

# Iron(III) Chelating Resins. V. Cross-Linked Copolymers of 1-( $\beta$ -Acrylamidoethyl)-3-hydroxy-2-methyl-4(1H)pyridinone (AHMP) and *N,N*-Dimethylacrylamide (DMAA) for Iron(III) Chelation Studies

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## SYNOPSIS

Iron(III) chelating resins containing 3-hydroxy-2-methyl-4(1H)pyridinone (HMP) groups were prepared from 1-( $\beta$ -acrylamidoethyl)-3-hydroxy-2-methyl-4(1H)pyridinone (AHMP) and *N,N*-dimethylacrylamide (DMAA), using *N,N'*-ethylene-bis-acrylamide (EBAA) as a cross-linking agent. The cross-linked AHMP-DMAA copolymer resins were very stable and exhibited a high hydrophilicity with water contents of the swollen resins of 85–90%. The iron(III) chelating capacity of the resins was determined at physiological pH, and the chelating efficiency was, in general, higher than 90%. The iron(III) chelation was about 80% after 2 h and almost complete in 8 h. The distribution ratio of iron(III) between the resin and iron(III) solutions was quite high (3700–25,000 mL/g) and depended on the initial iron(III) concentration. It was possible to remove iron completely from aqueous solutions by using excess amounts of the resin. The resin also showed a very high selectivity for iron(III), and selectivity coefficients for iron(III) in the range of  $9.6 \times 10^8$ – $1.4 \times 10^{12}$  were determined at pH 5.6 in binary solutions of  $\text{Fe}^{3+}$  with  $\text{Mg}^{2+}$ ,  $\text{Cu}^{2+}$ ,  $\text{Ca}^{2+}$ ,  $\text{Mn}^{2+}$ , and  $\text{Zn}^{2+}$ . The iron(III) chelating resins could be easily regenerated and used again without any considerable loss of their iron(III) chelating capacities. © 1994 John Wiley & Sons, Inc.

## INTRODUCTION

Chelating resins differ from ion-exchange resins in their high selectivity in sorption processes, and they have been used for water treatment, pollution control, and recovery of metals and in analytical chemistry.<sup>1–4</sup> The specific application of iron(III) chelating resins has been increasing recently because of their potential uses in the biomedical field.<sup>5–8</sup>

To study the synthesis, properties, and application of iron(III) chelating resins, we directed our research project to the design and development of new iron(III) chelating resins and the investigation of the relationship between structure and properties.

A suitable iron(III) chelating resin for chemical and biomedical applications should possess a high capacity and a favorable selectivity toward iron(III), combined with a good stability and reusability. The selectivity of a chelating resin is determined to a large extent by the nature of the chelating groups (ligands) such as the affinity of the ligands to the metal ions and the stability constant of the complexes. The chelating capacity is dependent on the ligand density and the chelating efficiency, which is strongly influenced by the nature of the polymeric matrix. It has been reported that the properties of the polymer backbone or support not only have a significant effect on the chemical and mechanical stability of the chelating resin, but also affect chelating properties such as capacity, kinetic behavior, and selectivity.<sup>9–12</sup> This is illustrated by the fact that, generally, the experimental capacity of a chelating

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resin is lower than the calculated value based on ligand density because part of the ligands is inaccessible for metal ions.<sup>11,13</sup> It is generally considered that a poor selectivity, low capacity, and a slow sorption rate are often caused by the low affinity of the ligands on the resin, steric hindrance, and insufficient hydrophilicity or swelling degree of the resin.<sup>9-13</sup>

In our previous work, synthesis and properties were studied for various iron (III) chelating systems prepared by the immobilization of natural and synthetic iron (III) chelators or by the polymerization of a functional monomer with a comonomer.<sup>14-16</sup> Desferrioxamine (DFO), 1-( $\beta$ -aminoethyl)-3-hydroxy-2-methyl-4(1H)pyridinone (HP), and L-mimosine were immobilized by the coupling of DFO, HP, or L-mimosine to CNBr-activated Sepharose gels. The ligand-Sepharose gels showed a high affinity for iron (III) and were used for removing iron from milk, wine, whey, and lactoferrin. However, the stability of the gels in water was not so high, and about 0.5–1.0% ligands were released from the gels in 24 h, which might be due mainly to the hydrolysis of isourea bonds.<sup>14,15</sup>

To obtain a chelating system with a higher stability, iron (III) chelating beads were synthesized by the copolymerization of 1-( $\beta$ -acrylamidoethyl)-3-hydroxy-2-methyl-4(1H)pyridinone (AHMP) with 2-hydroxyethyl methacrylate (HEMA) and cross-linked with ethylene glycol-bis-methacrylate (EGBMA). The resulting AHMP-HEMA copolymer beads had 3-hydroxy-2-methyl-4(1H)pyridinone (HMP) groups as iron (III) chelating ligands connected to the polymeric matrix through amide bonds. The beads were found to be more stable in water and also had a sufficiently high ligand density. However, the AHMP-HEMA copolymer beads exhibited low iron (III) chelating efficiencies, which was probably due to the poor hydrophilicity of the polymer backbone.<sup>16</sup>

Therefore, similar to the preparation of AHMP-HEMA resins, new iron (III) chelating resins were synthesized by the polymerization of a functional monomer, a comonomer, and a cross-linking agent. AHMP was used as the functional monomer because the monomer is a strong iron (III) chelator.<sup>17</sup> *N,N*-Dimethylacrylamide (DMAA) and *N,N'*-ethylenebis-acrylamide (EBAA) are both more hydrophilic than are HEMA and EGBMA, respectively, and were therefore chosen as comonomer and cross-linking agent to improve the hydrophilicity of the resin.

In this article, the synthesis of cross-linked AHMP-DMAA copolymer resins by polymerization

of AHMP, DMAA, and EBAA with various compositions is reported. In addition, results are given about the stability, water content, iron (III) chelating capacity, chelation rate, iron (III) removal ability, selectivity coefficient for iron (III), regeneration, and reusability of the iron (III) chelating resins.

## EXPERIMENTAL

### Materials

1-( $\beta$ -Acrylamidoethyl)-3-hydroxy-2-methyl-4(1H)pyridinone (AHMP) was prepared from 3-hydroxy-2-methyl-4-pyrone as described previously.<sup>17</sup> *N,N*-Dimethylacrylamide (DMAA) and *N,N'*-ethylenebis-acrylamide (EBAA) were obtained from Aldrich Chemical Co. and used without purification. Ammonium persulfate (Merck), sorbitan monostearate (Aldrich), and *N,N,N',N'*-tetramethylethylenediamine (Merck) were used as received. All other compounds such as  $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ ,  $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ ,  $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}$ ,  $\text{MnCl}_2 \cdot 2\text{H}_2\text{O}$ ,  $\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$ ,  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ , and iron (III) citrate were obtained from Aldrich or Merck.

### Methods

#### Preparation of Cross-linked AHMP-DMAA Copolymer Resins

AHMP-DMAA resins were prepared by the reverse-phase suspension polymerization of AHMP, DMAA, and EBAA (Table I). In a typical experiment, AHMP (3.0 mmol), DMAA (54 mmol), EBAA (3.0 mmol),  $(\text{NH}_4)_2\text{S}_2\text{O}_8$  (0.6 mmol), water (40 mL), hexane (100 mL), and  $\text{CCl}_4$  (59 mL) were placed into a reaction vessel fitted with a mechanical stirrer. While stirring, 100 mg of sorbitan monostearate was

**Table I** Preparation of Cross-linked AHMP-DMAA Resins

Resin Code	Monomer Composition (Mol %)			Weight Recovery (%)
	AHMP	DMAA	EBAA	
1	0.0	95.0	5.0	98
2	2.0	93.0	5.0	98
3	3.7	91.3	5.0	98
4	5.0	90.0	5.0	98
5	10.0	85.0	5.0	96
6	20.0	75.0	5.0	83
7	95.0	0.0	5.0	0

added, and the mixture was flushed with N<sub>2</sub> for 20 min. Then, N,N,N',N'-tetramethylethylenediamine (0.20 mL) was added and stirring was continued for 2 h at 40°C. Resin 4 was filtered; washed with 2-propanol, methanol, water, ethanol, carbon tetrachloride, and ethyl acetate; and dried in vacuum at 80°C for 48 h.

### Swelling Measurements

The water content in swollen AHMP–DMAA resins was determined as follows: Approximately 0.5 g of dry resin was weighed into a flask and swollen with 40 mL of distilled water for 24 h at 25°C. The swollen resin was weighed and the water content (%) was calculated according to the formula

Water content (%)

$$= \frac{\text{wet weight} - \text{dry weight}}{\text{wet weight}} \times 100$$

### Stability of AHMP–DMAA Resins

As an indication for the stability of the AHMP–DMAA resins, the release of AHMP groups from the resins in water was determined at 25°C or at 121°C. About 0.2 g of dry resin was placed into a flask with 20 mL of water. The flask was rotated at 25°C or incubated in an autoclave at 121°C for a determined time. The amount of released AHMP in the supernatant was measured by its maximal absorbance at 280 nm.

### Spectrophotometric Measurements

UV-visible absorbance measurements were made with a Uvikon 930 spectrophotometer. Concentrations of iron and other metal ions in aqueous solutions were determined with the UV-visible spectrophotometer or with a Perkin-Elmer Zeeman 5000 atomic absorption spectrophotometer (AAS). For measuring the iron(III) contents in aqueous solutions by UV-visible spectrophotometry, a procedure with 1,10-phenanthroline<sup>18</sup> was used. Infrared spectra were recorded with a Bio-Rad FTS-60 FTIR spectrophotometer.

### Measurement of Iron(III) Chelation

Experiments for the determination of the iron(III) chelation of the resins were carried out with an iron(III) citrate solution at physiological pH except where otherwise indicated. Iron(III) citrate solutions (1.8 mM) were freshly prepared by dissolving appropriate amounts of iron(III) citrate in phos-

phate-buffered saline (PBS, pH 7.4), and the final pH of the solution was 6.8.

**Batch Chelating Capacity.** In a stoppered vessel were placed 30–200 mg of dry resin and 25 mL of the iron(III) citrate solution, and the mixture was rotated for 24 h at 25°C. The amount of iron(III) chelated on the resin was calculated from the change in the iron(III) concentration. For comparison, the procedure was carried out using a blank control.

**Column Chelating Capacity.** About 100 mg of the resin was packed in a glass column ( $\phi$  6 × 200 mm). The resin was swollen in water for 2 h, and the iron(III) citrate solution was passed through the resin at a speed of about 5 mL/h for 24 h. The iron(III) content in the effluent was determined. The amount of iron(III) chelated on the resin was calculated from the difference between the amounts of iron(III) added and remaining in the effluent.

**Iron(III) Chelation Rate.** About 250 mg of resin 4 was added to 50 mL of the iron(III) citrate solution and rotated at 25°C. The iron(III) concentration in the supernatant was measured at regular intervals, and the amount of iron(III) chelated on the resin was calculated from the changes of the iron(III) concentration in the solution.

**Distribution Ratio of Iron(III).** Two hundred milligrams of resin 5 was placed into 10 mL of iron(III) citrate solutions with various iron(III) concentrations (0.5, 1.0, and 1.5 mM) in PBS (pH 7.4). The mixtures were rotated for 24 h at 25°C. The amount of iron(III) remaining in the solution was determined by AAS, and the amount of iron(III) chelated on the resin was calculated from the change of the iron(III) concentration in the solution. The distribution ratio of iron(III) between the resin and the solution was calculated according to the following formula<sup>19</sup>:

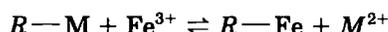
$$D = \frac{\text{mmol of iron(III) on the resin}}{\text{mmol of iron(III) in solution}} \times \frac{\text{volume of solution}}{\text{weight of resin}} \text{ (mL/g)}$$

**Removal of Iron(III).** Iron(III) citrate solution, 1.0 mM, in PBS (pH 7.4) was prepared. To 10 mL of the solution were added various amounts (100, 200, and 500 mg) of resin 4. After 24 h rotation at 25°C, the iron concentrations in the supernatants of the mixtures were determined by AAS.

### Selectivity Coefficient of the Resin for Iron(III)

FeCl<sub>3</sub> solutions and solutions of bivalent metal chlorides or sulfates (about 1 mM) were prepared

in a citrate buffer (10 mM) at pH 5.6 to prevent precipitation of the hydroxides of the metal ions. To a mixture of 10 mL of a  $\text{Fe}^{3+}$  solution and 10 mL of a  $\text{M}^{2+}$  solution was added about 60 mg of resin 4. The mixture was rotated at 25°C for 24 h. The contents of  $\text{Fe}^{3+}$  and  $\text{M}^{2+}$  remaining in the solution were determined by AAS. For determining the amounts of  $\text{Fe}^{3+}$  and  $\text{M}^{2+}$  chelated on the resin, the resin was filtered, added to 10 mL of 1 M HCl, and rotated for 24 h at 25°C to desorb the chelated metal ions. The amounts of desorbed  $\text{Fe}^{3+}$  and  $\text{M}^{2+}$  were measured by AAS. The selectivity coefficient,  $K_D$ , was calculated for the selective chelation reaction<sup>20</sup>:



$$K_D = ([\text{Fe}^{3+}]^2/[\text{M}^{2+}]^3)_R \times ([\text{M}^{2+}]^3/[\text{Fe}^{3+}]^2)_S$$

where  $R$  represents the resin;  $S$ , the solution; and  $\text{M}^{2+}$ , the bivalent metal ions.

### Regeneration of the Resins

The resins with iron (III) were washed with water and filtered, then added to an excess of 1 M HCl and rotated for 24 h at 25°C. The amount of iron (III)

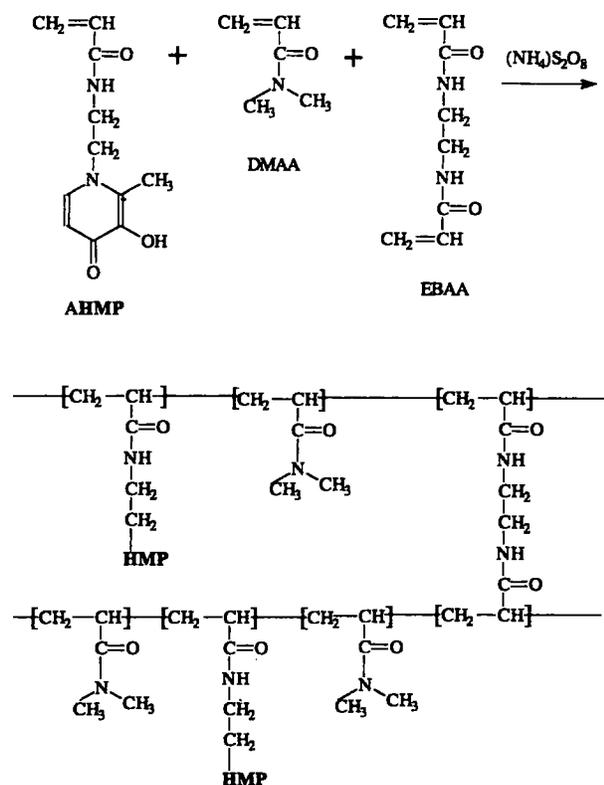
desorbed into the solution was measured. The resin was filtered and, before reuse, washed with 1 M HCl and water.

## RESULTS AND DISCUSSION

### Synthesis of Cross-linked AHMP-DMAA Copolymers

The synthesis of the cross-linked copolymers of AHMP with DMAA and the typical structure of the resins are shown in Scheme I. AHMP-DMAA resins were prepared by varying the initial AHMP percentage from 0 to 20, while keeping the content of the cross-linking agent at 5 mol % (Table I). It is to be noted that cross-linked poly(DMAA) was obtained almost quantitatively, whereas it was not possible to prepare cross-linked poly(AHMP) using the same conditions. Furthermore, the weight recovery of the AHMP-DMAA resins decreased with increasing AHMP content in the feed. These findings are in agreement with previous results for the suspension polymerization of AHMP, HEMA, and EGDMA,<sup>16</sup> also indicating that, possibly, the bulky and polar AHMP group makes the polymerization of AHMP difficult.

Figure 1(a) and (b) shows the IR spectra in the region of 2000–500  $\text{cm}^{-1}$  of cross-linked poly(DMAA) and of the AHMP-DMAA resin 4, respectively. Since the characteristic absorption bands of AHMP, DMAA, and EBAA were similar and because in the resin mainly DMAA units were present, the change in the IR spectrum due to the introduction of AHMP groups was small, except for a new peak at about 830  $\text{cm}^{-1}$ , which belongs to the stretching vibration of the two adjacent hydrogen atoms at the AHMP ring. Furthermore, in the spectrum of the AHMP-DMAA copolymer, a small absorption from the N—H deformation vibration of the AHMP groups occurred at 1560  $\text{cm}^{-1}$ . Two weak bands at 750 and 720  $\text{cm}^{-1}$ , only in the spectra of bonded secondary amides, are probably from out-of-plane deformations of AHMP N—H and EBAA N—H groups, respectively, because in the spectrum of the cross-linked poly(DMAA), only the band at 720  $\text{cm}^{-1}$  was present. In the IR spectra of the resins, a broad band near 1640  $\text{cm}^{-1}$  was assigned to the combined absorption of C=O groups in the amides and/or in the AHMP rings.



**Scheme 1** Synthesis of cross-linked copolymers of AHMP and DMAA.

### Stability of the Cross-linked Copolymers

The stability of the AHMP-DMAA resins in water was measured at 25°C as well as at 121°C in an

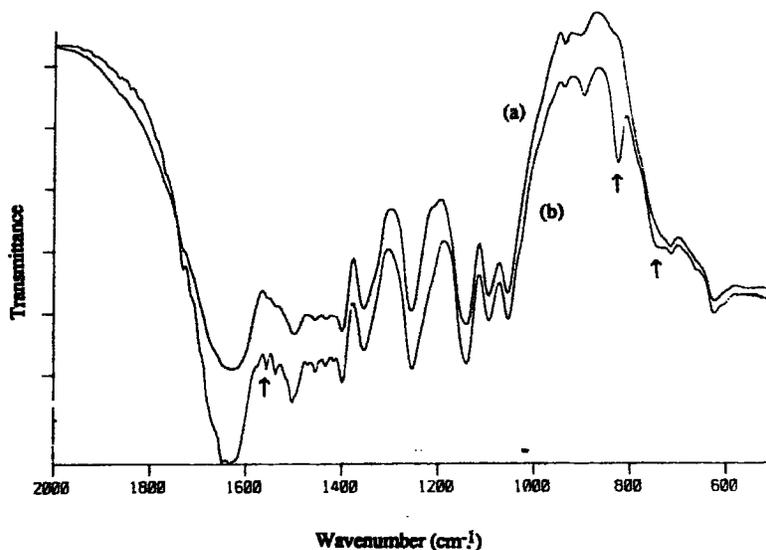


Figure 1 IR spectra of (a) cross-linked poly(DMAA) and (b) AHMP-DMAA resin 4.

autoclave, by determining the amount of AHMP groups released from the resins. The reason for measuring the stability at 121°C was to explore the possibility of using the resins in microbiological systems where it is generally necessary to sterilize biomedical materials at higher temperature before use. In the calculation of the percentages of AHMP groups released, the initial AHMP contents were used as the total contents of AHMP groups contained in the resins. As can be seen from Table II, the AHMP-DMAA resins were very stable in water and only less than 0.2% of the AHMP groups were released from the resins at 25°C for 48 h or at 121°C for 15 min. These resins are obviously more stable than are the iron(III) chelating systems that we studied earlier,<sup>14-16</sup> possibly indicating different effects of the polymeric matrices.

#### Water Content of Swollen Resins

The water contents of the swollen AHMP-DMAA resins were 85–88% (Table III), whereas swollen AHMP-HEMA beads contained only 40–45% water at the same conditions.<sup>16</sup> This difference has to be attributed mainly to the high hydrophilicity of DMAA. In fact, the water content of swollen cross-linked poly(DMAA) was 90%, whereas that of swollen cross-linked poly(HEMA) was 38%.<sup>16</sup>

The effect of the amount of the cross-linking agent on the water content and on the mechanical strength of the resins was also investigated for the AHMP-DMAA resins by using various initial EBAA concentrations. At the same AHMP concentration

(10 mol %), the water content increased from 80 to 93% when the concentration of EBAA was reduced from 9 to 1 mol % (not shown in Table III), but the resin with 1 mol % cross-linking agent showed a low mechanical strength after swelling. For this reason, the percentage of the cross-linking agent was set at 5 mol %, and the resins obtained using this concentration had a reasonable mechanical strength and a favorable hydrophilicity.

#### Iron(III) Chelating Capacity and Efficiency

The chelation of the AHMP-DMAA resins for iron(III) was studied with the iron(III) citrate solution in phosphate-buffered saline (PBS, pH 7.4) at physiological pH. The yellow AHMP-DMAA copolymers took a red color when added to the

Table II Stability of AHMP-DMAA Resins in Water

Resin Code	AHMP Groups Released at Different Conditions (%)		
	25°C, 24 h	25°C, 48 h	121°C, 15 min
1	—	—	—
2	0.02	0.02	0.08
3	0.14	0.12	0.17
4	0.03	0.02	0.09
5	0.02	0.02	0.15
6	0.03	0.03	0.14

**Table III** Water Content, Ligand Density, Iron(III) Chelating Capacity, and Efficiency of AHMP-DMAA Resins

Resin Code	Water Content (%)	Ligand Density ( $\mu\text{mol/g}$ ) <sup>a</sup>	Iron(III) Chelating Capacity ( $\mu\text{mol/g}$ ) <sup>b</sup>	Chelating Efficiency (%) <sup>c</sup>
1	90	0	0	0
2	88	191	67	105
3	88	343	105	92
4	88	460	146	95
5	87	871	277	95
6	85	1574	414	79

<sup>a</sup> Calculated from initial compositions.

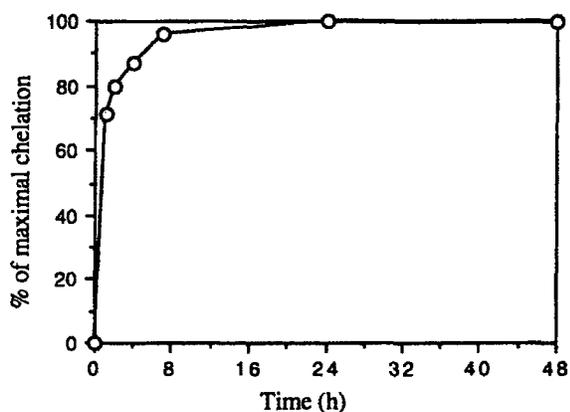
<sup>b</sup> Determined with the batch method.

<sup>c</sup> (Chelating capacity  $\times 3$ /ligand density)  $\times 100\%$ .

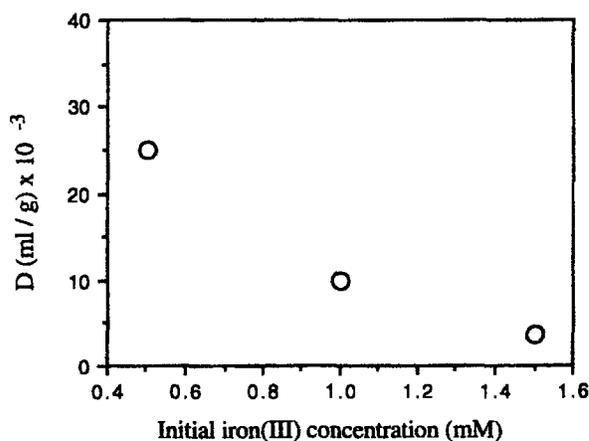
iron(III) solution, indicating the formation of a resin-iron(III) complex.

The iron(III) chelating capacity of the AHMP-DMAA resins was determined, and the results are given in Table III. It was found that all AHMP-containing resins were able to chelate iron(III) and the chelating capacity depended on the initial AHMP concentration. As already shown in Table I, the weight recovery in the preparation of the AHMP-DMAA resins was generally almost quantitative, which means that the resins contained a high amount of DMAA. For that reason, it was difficult to obtain reliable results for the composition of the resins from elemental analysis data. Therefore, considering a nearly quantitative weight recovery for most of the AHMP-DMAA resins, the composition of the cross-linked copolymers was assumed to be equal to the initial composition. Based

on the formation of 3 : 1 AHMP/iron(III) complexes at a physiological pH,<sup>17,21</sup> the chelating efficiency of the resins was calculated from their iron(III) chelating capacity and their ligand (AHMP) density (Table III). The chelating efficiency of the AHMP-DMAA resins was quite high except for resin 6. However, the value of the calculated ligand density for resin 6 is questionable because of the low weight recovery (Table I). In our opinion, the relatively low chelating efficiency for resin 6 was probably due to a low conversion of AHMP monomer (Table I). The chelating efficiency of the AHMP-DMAA resins was much higher than that of AHMP-HEMA beads, which might be explained by their difference in hydrophilicity (water contents), as discussed previously.<sup>16</sup>



**Figure 2** Iron(III) chelation of AHMP-DMAA resin 4 vs. time.



**Figure 3** Effect of initial iron(III) concentration on the distribution ratio ( $D$ ) of iron(III) between resin 5 and an iron(III) citrate solution.

**Table IV Removal of Iron(III) by AHMP–DMAA Resin 4<sup>a</sup>**

Resin		Iron(III) Added			Removal of Iron(III) (%)
Amount (mg)	Capacity ( $\mu\text{mol}$ )	Content (ppm)	Amount ( $\mu\text{mol}$ )	Iron Content Left (ppm) <sup>b</sup>	
100	15	55.85	10	1.1	98.03
200	30	55.85	10	0.09	99.84
500	75	55.85	10	0.05	99.91

<sup>a</sup> The experiments were carried out at 25°C for 24 h.

<sup>b</sup> Determined by AAS.

### Rate of Iron(III) Chelation

Iron (III) chelation was studied with resin 4 at 25°C to determine the rate of iron(III) chelation. As shown in Figure 2, more than 80% of the chelating capacity of the resin was utilized within 2 h and the chelation was almost maximal after 8 h.

### Distribution Ratio of Iron(III)

The effect of the initial iron(III) concentration on the distribution of iron(III) between the resin and the solution was investigated for AHMP–DMAA resin 5 with an iron(III) citrate solution at a physiological pH (Fig. 3). Distribution ratios of iron(III) in the region of 3700–25,000 mL/g were observed, which indicates that the resin was a much stronger chelator than was citrate. The ratio decreased with increasing iron(III) concentration because the ratio of ligand concentration to iron(III) concentration decreased.

### Complete Removal of Iron(III)

Whether complete removal of iron(III) is possible by the iron(III) chelating resins was studied by

treating iron(III) solutions with an excess amount of the resin. The experiments were carried out with AHMP–DMAA resin 4 and the results are given in Table IV. By using various amounts of the resin with capacities of 1.5, 3.0, and 7.5 times the iron(III) amount, respectively, 98.01–99.91% of the iron(III) could be removed. It is evident that the amount of iron(III) left in the solution could be decreased to a very low level by adding excess amounts of the resin. The data suggest that the resin may be useful in systems where complete removal of iron(III) is necessary.

### Selectivity of the Resin

Since the AHMP–DMAA resins have a high affinity for iron(III), it might be possible that the resins will show some selectivity between  $\text{Fe}^{3+}$  and other metal ions. The selectivity of resin 4 for  $\text{Fe}^{3+}$  and some other biologically important metal ions was investigated, and the results are given in Table V.

It can be seen that the affinity of the resin was much higher for iron(III) than for other metal ions tested. The selectivity coefficients observed for iron(III) ( $9.6 \times 10^8$ – $1.4 \times 10^{12}$ ) are the highest values found for iron(III) chelating resins.<sup>22–24</sup>

**Table V Selectivity Coefficients of Resin 4 for Iron(III) to Other Metal Ions<sup>a</sup>**

Metal Ions	$\text{Fe}^{3+}$ ( $\mu\text{mol}$ )		$M^{2+}$ ( $\mu\text{mol}$ )		$K_D$
	On Resin	In Solution	On Resin	In Solution	
$\text{Fe}^{3+}/\text{Cu}^{2+}$	8.10	0.885	0.041	9.25	$9.6 \times 10^8$
$\text{Fe}^{3+}/\text{Zn}^{2+}$	8.47	0.515	0.0061	10.86	$1.4 \times 10^{12}$
$\text{Fe}^{3+}/\text{Mg}^{2+}$	8.63	0.35	0.012	10.53	$4.1 \times 10^{11}$
$\text{Fe}^{3+}/\text{Mn}^{2+}$	8.53	0.45	0.011	10.27	$2.7 \times 10^{11}$
$\text{Fe}^{3+}/\text{Ca}^{2+}$	8.53	0.45	0.075	10.43	$9.6 \times 10^8$

<sup>a</sup> Determined after chelation experiments at pH 5.6 for 24 h at 25°C.

**Table VI Iron(III) Chelating Capacities of Resins After Repeated Use<sup>a</sup>**

No. Times Used (n)	Chelating Capacity ( $\mu\text{mol/g}$ ) <sup>b</sup>	
	Resin 2	Resin 5
1	67 (67)	277 (291)
2	70 (69)	274 (260)
3	66 (67)	255 (243)
4	60 (61)	—

<sup>a</sup> Chelation and regeneration were performed as mentioned in the batch capacity determination and the regeneration of the resins.

<sup>b</sup> Values in parentheses are desorption values.

### Reuse of the Resin

To be viable materials for use in chemical and biomedical systems, the resins must be chemically stable and reusable. As already mentioned, the resins are very stable at 25 and 121°C. In addition, the reusability of the AHMP-DMAA resins was also demonstrated (Table VI). The resins could easily be regenerated with 1M HCl and the chelated iron(III) was released almost quantitatively.

For testing the applicability of the resin in a continuous process, the iron(III) chelating capacity of resin 4 was also measured using a column. Some experiments were done, and it was found that the iron(III) chelating capacity was as high as in the batch process. Thus, the iron(III) chelating capacity of resin 4 was 146  $\mu\text{mol/g}$  in the batch process, whereas using the column a capacity of 144  $\mu\text{mol/g}$  was observed.

### CONCLUSIONS

Iron(III) chelating resins with covalently bound 3-hydroxy-2-methyl-4(1H)pyridinone (HMP) ligands were synthesized by copolymerization of AHMP and DMAA in the presence of a cross-linking agent (EBAA). The AHMP-DMAA resins showed a high hydrophilicity, stability, and iron(III) chelating efficiency. The resins may be very useful for iron(III) removal from various systems because of their high affinity, ability, and selectivity. In addition, the resins could easily be regenerated and reused.

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