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Comparison of chelating ligands on manganese (II) removal from aqueous solution

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ABSTRACT

The removal of manganese, Mn^{2+}, by chelating ion exchange resins of Lewatit TP 260 and Lewatit TP 207, which contain aminomethylphosphonic and iminodiacetic acid as functional groups, was studied under various experimental conditions such as solution pH and resin dosage. The ion exchange process indicated that the maximum removal could be achieved at pH ≥ 3. The results of adsorption equilibrium experiments fit well in Langmuir isotherm, and also indicated that the sorption was a monolayer process. Additionally, kinetic experiments showed that the pseudo-first-order model was suitable for TP 260 as well as pseudo-second-order for TP 207 resin. It was found out that the rate-determining step is the chemical reaction for both resins according to unreacted core model. Resins were also regenerated and obtained results showed that both resins can be reused after regeneration either with HCl or H_2SO_4.

Keywords: Aminomethylphosphonic acid; Iminodiacetic acid; Chelating resin; Lewatit TP 207; Lewatit TP 260; Manganese

1. Introduction

Manganese (Mn) exists in water as a groundwater mineral; however, it may also present due to pollution of underground sources. Manganese may become noticeable in tap water at concentrations higher than 0.05 mg/l of water by imparting a color, odor, or taste to the water [1]. In general, manganese enters natural waters from a variety of sources: (i) The rocks directly exposed to carbonated waters are the main natural source of Mn in geochemical cycle. (ii) Drainage of forest grounds contribute small amounts of metals to adjacent waters, particularly the coniferous trees accumulate such metals and are rained out into the river. (iii) Human-related activities (e.g. industrial activities) could introduce significant amounts of Mn to natural water sources [2]. It is an essential metal for human health and many enzymes are activated by Mn. Intake of high concentrations of Mn causes manganese

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psychosis, an irreversible neurological disorder. Similar to the most of the metallic species, excessive supply of Mn to plants has detrimental effects [3]. Many separation techniques have been proposed for the removal of Mn from various environmental samples, including solvent extraction, ion exchange, coprecipitation, membrane processes, and sorption [4–10]. Among the above-mentioned methods, ion exchange is highly popular and has been widely practiced in industrial wastewater treatment processes [11]. Chelating resins have ligands that can selectively bond to certain types or classes of metal cations. Ligand is the chemical term for an electron pair donor (Lewis base) that forms a bond with a metal cation that is a Lewis acid. Chelating resins are widely utilized to remove heavy metal ions from wastewater and to concentrate and retrieve noble metal ions. These resins have high thermal and chemical stability as well as the possibility of regeneration repeatedly. The chelates forming polymeric ligands are characterized by O, N, S, and P donor atoms containing reactive functional groups and are capable of coordinating to different metal ions [12].

In this study, two different chelating resins, Lewatit TP 207 and Lewatit TP 260, were used for the sorption of Mn^{2+} from aqueous solution. The effects of experimental parameters such as resin dosage, contact time, pH of the solution on the removal performance of resins were investigated and optimized for the most effective removal. In addition, the adsorption isotherms and kinetic models for Mn^{2+} were obtained and compared with different chelating ligands.

2. Experimental

2.1. Materials

Lewatit TP 207 and TP 260 are weakly acidic, macroporous cation exchange resins with chelating iminodiacetate and aminomethylphosphonic acid groups. The physicochemical properties of these resins were summarized in Table 1.

Mn^{2+} solutions of different concentrations were prepared from analytical grade $MnSO_4 \cdot H_2O$ (Merck, Germany). The solutions utilized for pH control were prepared from analytical grade HCl (Merck, Germany) or NaOH (Merck, Germany). pH meters were calibrated using pH 4.0 and 7.0 buffers (Merck, Germany).

The analysis of Mn^{2+} was performed spectrophotometrically by PG T80+ model spectrophotometer (PG Instrument, England).

2.2. Batch experiments

2.2.1. Effect of resin dosage

Batch experiments were conducted in 50-mL square plastic bottles using 25.0 mL of Mn^{2+} ion solutions (20.0 mg/L, pH 4.0) at room temperature. The various resin amounts (0.01–0.2 g) were added to the solution. The resulting suspension was stirred for 24 h in a temperature-controlled shaker. At the end of the experiments, the resins were separated by decantation. The solution was then analyzed for Mn^{2+} ions. The removal efficiency (*Re*) and ion exchange capacity (*q*) were calculated according to Eqs. (1) and (2), respectively.

$$q = V\frac{C_0 - C}{m} \tag{1}$$

$$Re = \frac{C_0 - C_\mathrm{i}}{C_0} \times 100 \tag{2}$$

In these equations, V is the volume of solution (L); C_0 and C_i are the initial and final concentrations of Mn^{2+} ions in the solution (mg/L), respectively; and m is the mass of resin (g).

2.2.2. Effect of solution pH

In this series of experiments, pH of solution varied from 1 to 7 and it was adjusted using HCl or NaOH solutions. The optimum resin amounts (0.05 g Lewatit TP 207 and 0.02 g Lewatit TP 260) were added to 25.0 mL of solution with initial concentration 20 mg-Mn^{2+}/L. The solutions were stirred for 24 h. At the end of the experiment, the resins were separated and Mn^{2+} concentration in the solution was found out by analyzing the solution.

2.2.3. Equilibrium experiments

Sorption studies of Mn^{2+} were carried out as follows: The optimum resin amounts were added to 25.0 ml of sample. The solutions were stirred in a temperature-controlled shaker. 24 h later, solutions were filtered and the Mn^{2+} ion concentration was measured. The initial Mn^{2+} concentrations varied from 20.0 to 1,000.0 mg Mn^{2+}/L (pH 4.0).

2.2.4. Ion exchange kinetic

Kinetic tests were performed by contacting optimum resin dosages (Lewatit TP 207 2.0 g/L, Lewatit TP 260 0.8 g/L) with 1.0 L of Mn^{2+} solution at pH 4.0.

Table 1
Physicochemical properties of chelating ion exchange resins

	LEWATİT TP 207	LEWATİT TP 260
Matrix	Cross-linked polystyrene	Cross-linked polystyrene
Functional group	Iminodiacetic acid	Aminomethylphosphonic acid
Ionic form	Na^+	Na^+
Total capacity	2.2 (min) eq/L	2.3 (min) eq/L
Water retention	53–58%	59–61%

The Mn^{2+} concentration in the solution was 20 mg-Mn^{2+}/L. Solution was stirred with an overhead mixer. Mn^{2+} concentrations were monitored by taking the samples at prescribed times.

2.2.5. The effect of interfering ion

The influences of diverse foreign ions such as Ca^{2+}, Mg^{2+}, and Cu^{2+} on the removal of Mn^{2+} were investigated. Mn^{2+} concentration in the solution was 20 mg/L and concentration of other ions adjusted as have same equivalent ratios (Equivalent ratio = charge of ion × concentration of ion).

2.2.6. Regeneration experiments

This part of experiments was carried out in two stages. In the first stage, 1.0 g resin was contacted 250 ml of Mn^{2+} solution (1,000 mg Mn^{2+}/L) for 24 h. Later, the resin was removed from solution and washed with pure water and then air dried. The Mn^{2+} concentration in feed and in effluent was analyzed and loaded amount of Mn^{2+} was calculated. In the second step of work, an amount of resin (0.1 g) loaded with Mn^{2+} was placed in a series of flasks containing 25 ml of different regeneration solutions; then, the mixture was shaken at room temperature for 24 h. Finally, Mn^{2+} concentration in the solution was determined.

2.3. Mn^{2+} speciation

We used Visual MINTEQ 3.1 program to calculate the predominant species of Mn^{2+} under the optimum experimental conditions (20 mg-Mn^{2+}/L, pH 4.0) The results showed that 95.8% of Mn^{2+} is predominant and 4.17% as MnSO4 (aq) and the rest is as $MnCl^+$.

3. Results and discussions

3.1. Effect of resin dosage on the removal of Mn^{2+}

The results of the residual Mn^{2+} concentration against amounts of different resin dosages are shown in Fig. 1. The removal of Mn^{2+} increased as resin amount increased. The reason is that the number of available ion exchange sites increases with increasing adsorbent amount. As can be seen in Fig. 1, the optimum resin amounts are 0.05 g Lewatit TP 207 and 0.02 g Lewatit TP 260).

3.2. Effect of solution pH on the removal of Mn^{2+}

The pH of the solution can alter the structure of ligands on ion exchange resins as well as the speciation of metal ions in aqueous solution [13]. Fig. 2 demonstrates the effect of pH on the removal of Mn^{2+}. It is clearly seen in Fig. 2 that increasing pH from 1 to 3 causes a significant increase in removal of Mn^{2+}. Additionally, when the pH of solution was increased further, no change was observed in the removal performance of Mn^{2+}.

The functional groups can act as tridentate ligands and the structure of formed complexes could be seen in Fig. 3.

In solutions with a pH about 2 (or less), carboxylic groups, phosphonic acid groups, and nitrogen atom occur in the protonated form [14]. At low pH, excess of hydrogen ions can compete effectively with Mn^{2+} for binding sites, resulting in a lower level of Mn^{2+} removal. When the pH was increased, the H^+ is

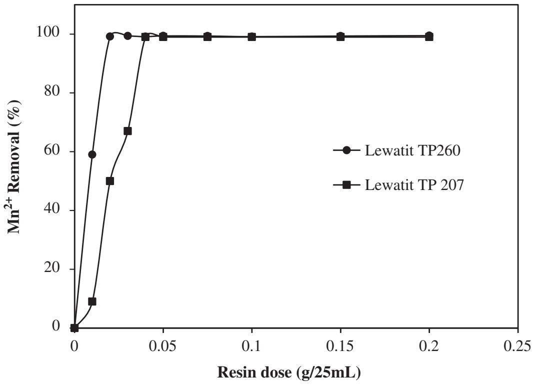

Fig. 1. Effect of resin dosage on the removal of Mn(II).

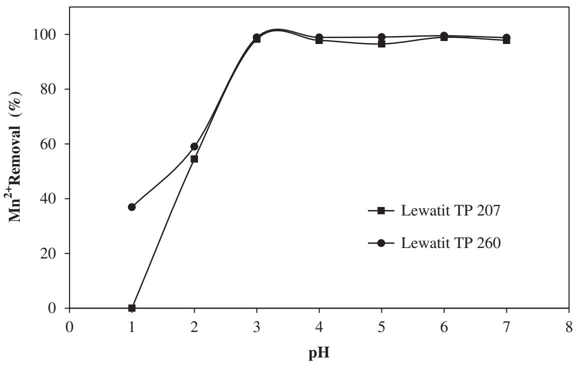

Fig. 2. Effect of solution pH on the removal of Mn(II).

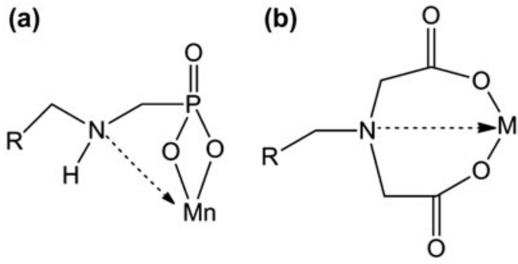

Fig. 3. Removal mechanism of Mn(II): (a) Lewatit TP 207 resin (b) Lewatit TP 260 resin.

displaced from the functional group of the resin, thus allowing the ion exchange of the Mn^{2+} to the resin [14,15].

In order to prevent the precipitation of $Mn(OH)_2$, the experiments were not performed in alkaline pH ($pH > 7.0$).

3.3. Equilibrium experiments

3.3.1. Langmuir model

The Langmuir isotherm is a commonly applied model for adsorption on a completely homogenous surface with negligible interaction between adsorbed molecules. The model assumes uniform adsorption energies onto the surface and maximum adsorption depending on saturation level of monolayer [16]. Langmuir model can be represented with the following linear Eq. (3):

$$\frac{C_e}{Q_e} = \frac{1}{b\,Q_0} + \frac{C_e}{Q_0} \tag{3}$$

where Q_e (mg/g) is the amount of Mn^{2+} sorbed per gram of dry resin at equilibrium, C_e is the equilibrium concentration of Mn^{2+} in the solution (mg/L), Q_0 (mg/g), and b (L/mg) are the Langmuir constants related to the capacity and energy of sorption, respectively.

3.3.2. Freundlich model

The Freundlich model is known as the earliest empirical equation and is shown to be consistent with exponential distribution of active centers, characteristic of heterogeneous surfaces [17] Freundlich Eq. (4):

$$\log Q_e = \log K_f + \frac{1}{n}\log C_e \tag{4}$$

where K_f and n are the Freundlich constants which are indicative of relative capacity and adsorption intensity, respectively. Adsorption isotherm curve for such resins was shown in Figs. 4 and 5. In addition, the calculated data are summarized in Table 2.

From the obtained results, the regression correlation coefficients (R^2) for Mn^{2+} complex with both resins are very high for the Langmuir model, which means that sorption of Mn^{2+} is monolayer. Also from the Figs. 4 and 5, it is clear that experimental results fit better with Langmuir model.

3.4. Ion exchange kinetics

Kinetic tests were performed by exposing 0.8 g for TP 260 resin and 2.0 g for TP207 resin to 1.0 L of Mn^{2+} solution (20.0 mg Mn^{2+}/L at pH 4). The obtained results are depicted in Fig. 6.

As can be seen from Fig. 6, kinetic of TP 207 is faster than TP260 resin. This may be because of the stability constant differences of functional groups–Mn^{2+} complex. The Mn–iminodiacetate complex formation constant is 2.51×10^5 [18] and Mn–aminomethylphosphonate is 3.16×10^2 [19]. The stability constant of iminodiacetate complex is larger than aminomethylphosphonate and

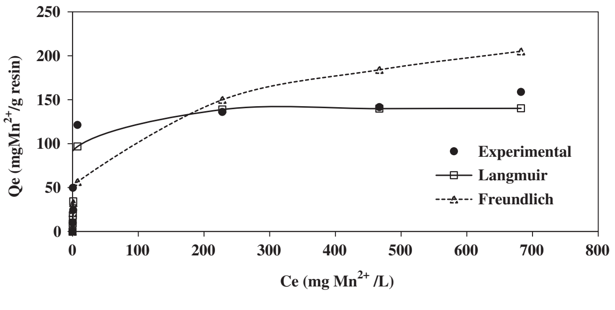

Fig. 4. Adsorption isotherm curve TP 207.

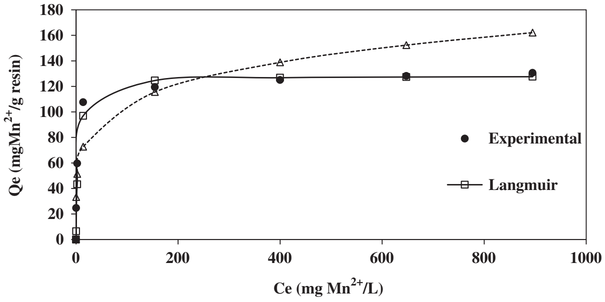

Fig. 5. Adsorption isotherm curve TP 260.

this may be the fact that kinetic of TP 207 is faster than TP 260.

The experimental results were also evaluated using the most common kinetic models, which are pseudo-first-order and second-order reactions. The linear form of pseudo-first-order rate equation was given by following Eq. (5) [20].

$$\log\,(q_e - q_t) = \log\,(q_e) - \frac{k_1\,t}{2.303} \tag{5}$$

Sorption rate constant k_1 (min^{-1}) can be calculated from the plot of log ($q_e - q_t$) vs. time.

If the rate of sorption is a second-order mechanism, the pseudo-second-order chemisorption kinetic rate equation is expressed as Eq. (6) [21].

$$\frac{t}{q_t} = \frac{1}{k_2\,q_e^2} + \frac{1}{q_e}\,t \tag{6}$$

If pseudo-second-order kinetics is applicable, the plot of t/q_t against t of Eq. (6) should give a linear relationship, from which q_e and k_2 (g/mgmin) can be determined from the slope and intercept of the plot. Constant k_2 is used to calculate the initial sorption rate r; at $t \rightarrow 0$, as shown in Eq. (7) [22].

$$h = k_2\,q_e^2 \tag{7}$$

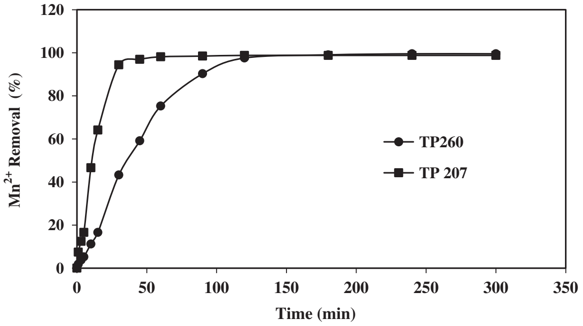

Fig. 6. Removal of Mn^{2+} vs. time.

The calculated parameters of kinetic models were summarized in Table 3. Removal of Mn^{2+} by TP 260 resin showed good compatibility with pseudo-first-order kinetic model and also R^2 value is found out to be 0.99. On the other hand, experimentally obtained q_e value (25 mg Mn^{2+}/g resin) is smaller than the calculated value from first-order model (31.70 mg Mn^{2+}/g resin). The calculated q_e from second-order reaction value is larger than the experimentally obtained q_e.

When the TP 207 resin was used, different results were obtained. According to the R^2 values, it was observed that pseudo-second-order reaction model is more suitable for TP 207 resin, and in this case, experimentally obtained q_e value for TP 207 resin is 10 mg Mn^{2+}/g resin. It could be stated that such value is very close to q_e value which was obtained from second-order reaction model.

Batch kinetic studies have also been evaluated using diffusion and reaction models. One approach uses the infinite solution volume (ISV) model, whereas the other method uses the unreacted core model (UCM) to describe the rate-determining steps in the ion exchange process [15,23].

According to the ISV model, the rate is controlled by film diffusion process. However, according to the UCM, chemical reaction is the rate-determining step in both resins (See Table 4). The reason for that could be complex formation of functional groups with Mn^{2+} ions.

Table 2
Isotherm constants for Mn^{2+} sorption on the ion exchange resin

	Langmuir isotherm constants			Freundlich isotherm constants		
Resin	Q_0 (mg g^{-1})	b (L/mg)	R^2	K_f (mg/g)	n	R^2
Lewatit TP 260	128.21	0.22	0.99	43.78	5.19	0.86
Lewatit TP 207	140.85	0.29	0.99	31.77	3.50	0.71

Table 3
The calculated parameters of pseudo-first- and pseudo-second-order kinetic models

Kinetic model	Parameter	TP 207	TP 260
Pseudo-first-order	k_1 (min^{-1})	0.07	0.03
	q_e (mg/g)	8.13	31.70
	R^2	0.94	0.99
Pseudo-second-order	k_2 (g/mg min)	7.9×10^{-3}	2.49×10^{-4}
	q_e (mg/g)	10.24	40.16
	h (mg/gmin)	0.83	0.40
	R^2	0.99	0.87

3.5. Selectivity of resins

Foreign ions are one of the key factors affecting the removal performance of ion exchange resins. In order to find out the effect of foreign ion on Mn^{2+} removal, different metal ions were added to solution and obtained results were summarized in Table 5.

As summarized in Table 5, removal performance of resins was slightly influenced in the presence of foreign ions. Especially, in the presence of Cu^{2+}, removal of Mn^{2+} decreased from 99 to 90%. In the presence of Ca^{2+} and Mg^{2+}, removal of Mn^{2+} was not influenced. According to the manufacturer's data sheet [24], divalent cations are removed from neutralized water in the following order: $Cu^{2+} > VO^{2+} > UO_2^{2+} > Pb^{2+} > Ni^{2+} > Zn^{2+} > Cd^{2+} > Fe^{2+} > Be^{2+} > Mn^{2+} > Ca^{2+} > Mg^{2+} > Sr^{2+} > Ba^{2+}$ for TP 207 (iminodiacetate resin) and $UO_2{}^{2+} > Pb^{2+} > Cu^{2+} > Zn^{2+} > Ni^{2+} > Cd^{2+} > Co^{2+} > Mg^{2+} > Sr^{2+} > Ba^{2+}$ for TP 260 [25]. The obtained results are compatible with manufacturer's data sheet.

Table 5
Removal of Mn^{2+} (%) in the presence of interfering ions

	Interfering ion in the solution		
Resin	Cu^{2+}	Ca^{2+}	Mg^{2+}
Removal by TP 207	89	99	99
Removal by TP 260	91	99	99

The choice of an effective chelating resin is dictated by the physicochemical properties of the resin materials. These are the acid–base properties of the metal species and the resin materials, and the polarizability, selectivity, sorption capacity, kinetic and stability characteristics of the resin. The functional groups in the chelating polymer materials usually act as bases. The oxygen-containing functional groups are hard and sulfur-containing groups are soft. Functional groups with a basic nitrogen atom have an intermediate character. According to Pearson's hard–soft–acid–base principle, soft metal ions (Lewis acid) prefer to bind with soft ligands (Lewis base) and hard metal ions prefer to bind with hard ligands [26,27]. The iminodiacetate and aminomethylphosphonic acid have two oxygen (O) and one nitrogen (N) for bonding the metal ions.

The order of increasing hardness parameters for selected metal ions are: $Ca^{2+} > Mg^{2+} > Mn^{2+} > Cu^{2+}$ [28]. However, the obtained results were different than this arrangement. It should be noted that variations in the synthesis procedure which resulted in variations in the structure of the matrix, degree of cross-linking, density of functional groups, proportion of functional groups, and also the particle size also effects the removal performance of resin [26].

Table 4
Linear regression analysis data related to diffusion models on sorption kinetics of Mn^{2+} ions

				R^2 value	
Method	Equation	k	Rate-controlling step	Lewatit TP-260	Lewatit TP-207
ISV	$-\ln(1-X) = k_{li}t$	$k = 3DC/r_o\delta\, Cr$	Film diffusion	0.96	0.96
	$-\ln(1-X^2) = kt$	$k = Dr\pi^2/r_o^2$	Particle diffusion	0.91	0.91
UCM	X	$k = 3C_{Ao}K_{MA}/ar_o\, C_{So}$	Liquid film	0.95	0.96
	$3–3(1-X)^{2/3}–2X = kt$	$k = 6D_{eR}C_{Ao}/ar_o^2\, \mathrm{C}_{So}$	Reacted layer	0.98	0.91
	$1-(1-X)^{1/3} = kt$	$k = K_sC_{Ao}/r_o$	Chemical reaction	0.99	0.97

Table 6
Percentage recovery of Mn^{2+} by different regeneration solutions

	Regeneration (%)	
Regenerated by	TP 207	TP 260
1 M HCl	96	73
1 M HNO_3	81	90
1 M H_2SO_4	99	93
2 M HCl	99	99
2 M HNO_3	89	86
2 M H_2SO_4	99	99

3.6. Regeneration of ion exchange resins

In the regeneration studies, the Mn-loaded resins were exposed to HCl, HNO_3, and H_2SO_4 solutions with different concentrations. Regeneration efficiency was calculated using the following Eq. (8) and obtained results were summarized in Table 6.

$$\text{Regeneration efficiency } (\%) = \frac{\text{Amount of eluated } Mn^{2+}}{\text{Amount of loaded } Mn^{2+}} \times 100 \quad (8)$$

The data of Table 6 showed that 2 M HCl or 2 M H_2SO_4 regenerated about 99% of such resins.

4. Conclusions

In this study, the removal of Mn^{2+} ions from aqueous solutions onto chelating ion exchange resins was investigated under various experimental conditions. The ion exchange process was relatively fast and the removal of Mn^{2+} completed in 2 h. Obtained results showed that kinetic of TP 207 was faster than TP 260. The optimum operating conditions for the removal of Mn^{2+} ions from aqueous solutions were $pH \geq 3$, 2 g resin/L for TP 207 and 0.8 g resin /L for TP 260 of resin dosage. The linear Langmuir and Freundlich isotherm models were used to represent the experimental data and experimental data could be relatively well interpreted by the Langmuir isotherm. By applying the kinetic models to the experimental data, it was found out that aminomethylphosphonic acid containing resin (TP 260) follows the pseudo-first-order rate kinetic and iminodiacetic acid containing resin (TP 207) follows the pseudo-second-order rate kinetic. The Mn^{2+} iminodiacetic acid interaction is stronger than Mn^{2+} aminomethylphosphonic interaction, therefore kinetic of TP 207 is faster than TP 260. Resins can be regenerated with 2 M HCl or 2 M H_2SO_4 with 99% recovery rate.

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