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Evaluation of Selective Ion Exchange for Nickel and Cadmium Uptake from the Rinsewaters of a Plating Shop

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ABSTRACT

The use of selective ion exchange to recover metals from the rinsewaters of various plating baths has been studied. Two chelating resins possessing aminophosphonate or iminodiacetic functionalities were compared in nickel- and cadmium-bearing rinsewaters. In addition, an inorganic ion exchanger based on sodium titanate was studied against nickel containing rinsewaters. Breakthrough capacities of 5% and decontamination factors were calculated from column tests. The aminophosphonate resin proved to be the best material for both nickel and cadmium recovery. Ammonium nitrate was observed to enhance the adsorption performance of the aminophosphonate resin.

INTRODUCTION

The plating process involves several rinsing steps. As a consequence, metal ions are transferred from a plating bath and accumulate in rinsewater. A typical plating bath contains up to several hundred grams per liter of dissolved metals. The concentration of metal ions in the rinsewater is less than 0.5% of the original plating bath concentration.

Nickel is among the most common plating metals, whereas cadmium is predominantly used by the aircraft industry for flight safety reasons. Recycling of rinsewater and zero discharge are considered as the ideal solution for the wastewater problem. Technical solutions, however, have been so expensive

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that a closed loop is possible only in special cases. Recovering metals from rinsewater with automated selective ion exchange (SIX) might provide a cost-effective solution to recycling problem of metals and rinsewater. This is valid for the case of a plating shop with a large work load and high water consumption per day. A SIX-based process combined with deionization may provide savings in operational costs when compared with water recycling by membrane filtration. For example, Hewlett-Packard (1) designed a process for the recovery of nickel. Meyers (2) wrote some practical information about the behavior of chelating resins and their applicability to plating rinsewaters.

A possible use of the SIX process for the maintenance of pretreatment baths (etching and coating removal) has been studied (3). The results of the investigations showed that the SIX process was impractical for the solutions tested since the resin could not be adequately regenerated.

The objective of the current study (funded by the European Commission) is to identify ion-exchange selectivity for cadmium or nickel instead of for other cationic impurities and competitive ligands present in plating shop rinsewaters.

EXPERIMENTAL

An aminophosphonate resin (Purolite S950) and iminodiacetic resin (Purolite S930) were tested in nickel- and cadmium-bearing rinsewaters of plating/preplating baths. Also, an inorganic ion exchanger (commercial name SrTreat) was tested in nickel-bearing rinsewaters. The selective ion-exchange materials were screened from a wide range of commercial chelating ion exchangers. Lehto et al. (4, 5) carried out ion-exchange studies in synthetic simulant solutions of rinsewaters and concluded that the aminophosphonate resin is effective for purifying rinsewater solutions. Laboratory- and bench-scale column experiments were performed. The laboratory-scale experiments were performed against simple rinsewater and a combination of four different nickel-bearing rinsewaters was tested on the bench scale.

Resins and Materials Used

The organic ion exchangers Purolite S930 and S950 are chelating resins with a macroporous polystyrene matrix crosslinked with divinylbenzene (see Table 1). The resins were received from Purolite Int. The inorganic ion exchanger SrTreat is crystallized sodium titanate (manufactured by Selion Oy, Finland). It was received from the Laboratory of Radiochemistry of Helsinki University.

Test Solutions

The test solutions used in all experiments were prepared from plating bath solutions; these were diluted with deionized distilled water (see Table 2 for de-

TABLE 1
Characteristic Data of Tested Purolite Resins (6)

Resin	Purolite S930	Purolite S950
Functional group	Iminodiacetic	Aminomethylene phosphate
Capacity (Na+ form), eq·L^{-1}	0.94	1.2
Operating pH range	2–11	2–11
Particle size, mm	0.3–1.2	0.3–1.2
Moisture retention, %	45–50	60–65

tails). In the following text the numbers as defined in Table 2 will refer to the test solutions.

The feed pH of the nickel-bearing test solutions was adjusted to between 4.3 and 4.5 with 1 M hydrochloric acid or 1 M sodium hydroxide. pH adjustment of the test solution from the cadmium cyanide bath was not done due to the

TABLE 2
Test Solutions and Metal Concentrations[a,b]

Test solution no.	Rinse water type	Nickel concentration (mg·L^{-1})	Cadmium concentration (mg·L^{-1})
1	Nickel coating removal bath (HNO_3 bath)	69.2	—
2	Complexing nickel coating removal bath (contains ethylenediamine)	82.3	—
3	Nickel chloride + nickel sulfamate bath	50.8	—
4	Electroless nickel bath	49.4	—
5	Combination of nickel-bearing solutions	46.8	—
6	Cadmium cyanide bath	—	111.8
7	Cadmium coating removal bath (ammonium nitrate bath)	3.2	123.9
8	Cadmium coating removal bath	—	798.1

[a] Additional notes concerning the pretreatment baths: *Nickel coating removal bath* is a concentrated nitric acid solution; the bath contains mainly nickel and small amounts of iron and aluminum. *Complexing nickel coating removal bath* contains nitrobenzol acid, ammonia, ethylenediamine (EDA), and dithiocarbamat compound; this bath is alkaline. *Cadmium coating removal bath* contains ammonium nitrate; this bath is alkaline.

[b] Additional notes concerning the plating baths: *Nickel chloride bath* contains nickel chloride and sulfuric acid. *Nickel sulfamate bath* contains nickel sulfamate, boric acid, and surface-active agents. The main components of the *Electroless nickel bath* are sodium hypophospite and nickel sulfamate; this solution is acidic. *Cadmium cyanide bath* contains cadmium, sodium cyanide, sodium hydroxide, and sodium carbonate.

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danger of generating hydrogen cyanide; its feed pH was thus 10.8. The pH of the test solution from the cadmium coatings stripping bath was 7.8 (the pH was not adjusted).

Screening Experiments

The objective of the screening experiments was to find plating and rinse-water baths where selective ion exchange may have a potential application for metal recovery. Ten milliliters of wet resin (Na^+ form) or dry SrTreat ion-exchange material was packed in Pharmacia Biotech XK 16 columns (column diameter 1.6 cm, height 20.0 cm). The column had two distributors that could be used to adjust the bed size within a 1–24 mL range. A schematic drawing of the experimental setup is presented in Fig. 1. Test solutions were passed down through the column at ambient temperature, and the breakthrough profiles were recorded. The flow rate was maintained between 9 and 11 bed volumes per hour ($BV \cdot h^{-1}$). Following metal breakthrough, the ion exchangers were washed with 5 BV of deionized distilled water, and the sorbent bed was regenerated with 1 M hydrochloric acid (the eluant was pumped up through the column in the countercurrent mode).

Bench-Scale Experiments

Bench-scale column breakthrough experiments were carried out with test solution 5. The idea behind test solution 5 was to try and treat all the nickel-bearing rinsewaters together. According to the screening experiments, possible solutions were test solutions 1, 3, and 4. The solutions were mixed together in a ratio of 1:10:10, respectively (based on rinsewater flow of each bath at the plating shop). The Purolite S950 aminophosphonate resin (Na^+ form) was tested against the solution. This resin had shown the best selectivity, decon-

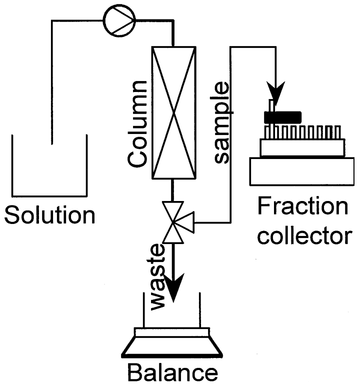

FIG. 1 Schematic drawing of the test equipment used in the laboratory scale tests.

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tamination factor, and regeneration characteristics of all the sorbents studied. The dimensions of the bench-scale column were: diameter 6.8 cm, height 40 cm and bed volume 1.25 L. The column had no flow distributors, a solution flow rate of 15.0 $BV\cdot h^{-1}$ was used (the solution was pumped downflow through the column), and the feed pH was adjusted to 3.9. Experiments were performed at ambient temperature.

Further Experiments

To enhance the performance of the aminophosphonate resin in breaking up the ethylenediamine (EDA)–nickel complexes found in test solution 2, preconditioning of the ion exchanger (pH adjustment of the resin bed to the feed pH) was done. The following procedure was carried out: 10 mL of H^+ form resin was measured into an Erlenmeyer flask, small doses of 1 M sodium hydroxide were added, and the pH was measured to obtain a titration curve. Wet aminophosphonate resin (Na^+ form) was loaded into a minicolumn (10 mL nominal capacity) and converted to the H^+ form with an excess of hydrochloric acid followed by washing with deionized distilled water until the solution pH remained unchanged. The preconditioning was continued by washing the resin with a slight excess of sodium hydroxide based on the titration curve achieved in the batch treatment, so that the sorbent bed reached the required pH.

Analysis

Metal determination was carried out using a Perkin-Elmer atomic absorption spectrophotometer in either a flame or graphite furnace (Zeeman 5000) mode. The solution pH was recorded using a Knick pH meter.

To define the resin operational capacity, 5% breakthrough capacity and units of equivalents per liter ($eq\cdot L^{-1}$) were chosen. Decontamination factors (DF) were calculated to represent the purification efficiency before breakthrough (see Eq. 1).

$$\mathrm{DF} = \frac{\text{Metal concentration}_{\text{feed}}}{\text{Metal concentration}_{\text{effluent}}} \qquad (1)$$

RESULTS AND DISCUSSION

Nickel-Bearing Solutions

SrTreat showed the most promising 5% breakthrough capacities in all the nickel-bearing solutions. The aminophosphonate resin performed better than the iminodiacetic resin (see Fig. 2). A comparison of the 5% breakthrough capacities of S950 and SrTreat shows that the difference between the sorbent

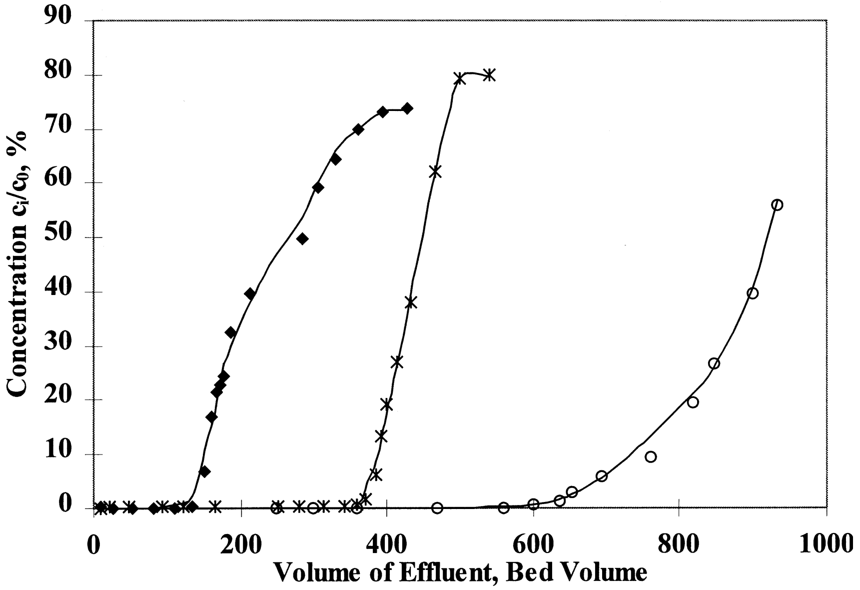


FIG. 2 Nickel breakthrough curves for Purolite S930 (◆), Purolite S950 (∗), and inorganic ion exchanger SrTreat (○) in combined nickel sulfamate and nickel chloride plating bath rinsewater. Feed rate, 10.5 BV/h; feed pH, 3.9.

performance is solution dependent (see Table 3). SrTreat shows an excellent nickel sorption capacity against test solution 3, but in solutions that contain complex forming or oxidizing agents (test solutions 1 and 4) the difference between SrTreat and S950 is small. None of the tested sorbents removed nickel from test solution 2 at feed pH 4.5.

TABLE 3
5% Breakthrough Capacities and Decontamination Factors for Purolite S930 and S950 Resins and Inorganic Ion-Exchanger SrTreat When Treating Rinsewaters after Nickel-Bearing Plating/Pretreatment Baths

Test solution no.	Capacity at 5% breakthrough ($eq \cdot L^{-1}$)			Decontamination factor (—)		
	S930	S950	SrTreat	S930	S950	SrTreat
1	0.1	0.2	0.2	920	380	1,100
2	0.0	0.0	0.0	1	1	1
3	0.3	0.7	1.2	930	440	2,770
4	0.5	0.5	0.6	1,650	210	2,100
5	—	0.7	—	—	5,500	—
6	0.0	0.0	—	1	1	—
7	0.5	0.8	—	4,880	5,990	—
8	—	1.4	—	—	51,400	—

There is a significant difference in favor of S950 and S930 when the regenerabilities of the sorbents are compared (see Fig. 3). The aminophosphonate resin is regenerated completely after six bed volumes of eluant (1 M HCl) whereas SrTreat has a poor regeneration profile. It also showed weak resistance to mechanical stress. From an economic point of view, Bolto and Pawlowski (7), for example, stated that it is crucial to obtain as high a metal concentration in the regenerant as possible. This can be achieved by utilizing concentrated acid solutions and employing the appropriate arrangement of ion-exchange unit operations.

Resin S950 was chosen for the bench-scale experiments because it performed best although resin S930 has a similar 5% breakthrough capacity and its decontamination factor was far better in comparison with S950 for test solution 4 (see Table 3). Low decontamination factors in the screening tests may be due to the small height/diameter ratio of the resin bed and the consequent short retention times. The bench scale experiments showed that it is possible to combine almost all the nickel-bearing rinsewaters generated from various plating baths and treat them as a simple effluent solution without loss of capacity of the ion exchanger. Compared with the screening tests, the decontamination factors increased due to the larger sorbent bed height and better flow characteristics of the column.

Metal concentration in rinsewater is a function of numerous factors related to the process type, shape of parts processed, production equipment, bath concentration, bath temperature, bath viscosity, part orientation, and the length of

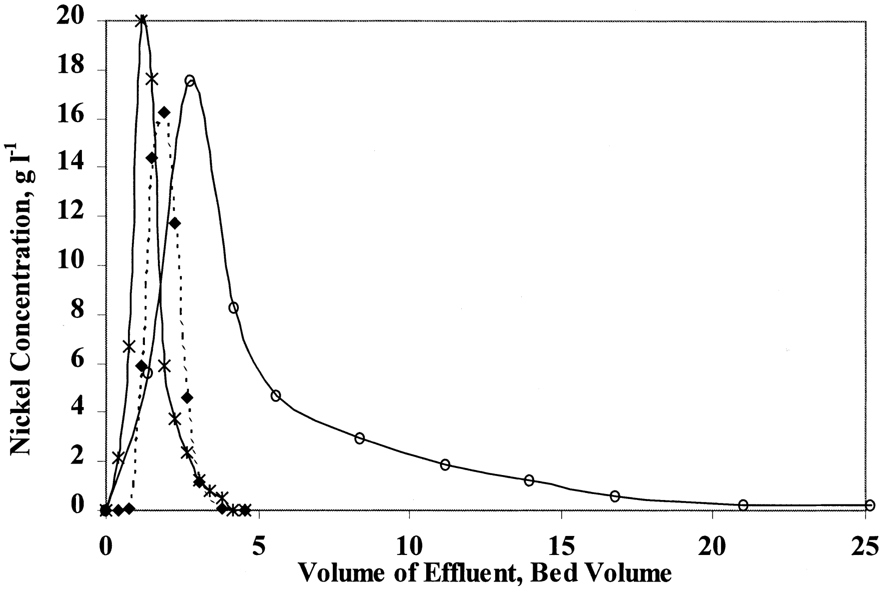


FIG. 3 Elution of nickel from Purolite S950 (◆), Purolite S930 (*), and SrTreat (○) with 1 M HCl. Feed rate, 10.0 BV/h.

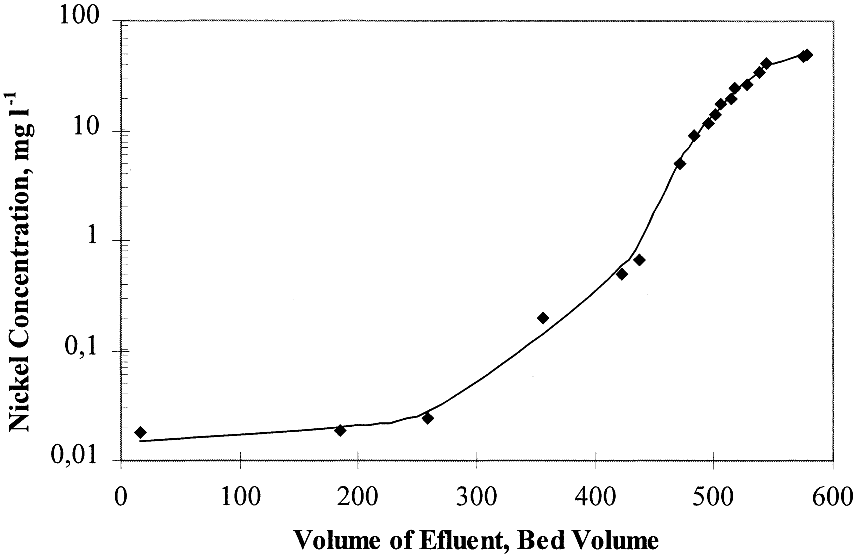


FIG. 4 Nickel concentration in effluent for Purolite S950 in combined nickel-bearing rinsewater of plating shop (test solution 5, bench scale column). Feed rate, 13.0 BV/h; feed pH, 3.9.

drain time provided. Requirements for the purity of the last rinsewater vary depending on the desired quality of the product. As can be seen in Fig. 4, the nickel concentration can be reduced below 0.03 mg·L^{-1} with selective ion exchange. This level fulfills most rinsewater purity and effluent discharge requirements.

Cadmium-Bearing Solutions

The cadmium-coating removal bath contains ammonium nitrate which forms ammonium complexes with cadmium. Some of the coatings removed also include small amounts of nickel. Aminophosphonate easily breaks down the ammonium complexes and removes the cadmium by chelation. According to Purolite's specifications, the S950 resin capacity is 1.2 eq· L^{-1} (Ca^{2+}). For test solution 7 (cadmium 1.1 mmol·L^{-1}, ammonium nitrate 7.0 mmol·L^{-1}) the total capacity of the resin calculated at 50% breakthrough was near the quoted ion-exchange capacity of the resin (see Table 3). A high service capacity was not achieved with iminodiacetic resin S930 (see Fig. 5 and Tables 1 and 3). Ammonium nitrate was observed to enhance the performance of the aminophosphonate resin. When cadmium was stripped straight from the bath (cadmium 7.1 mmol·L^{-1} ammonium nitrate 1.5 mol·L^{-1}), breakthrough capacity was increased by 75%. Also, the purity of the effluent was better. The ion-exchange behavior is not clear, but indications are that cadmium can be

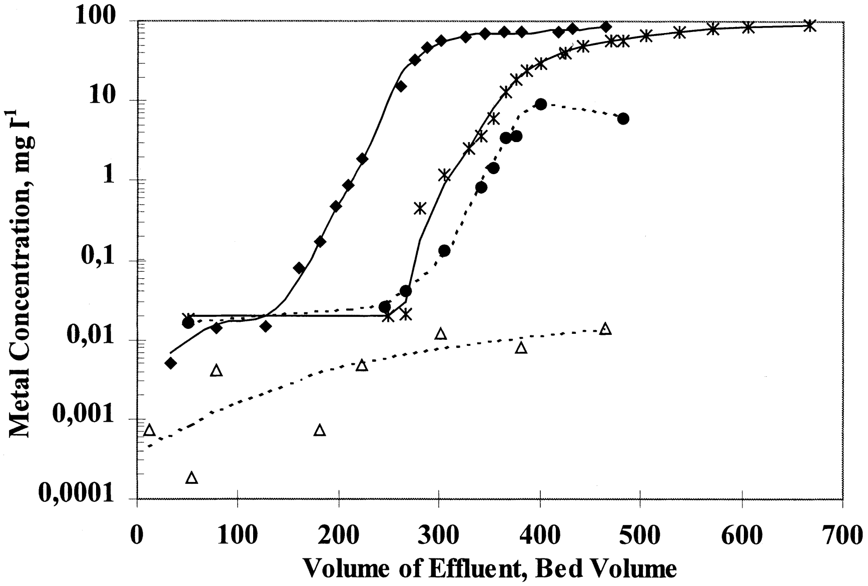


FIG. 5 Cadmium and nickel concentrations in effluent for Purolite S950 and S930 resins when treating rinsewater of cadmium coating removal bath (ammonium nitrate bath). (◆) Purolite S930, Cd; (∗) Purolite S950, Cd; (△) Purolite S930, Ni; (•) Purolite S950, Ni.

removed more efficiently from a concentrated ammonium nitrate solution than from a dilute one. Reedijk et al. (8) made spectral studies on aminophosphonate resin complexes with transition metals. They used metal chloride and nitrate salts in their experiments and concluded that these anions do not play a significant role in ion exchange as far as the capacity of the resin is concerned. This and the results achieved in this study indicate that the ammonium ion alone or together with the nitrate ion might enhance the adsorption of cadmium on aminophosphonate resin.

The aminophosphonate resin prefers cadmium to nickel, whereas the selectivity of the iminodiacetic resin (7) is the reverse. Nickel breakthrough occurs with the S950 resin before cadmium breakthrough, whereas nickel breakthrough is not observed in the case of the S930 resin. It may be seen from Fig. 5 that the nickel concentration rises well above the initial feed solution value (3.2 mg·L^{-1}). This indicates nickel desorption and cadmium sorption in the upper layers of the S950 column. This kind of behavior is not observed with the S930 resin. The regeneration of the resins supports the selectivity sequence discussed above (see Fig. 6). Separation of nickel and cadmium could be achieved by using both iminodiacetic and aminophosphonate resins.

The rinse water of the cadmium cyanide bath was not successfully treated with the resins tested since the cadmium cyanide complex can not be com-

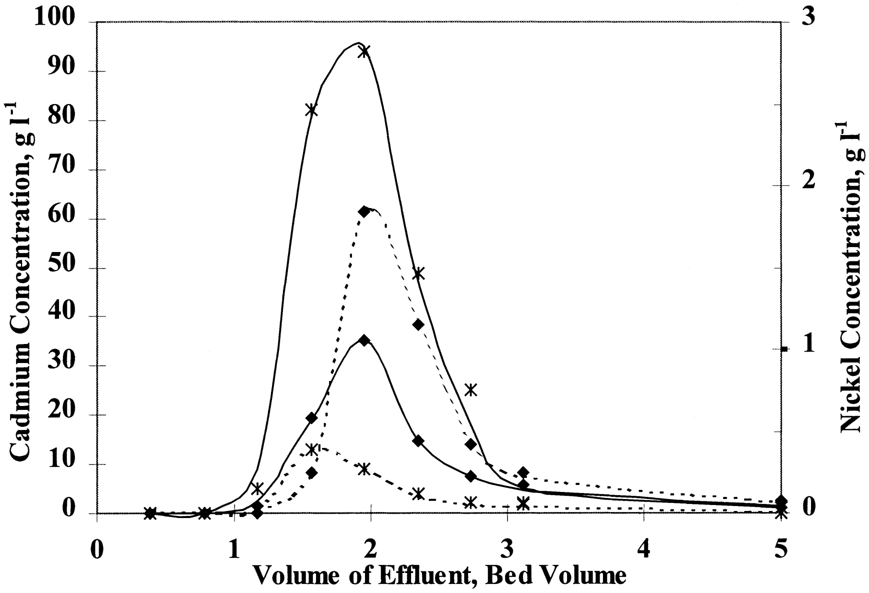


FIG. 6 Elution of cadmium (left ordinate, solid line) and nickel (right ordinate, dashed line) from Purolite S930 (◆) and S950 (*) resins with 1 M HCl. Feed rate, 10.0 BV/h.

peted for by the resins. The complexes must be broken down before SIX treatment.

Further Experiments

Nickel removal from the complexing nickel coating stripping bath (test solution 2) with SIX proved to be rather difficult. At a feed pH of 4.5 and a flow rate of 13.0 $BV \cdot h^{-1}$, none of the sorbents tested removed nickel from the test solution. In these screening tests the height–diameter ratio of the resin bed was only 2.5. Due to the slow kinetics displayed by the complexing resins and the presence of competing complex-forming agents in the solution (7 $mmol \cdot L^{-1}$ EDA), further tests were made with a reduced flow rate of 3.5 $BV \cdot h^{-1}$, a higher height–diameter ratio of 29, and a preconditioned resin bed. The concentration of nitrobenzol acid was decreased to prevent resin fouling. By adjusting the solution pH to 2.5, the nitrobenzol acid precipitated and it was removed by filtration. The permeate was treated with activated carbon. Column experiments were then performed at three different feed pH values: 4.0, 5.0, and 7.0. The resin was preconditioned according to the procedure mentioned above to match the pH of the test solution being treated.

The pH dependence of EDA–nickel complexes is presented in Fig. 7 when the EDA concentration is about 5 times higher than the nickel concentration.

Calculations for different species of nickel were made with the species distribution program SPE developed by Motekaitis (9) using tabulated stability constants of hydrolysis species (10).

Figure 7 shows that solution pH has a significant effect on the speciation of the EDA–nickel complex. Reduced feed pH improves the ability of the aminophosphonate resin to break down the complexes. In SIX the pH of the resin bed plays an important role when treating EDA-containing solution. When the resin bed is not conditioned, the bed is alkaline (pH > 10). In such cases the EDA coordinates around the nickel, thus preventing its adsorption on the resin. At feed pH 5.0 the unconditioned S950 resin leaks nickel at greater than 40% and breakthrough occurs almost instantaneously (see Fig. 8a). When the pH of the exchanger approaches the feed pH, nickel leakage reduces to around 15% of the influent value. Preconditioned resin leaks less than 0.5% of the feed concentration of nickel (see Fig. 8b).

The optimum feed pH for treating the EDA solution can be seen from Fig. 9 and Table 4. Reduced leakage of nickel is observed at feed pH 5 (i.e., a higher decontamination factor is obtained) in comparison with nickel breakthrough at feed pH values of 4.0 and 7.0. Reduction in the decontamination factor and sorption capacity of the resin bed at a pH lower than 5 might result from preconditioning the resin to too low a pH when the resin is not com-

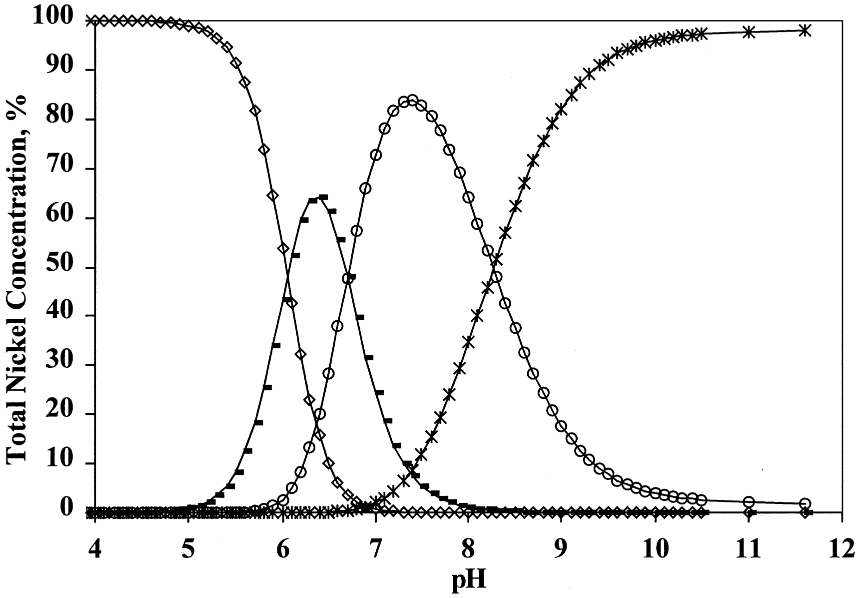
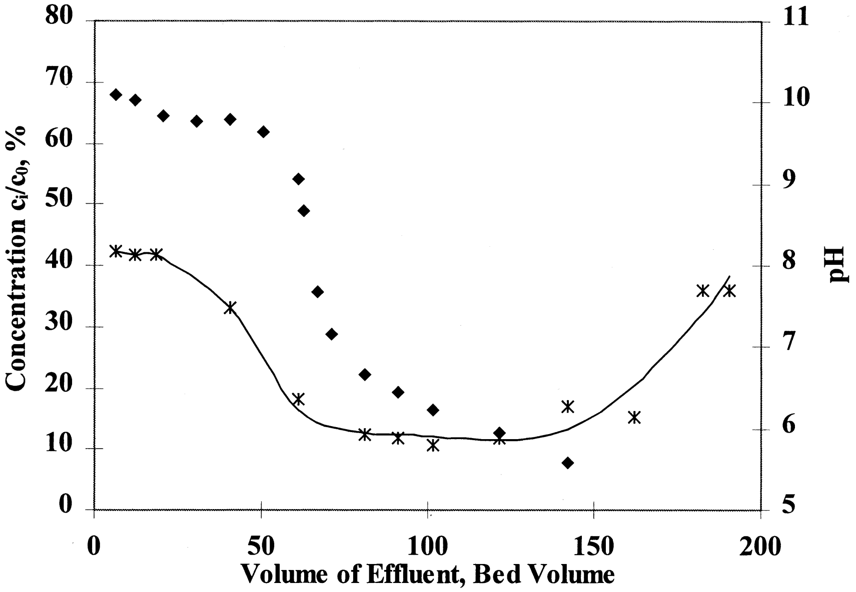


FIG. 7 pH dependency of the ethylenediamine coordination around nickel when EDA concentration is 7 mmol·I^{-1} and nickel concentration is 1.5 mmol·I^{-1}: (◊) Ionic Ni, (■) Ni-EDA, (○) Ni-2EDA, (∗) Ni-3EDA.

a)



b)

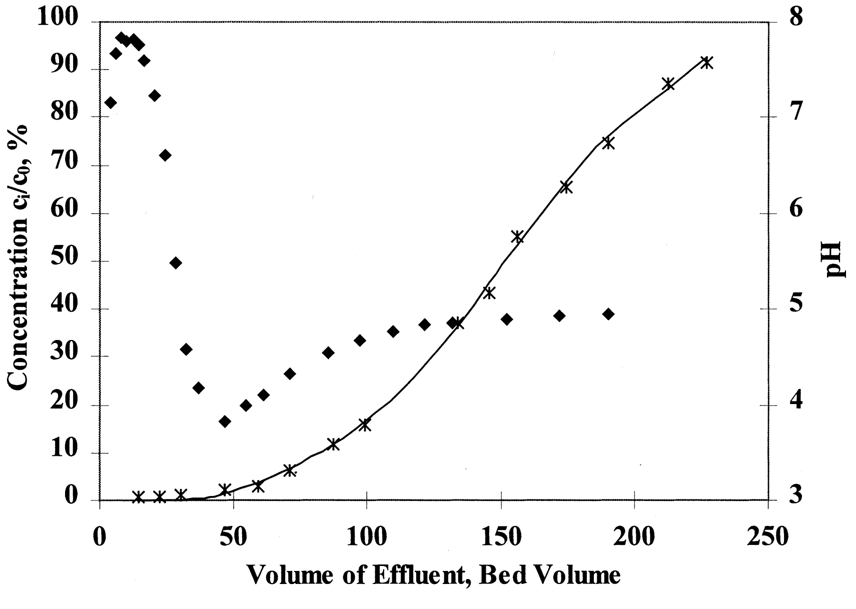


FIG. 8 Breakthrough curves and effluent pH for (a) nonconditioned and (b) preconditioned Purolite S950 resin in a solution which contains ethylenediamine. Feed rate, 7.5 BV/h; feed pH, 5.0. (*) Ni, (◆) pH.

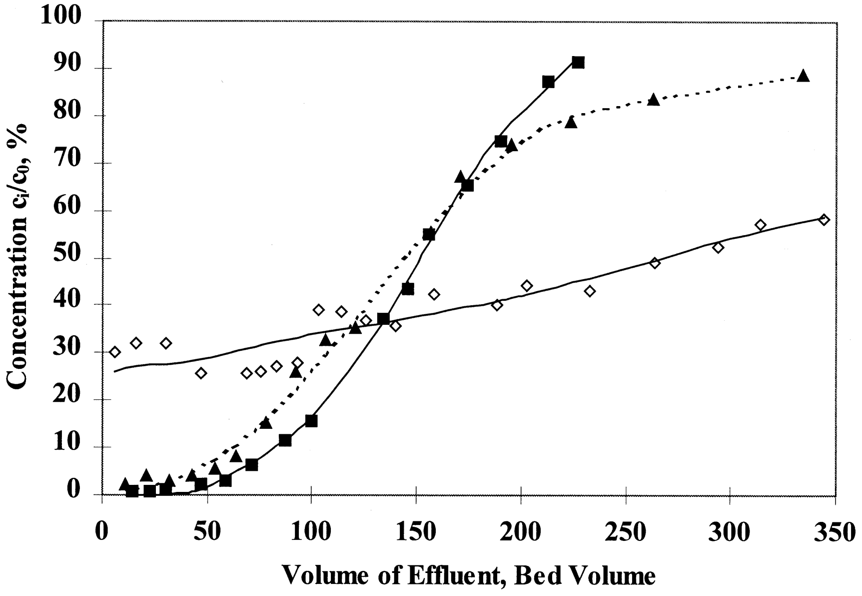


FIG. 9 Nickel breakthrough curves for preconditioned Purolite S950 resin in various feed pH's in a solution which contains ethylenediamine. Feed rate, 7.5 BV/h. Feed pH: (▲) 4.0, (■) 5.0, (◊) 7.0.

pletely in sodium form. This results in a decrease in the selectivity of the resin toward nickel.

Difficulties in conditioning the SIX resin bed to the optimal pH, the fact that conventional ion exchangers have better capacities, and also that EDA complexes can be broken down by pH adjustment makes the use of SIX in this kind of application unfeasible. However, the presence of EDA can influence the relative selectivity of the two metal ions (11) for the resin. Separation of various metals can thus be achieved with selective ion exchange and EDA addition.

TABLE 4
5% Breakthrough Capacities and Decontamination Factors for Preconditioned and Nonconditioned Purolite S950 Resin at Three Different Feed pH Values When Treating Rinsewater after Complexing Nickel Coating Removal Bath

Feed pH	Capacity at 5% breakthrough ($eq \cdot L^{-1}$)		Decontamination factor (—)	
	Preconditioned	Nonconditioned	Preconditioned	Nonconditioned
4.0	0.2	—	32	4
5.0	0.2	—	120	4
7.0	—	—	3	3

CONCLUSIONS

In this study selective ion exchange was examined for the removal of nickel and cadmium from rinsewaters generated in a plating shop. Two complex-forming organic resins and one inorganic-ion exchange material were tested: aminophosphonate resin Purolite S950, iminodiacetic resin Purolite S930, and a sodium-titanate-based ion exchanger SrTreat.

The best 5% breakthrough capacities and decontamination factors for nickel-bearing solutions which did not contain complex forming agents was achieved with SrTreat. However, the regeneration and durability of this exchanger was not good. The most promising overall performance in treating nickel- and cadmium-bearing rinsewaters was achieved with the chelating aminophosphonate resin Purolite S950. Regeneration of this resin with acid was very efficient.

The aminophosphonate resin can be used for the selective stripping of nickel from the rinsewaters of nickel chloride baths, nickel sulfamate baths, electroless nickel baths, and nitric-acid-based nickel coating removal baths. These applications need pH feed adjustments.

The performance of chelating resins is solution pH dependent. Preconditioning the resin bed to match the feed pH can enhance the removal of nickel with the selective ion exchanger from a solution which contains ethylenediamine as a complex forming agent. Preconditioning of the selective ion exchanger, however, is not simple.

Cadmium could be stripped very efficiently from the cadmium coating removal bath (ammonium nitrate solution) with the aminophosphonate resin Purolite S950. Iminodiacetic resin performed less well than the aminophosphonate resin. Ammonium nitrate enhances the metal adsorption performance of the aminophosphonate resin. Cadmium could not be adsorbed from cyanide solution with the tested resins because the cadmium cyanide complex could not be broken.

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