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Separation Science and Technology

Publication details, including instructions for authors and subscription information:
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Online publication date: 08 July 2010

To cite this Article Zhuravlev, Igor Z. , Kanibolotsky, Valentin A. , Strelko, Vladimir V. and Gallios, George P.(2005) 'Novel Spherically Granulated Inorganic Ion Exchangers Based on Aluminophosphatesilicate and Ferrophosphatesilicate Gels', Separation Science and Technology, 39: 2, 287 — 300
To link to this Article: DOI: 10.1081/SS-120027559
URL: http://dx.doi.org/10.1081/SS-120027559

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SEPARATION SCIENCE AND TECHNOLOGY
Vol. 39, No. 2, pp. 287–300, 2004

Novel Spherically Granulated Inorganic Ion Exchangers Based on Aluminophosphatesilicate and Ferrophosphatesilicate Gels

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ABSTRACT

Novel porous sodium aluminophosphatesilicate (APS) with the empirical formula $Na_{4.01}Al_{4.02}PSi_{17.99}O_{46.5} \cdot 18H_2O$ and sodium ammonium ferrophosphatesilicate (FPS) with the empirical formula $Na_{0.32}(NH_4)_{2.44}$-$Fe_{6.17}PSi_{8.9}O_{30.9} \cdot 14.3H_2O$ were synthesized by the sol–gel method as the spherically granulated beads. The APS and FPS were characterized by elemental analysis, x-ray diffraction, ^{27}Al, ^{31}P, ^{29}Si MAS NMR, ^{57}Fe

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DOI: 10.1081/SS-120027559 0149-6395 (Print); 1520-5754 (Online)

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Mössbauer spectroscopy, and thermal gravimetric analysis (TGA). Ion exchange of some divalent metal cations (Pb^{2+}, Cd^{2+}, Cu^{2+}, Ni^{2+}, Zn^{2+}) and sodium by APS and FPS was studied in batch conditions. It was found that APS and FPS have a cation exchange capacity of about 2.5 and 2.0 mEq/g respectively. The ion exchange isotherms for divalent cations were determined. The investigated APS exhibits a high affinity toward lead and copper ions; moderate affinity towards zinc, nickel, and cadmium ions; FPS exhibits a high affinity towards lead and copper cations; moderate affinity to zinc and cadmium cations; and a low affinity to nickel cations in 0.1-M $NaNO_3$ solutions. Due to low cost and high process ability of manufacture and the spherical form of the particles, we can suppose that these sorbents could be used for purification of some industrial waste waters from toxic heavy metals.

Key Words: Sol–gel technique; Ion exchangers; Amorphous gels; Waste waters purification; Inorganic materials; Porous materials.

INTRODUCTION

Purification of water in the biosphere and removing toxic metal ions is an area of high priority research in environmental science. The basic problem is that some ions, such as cadmium and lead, are very toxic in low concentrations, but they present in water containing much greater concentrations of benign metal ions, such as sodium and calcium. Any attempts to remove the toxic ions by reagent methods are not successful, usually because the reagents used are not selective enough to form complexes only with the target ions and remain transparent to the benign ions. An active search for new selective ion exchangers and adsorbents undertaken in last several decades resulted in discovery of a wide variety of synthetic inorganic compounds exhibiting ion-exchange properties.[1–4] Some of these compounds (hydrated oxides and insoluble acidic salts of polyvalent metals, heteropolyacids, metal ferrocyanides, etc.), due to their high thermal stability, resistance to oxidation and irradiation, and high affinity to certain ions, are regarded as prospective materials for operating in extreme conditions (high radioactivity, temperature, pressure, presence of oxidants, and organic solvents, high concentration of background electrolytes, etc.) where organic ion exchange resins fail to work efficiently. However, synthetic inorganic ion exchangers, with the exception of silica gel and some zeolites, have not found such a wide application yet as organic ion exchange resins. The main reason for this is connected with the technical problems of preparation in a spherical form acceptable for industrial application. Recently, we reported data about synthesis of spherically granulated titanium and zirconium phosphates, hydroxophosphate, hydroxides[5,6] via a sol–gel method and characterization of their ion exchange properties.[5–9] It was shown that these sorbents exhibit a high affinity toward some heavy metal cations. However, the reagents used for titanium and zirconium phosphates and hydroxides syntheses are quite expensive. For this reason we recently elaborated a new hybrid gel of inorganic ion exchangers—sodium aluminophosphatesilicate via a sol–gel method[8,9] using simple and quite inexpensive inorganic reagents, such as sodium silicate, sodium aluminate, and phosphoric acid. The amorphous character of some gel ion exchangers allows smooth adjustment of such important physical and chemical properties as porosity and selectivity of sorption. In our recent research, the opportunity of variation of selectivity of the aluminophosphate-silicate (APS) toward radio cesium ions was shown. The APS ion exchanger described in this article has the biggest selectivity toward cesium and has been recognized by us as the most prospective. As a continuation of these efforts, the results of further research of the APS and synthesis of another spherically granulated inorganic ion exchanger—sodium–ammonium ferrophosphatesilicate (FPS) by similar technique and investigation of their properties are also presented in this article.

EXPERIMENTAL

Syntheses of Sodium Aluminophosphatesilicate and Sodium–Ammonium Ferrophosphatesilicate

Spherically granulated APS and FPS were prepared with a sol–gel method, similar to that described earlier.[8] According to this method, for APS synthesis, 140 mL of a 5-M H_3PO_4 solution, 490 mL of a 1-M $Na_2Si_{2.27}O_{5.54}$ solution, and 330 mL of a 1-M $[NaAl(OH)_4 + NaOH]$ solution were mixed efficiently in a mixer.

FPS was synthesized by a similar technique: 20 mL of a 5-M NaH_2PO_4 solution, 317 mL of a 1-M $Na_2Si_{2.27}O_{5.54}$ solution, 160 mL of a 3-M $FeCl_3$ solution, and 150 to 300 mL of a 6-M NH_4OH solution were mixed efficiently in a mixer. In both cases the obtained reaction mixtures were dispersed into a vertical column filled with an organic solvent (aliphatic hydrocarbons C_9H_{20}–$C_{11}H_{24}$ at ambient temperature). In the column, formation of spherical granules took place. After separation from the organic solvent the granules of gel were thoroughly washed, aged for 48 hours at ambient temperature, and then dried in air. The APS and FPS are mechanically strong, glass-like, colorless and brown, respectively, transparent spherical beads with a diameter within the range of 0.5 to 2 mm. The H^+-form of the APS (APS-H) was prepared by treatment with an excess of a 0.1-M solution of nitric acid.

Analytical Procedure

The difractometer used was a Seifert-Scintag PAD5 with CuK$_\alpha$ radiation. Thermal analysis was performed with a DuPont Instruments TA 4000 unit (under nitrogen, at a heating speed of 10°C/min). The phosphorus, silicon, iron, aluminum, and sodium content in the APS and FPS were determined by using a Spectra Spec spectrometer DCP-AEC after dissolving an amount weighted in HF. The pH of solutions were measured using a Corning-340 pH meter. The concentrations of ions in the solutions were measured using a Varian Spectr AA 300 atomic absorption spectrometer. ^{27}Al, ^{31}P, ^{29}Si NMR spectra at magic angle of air-dried APS samples were obtained on a BRUCER CXP-200 spectrometer. The Mössbauer spectrometer was used ЯГРС-4м (USSR).

Ion Exchange Study

The ion-exchange sorption of sodium cations and chloride anions with spherically granulated APS and FPS was studied using a model of 0.1-M (NaCl + NaOH or HCl) solutions at V : m = 100 : 1 (mL/g). Exchange of all divalent cations (Pb^{2+}, Cd^{2+}, Cu^{2+}, Ni^{2+}, Zn^{2+}) by APS and FPS was determined in (0.001–0.05) molar $M(NO_3)_2$ aqueous 0.1 mole $NaNO_3$ containing solutions at V : m ratio = 100 : 1 (mL/g). In all cases, the agitation was performed by constant, intensive shaking 8 hours per day for 7 days at 25°C. Exchange of heavy metals was carried out by the batch technique at equilibrium pH of 3 to 7. The second series of cadmium sorption experiments were performed under constant pH. Volume to mass ratios 250 : 1 for APS and 1000 : 1 for FPS were used.

The affinity of the exchangers toward the ions of interest was expressed as the uptake in milliequivalent per gram (mEq/g) vs. equilibrium concentration of M^{2+}.

RESULTS AND DISCUSSION

X-ray diffraction and ^{57}Fe Mössbauer spectroscopy analyses of the spherically granulated sodium APS and sodium–ammonium FPS were performed and an amorphous nature was found for both samples. Using previous ^{57}Fe Mössbauer spectroscopy investigation, the doublet signal was obtained for some similar FPS samples. It is an indication of Fe^{3+} high spin state in octahedral coordination by oxygen. With the help of ^{57}Fe Mössbauer spectroscopy, we could not find a distinction in the condition or coordination of iron among similar FPS samples. The optical transparency of the APS and FPS materials unequivocally testifies in favor of homogeneity. Using a solid state MAS NMR ^{27}Al, ^{31}P,[8,9] ^{29}Si[8] investigation, we studied the chemical nature of the aluminum–phosphorus-containing compounds synthesized by a sol–gel technique with a different pH in $NaOH-HCl-AlCl_3-H_3PO_4-Na_2SiO_3$ system.[9] It was shown that in acidic media, aluminum phosphates formation takes place (octahedral coordinated aluminum: chemical shift $\delta = -16.4$ ppm; phosphorus $\delta = -11.9$ ppm). In alkaline media, the aluminosilicates were prepared (aluminum in tetrahedral coordination, ^{27}Al $\delta = +55.04$ ppm; ^{31}P—two resonance signals: $\delta_1 = +2.5$ ppm, $\delta_2 = -14.3$ ppm). In neutral media the hybrid APS compounds were synthesized: ^{27}Al $\delta_1 = +54.8$ ppm, $\delta_2 = -10.8$ ppm; ^{31}P $\delta = -12.8$ ppm. In other words, in this case, a continuous inorganic polymer with –Si–O–Al–O–P–O– chemical bonds is formed. It is reasonable to suppose that APS is a dispersion of APS in silica. This statement is reinforced, first of all, by the existence of a broad ^{29}Si NMR signal with a maximum at -102 ppm,[8] which partly arises from the presence of a large quantity of amorphous SiO_2 in the compound.

Elemental analysis gave: Na 5.09%, Al 5.98%, P 1.71%, Si 27.89% (APS); and Na 0.53%, Fe 25.06, P 2.26%, Si 18.15%, N 0.63% (FPS), which yields the empirical formulae $Na_{4.01}Al_{4.02}PSi_{17.99}O_{46.5} \cdot 18H_2O$ (F · Wt · 1805) and $Na_{0.32}(NH_4)_{2.44}Fe_{6.17}PSi_{8.9}O_{30.9} \cdot 14.3H_2O$ (F · Wt · 1428.7), respectively.

Thermal gravimetric analysis (TGA) weight loss was found: 15.9% (APS) and 23.0% (FPS) [Figs. 1(a) and 2, accordingly].

Thermal analysis of the APS [see Fig. 1(a)] shows that its thermal decomposition occurs in one step within the temperature range of 50 to 550°C. In this range, there is a removal of physically and chemically bound water, and also condensation of free M–OH groups.

The H-form of the APS, in the process of thermal transformation to $Al_{4.02}PSi_{17.99}O_{44.5}$ (F · Wt · 1356), yielded a total weight loss of 17.40% [see Fig. 1(b)]. Thus, the formula weight of the original protonated compound was 1642 and its formula was $H_{4.01}Al_{4.02}PSi_{17.99}O_{46.5} \cdot 13.8H_2O$. The weight loss occurs in two steps as described below:

The first step

$$H_{4.01}Al_{4.02}PSi_{17.99}O_{46.5} \cdot 13.8H_2O \xrightarrow{50-200^{\circ}C} H_{4.01}Al_{4.02}PSi_{17.99}O_{46.5}2.2H_2O + 11 \cdot 6H_2O$$

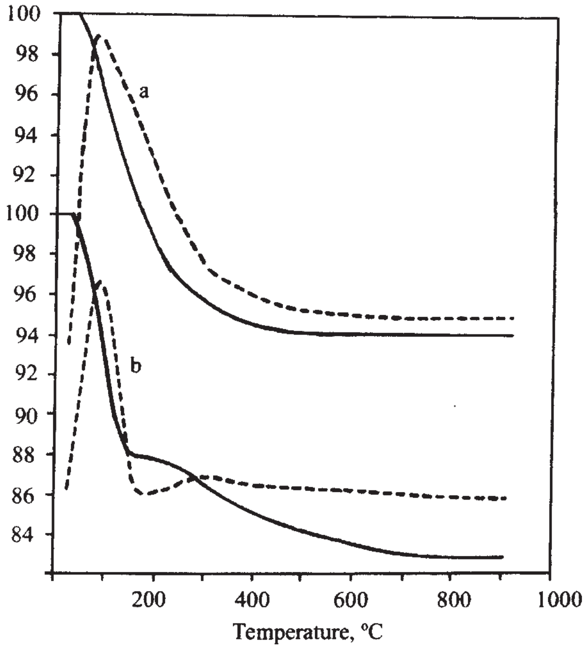


Figure 1. TG and DTG curves for APS in sodium (a) and H-forms (b).

The second step

$$H_{4.01}Al_{4.02}PSi_{17.99}O_{46.5} \cdot 2.2H_2O \xrightarrow{200-780^\circ C} Al_{4.02}PSi_{17.99}O_{44.5} + 4.2H_2O$$

Thermal analysis of FPS (Fig. 2) shows that its thermal decomposition occurs in two steps in the temperature ranges of 20 to 195°C and 195 to 605°C. The total weight loss within the range of 20 to 605°C was 22.5%. The first step in the range of 20 to 195°C corresponds to release of physically bound water. The weight loss was 17.8%, against 18.0% calculated for water release from $Na_{0.32}(NH_4)_{2.44}Fe_{6.17}PSi_{8.9}O_{30.9} \cdot 14.3H_2O$. In the second stage, decomposition takes place within the temperature range of 195 to 605°C and the weight loss was 4.7%. These changes are assigned to ammonia release and to condensation of arising acid and base groups.

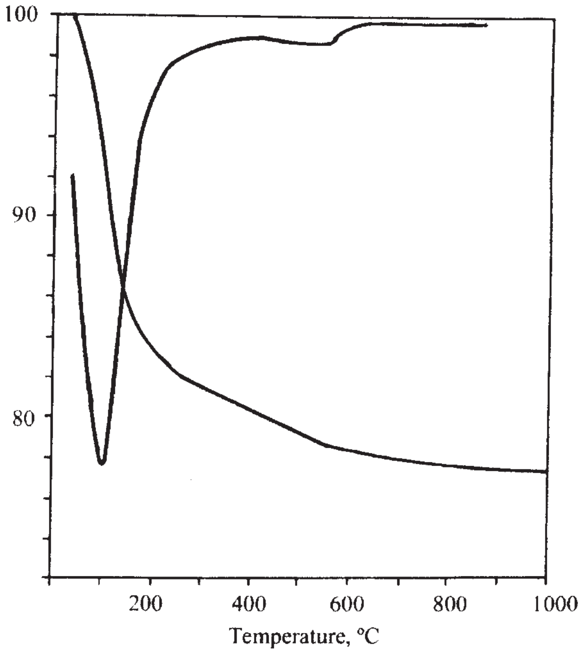


Figure 2. TG and DTG curves for FPS in sodium–ammonium form.

The reactions then can be represented as:

The first step

$$\mathrm{Na_{0.32}(NH_4)_{2.44}Fe_{6.17}PSi_{8.9}O_{30.9} \cdot 14.3H_2O \xrightarrow{20-195^\circ C} Na_{0.32}(NH_4)_{2.44}Fe_{6.17}PSi_{8.9}O_{30.9} + 14.3H_2O}$$

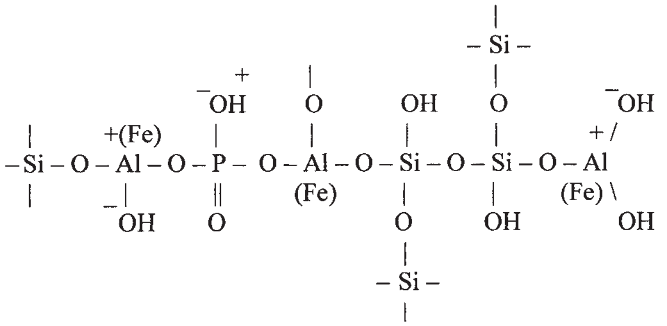
The second step

$$\mathrm{Na_{0.32}(NH_4)_{2.44}Fe_{6.17}PSi_{8.9}O_{30.9} \xrightarrow{195-605^\circ C} Na_{0.32}H_{2.44}Fe_{6.17}PSi_{8.9}O_{30.9} + 2.44NH_3}$$

$$\mathrm{Na_{0.32}H_{2.44}Fe_{6.17}PSi_{8.9}O_{30.9} \xrightarrow{195-605^\circ C} Na_{0.32}Fe_{6.17}PSi_{8.9}O_{29.68} + 1.22H_2O}$$

Based on the detailed MAS NMR investigation (see above) of some sodium APS[9] and on the described data, the fragment of skeleton formula of

$H_{4.01}Al_{4.02}PSi_{17.99}O_{44.5}(OH)_{4.4} \cdot 11.6H_2O$ without specifying water, may be represented as follows:



$$-\overset{|}{\underset{|}{Si}}-O-\overset{+(Fe)}{\underset{\overset{-|}{^{-}OH}}{Al}}-O-\overset{\overset{+}{^{-}OH}}{\underset{\overset{\|}{O}}{P}}-\ O-\overset{\overset{|}{O}}{\underset{(Fe)}{Al}}-O-\overset{OH}{\underset{\overset{|}{\underset{|}{\overset{|}{O}}}\,\underset{|}{-Si-}}{Si}}-O-\overset{\overset{\overset{|}{-Si-}}{\overset{|}{O}}}{\underset{OH}{Si}}-O-\overset{\overset{^{-}OH}{+/}}{\underset{\underset{OH}{(Fe)\backslash}}{Al}} \qquad (1)$$

Despite the seeming differences in chemical compositions of APS and FPS, both compounds have a common chemical nature. APS and FPS are the solid solutions of phosphorus–metal–silicon–oxide inorganic polymers in amorphous silica. We have proved the formation of such a polymer for APS. FPS was synthesized by the same technique in the same conditions and it has similar chemical properties. Therefore, in our opinion, the skeletal formulas of both compounds are identical. The difference consists only in that APS contains a little bit more silica, which influences the chemical nature a little. Sodium and ammonium do not participate in formation of a skeleton. The hydrate water participates in formation of the structure, but in both cases, its action is identical. So, the skeleton structure of FPS is similar to that of APS (formula 1) without specifying the sodium and ammonium cations and water.

Potentiometric titration curves of the APS in H-form, $H_{4.01}Al_{4.02}PSi_{17.99}O_{44.5}(OH)_{4.4} \cdot 11.6H_2O$, with sodium hydroxide are presented in Fig. 3(a). Based on the proposed formula, the theoretical cation exchange capacity of this material is 2.44 mEq/g. We assume that only protons in positions previously occupied by sodium ions are exchangeable. It does not exclude the possibility of the ions exchange of some protons in the M–OH groups (M=Al, Fe, Si) for alkali metal cations in alkaline media as well as free hydroxide groups for anion in acid media.[1,2] However at $pH = 10$, the exchange capacity reaches only 1.77 mEq/g. The filling of other ion-exchangeable groups occurs in alkaline media with $pH > 10$ and is accompanied by a partial sorbent decomposition.

It was found that APS is a moderately weak anion exchanger. The presence of free hydroxide groups enables APS to behave as an anion exchanger in acidic media. The APS begins to exchange chloride anion at $pH < 6$ to 7 and its exchange capacity for Cl^- reaches 2.5 mEq/g at pH 3 [see Fig. 3(b)]. Any attempt to transform the sodium ammonium form of FPS into

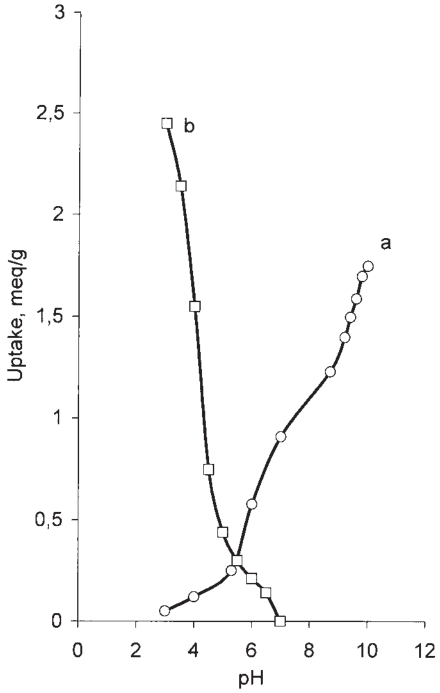


Figure 3. Potentiometric titration of APS (H-form) with 0.1-M solutions of NaOH (a) and HCl (b).

H-form was not successful because of its hydrolytic decomposition in acidic media at $pH < 3$.

According to the proposed initial formula, the theoretical cation exchange capacity of the APS in sodium form is 2.2 mEq/g. Potentiometric titration curves of the FPS in the sodium–ammonium form with sodium hydroxide and hydrochloric acid are presented in Fig. 4 [(b) and (c) respectively]. It is necessary to note that the sodium cations substitute the ammonium cations (NH_4^+) of FPS in the process of potentiometric titration. In the case of sodium–ammonium FPS both NH_4^+ and Na^+ cations are exchangeable by polyvalent cations. The theoretical cation exchange capacity of FPS is 1.93 mEq/g. The exact total ion exchange capacities estimation based on potentiometric titration curves for APS and FPS are quite difficult because they hydrolyze and decompose in alkali media with $pH > 10$ (APS and FPS) and in acidic media with $pH < 3$ (FPS). This statement based on the well-known fact of the decomposition of amorphous silica-containing gels in

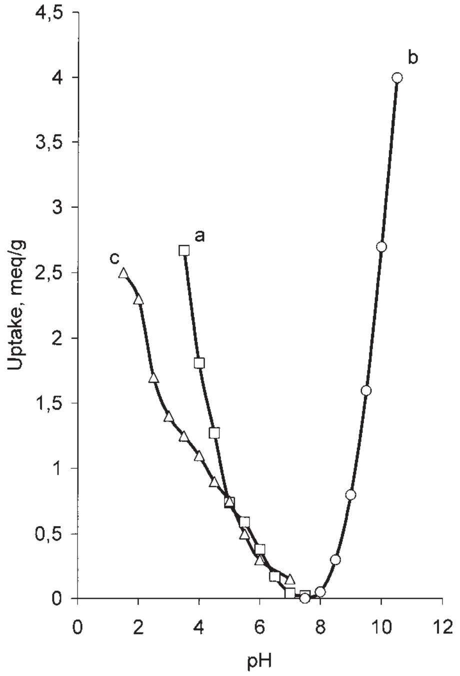


Figure 4. Potentiometric titration of APS (sodium form) with 0.1-M solution of HCl (a) and FPS (sodium–ammonium form) with 0.1-M solutions of NaOH (b), and HCl (c).

alkaline media with pH > 10[10] and on our own results of the FPS hydrolytic stability study.

The ion exchange determination of the affinity of the APS and FPS toward some divalent cations (Pb^{2+}, Cd^{2+}, Cu^{2+}, Ni^{2+}, Zn^{2+}) was carried out in aqueous 0.1-M $NaNO_3$ containing solutions of the corresponding 0.001 to 0.05-M metal nitrates using the batch technique at a constant volume to mass ratio. After reaching equilibrium, the solid and liquid phases were separated by filtration and the composition of the solutions was determined. Based on these data, the uptakes of cations of interest were calculated. These uptakes are presented on Figs. 5 and 6 as functions of cations equilibrium concentrations in solution. The pH values of equilibrium solutions after the M^{2+} ion exchange adsorption were for FPS: Cu—4.6; Pb—4.5; Cd—6.05; Zn—5.5; Ni—6.4; for APS : Cu—4.7; Pb—4.3; Cd—5.8; Zn—5.5; Ni—6.0. None of the investigated bivalent cations hydrolyze in the conditions of the experiment and they do not form anionic complexes.[11]

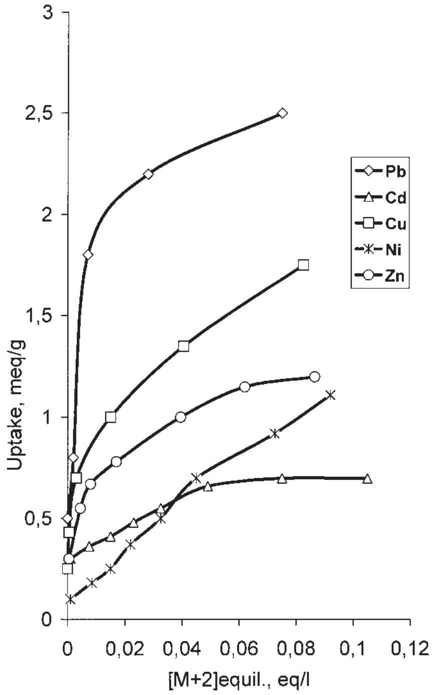


Figure 5. Pb^{2+}, Cd^{2+}, Cu^{2+}, Ni^{2+}, Zn^{2+} ion exchange on APS (sodium form) in 0.1-M $NaNO_3$ solution.

It was found that APS exhibits a high affinity to lead and copper ions, and moderate affinity to zinc, nickel, and cadmium ions (see Fig. 5); FPS exhibits a high affinity to lead and copper ions, moderate affinity to zinc and cadmium ions, and a low affinity to nickel ions in solutions with moderate initial concentrations of M^{2+} (see Fig. 6). The data indicate that for APS and FPS, the ion exchange uptake reaches 1.5 to 2.5 mEq/g. These values coincide with the amount of sodium and ammonium ions in the solids, which suggests that predominantly $M^{2+} \rightarrow Na^{+}(NH_4^{+})$ ion exchange takes place. In a series of investigated cations, the ions of nickel have the smallest ionic radius (0.78 Å), the greatest hydration,[11] and, therefore, the worst diffusion into a sorbent. With some phosphatic ion exchangers the nickel adsorption is bad.[1,3,5] The investigated FPS is not an exception. In the range of micro concentration, opposite to APS, on FPS the nickel practically does not adsorb. This circumstance can be used for separation of other d-metals from nickel at its analytical definition and for purification of nickel electrolytes.

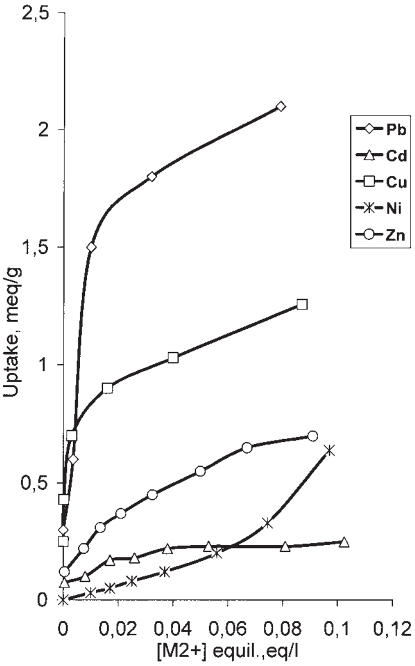


Figure 6. Pb^{2+}, Cd^{2+}, Cu^{2+}, Ni^{2+}, Zn^{2+} ion exchange on FPS (sodium–ammonium form) in 0.1-M $NaNO_3$ solution.

An ion exchange adsorption of cadmium cations without background electrolyte on APS and FPS was studied using batch technique at constant volume to mass ratio 250 : 1 (APS) or 1000 : 1 (FPS) and the results are shown on Fig. 7. The initial cadmium concentrations were 50 to 600 ppm. The pH of the solutions were kept constant with HNO_3 or NaOH as necessary, automatically. Three different pH values were studied: 4, 6, and 7 (APS) and 6 (FPS). It was shown that APS and FPS exhibit a high affinity toward cadmium cations in dilute solutions at pH 4 to 7 (APS) and at pH 6 (FPS). The exchange capacity of APS by Cd^{2+} at V : m = 1000 : 1 (data are not presented here) reaches 2.1 mEq/g (pH 7), and 1.8 mEq/g at V : m = 250 : 1 (pH 7) (see Fig. 7). The ion exchange capacity of FPS by cadmium is a little bit less than that of APS. It is clear that these results are better than those obtained for cadmium uptake from 0.1 $NaNO_3$ solutions (see Figs. 5 and 6) due to the absence of competition between Na^+ and Cd^{2+} cations.

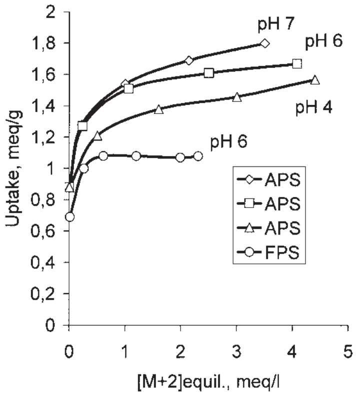


Figure 7. Cd^{2+} ion exchange on APS and FPS without background electrolyte.

CONCLUSION

The study of synthesized APS ion exchangers, described by us earlier was continued. For the new FPS ion exchanger, the synthesis, structure, behavior at heating, and ion-exchange properties are described. APS and FPS ion exchangers have the theoretical cation exchange capacities 2.2 mEq/g and 1.93 mEq/g, respectively. Experimentally found values of these capacities are close to the theoretical. It turns out that both ion exchangers show a high affinity to ions of cadmium in pure cadmium solutions and moderate affinity on a salt background. APS and FPS can be recommended for purification of various water solutions from cadmium. The greatest affinity of both ion exchangers was found toward ions of lead and that is typical for inorganic phosphatic ion exchangers.[3–6] It was found that in the region of low concentrations, the FPS practically does not adsorb the nickel. It allows us to recommend FPS sorbent for the purposes of analytical definition of nickel and for the nickel electrolytes purification from undesirable cations.

ACKNOWLEDGMENT

This research was supported partly by NATO Grant SST CLG 976355.

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Received November 2002
Revised April 2003

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