

# Interaction of Poly(maleic anhydride-*alt*-acrylic acid) with Transition Metal Cations, Ni<sup>2+</sup>, Cu<sup>2+</sup>, and Cd<sup>2+</sup>: A Study by UV-Vis Spectroscopy and Viscosimetry

Serap Kavlak, Hatice Kaplan Can, Ali Güner

Hacettepe University, Faculty of Science, Chemistry Department, Polymer Chemistry Division, 06532, Beytepe, Ankara, Turkey

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**ABSTRACT:** Interactions between poly(maleic anhydride-*alt*-acrylic acid), [poly(MA-*alt*-AA)] and Cu<sup>2+</sup>, Ni<sup>2+</sup>, and Cd<sup>2+</sup> ions were studied by UV-vis spectroscopy and viscosimetry. Effects of nature and the concentrations of the metal ions on the complex formation were investigated and the formation constants of each complex were determined by the mole-ratio method. UV-vis studies showed that the complex formation tendency increased in the followed order: Cd(II) < Ni(II) < Cu(II). This order was confirmed by the Irving-William series and the Pearson's classification. The

influence of metal ions on the reduced viscosity of poly(MA-*alt*-AA) increased in the following order: Cu(II) < Ni(II) < Cd(II), and this result was explained by the concentration effect. © 2004 Wiley Periodicals, Inc. *J Appl Polym Sci* 92: 2698–2705, 2004

**Key words:** poly(maleic anhydride-*alt*-acrylic acid) [poly(MA-*alt*-AA)]; polyelectrolytes; metal-polymer complexes; UV-vis spectroscopy; viscosimetry

## INTRODUCTION

Polymer-metal complexes are composed of polymeric ligand and metal ions. In solution, polymer-metal complexes form microheterogeneous regions occupied by the polymer backbone, where physicochemical properties differ from those of the bulk solution. Most significant reaction patterns of polymer-metal complexes are attributed to the characteristic nature of these microheterogeneous regions. Polymer-metal complexes show unique characteristics in absorption spectra, coordination structures, stability, redox reactions, catalytic activities, electrochemical reactions, and other areas compared to those of corresponding low molecular metal complexes. They often show a specific catalytic activity based on the large ligand comparable to metal enzymes; they are considered to be analogues of synthetic enzymes. Complexation of polymeric ligand with metal ions and ligand substitution reaction of polymer-metal complexes are used to separate metal ions and/or small molecules.<sup>1</sup>

Complexation of metal ions in solution is an important process in several areas, for example, design of ligands as therapeutic reagents for the treatment of metal intoxication,<sup>2,3</sup> design of antibiotics that owe their antibiotic action to specific metal complexation,<sup>4</sup> design of complexes to act as imaging agents<sup>5</sup> in the

body design of functional groups for chelating ion-exchange materials,<sup>6</sup> and catalysts.<sup>7,8</sup> The scope of applications of metal complexation polymers should increase considerably in the future.

The formation of metal complexes between poly(acrylic acid) and divalent, trivalent, and tetravalent metal ions was previously studied and the stability constants of the complexes were also determined.<sup>9–11</sup> Polychelates, obtained by the addition of an aqueous solution of the copolymer poly(acrylic acid-*co*-maleic acid) to the aqueous solutions of Cu(II), Co(II), and Ni(II) chlorides, were investigated by elemental analysis, magnetic and spectral thermal properties, and electrical conductivities.<sup>12</sup> Recently, crystalline structure and the temperature-dependent electrical conductivity of the poly(maleic anhydride-*alt*-acrylic acid), [poly(MA-*alt*-AA)]-Cu(II), poly(MA-*alt*-AA)-Ni(II), and poly(MA-*alt*-AA)-Cd(II) polychelates at different pH values were determined by X-ray diffraction spectroscopy<sup>13,14</sup> and electrical conductivity measurements<sup>15</sup> in our group.

In this study, the compositions of metal complexes between poly(MA-*alt*-AA) and divalent metal ions (Cu<sup>2+</sup>, Ni<sup>2+</sup>, Cd<sup>2+</sup>) were studied by UV-visible spectroscopy and the stability constants of the complexes were carried out according to the mole-ratio method. The interaction of these transition metal ions with poly(MA-*alt*-AA) in aqueous solutions was also studied by viscosity measurements, and the effects of the type and the concentration of the cation on the interaction mechanisms were investigated.

Correspondence to: A. Güner (agun@hacettepe.edu.tr).

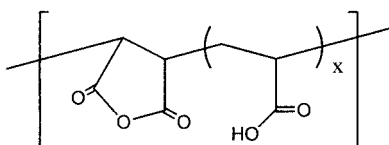
## EXPERIMENTAL

### Materials

Acrylic acid (AA; Fluka Chemie, Buchs, Switzerland) was distilled before use. It had the following characteristics: bp 141.6°C, mp 13.5°C,  $d_4^{20}$  1.0510,  $n_D^{20}$  1.4215. Maleic anhydride (MA; Fluka) was purified before use by recrystallization from anhydrous benzene and by sublimation in vacuum (mp 52.8). Benzoyl peroxide (BP; Fluka) was purified by recrystallizing twice from chloroform solution by methanol and dried under vacuum (mp 106°C). Inorganic salts (CuCl<sub>2</sub>·2H<sub>2</sub>O, CdCl<sub>2</sub>·2H<sub>2</sub>O, NiCl<sub>2</sub>·6H<sub>2</sub>O; BDH, Poole, UK) were used without further purification and other reagents including organic solvents were purified by ordinary methods. The water used for the solutions was deionized and bidistilled.

### Copolymerization

Copolymerization of MA with AA was carried out in *p*-dioxane in the presence of BP (0.1%) as an initiator at 70°C under a nitrogen atmosphere. Copolymer was purified by twice precipitating from dioxane solution with *n*-hexane and by washing with several portions of *n*-hexane, benzene, and diethyl ether, after which it was dried in vacuum at 50°C to a constant weight with almost quantitative yield (~75%). The copolymer poly(MA-*alt*-AA), synthesized by the use of 1:1M ratios of initial monomers, respectively, is represented schematically as



where  $x = 1.12$  (AA unit = 52.83), and had the following characteristics<sup>16</sup>: glass-transition temperature ( $T_g$ ) 111°C and melting temperature ( $T_m$ ) 153°C [by DSC analysis, DuPont V4.1 2000 (Boston, MA), in nitrogen atmosphere at a heating rate of 5°C/min], intrinsic viscosity  $[\eta]_{\text{inv}}$  in *p*-dioxane at 25°C, 1.25 dL/g [by Ubbelohde viscometer (Cannon-Ubbelohde, State College, PA)], acid number (AN) = 878 mg KOH/g (by known nonaqueous titration method), and monomer unit ratio in copolymer ( $m_1 : m_2 = 1 : 1.12$ ).

FTIR spectra [by Nicolet 510 spectrometer (Nicolet Analytical Instruments, Madison, WI)], cm<sup>-1</sup>:  $\nu_{\text{OH}}$  3060 (broad, in -COOH),  $\nu_{\text{CH}_3}$  2950 (as), 2880 (s),  $\nu_{\text{CH}_2}$  2930 (as),  $\nu_{\text{C=O}}$  2870 (s),  $\nu_{\text{COOH}}$  2545 (broad),  $\nu_{\text{C=O}}$  1836 (as) and 1766 (s) (C=O in anhydride unit),  $\nu_{\text{C=O}}$  1585 (as) (in COO-),  $\delta_{\text{CH}_2}$  1478 and 1443 (doublet),  $\delta_{\text{CH}_3}$  1385 and 1357 (doublet),  $\delta_{\text{C-O}}$  1170 (carboxyl),  $\delta_{\text{C-O-C}}$  1035 (in anhydride unit),  $\delta_{\text{OH}}$  943 (out-of-plane OH bending),  $\delta_{\text{CH}}$  886 and 871

(doublet),  $\delta_{\text{CH}_3}$  842 (rock),  $\delta_{\text{CH}_2}$  720 (rock),  $\delta_{\text{CH}}$  645 (in main chain from anhydride unit), and  $\delta_{\text{C-O=C}}$  560 (s) (bend of COOH), 645 (in the main chain from anhydride unit). In the FTIR spectrum of hydrolyzed copolymer, characteristic bands for anhydride units disappeared, and new bands appeared in the field of 1970, 1585, and 1630 cm<sup>-1</sup> relating to -COOH groups, and increased in intensity of 3060 and 2545 cm<sup>-1</sup> broad bands.

### UV-visible spectroscopy

Spectrophotometric measurements were carried out by a Hitachi UV-vis double beam spectrophotometer (Model 100-60; Hitachi, Osaka, Japan). Poly(MA-*alt*-AA)/Cu<sup>2+</sup>, poly(MA-*alt*-AA)/Cd<sup>2+</sup>, and poly(MA-*alt*-AA)/Ni<sup>2+</sup> solutions were kept in shakers after preparation at 37°C for 2 h to become fact polymer-metal complexations.

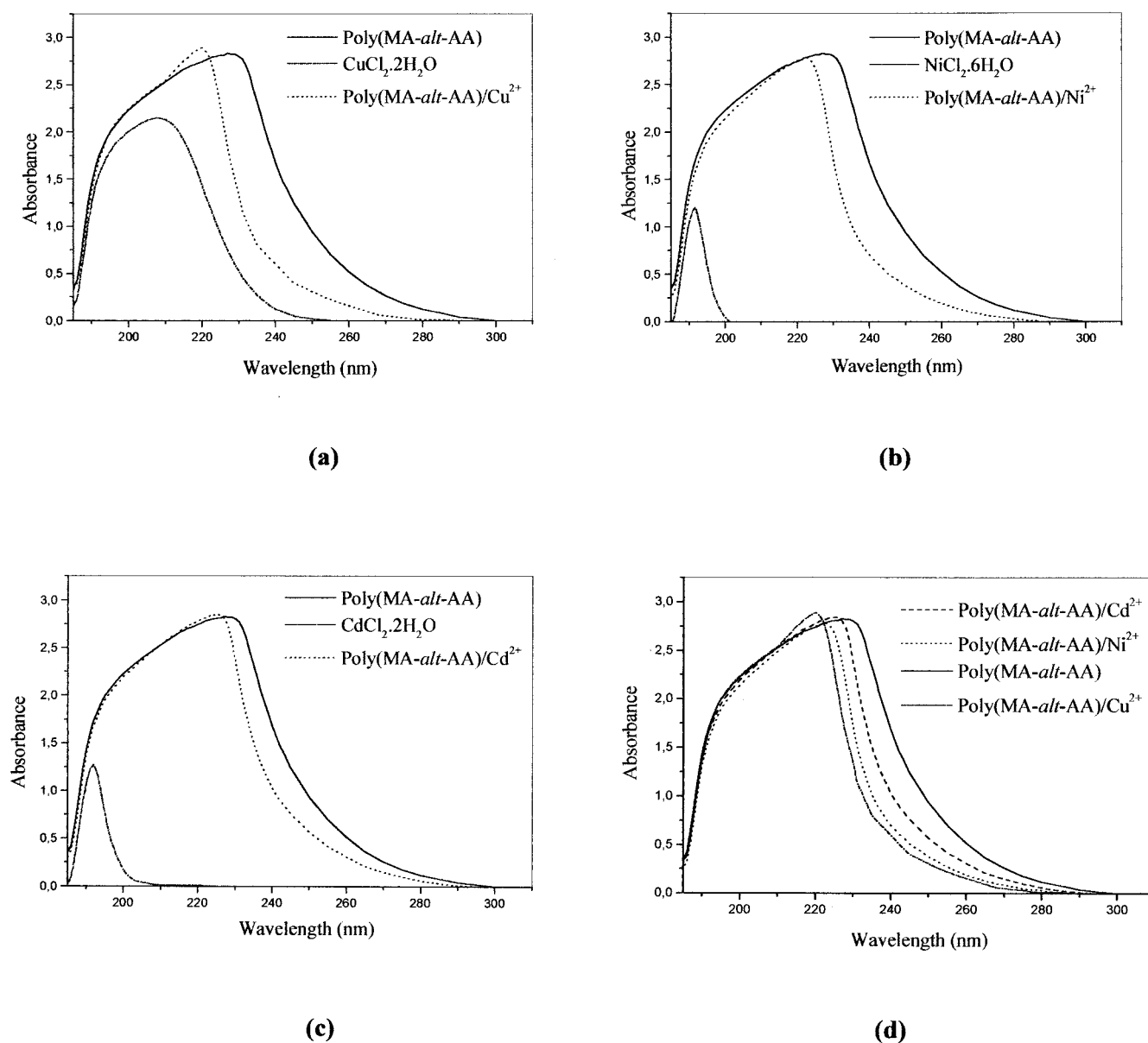
### Viscosity measurements

The viscosimetric behavior of poly(MA-*alt*-AA)/water and poly(MA-*alt*-AA)/Cu<sup>2+</sup>, poly(MA-*alt*-AA)/Cd<sup>2+</sup>, and poly(MA-*alt*-AA)/Ni<sup>2+</sup> solutions were performed at 37°C by using a Ubbelohde-type capillary viscometer. The temperature of the thermostat was controlled within the range of  $\pm 0.1^\circ\text{C}$  and the flow times were measured with a digital accuracy of  $\pm 0.1$  s. The concentration of poly(MA-*alt*-AA) in solutions was changed in the range of 0.016–0.0082 g/dL.

## RESULTS AND DISCUSSION

### Effect of metal ions on the absorption spectrum of poly(MA-*alt*-AA)

Complex formation is made through the complexation of a polymeric ligand with a metal ion by the reaction of a polymer containing a donating group such as -COO<sup>-</sup> with metal ions. Absorption spectra of poly(MA-*alt*-AA), Cu<sup>2+</sup>, Ni<sup>2+</sup>, Cd<sup>2+</sup>, and poly(MA-*alt*-AA)-metal complex solutions are shown in Figure 1 and the  $\lambda_{\text{max}}$  values of each solution are also given in Table I. The  $\lambda_{\text{max}}$  values of Cu<sup>2+</sup>, poly(MA-*alt*-AA), and poly(MA-*alt*-AA)-Cu<sup>2+</sup> complex were obtained at 207.5, 227.0, and 220.0 nm, respectively. The  $\lambda_{\text{max}}$  value of poly(MA-*alt*-AA) represented the  $n \rightarrow \pi^*$  transition of -C=O groups on the copolymer. According to Figure 1(a), when Cu<sup>2+</sup> ions were added to poly(MA-*alt*-AA) solution, the  $\lambda_{\text{max}}$  value of poly(MA-*alt*-AA) shifted to a shorter wavelength (220.0 nm) because the electron density of the -COO<sup>-</sup> group decreased. Figure 1(b) and (c) show absorption spectra of Ni<sup>2+</sup> and Cd<sup>2+</sup> ions, respectively, and the polymer complexes of these ions. The  $\lambda_{\text{max}}$  value of poly(MA-*alt*-AA) also shifted to shorter wavelengths in the pres-



**Figure 1** Absorption spectra of (a) poly(MA-*alt*-AA), Cu<sup>2+</sup>, and poly(MA-*alt*-AA)/Cu<sup>2+</sup>; (b) poly(MA-*alt*-AA), Ni<sup>2+</sup>, and poly(MA-*alt*-AA)/Ni<sup>2+</sup>; (c) poly(MA-*alt*-AA), Cd<sup>2+</sup>, and poly(MA-*alt*-AA)/Cd<sup>2+</sup>; (d) poly(MA-*alt*-AA), poly(MA-*alt*-AA)/Cu<sup>2+</sup>, poly(MA-*alt*-AA)/Ni<sup>2+</sup>, and poly(MA-*alt*-AA)/Cd<sup>2+</sup> complexes.

ence of Ni<sup>2+</sup> (222.8 nm) and Cd<sup>2+</sup> (225.3 nm) ions. This means that complexation occurred between poly(MA-*alt*-AA) and these metal ions. Figure 1(d) shows the

**TABLE I**  
λ<sub>max</sub> (nm) Values of Poly(MA-*alt*-AA), Metal Ions (Me<sup>2+</sup>), and Poly(MA-*alt*-AA)-Me<sup>2+</sup> Complexes

Metal Ion	λ <sub>max</sub> (nm)		
	Poly(MA- <i>alt</i> -AA)	Me <sup>2+</sup>	Poly(MA- <i>alt</i> -AA)-Me <sup>2+</sup> Complex
Cu <sup>2+</sup>	227.0	207.5	220.0
Ni <sup>2+</sup>	227.0	191.6	222.8
Cd <sup>2+</sup>	227.0	192.0	225.3

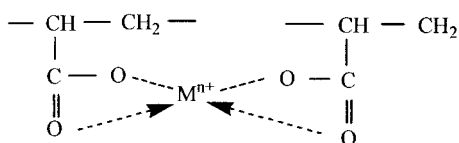
effect of metal ions on the absorption spectra of poly(MA-*alt*-AA). From this figure it is possible to establish the following order for the metal ions to form increasingly stable complex species with poly(MA-*alt*-AA): Cd(II) < Ni(II) < Cu(II), which is confirmed by the order of chelating stability of metal ions.<sup>17</sup>

#### Equilibrium constants

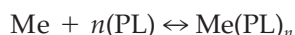
The mole-ratio method<sup>18,19</sup> is the most common technique used for complex-ion studies. To determine the equilibrium constants, a series of solutions were prepared in which the analytical concentration of metal ions was held constant, whereas that of others was

varied. Plots of absorbance versus mole ratio of the reactants were then prepared and two lines with different slopes were obtained (Fig. 2). The two intersected at a mole ratio that corresponded to the combining ratio in the complex. Coordination numbers of all poly(MA-*alt*-AA)-metal complexes appeared as 4, from Figure 2, that is one metal ion is coordinated by four chelating groups.

The chelating process of a metal ion by a polymer may be preceded by long-range attractive electrostatic interactions, and once the metal ion is condensed on the polymer surface, its site is fixed by the polymer ligands. The most accepted mechanism for carboxylic type complexes is shown in the following schematic:



The carboxylic groups can act as mono- and bidentate ligands, depending on the pH values.<sup>20</sup> The complex-forming reaction is expressed as

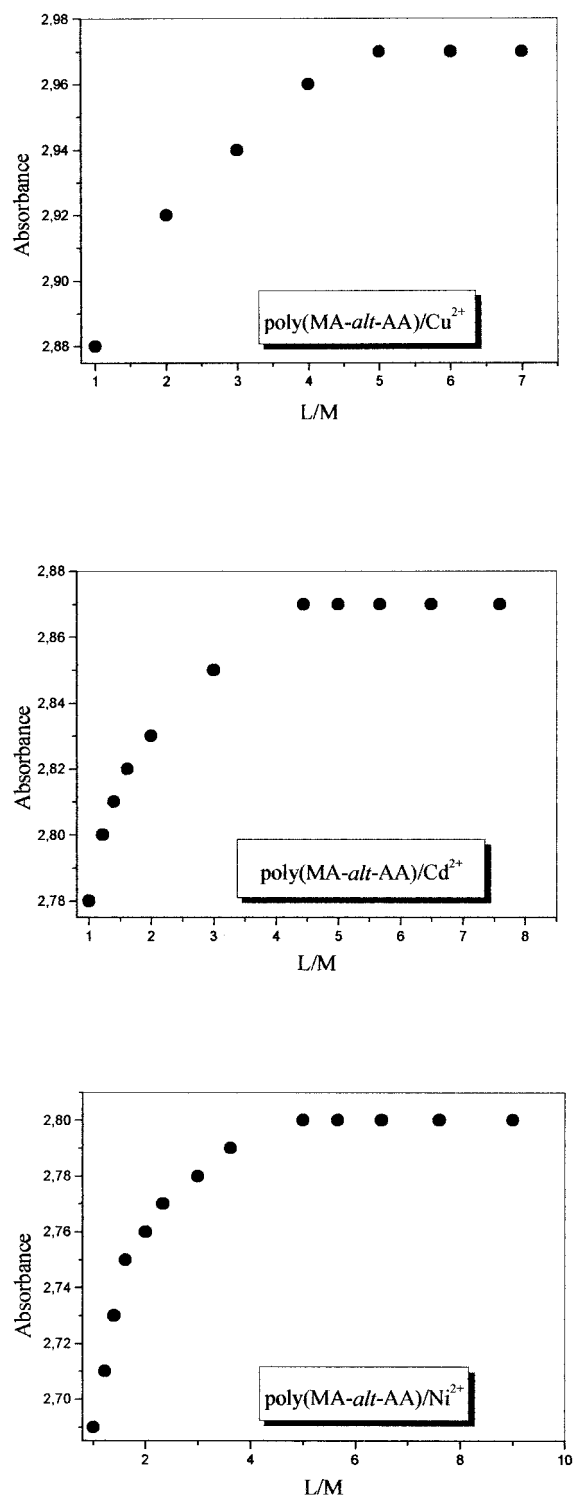


where Me represents metal, PL is the chelating unit of the polymer ligand, and  $n$  is the coordination number of the metallic ion. The formation constant  $K_f$  can be expressed by the following equation<sup>21,22</sup>:

$$K_f = (1 - \alpha) / \alpha(\alpha n C)^n \quad (1)$$

where  $C$  is the concentration of complex and  $\alpha$  is the dissociation constant, which is calculated by the ratio of the absorbance for a defined coordination number to the theoretical absorbance value for maximum coordination. Complex formation constants from eq. (1) for all metal ions are given in Table II.  $K_f$  values of poly(MA-*alt*-AA)-Cu<sup>2+</sup>, poly(MA-*alt*-AA)-Ni<sup>2+</sup>, and poly(MA-*alt*-AA)-Cd<sup>2+</sup> complexes were obtained as  $11.6 \times 10^{16}$ ,  $3.81 \times 10^{16}$ , and  $2.20 \times 10^{16}$ , respectively. The increasing order of the stability constants is given as Cd(II) < Ni(II) < Cu(II). This order was confirmed by the order of the efficiency of metal ions on the absorption spectrum of poly(MA-*alt*-AA) and can be explained by Pearson's classification.<sup>23</sup>

To explain the differences in basic ligand toward various acidic metal ions, Pearson proposed that a hard acid prefers to combine with a hard base, and a soft acid prefers to combine with a soft base. According to this classification Cd<sup>2+</sup> is a soft acid, Ni<sup>2+</sup> and Cu<sup>2+</sup> are in the borderline acid region, and the negatively charged oxygen donor (occurs commonly in the



**Figure 2** Absorption curves of poly(MA-*alt*-AA)/Cu<sup>2+</sup>, poly(MA-*alt*-AA)/Cd<sup>2+</sup>, and poly(MA-*alt*-AA)/Ni<sup>2+</sup> complexes for the determination of the dissociation constants according to the "mole-ratio" method.

form of the carboxylate groups) is a hard base. Because of that, -COO<sup>-</sup> group must complex effectively with Ni<sup>2+</sup> and Cu<sup>2+</sup> ions. In addition, the strength of coordination of negatively charged groups is related

**TABLE II**  
Complex Formation Constants ( $K_f$ ) for the Poly(MA-*alt*-AA)-Metal Complexes

Poly(MA- <i>alt</i> -AA)-Me <sup>2+</sup> Complex	$K_f \times 10^{-16}$
Poly(MA- <i>alt</i> -AA)-Cu <sup>2+</sup>	11.6
Poly(MA- <i>alt</i> -AA)-Ni <sup>2+</sup>	3.81
Poly(MA- <i>alt</i> -AA)-Cd <sup>2+</sup>	2.20

to the acidity (affinity for the OH<sup>-</sup> ion) of the metal ion and proton basicity of the oxygen donor. The selectivity of the polymeric ligand for a more acidic metal ion over a less acidic metal ion will be increased by an increase in the number and basicity of the charged oxygen groups. Steric effects usually lead to a decrease in complex stability of smaller metal ions.<sup>24</sup>

### Polyelectrolyte behavior of poly(MA-*alt*-AA)

The water-soluble poly(MA-*alt*-AA) exhibits typical polyelectrolyte behavior, that is, a decrease in the reduced viscosity with an increase in polyelectrolyte concentration (Fig. 3). This behavior may be explained that, as the polyelectrolyte concentration decreases, the ionization degree increases and the produced ions form an ionic atmosphere greater than the diameter of the polymer coil. The repulsion among the ions increases the rigidity of the chain, expanding the polymeric coil, with a consequent increase of the viscosity.<sup>20</sup>

### Effect of metal ions on the reduced viscosity of poly(MA-*alt*-AA)

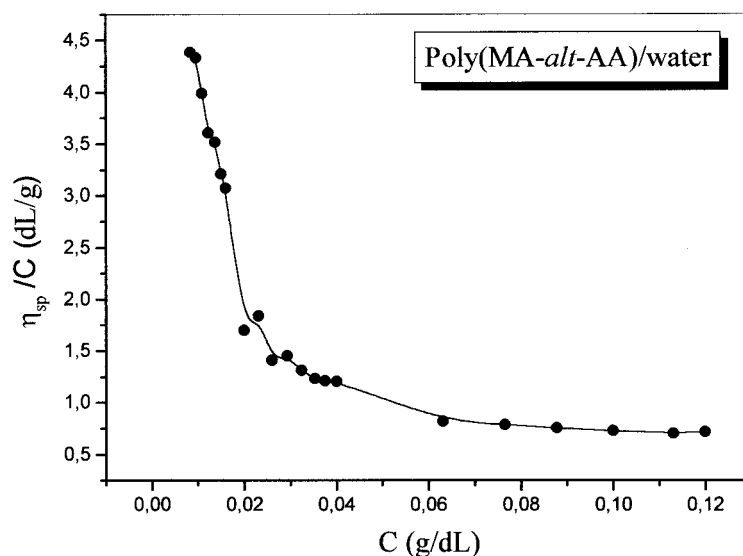
Viscosimetric measurements may help in the understanding of the polymer-metal interaction phenomena

in aqueous solution, including polymer-metal complex formation. The intrinsic viscosity values of water-soluble polymer and the water-soluble polymer-metal complexes may indicate the type of polymer-metal ion interaction. In this sense, for a better understanding of these phenomena, it is necessary to study the behavior, in aqueous solution, of polymers and polymer-metal complexed metal ions.<sup>20</sup>

At a constant concentration of poly(MA-*alt*-AA), an increase in the amount of Cu(II), Ni(II), and Cd(II) ions caused a decrease in the viscosity (Figs. 4, 5, and 6, respectively) because the complex formation causes the macromolecular coil to become tighter by reason of the intrapolymer complex formation.<sup>25,26</sup> In general all polymer-metal complexes studied (with Fe<sup>3+</sup>, Co<sup>2+</sup>, Ni<sup>2+</sup>, Cu<sup>2+</sup>, and Zn<sup>2+</sup>) showed a decrease in the reduced viscosity, compared to the reduced viscosity of the polymers, indicating that the complex formation is preferentially intrachain.<sup>20,25</sup>

According to Figures 7 and 8, at constant copolymer concentration, the Cd<sup>2+</sup> ion showed the strongest relative decrease in reduced viscosity of poly(MA-*alt*-AA). In other words, the Cd<sup>2+</sup> ion was the most effective metal ion in promoting intramolecular association and complex formation. As can be seen in Figures 7 and 8, reduced viscosity of poly(MA-*alt*-AA) decreased in the following order: Cu(II) < Ni(II) < Cd(II).

Viscosimetric observations mainly represent the interaction/complexation between poly(MA-*alt*-AA) and metal ions, depending on the nature of the metal ions in aqueous system. From Pearson's classification, the maximum reduction of the reduced viscosity should be expected in the presence of Cu<sup>2+</sup> ion, as mentioned earlier, although the reduction of the reduced viscosity was higher in the presence of Cd<sup>2+</sup>



**Figure 3** Plot of reduced viscosity versus the concentration of poly(MA-*alt*-AA) in water.

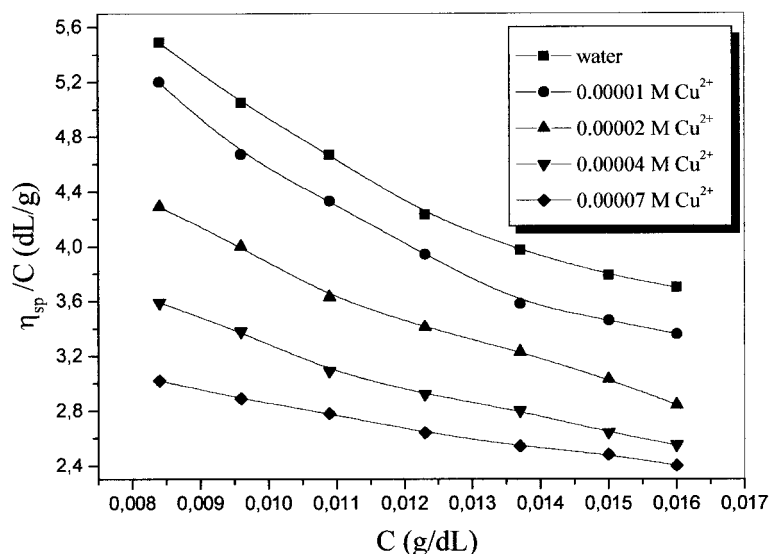


Figure 4 Effect of Cu<sup>2+</sup> ion concentration on the reduced viscosity of poly(MA-*alt*-AA).

ion. This unexpected result can be explained by the concentration effect. In viscosity measurements the concentrations of poly(MA-*alt*-AA) were two times, and of metal ions were 100 times, smaller than concentrations used in the UV studies because, at high concentration, precipitation occurred by the addition of metal ions. Literature data suggested that the formation constants depend not only on the ligand/concentration ratio but also on the ligand concentration. The metal ions were divided into four groups, depending on their effect on the pH decrease of the solution. These groups differ from one another by (1) the speed of water exchange or (2) by the speed of ligand exchange in the coordination sphere of the

metal. The increase of the ligand concentration or of the ligand to metal ratio always led to an increase of the exchange speed of both water and carboxyl groups.<sup>10</sup>

The interactions of monofunctional polyelectrolyte structures, such as poly(acrylic acid), with cations were reported from a number of studies in the literature and different complexation orders were observed. In this study poly(MA-*alt*-AA) has three ligand groups. The linear concentration region of poly(MA-*alt*-AA) and metal ions for the viscosity measurements were also appropriate for electronic spectroscopy. In other words, in this concentration region polyelectrolytic behavior was not clearly observed. Intermolecu-

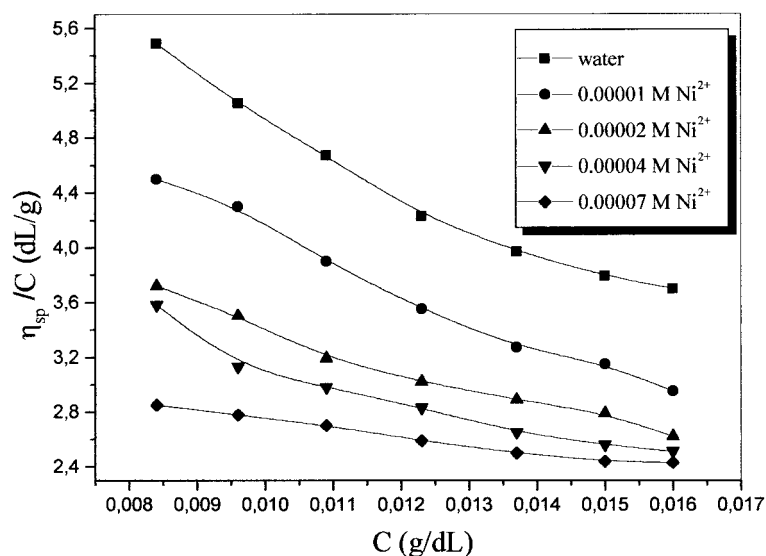


Figure 5 Effect of Ni<sup>2+</sup> ion concentration on the reduced viscosity of poly(MA-*alt*-AA).

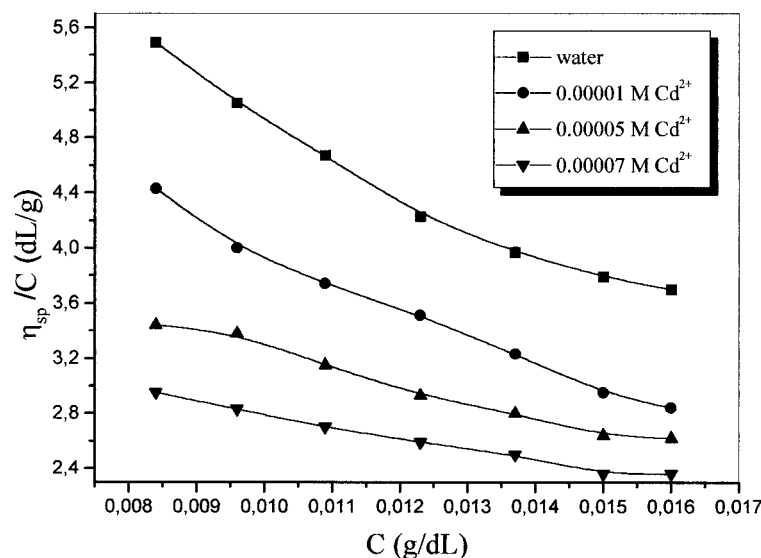


Figure 6 Effect of  $\text{Cd}^{2+}$  ion concentration on the reduced viscosity of poly(MA-*alt*-AA).

lar associations were dominant in this high-concentration region and the ions with small ionic radius were active in these interactions and this behavior was observed in the electronic spectroscopy measurements. However, the dynamism of complexation was determined with the  $\text{Cd}^{2+}$  ion, which has a high ionic radius, in the dilute region because of polyelectrolytic behavior.

### CONCLUSIONS

By the results presented in this article new lines of evidence of the effect of metal ions ( $\text{Cu}^{2+}$ ,  $\text{Ni}^{2+}$ , and  $\text{Cd}^{2+}$ ) on the complexation dynamism of poly(MA-*alt*-

AA)-metal complexes were explained. Complexation occurred between polymer and metal ions and it is possible to establish the following order for the metal ions to form increasingly stable complex species with poly(MA-*alt*-AA):  $\text{Cd(II)} < \text{Ni(II)} < \text{Cu(II)}$ , which was confirmed by the order of chelating stability of metal ions according to the Irving-Williams series. The increasing order of stability constants (by the mole-ratio method) of the polymer-metal complexes also followed the same order. Interactions of poly(MA-*alt*-AA)-metal ions were also investigated by viscosimetry. Poly(MA-*alt*-AA) and its metal complexes behaved in the electrostatic manner of polyelectrolytes in aqueous solutions. Reduced viscosity of poly(MA-

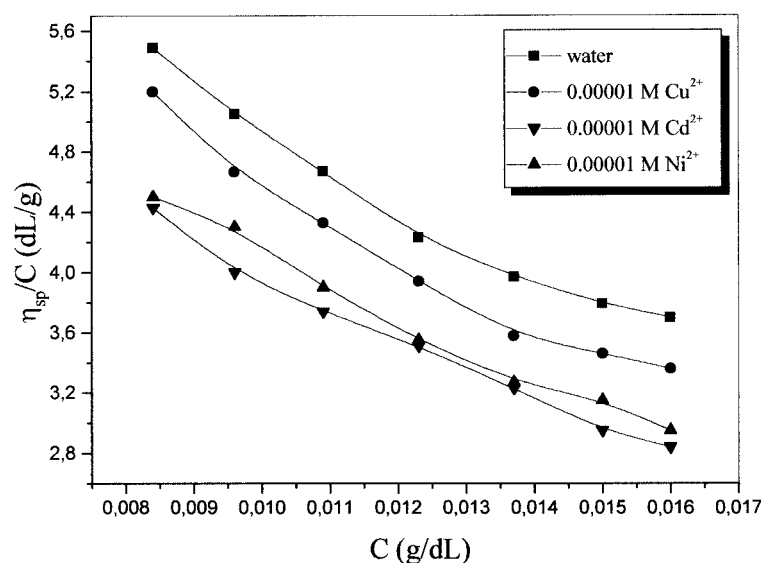
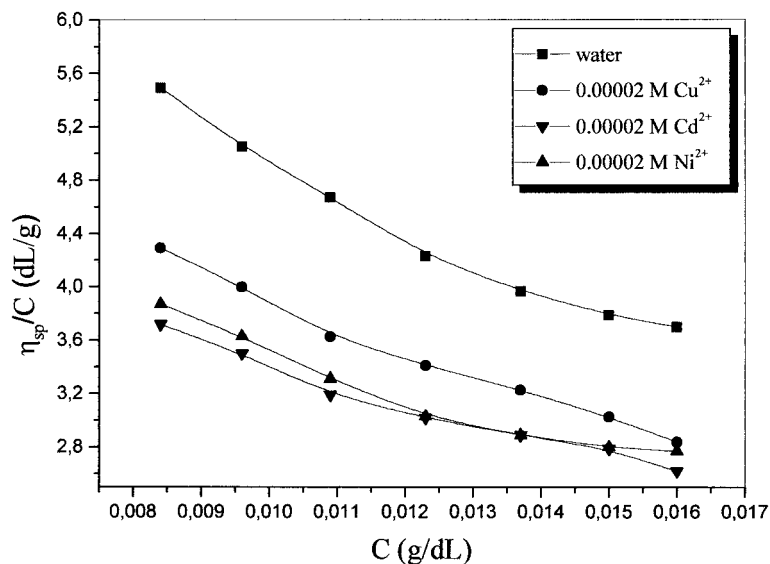


Figure 7 Variation of the reduced viscosity of poly(MA-*alt*-AA) in the presence of  $1 \times 10^{-5}$  M  $\text{Cu}^{2+}$ ,  $\text{Ni}^{2+}$ , and  $\text{Cd}^{2+}$  ions.



**Figure 8** Variation of the reduced viscosity of poly(MA-*alt*-AA) in the presence of  $2 \times 10^{-5}$  M Cu<sup>2+</sup>, Ni<sup>2+</sup>, and Cd<sup>2+</sup> ions.

*alt*-AA) in the presence of metal ions decreased in the following order: Cu(II) < Ni(II) < Cd(II), and this order was explained by the concentration effects.

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