Synthesis and Analytical Study of New Chelating Resin Containing Sulfadiazine Drug

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Abstract: A new chelating resin was prepared by mixing sulfadiazine drug and TMP (trimethylolphenol). It was polymerized by heating to 90 °C then it was post cured to 100 °C after that it was grinded. The chelating behavior was examined against Cu^{2+} , Ni^{2+} using patch method in deferent conditions like treatment time and pH at room temperature. The resin show a good loading capacity toward Cu^{2+} (in treatment time = 3 h & pH=4) = 0.2174 mg ion / 100 mg resin and it show good loading capacity toward Ni^{2+} (in treatment time = 24 h & pH=4) = 0.14 mg ion / 100 mg resin.

Keywords: Chelating resins, Sulfadiazine, Chelating behavior.

Introduction

Chelating resins has wide range of interest in pre-concentration of trace metal ions and separation of selective metal ions from solution that contain many other metal ions. Atsushi *et al*¹ have prepared a chelating resin contain poly (4-vinylpyridine) cross-linked with oligo(ethylene glycol dimethacrylates), the resin show high sorption rates towards Cu^{2+} in pH=4.

Uchiumi² has prepared a number of chelating resins by reaction of formazane derivatives with many acid groups like COOH , OH and AsO_3H_2 , and the distribution coefficients, pH dependency and exchange capacity for the resins was examined. The resins show high selectivity towards Cd^{2+} , Zn^{2+} and Cu^{2+} . A chelating resin was prepared by Egawa *et al*³ contains 1, 4, 8, 11-tetraazacyclotetradecane-5,7 dione with two cross linked copolymers beads styrene-divinylbenzene (RCS) and glycidyl methacrylate-divinylbenzene (RG) copolymers. The RG show high selectivity towards Cu(II) and the loading capacity of Cu(II) in RG was more than RCS. Suzuki *et al*⁴ have prepared a selective chelating resin complexes having iminodiacetic acid (IDA) as the functional group with oxomolybdenum(VI) or oxotungsten(VI). The ^{13}C and ^1H NMR spectra of the prepared complexes in the pH rang 4-7 was examined and they found that the complex ratio was 1:1 and the complex formation of IDA was much favorable with Mo(VI) than W(VI) .

Ohashi⁵ has prepared a chelating resin by bonding 8-quinolinol to macroporous poly (styrene-divinylbenzene) copolymer. The adsorption behavior of resin towards copper(II),

manganese(II), cobalt(II), nickel(II), zinc(II), lead(II), palladium(II), platinum(II) and iron(III) by batch method was studied. Gudasi *et al* have prepared⁶ a macrocyclic amide ligand *N,N'*-bis(2-benzothiazolyl)-2,6-pyridinedicarboxamide (BPD) and their complexes with Cu(II), Ni(II), Co(II), Mn(II), Zn(II) and Cd(II).

RAO *et al* have studied⁷ the sorption behavior of bombax malabaricum towards Mn(II), the sorption capacity was increased with increasing pH as well as the optimum treatment time and they found that the time 50 minutes has the maximum loading capacity. El-Ashgar⁸ was prepared a chelating resin diethylenetriamine polysiloxane and the separation of Co(II), Ni(II), Cu(II) from aqueous solutions was also studied.

Experimental

Stock solutions of Ni(II), Cu(II) were prepared using demineralized water and (pH= 2,4,6) HNO₃. The loading capacity of the resin towards Ni(II), Cu(II) were determined by using (ChromTech UV-1100) UV-Visible spectrophotometer. The wavelengths of maximum absorption (λ_{max}) were identified for both of Cu(II) and Ni(II). Then all absorption measurements were carried out in the same wave length for each element.

The standard calibration curve for each element was measured by taking the absorption for series of concentrations (0.5, 1, 2, 4, 6, 8 and 10 mg/L) of standard solution for each element (Table 1). The standard calibration curve for Cu(II) is shown in the Figure 1. Trimethylol phenol (TMP) was prepared according to Iraqi Patent⁹. Then the TMP was mixed with sulfadiazine drug, and it was allowed to polymerize by heating to 90 °C then it was post cured to 100 °C and was crashed.

The study was carried out by using the patch method by treating 0.1 g of resin with 10 mL of 100 mg/L of aqueous solution of the metal in pH rang (2,4 and 6) and treatment time (1,2,3 and 24 hour) by using electric shaker at room temperature. The solutions were filtered and the remaining concentration for the filtrate was measured by taking the absorption using the standard calibration curve for each element. The loading capacity for each treatment was measured by calculating the deference between the primary concentration and the concentration of the filtrate.

Table 1. The calibration curve for Cu(II).

Absorbance	Concentration, mg/L
0	0
0.002	0.5
0.0041	1
0.01	2
0.019	4
0.025	6
0.0332	8
0.049	10

Figure 1. The calibration curve for Cu(II).

Results and Discussion

The effect of treatment time on the loading capacity

The effect of treatment time on the loading capacity was studied by making all other effecting parameters constant such as the pH and temperature. Table 2 and Figure 2 show the relationship between the treatment time and the loading capacity for Cu(II) ions. The values in Table 2 and the curves in Figure 2 show that the increasing in loading capacity with increasing of treatment time until the treatment time be 3 hours, after that the increasing of treatment time

will lead to decreasing in the loading capacity in each studied pH. This may be explained after 3 hours of treating the resin with Cu(II) ions, the Cu(II) ions reach equilibrium in it's concentration between the resin and the solution then after that treatment time the equilibrium will be shifted toward the solution so the loading capacity of the resin will decrease.

The chelating site in the chelating resin will be able to make more coordination bonds with Cu(II) ions. This coordination bonds will be increased with treatment time increasing, so the resin loading capacity of Cu(II) ions will be increased until the treatment time reach 3 hours. After 3 hours of treating the resin with Cu(II) ions some of formed coordination bonds will be broken and that broken bonds will be increased with increasing of treatment time, so the resin loading capacity of Cu(II) ions will decrease.

Table 2. The effect of treatment time on the loading capacity.

mg ion / 100 mg resin	Time, h	pH
0.0217	1	2
0.0652	2	
0.1522	3	
0.1129	24	
0.0652	1	4
0.1087	2	
0.2174	3	
0.1196	24	
0.0326	1	6
0.0869	2	
0.1696	3	
0.0957	24

Figure 2. The effect of treatment time on the loading capacity for Cu(II) ion in different pH.

Study the effect of pH on the loading capacity

The effect of pH on the loading capacity was studied by making all other effecting parameters constant such as the treatment time and temperature. Table 3 and Figure 3 show the relationship between the pH and the loading capacity. The maximum loading capacity is at pH=4.

In the strong acidic media such as pH=2, some of chelating atoms in the chelating site in the resin might be ionized with protons, so it will not be able to make coordination bonds with Cu(II) ions that lead to decreasing in chelating efficiency. So the loading capacity will be decreased.

In theoretically, at pH=6 the loading capacity should be the best or the maximum loading capacity. This may be explained that, pH=6 is very near to neutralization pH, so the other atoms near the chelating site will be available to chelate with Cu(II) ions.

At pH=4, the chelating site in the chelating resin may be in its best steric shape or the chelating atoms in the chelating resin may be more available to make more coordination bonds with Cu(II) ions that will lead to make the resin more effective or more able to withdraw the Cu(II) ions from the solution. So the loading capacity of the resin towards Cu(II) ions in the maximum level in all studied treatment times.

Table 3. The effect of pH on the loading capacity for Cu(II) ions.

mg ion / 100 mg resin	pH	Time, hour.
0.0217	2	1
0.0652	4	
0.0326	6	
0.0652	2	2
0.1087	4	
0.0869	6	
0.1522	2	3
0.2174	4	
0.1696	6	
0.1129	2	24
0.1196	4	
0.0957	6

Figure 3. The effect of the pH on the loading capacity toward Cu(II) ion.

The analytical study for the resin towards Ni(II) ion

The effect of treatment time on the loading capacity

The effect of treatment time on the loading capacity was studied by making all other effecting parameters constant such as the pH and temperature. Table 4 and Figure 4 shows the maximum loading capacity on treatment (time = 24 hour) in every studied pH. This may be explained after 24 hours of treating the resin with ion, the ion reach equilibrium in it's concentration between the resin and the solution then after that time there is no more effect of increasing time on the loading capacity.

The chelating atoms in the chelating resin will be able to make coordination bonds with Ni(II) ions, and this ability will increase with increasing of treatment time. So the loading

capacity will increase as the treatment time increases until the treatment time is 24 hours. It might be due to all the chelating sites in the chelating resin will be saturated with Ni(II) ions, so the resin can not withdraw more Ni(II) ions from the solution.

Table 4. The effect of treatment time of the loading capacity for Ni(II) ion in different pH.

mg ion / 100 mg resin	Time, hour	pH
0.0100	1	2
0.0600	2	
0.0950	3	
0.1150	24	
0.0250	1	4
0.0700	2	
0.1050	3	
0.1400	24	
0.0200	1	6
0.0450	2	
0.0850	3	
0.0950	24

Figure 4. The effect of treatment time of the loading capacity for Ni(II) ion in different pH.

Study the effect of pH on the loading capacity

The effect of pH on the loading capacity was studied by making all other effecting parameters constant such as the treatment time and temperature. Table 5 and Figure 5 show the relationship between the pH and the loading capacity. The maximum loading capacity is at pH=4.

At pH=2, some of chelating atoms in the chelating site in the resin might be ionized with protons, so it will not be able to make coordination bonds with Ni(II) ions that lead to decreasing in chelating efficiency. Therefore the loading capacity will be decreased.

In theoretically, at pH=6 the loading capacity should be the best or the maximum loading capacity. This may be explained that, pH=6 is very near to neutralization pH, so the other atoms near the chelating site will be available to chelate with Ni(II) ions.

At pH=4, the chelating site in the chelating resin may be in its best steric shape or the chelating atoms in the chelating resin may be more available to make more coordination bonds with Ni(II) ions that will lead to make the resin more effective or more able to withdraw the Ni(II) ions from the solution. So the loading capacity of the resin towards

Ni(II) ions in the maximum level in all studied treatment times. From Table 3 & 5 and Figure 3 & 5, it can be concluded that the best pH environment to this resin is pH=4 to work as a chelating resin.

Table 5. The effect of pH on the loading capacity for Ni(II) ions.

mg ion / 100 mg resin	pH	Time, hour
0.0100	2	1
0.0250	4	
0.0200	6	
0.0600	2	2
0.0700	4	
0.0450	6	
0.0950	2	3
0.1050	4	
0.0850	6	
0.1150	2	24
0.1400	4	
0.0950	6

Figure 5. The effect of the loading capacity for Ni(II) ion.

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