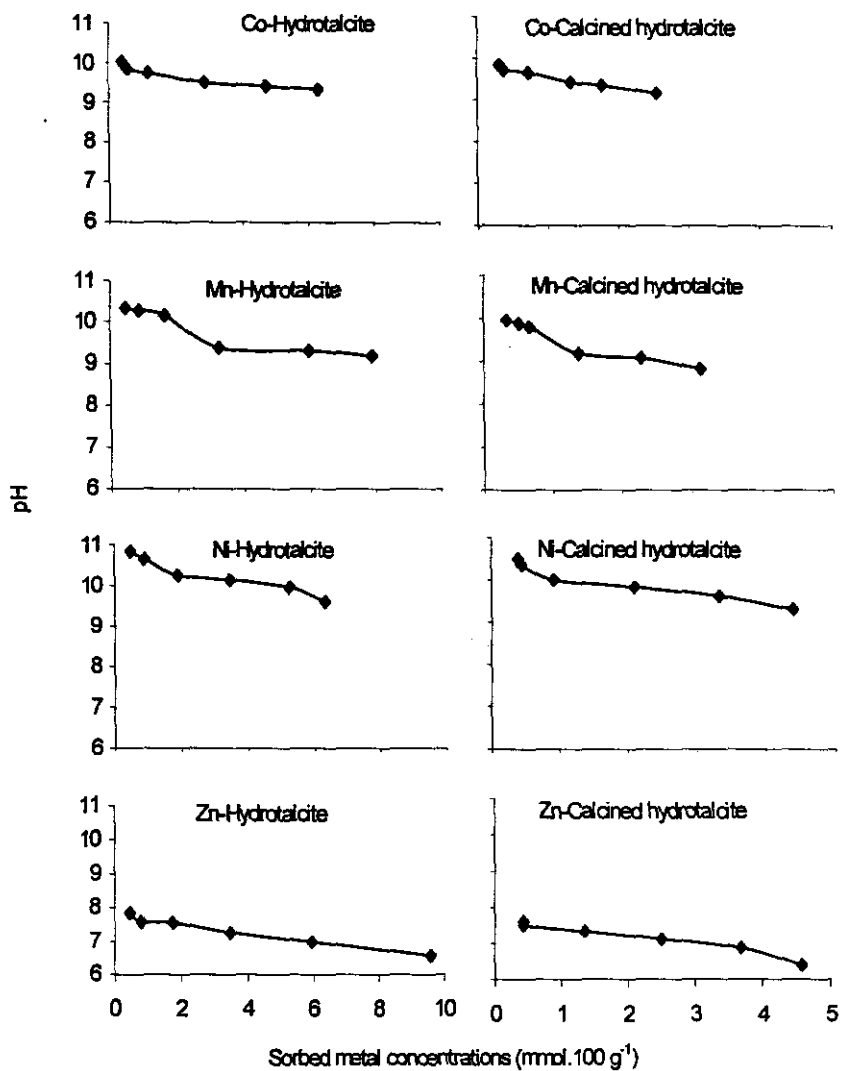


Fig. 2. Relationships between sorbed metals and pH value of equilibrium solutions



Table 2. Cobalt, Mn, Ni and Zn extracted from both hydrotalcite and calcined hydrotalcite synthesized minerals (as percent of the adsorbed amount) using DTPA extractant

Extracted value (mg/100g)	Extraction (%)	Extracted value (mg/100g)	Extracted (%)	Extraction value (mg/100g)	Extraction (%)	Extracted value (mg/100g)	Extraction (%)
Co		Mn		Ni		Zn	
Hydrotalcite							
1.77	7.70	0.55	2.32	1.18	6.05	1.30	6.25
2.95	11.4	1.10	4.64	1.77	5.88	3.25	13.2
4.13	7.69	1.65	2.22	2.36	6.07	3.90	8.21
7.08	5.76	2.75	1.99	5.31	6.57	9.10	10.5
12.4	6.33	3.30	1.63	5.90	4.40	13.0	11.3
20.7	7.99	6.60	2.63	8.85	4.81	14.3	8.61
Calcined hydrotalcite							
1.18	4.17	0.55	2.17	1.18	4.35	0.65	2.43
1.77	3.33	0.55	1.25	1.77	3.45	1.30	3.45
2.95	2.66	1.10	1.14	1.77	1.83	1.95	2.52
6.49	3.21	2.20	1.16	4.72	2.47	3.90	2.10
10.0	3.25	3.30	1.01	5.90	1.68	5.85	1.91
14.8	3.98	4.95	0.94	8.85	1.90	9.75	2.38

several mechanisms. In fact, Suzuki *et al* (1981 and 1982) concluded that the removal of Pb, Cu, Mn, Co, Cd, Zn, Ni, and Ba from their solutions was due not only to an adsorption effect but also to an ion-exchange reaction between the cations in solution and the  $Ca^{2+}$  ions of the apatite under alkaline conditions.

In general, it can be concluded that either hydrotalcite or calcined hydrotalcite has a good sorption properties towards Co, Mn, Ni and Zn. Different heavy metals under study have a similar behavior for extraction by the DTPA solution. The DTPA extractant solution doesn't serve for removal of adsorbed heavy metals with either hydrotalcite or calcined hydrotalcite. Calcination of the hydrotalcite-

like compounds increases the reacting surface and enhances their adsorption capacities. This in turn will help in cleaning the polluted water from toxic heavy metals.

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## تأثير شببيات الهيدروتالسيت على ادمصاص بعض العناصر الثقيلة من المحاليل المائية

[٥٤]

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أوضحت النتائج أيضا أن قيم معامل الارتباط ( $R^2$ ) لادمصاص العناصر الثقيلة على شببيات المعادن موضع الدراسة متوافقا مع معادلة لانجمير، فقد كانت قيم معامل الارتباط تساوى أو اكبر من ٠,٨٢ لكل العناصر. كذلك سجل الهيدروتالسيت العادي أعلى القيم بالنسبة للسعة الادمصاصية العظمى مع النيكل يليه الزنك يليه المنجنيز ثم الكوبالت بينما سجل الهيدروتالسيت المنكلس أعلى قيمة للسعة الادمصاصية العظمى مع الزنك يليه المنجنيز يليه الكوبالت ثم النيكل. أتضح من النتائج أيضا أن أعلى قيم لطاقة الارتباك (K) كانت عند استخدام الهيدروتالسيت المنكلس. وبدراسة تأثير ادمصاص الكاتيونات المختلفة على قيم (pH) فى محلول الاثران أتضح أن تركيز ايون الهيدروجين يقل بشكل ملحوظ خاصة مع استخدام الهيدروتالسيت العادي بدراسة عملية الاستخلاص للعناصر الثقيلة من الهيدروتالسيت العادي والهيدروتالسيت المنكلس باستخدام

استخدام المواد سهلة التحضير والأكثر أمانا على البيئة هي من أهم أهداف البحث العلمي، وتعتبر شببيات المعادن والممتلة في الهيدروتالسيت والشببية المنكلس والتي تتبع الطين الانيونى من أهم هذه المواد والتي تستخدم في إزالة الانيونات من الوسط. وفى هذا البحث، تم دراسة ديناميكية ادمصاص وانطلاق بعض العناصر الثقيلة (الكوبالت، المنجنيز، النيكل، الزنك) من المحاليل مختلفة التركيز على سطوح شببيات المعادن الهيدروتالسيت العادي والهيدروتالسيت المنكلس.

أوضحت النتائج إن قدرة الهيدروتالسيت المنكلس على ادمصاص العناصر الثقيلة موضع الدراسة تفوق قدرة الهيدروتالسيت العادي حيث وصلت نسبة الادمصاص من الكوبالت، المنجنيز، النيكل، الزنك ٨٥,٨، ٨٦,٥ ، ٨١,٠ ، ٧١,٥ % مع استخدام الهيدروتالسيت المنكلس بينما كانت هذه النسبة ٥٢,٣ ، ٦٢,٥ ، ٣٤,٣ ، ٣٣,٣ % عند استخدام الهيدروتالسيت العادي مع نفس العناصر بالترتيب.

الهيدروتالسيت العادي. وهذا يوضح إن مستخلص (DTPA) غير قادر على استخلاص العناصر المدمصة على الهيدروتالسيت العادي والمتكس لإعادة تنشيطهم ولا بد من البحث عن مستخلص يصلح لهذا الغرض. من هذه النتائج يتضح أنه يمكن استخدام هذه المركبات في تنقية المياه الملوثة من العناصر الثقيلة السامة والتي تعتبر من أهم المشاكل البيئية.

محلول (DTPA) أتضح إن المحلول أدى إلى زيادة انطلاق العناصر الثقيلة من الهيدروتالسيت العادي أكثر من الهيدروتالسيت المتكس. أوضحت النتائج أن عنصر المنجنيز كان أقل العناصر انطلقا فقد تراوحت نسبة المنطلق بين ٠,٩٤- ٢,١٧% من المدمص على الهيدروتالسيت المتكس بينما كان الزنك أكثر العناصر انطلقا واقلهم مسكا فقد تراوحت نسبة المنطق بين ٦,٢٥-١٣,٢% من على

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