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Aspects of the Adsorption Behavior of Cu^{2+}, Zn^{2+}, and Ni^{2+} Ions on Lithium Titanate Ion Exchanger

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ABSTRACT

Lithium titanate was prepared by hydrolysis of $TiCl_4$ using LiOH. The sorption of Cu^{2+}, Zn^{2+}, and Ni^{2+} ions on lithium titanate $[(LiO_x)(TiO)_y(OH)_z]$ has been investigated as a function of nitric acid concentrations in the presence of different organic solvents. Capacity, values of distribution coefficients (K_d), and selectivity patterns of the studied cations are determined on the matrix and a selectivity series $Cu^{2+} > Zn^{2+} > Ni^{2+}$ is established. The retention of the studied cations was increased by increasing the organic solvents up to 60% of the bulk. The sorption of different ions was increased by increasing the reaction temperatures,

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which enable us to calculate the enthalpy changes (ΔH^0) and then determine the type of adsorption process.

Key Words: Ion exchange; Lithium titanate; Organic solvents; Adsorption.

INTRODUCTION

Lithium titanate belongs to the family of alkali metal titanates, and most of these compounds can be represented as composition series $A_2O \cdot nTiO_2$ ($1 < n < 6$),[1] where A is an alkali metal ion. The isomorphism of tetratitanates $M_2Ti_4O_9$ (M = Tl, Li, Na, Rb, Cs, Ag) has been clearly demonstrated.[2] Electrical and magnetic properties of Li_xTiO_2 have been investigated by Marchand et al.[3] Alkali metal titanates possess important properties useful for various separation processes or analytical determination. Ion-exchange properties of $Na_2Ti_3O_7$, $K_2Ti_2O_5$, and $Na_2Ti_4O_9$ have been reported.[4–6] These materials have good stability against high temperatures and radiation.[7] Alkali metal (potassium and lithium) titanates were synthesized and employed for the efficient removal of cadmium(II) ions from aqueous solutions using radiotracer technique.[8] Comparative characteristics of ion-exchange properties of alkali metal titanates prepared by $TiCl_4$-alkali hydrolysis in relation to cations of some metals (Cd, Sr, etc.) have been reported and it has been show that lithium titanate possesses the highest sorption capacity.[9]

With regard to the ion-exchange behavior of lithium titanate in aqueous and mixed solvents, very little work has been done. In this regard, the present paper reports the adsorption behavior of Cu^{2+}, Zn^{2+}, and Ni^{2+} ions, representing alkaline earth metals, heavy metal ions, and/or corrosive materials as a function of hydrogen ion concentration at different reaction temperatures as well as in the presence and absence of organic solvents.

EXPERIMENTAL

All chemicals used were BDH (British Drug House, UK) analytical-grade reagents and used without further purification.

Preparation of Lithium Titanate

Lithium titanate was prepared by normal hydrolysis reaction using the method given by Mishra and Srinivasu,[10] by adding 300 mL of 1 M $TiCl_4$

(in 4 M HCl) to 700 mL of 2.8 M LiOH with constant stirring in a thermostated shaker water bath at 30°C ± 1°C at pH Ca. 12.3. The gelatinous precipitate was then digested by steam bath and standing for 48 hr, respectively. After standing, the precipitate was filtered and washed with double-distilled water until pH ca. 11. Finally, the precipitate was oven-dried at 60°C ± 1°C for 24 hr. The resulting granulates were crushed, sieved, and stored at room temperature.

Distribution Studies

The distribution coefficient values of Cu^{2+}, Zn^{2+}, and Ni^{2+} ions on lithium titanate were determined by shaking 0.05 g of the exchanger with 5 mL of 10^{-4} M of XCl_2 ($X = Cu^{2+}$, Zn^{2+}, and Ni^{2+} ions) in aqueous and organic media in a thermostated shaker water bath at 25°C, 40°C, and 60°C ± 1°C. After equilibration (6 hr sufficient to attain equilibrium), the mixture was centrifuged, and the filtrate concentrations of the Cu^{2+} and Ni^{2+} ions were analyzed using atomic absorption spectrophotometer model AA-6701F, Shimadzu, Japan, while Zn^{2+} was determined radiometrically as ^{65}Zn by measuring its γ rays using a NaI (Tl) scintillation detector connected to an ORTEC counting assembly, product from Enterprises, USA. Distribution coefficient values (K_d) were calculated from the following relation:

$$K_d = \frac{(C_0 - C)}{C} \cdot \frac{V}{m}, \quad \text{mL/g}$$

where C_0 and C are the concentration of metal ions before and after equilibration, respectively; V is the volume of solution in cm^3; and m is the weight of the exchanger per g.

All the experiments showed that the reproducibility measurements have a standard deviation less than ±5%.

Capacity Measurements

The ion-exchange capacity of lithium titanate in Li form for Zn^{2+}, Cu^{2+}, and Ni^{2+} ions at $[H^+] = 10^{-3}$ M in aqueous form and 30–90% by weight of acetone and methanol was determined by repeated batch equilibration of 0.05 g of solid and 5 mL of 0.01 M of the desired solution in shaker water bath adjusted at 25°C ± 1°C until equilibration was achieved. After equilibration, the phases were separated and analyzed for each element. On the other hand, the capacity of H, Cu, and Ni forms of lithium titanate have been determined for each Na^+ and Cs^+ ion.

X-Ray, IR, and Thermal Analysis

X-ray diffraction (XRD) patterns were carried out using a Shimadzu x-ray diffractometer, Model XD 490 (Japan), with nickel filter and Cu-K_α radiation. The IR spectra were measured within the KBr disc technique (400–5000 cm^{-1}) using BOMEM FT-IR model MB 157 (product from Canada). Differential thermal analysis (DTA) and thermogravimetric (TG) analyses were performed at a heating rate of 10°C min^{-1} using a Shimadzu DTG-40 thermal analyzer (Shimadzu, Kyoto, Japan).

RESULTS AND DISCUSSION

The IR spectra of lithium titanate showed that broadband in the range of 3200–3500 cm^{-1} may be due to the stretching mode of water molecules, while the absorption band at $\approx$1630 cm^{-1} may be due to their bending modes.[11] The absorption band at $\approx$1390 cm^{-1} may be characteristic of the deformation vibration bond of Ti–OH. The broadbands around 600–900 cm^{-1} may be assigned to M–O interaction vibrations.[10] The decrease in water content of the sample, as demonstrated by the decrease in band intensities of molecular water at about 3500 and 1630 cm^{-1}, is parallel with the increasing heating temperature from 50°C to 600°C.

Both TG and DTA curves for lithium titanate are shown in Fig. 1. Figure 1 manifested that the first weight loss at ~75°C corresponds to loss of free water. The weight loss is continuous up to ~600°C. Furthermore, the thermograms for different lithium titanate phases (H, Ni Zn, and Cu) are studied up to 800°C, as shown in Fig. 2. The values of water content (%) for different phases are calculated and summarized in Table 1.

The XRD patterns of lithium titanate in initial Li form heated up to 400°C; proton and Cu phases show semicrystalline nature, which means that structure does not change under treatments by heating (up to 400°C), acids, or cations adsorption, as shown in Fig. 3. However, when lithium titanate heated up to 850°C, it reveals complete inversion to crystalline product corresponding to TiO_2 (rutile) patterns, as shown in Fig. 3(d) which was indexed and matched from library charts.[12]

These criteria, which are expected from the results of IR spectra, XRD patterns, and thermal and elemental analyses of the lithium titanate matrix, led to the proposed molecular formula as given in Table 1.

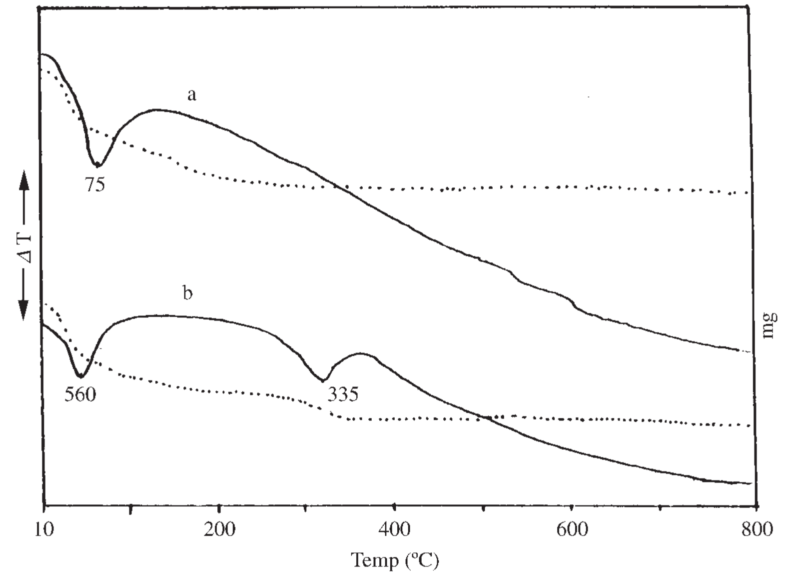

Figure 1. The DTA-TG curves of lithium titanate in initial (a) and Cu (b) forms.

Capacity Measurements and Distribution Studies

The effect of nitric acid concentrations on batch adsorption of Cu^{2+}, Zn^{2+}, and Ni^{2+} ions on lithium titanate revealed that quantitative adsorption occurred in the nitric acid molarity range of $10^{-4} - 10^{-1}$ M although the maximum adsorption occurred at concentrations of $10^{-3} - 10^{-2}$ M, as shown in Fig. 4. From the values of distribution coefficients, a selectivity series, $Cu^{2+} > Zn^{2+} > Ni^{2+}$, was established.

The values of ion-exchange capacity of lithium titanate for the studied cations from 10^{-3} M of HNO_3 in the absence and presence of certain organic solvents are investigated and given in Table 2. Table 2 clearly shows that the capacity sequence for the studied ions is parallel to the distribution coefficient order:

$$Cu^{2+} > Zn^{2+} > Ni^{2+}$$

Many authors reported the trend of selectivity for transition metal ions on different inorganic exchanger.[13,14] In this regard, two different assumptions describe the nature of the adsorbed ions. First is the adsorption of ions in dehydrated state, as reported for the adsorption of Cu^{2+} and Ni^{2+}

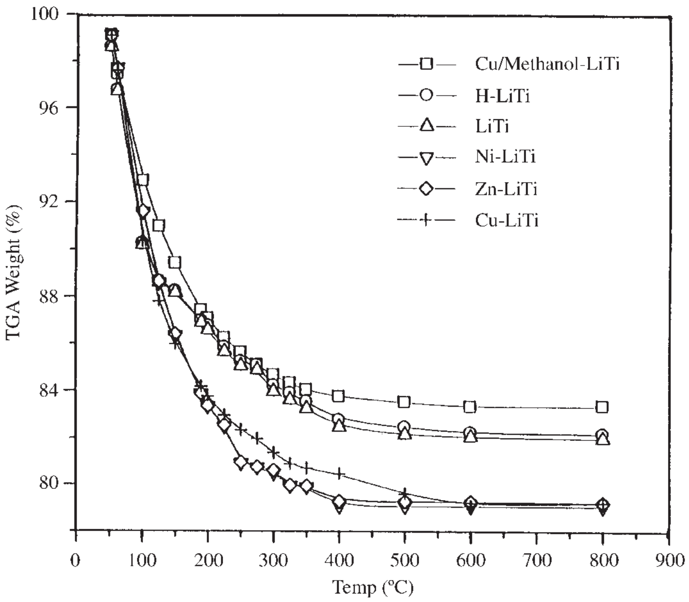

Figure 2. The TG curves of lithium titanate in initial form (LiTi) and in different loaded forms.

ions on sodium titanium silicate.[13] The second is explained by the fact that the alkaline metal ions are retained by the exchanger as essentially hydrated (or solvated) ions and thus the affinity is higher for the smaller solvated ion (large ionic radii) for which more exchange sites are accessible.[15] The last behavior was observed for adsorption of Cs^+ and Na^+ ions on amorphous

Table 1. Expected molecular formulas, water content, color, and XRD analysis of lithium titanate in different phases.

Exchanger	Phase	Water content (%)	Color	XRD analysis
Lithium titanate	Li_2TiO_3 $1.3H_2O$	17.96	White glass	Semicrystalline
	$H_{0.3}Li_{1.7}TiO_3$ $1.31H_2O$	17.76	White glass	Semicrystalline
	Cu phase	20.70	Hydrated copper	Semicrystalline
	Zn phase	20.75	White glass	Semicrystalline
	Ni phase	20.91	Hydrated nickel	Semicrystalline

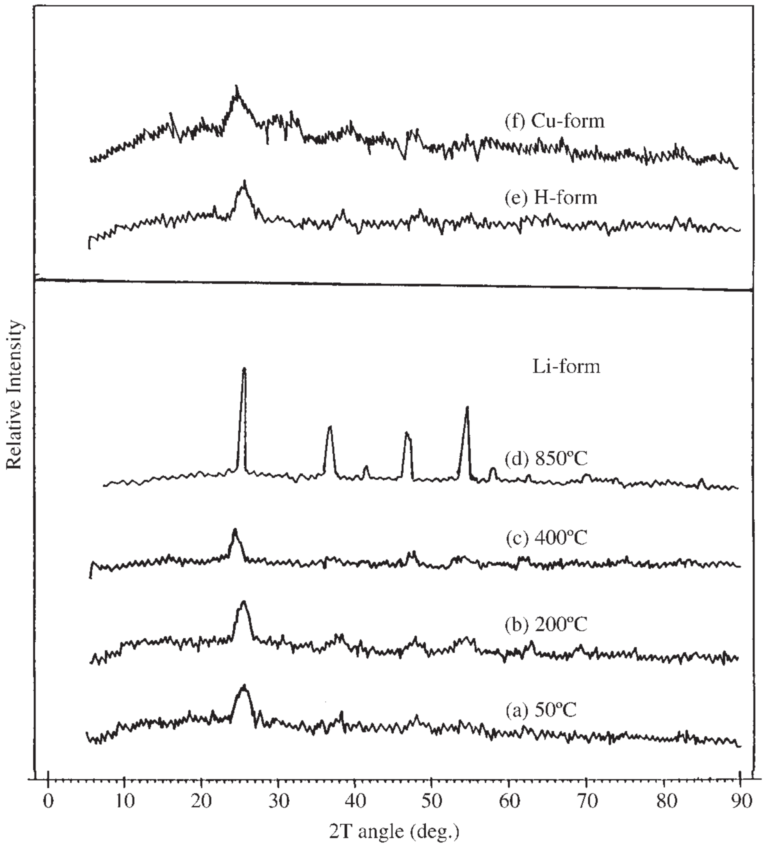

Figure 3. The XRD patterns of titanate in Li form dried at different temperatures (a)–(d), H form (e), and Cu form (f).

zirconium phosphate[15] and for Cu^{2+}, Zn^{2+}, Co^{2+}, Ni^{2+}, and Mn^{2+} ions on hydrous tin(IV) oxide.[16] Also, Nilchi et al. reported the sequence $Ba^{2+} > Sr^{2+} > Ca^{2+} > Mg^{2+}$ for cerium phosphate (disodium) exchanger.[17]

In our study, the trend of selectivity can be discussed from the viewpoint of thermal analysis (TG-DTA) of different alkaline metals and Li forms of lithium titanate. By TG analysis, the hydration water in the samples of lithium titanate have been determined before and after loading the divalent ions. (Cu^{2+}, Zn^{2+}, and Ni^{2+}). The results, which were obtained from TG thermograms (Fig. 2), are listed in Table 1. In all cases, the quantity of hydration water is higher for loaded samples. Also, the TGA curves (Fig. 2)

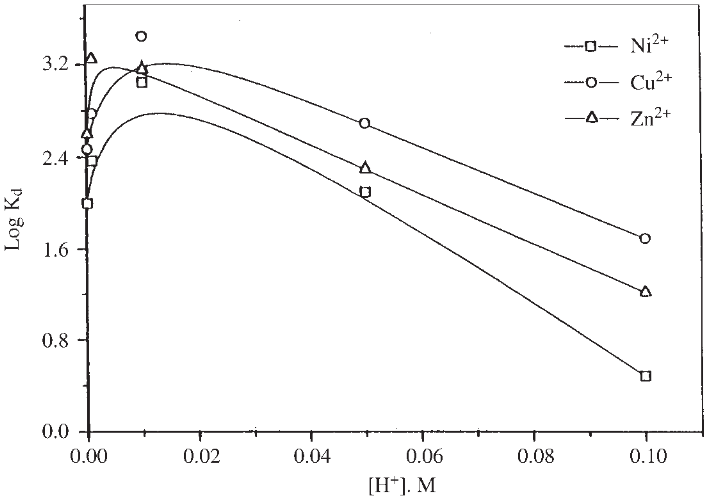

Figure 4. Distribution coefficient values of Cu^{2+}, Zn^{2+}, and Ni^{2+} ions as a function of nitric acid concentrations on lithium titanate at 25°C ± 1°C.

for the salt forms of lithium titanate are nearly similar, with a slight difference observed in percent of water contents:

Cu phase (20.73%) $<$ Zn phase (20.8%) $<$ Ni phase (20.9%)

The above results support the observed selectivity order and correlated very closely with the increase in the effective ionic radii, indicating that the

Table 2. Capacity of lithium titanate for Cu^{2+}, Zn^{2+}, and Ni^{2+} ions from various media at 10^{-3} M HNO_3.

		Capacity (meq g^{-1})		
Medium	Percentage	Cu^{2+}	Zn^{2+}	Ni^{2+}
Aqueous	100	3.82	3.62	2.86
Acetone	30	4.00	3.60	3.27
	60	4.08	4.01	3.32
	90	3.45	3.61	2.23
Methanol	30	3.80	3.80	3.03
	60	4.01	3.38	3.15
	90	3.10	3.03	2.90

radii of the ions are the dominant factor in the retention behavior of the studied cations.

Continually, as shown in Fig. 2, all samples of lithium titanate in salt forms (Cu, Zn, and Ni) lose their weights in two partially overlapping steps in the temperature range 50–600°C. The observed weight losses are due to both interlayer crystal water release and water release from the hydrated cations (Cu^{2+}, Zn^{2+}, and Ni^{2+}). At the same time, Fig. 1(a) and (b) shows the DTA thermograms of lithium titanate in both lithium and Cu forms (taken as example), confirming the TG results. Lithium titanate in Cu form [Fig. 1(b)] shows two endothermic peaks at 56°C and 335°C, corresponding to two weight losses. The first peak may be due to lithium titanate dehydration and the second may be ascribed to the dehydration of Cu^{2+} ions loaded on the exchanger. While in case of pure Li form [Fig. 1(a)], there is only one endothermic peak at 75°C, which may be due to water dehydration of lithium titanate.

The above results confirm that the studied ions adsorbed in hydrated Form and the selectivity order decreased as the size of the hydrated ion increased ($Cu^{2+} < Zn^{2+} < Ni^{2+}$).[18] It is worth noting that after sorption, lithium titanate assumes the color of the hydrated ions, which supports the results of DTA-TG thermal analysis.

Effect of Organic Solvents

A higher Cu^{2+} selectivity on lithium titanate than Zn^{2+} and Ni^{2+} ions in aqueous medium is also observed for mixed solvents (methanol and acetone), as shown in Figs. (5)–(7). Figures 5 through 7 illustrate that at the same condition of acidity, the selectivity sequence takes the order $Cu^{2+} > Zn^{2+} > Ni^{2+}$ from aqueous, methanolic, and acetone solutions, which is parallel with the capacity sequence under the same conditions. However, by increasing the amount of organic solvents up to 60% of the bulk solution (Table 2), there is a slight increase in ion-exchange capacity for different ions. The increase in organic solvents primarily led to the step-wise dehydration of hydrated ions, i.e., to a progressive increase of ionic interactions and hence to association in mixed solutions. This trend can be confirmed from the results of TG-thermal analysis for lithium titanate samples loaded with Cu^{2+} ions in the presence of 60% of alcoholic solution (see Fig. 2). The TG results reveal that the percentage loss in the total water content is the lower value (16.58%) relative to other value (20.7%) under identical conditions but in the absence of alcoholic solution, which, in turn, reinforces the idea of ion dehydration. Consequently, there is a low number of water-molecule penetration to the

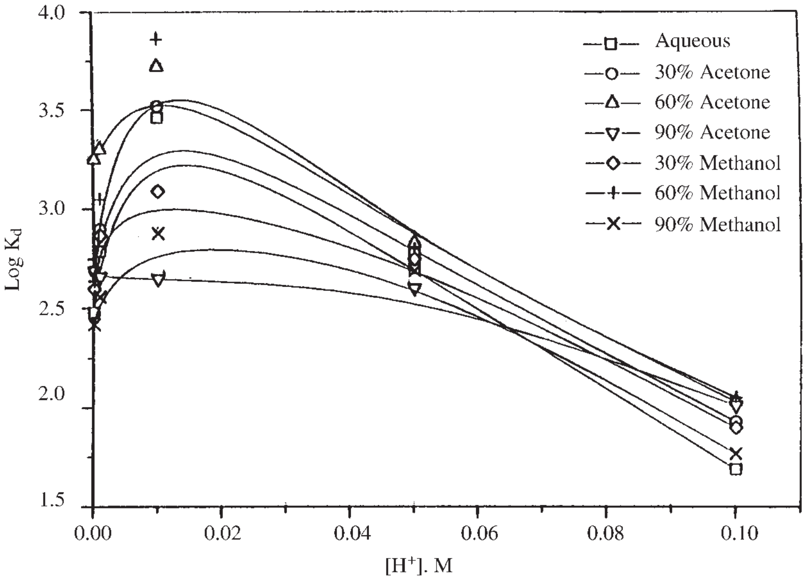

Figure 5. Distribution coefficient values of Cu^{2+} ions as a function of nitric acid concentration on lithium titanate at various organic solvents contents.

solid phase. The last observation gives higher probabilities for higher capacity values in the presence of organic solvents up to 60%, as given in Table 2.

Figures 5 through 7 reveal that at higher acid concentrations, the uptake of the studied cations decreases, which may be connected with the high energy requirement for dehydration of the cations,[13] or may be due to the high competiting effect of H^+ ions with the exchange sites.[14] A concave condition occurred in K_d curves for the studied cations at lower $[H^+]$, which may be due to the hydrolysis at higher pH values.

In order to establish a procedure to separate different studied ions, the separation factors (K_α) are calculated at certain conditions (60% organic solvents at 0.001 and 0.01 M of HNO_3) for Cu^{2+}, Zn^{2+}, and Ni^{2+} ions. Hence, at these conditions, the K_d have been improved and show optimum values, as shown in Figs. (5)–(7). Table 3 gives the K_α values, which show considerable differences from solvent to solvent and from one concentration to another. Among these conditions, methanol showed the highest separation factor at 0.01 M HNO_3, giving a value of 22.42 ($K_{d_{Cu}}/K_{d_{n}}$). However, this value is lower in the case of aqueous or 0.001 M HNO_3. Also, the highest value 3.34 ($K_{d_{Zn}}/K_{d_{Ni}}$) is found in the presence of 60% acetone at 0.01 M HNO_3.

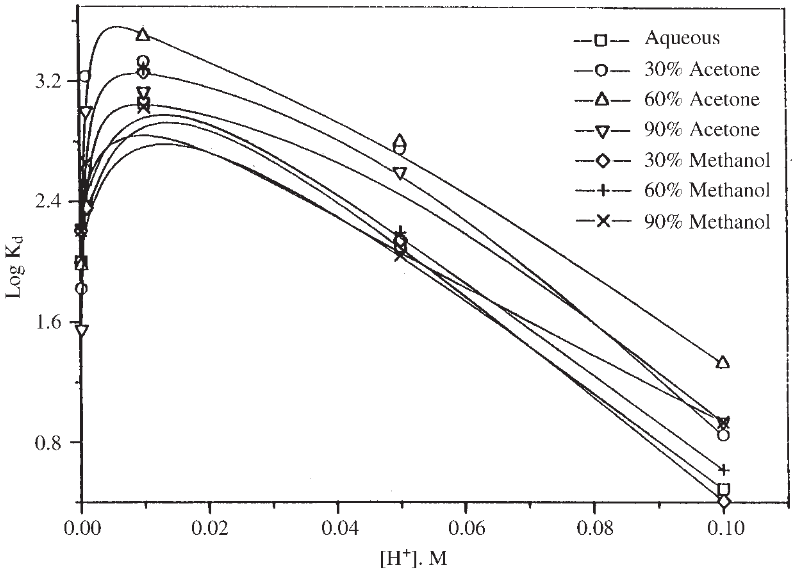

Figure 6. Distribution coefficient values of Zn^{2+} ions as a function of nitric acid concentration on lithium titanate at various organic solvents contents.

Generally, Table 2 showed that with the exception of 90% of organic solvents, all capacity values of the studied ions are higher in the mixed solvents than in aqueous media. This trend may be due to the above-mentioned results as well as the higher basisity of the solvents used,[16] which may lead to the replacement of more weakly bonding Li^+ in lithium titanate. In addition, the replacement of part of the aqueous phase by an organic solvent often enhances the ion-exchange adsorbabilities of inorganic ions.[19] The change in adsorption brought about by introducing alcohol in the system is in part due to modifications in properties of the aqueous phase and in part due to possible modifications in properties of the exchanger. Thus, the addition of alcohol to an ion-exchange system can be expected to influence swelling of the resin, dissociation, solvation, complex formation, and ionic and molecular interaction.[19] Also, solvation and complex formation play an important role in ion-exchange equilibria in mixed solvents.

The capacity of lithium titanate (in hydrogen and salt phases) for both Na^+ and Cs^+ ions has been determined (Table 4). Analysis of the data presented in Table 4 shows that the capacity of lithium titanate in the hydrogen form (pretreated with 0.1 M HNO_3 and with a water content ~17.76%) for

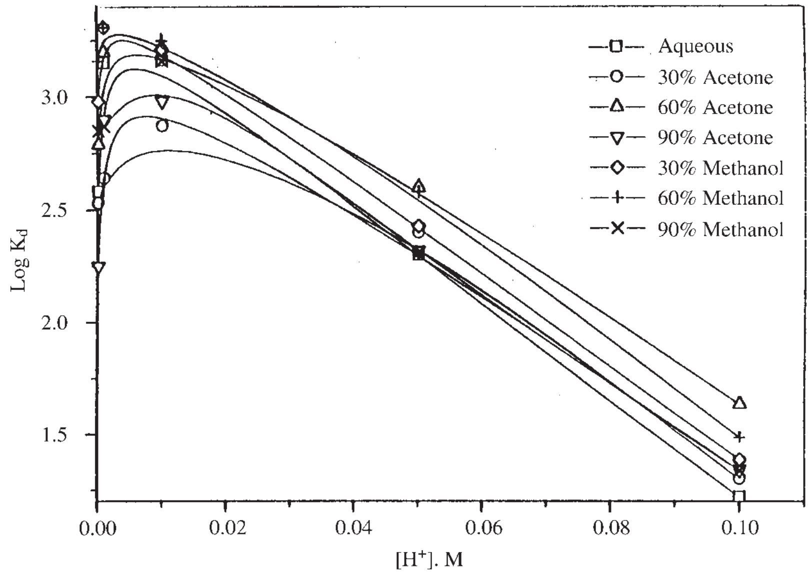

Figure 7. Distribution coefficient values of Ni^{2+} ions as a function of nitric acid concentration on lithium titanate at various organic solvents contents.

Table 3. Distribution coefficients (K_d) and separation factors $(K_\alpha)^a$ of Cu^{2+}, Zn^{2+}, and Ni^{2+} ions on lithium titanate from aqueous and organic media at 25°C ± 1°C.

	0.001 M HNO_3			0.01 M HNO_3		
Medium	Ni^{2+}	Zn^{2+}	Cu^{2+}	Ni^{2+}	Zn^{2+}	Cu^{2+}
Aqueous	1412	1122 (0.79)	602 (0.42) (0.53)	1445	234 (0.52)	2884 (2.00) (12.3)
60% acetone	1584	3162 (1.99)	1995 (1.26) (0.63)	1660	5377 (3.24)	5495 (3.80) (1.08)
60% methanol	1445	1905 (1.32)	1122 (0.78) (0.58)	1778	323 (0.18)	7244 (4.07) (22.42)

[a] $K_\alpha = K_d(Zn)/K_d(Ni)$ or $K_d(Cu)/K_d(Zn)$.

Table 4. Values of saturation capacity of Na^+ and Cs^+ ions on different forms of lithium titanate at 25°C ± 1°C.

	Capacity ($meq\,g^{-1}$)	
Ion-exchanger phase	Na^+	Cs^+
H phase	0.11	0.10
Li phase	2.32	1.96
Cu phase	2.62	2.30
Ni phase	2.58	2.27

Na^+ and Cs^+ ions is very low in comparison with Li form, which means that there are higher ion-exchange properties toward H^+ ions. The same result was reported for adsorption of Na^+ and Cs^+ ions on thermally prepared sodium titanate in protonated form.[20] The decrease of cation-exchange capacity of lithium titanate in protonated form was also observed by Izawa,[21] where $H_2Ti_3O_7$ showed ion-exchange properties only in the higher pH region because of Bronested acidic nature.

The conversion of lithium titanate to the salt phases (copper and nickel) and reusing it as adsorbent was investigated. The results show a drastic increase of the capacity for Na^+ and Cs^+ ions with respect to the Li form (Table 4). The above results confirm that the Cu^{2+} and Ni^{2+} ions may cause a slight increase in the interlayer distance of the semicrystalline lithium titanate matrix (Fig. 3). In this regard, Bortun et al.[22] showed higher affinity for Co^{2+}, Cd^{2+}, Pb^{2+}, Cu^{2+}, and Cr^{3+} ions on Ψ-zirconium phosphate (sodium or potassium form). However, Möller et al.[23] reported that the selectivity or capacity of sodium titanium silicate were not improved by doping with Nb ions. Generally, the adsorption of Cu^{2+} and Ni^{2+} ions as hydrated cations will produce high water content exchangers, which gives promise for using the new alkaline metal phases of lithium titanate as ion-exchange materials.

Effect of Reaction Temperature

The effect of temperature on the K_d values for Cu^{2+}, Zn^{2+}, and Ni^{2+} ions has been studied at a constant adsorptive concentration (10^{-4} M). The K_d values of different studied cations were increased by increasing the temperature from 25°C to 60°C ± 1°C. From the slope of the linear plot of the log K_d against $1/T$ (Fig. 8), the standard enthalpy changes (ΔH^0) of these reactions were 13.88, 24.69, and 20.79 $kJ\,mol^{-1}$ for Cu^{2+}, Zn^{2+}, and Ni^{2+}

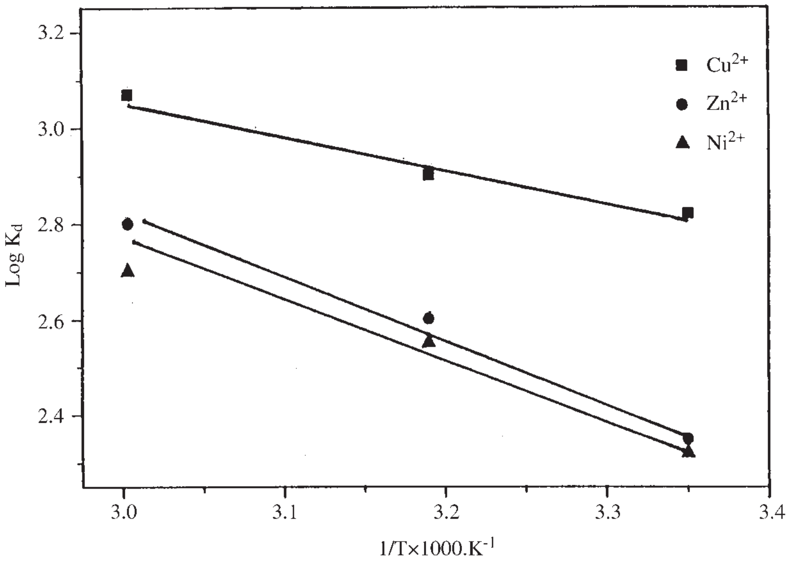

Figure 8. Plot of log K_d with $1/T$ for adsorption of Cu^{2+}, Zn^{2+}, and Ni^{2+} ions on lithium titanate.

ions, respectively. Numerically, the slightly higher values of ΔH obtained in the studied systems, compared to those usually reported[24] for ion-exchange mechanisms (8.4–12.6 kJ mol^{-1}), clearly suggests that our adsorption process of the studied ions is endothermic and chemosorptive in nature.[24,25]

CONCLUSION

The framework lithium titanate has selectivity sequence that is characteristics of the effective ionic radii (EIR) of divalent cations. The cations Cu^{2+}, Zn^{2+}, and Ni^{2+} have been adsorbed on lithium titanate in hydrated form. However, due to the dehydration effect of organic solvents on Cu^{2+}, Zn^{2+}, and Ni^{2+} ions, the sorption affinity was increased by increasing the percentage of organic solvents up to 60% in the bulk. The separation factors of the studied cations have been improved for Cu^{2+} and Zn^{2+} ions by the effect of organic solvents. Lithium titanate in a new form (Cu, Zn, and Ni forms) showed higher affinity for Na^+ and Cs^+ ions with respect to its Li form which suggests promising applicability as a new cation exchanger. Based on the calculation of ΔH^0 values for the exchange systems, the nature of adsorption can be evaluated processes.

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