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Synthesis of a New Granulated Polyampholyte and its Sorption Properties

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Abstract. In this paper, characteristics of the interaction of an anion-exchange resin PPE-1 formed using phosphonic acid and granular polyvinyl chloride were investigated. In addition, a reaction order of 1.43 and an activation energy of 47.8 kJ/mol for phosphorylation were determined. The as-obtained polyampholyte was characterized by scanning electron microscopy (SEM), energy-dispersive X-ray (EDX) analysis, and Fourier transform infrared (FTIR) spectroscopy. Optimum conditions for obtaining a new polyampholyte that selectively adsorbs non-ferrous metal ions on the basis of granulated polyvinyl chloride were determined. Sorption isotherms for non-ferrous metal ions were constructed, which were reasonably explained by Langmuir isotherms. Various thermodynamic parameters, such as isobaric-isothermal potential (ΔG), enthalpy (ΔH), and entropy (ΔS), were calculated to understand the nature of sorption. The examined polyampholyte selectively absorbed copper(II) ions.

Keywords: Metals; Modification; Polyampholyte; Sorption

1. Introduction

Currently, among various methods, polymer adsorbents are effective and widely used for the removal of non-ferrous and rare metal ions (Saad et al., 2011). Hence, polymer adsorbents are comparable to other recovery methods in terms of technical and economic efficiency, feasibility, and environmentally friendly technologies. Saad et al. (2011) crosslinked polyethylenimine (PEI) with epichlorohydrin (ECH) to afford a water-insoluble form and it was subsequently used as an adsorbent. As an extension of their study, (Saad et al., 2011; Saad et al., 2012), crosslinked polyethylenimine (CPEI) was phosphonated with phosphoric acid and formaldehyde for the selective removal of uranium ions. The binding affinity of phosphonated cross-linked polyethylenimine (PCPEI) with uranium ions as well as its regeneration for reuse were evaluated (Saad et al., 2012). The obtained polyampholyte exhibits up to 99% selectivity for uranium ions in the presence of competing metal ions (e.g. Mn, Ni, As).

Jeon and Kwon (2012) investigated the desorption characteristics of indium ions previously adsorbed on phosphorylated sawdust using various reagents such as HCl, HNO_3, NaCl, ethylenediaminetetraacetic acid, and nitrilotriacetic acid. Results revealed that HCl is the best desorption agent from an economic viewpoint, with an ~97%

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desorption efficiency for indium ions at an HCl concentration of 0.5 M. Moreover, an extremely high desorption efficiency (~94%) is observed for indium ions at a solid-to-liquid of 10.0, and desorption is rapidly completed in 60 min. Recycled phosphorylated sawdust maintains an 85% removal efficiency of indium ions in the 4th cycle.

Elsharma et al. (2019) prepared a polyampholyte of nanocomposite bio-polymers, i.e., poly (N,N-diallyldimethylammonium chloride-co-acrylamide) grafted onto carboxymethyl cellulose/iron(III) oxide [P(DADMAC-AAM) CMC/Fe_2O_3] and poly(N,N-diallyldimethylammonium chloride-co-sodium acrylate grafted onto carboxymethyl cellulose/iron(III) oxide [P(DADMAC-SA)CMC/Fe_2O_3]], with various molar ratios of anionic groups and cationic groups using gamma radiation. The structure and morphology of the obtained materials were examined by Fourier transform infrared spectroscopy and scanning electron microscopy. Sorption batch sorption experiments were conducted using a radioactive indicator such as ^{60}Co to remove Co(II) P(DADMAC-AAm) CMC/Fe_2O_3 and P(DADMAC-SA) CMC/Fe_2O_3 were evaluated from aqueous solutions. Experimentally, P(DADMAC-AAm) CMC/Fe_2O_3 and P(DADMAC-SA) CMC/Fe_2O_3 exhibit high sorption capacities of 69.67 mg g^{-1} and 75.17 mg g^{-1} for Co(II), respectively, making them potential sorbents for the removal of Co(II) from water or wastewater.

Zeng and Li (2014) employed an ion-exchange resin method for the purification of heavy metal ions such as Cu^{2+} from chemical wastewater and investigated the effects of flow rate, pH, and temperature on Cu^{2+} removal using a microporous, strongly acidic cation exchanger of styrene type D001. Results revealed that at a flow rate of 1.5 mL/min, a pH of 6.0, and a temperature of 30°C, a 99.8% removal rate of Cu^{2+} is reported over D001. Chemical wastewater can reach wastewater discharge standard.

Smanova et al. (2011) investigated the efficiency of fibrous materials based on polyacrylonitrile (PAN) modified with hydroxylamine in organic and aqueous carriers, as well as with hexamethylenediamine and ethylenediamine. In addition, properties of the as-obtained fibrous sorbent for the sorption of iron(III) ions in an aqueous solution were examined.

Kiefer and Höll (2001) investigated the ion exchange of heavy metal ions (such as Cu^{2+}, Ni^{2+}, Cd^{2+}, Zn^{2+}, and Co^{2+}) as well as Ca^{2+}, Na^{+}, and NH_4^{+}, by two industrial processes using ion-exchange complex-forming resins comprising amino- and phosphate-containing functional groups (Purolite S 940 and S 950). The authors estimated sorption according to the theory of complexation of external surfaces, leading to a set of binary equilibrium sorption of ions, which remains unchanged in multi-ion systems. Kinetic parameters of the secondary, tertiary, and quaternary equilibria for sorption and the balance of complex agents in the industrial effluent formed during the processing of metal surfactants are proposed on the basis of equations that calculated theoretical values of sorption and compared with experimental data (Kiefer and Höll, 2001).

Deepatana and Valix (2006) and Altun and Pehlivan (2007) independently investigated the release of trace amounts of non-ferrous and toxic metals using ion-exchange materials. Experiments were conducted using well-known industrial ion-exchange materials such as Lewatit CNP 80 and Lewatit TP 207 (Altun and Pehlivan, 2007). Authors investigated the effect of pH among solutions, sorption duration, metal ion concentration, and ion-exchanger amount during sorption. The examined complex media (Deepatana and Valix, 2006) exhibit a higher and more rapid sorption capacity for metal ions such as Pb(II), Cu(II), Zn(II), Cd(II), and Ni(II). The optimal pH range of solutions for the ion exchange of the above metal ions on Lewatit CNP 80 and Lewatit TP 207 is 7.0–9.0 and 4.5–5.5, respectively (Deepatana and Valix, 2006).

Olufemi and Eniodunmo (2018) examined the comparative adsorption removal of Ni(II) ions from an aqueous solution using coconut shells and a banana peel. The Adsorbate dose, adsorbent dose, pH, contact time, particle size, and temperature were varied, and their effects on the percentage removal of Ni(II) ions were evaluated. By using both adsorbents, the Maximum removal rate is observed at pH 8.0. The optimum conditions for both adsorbents include an adsorbent dose of 4.5 g, a contact time of 30 min, and a temperature of 25°C for the coconut shell, and an adsorbent dose of 4.5 g, a contact time of 120 min, and a temperature of 25°C for the banana peel.

Previous studies reported the Preparation and identification of amino- and phosphorus-containing ion-exchange materials, as well as the study of selectivity for non-ferrous metal ions with the formation of stable chelates with these materials. In this study, the sorption of Cu(II), In(III), and Ni(II) ions from aqueous solutions of amino- and phosphite-containing polyampholytes is examined.

2. Materials and Methods

2.1. Materials

PVC plastic compound and polyethylenepolyamine corresponding to GOST 23672–79 and Technical Specification (TS) 2413-010-75678843–2012, respectively, are used. To modify the anion of the exchange material, formalin and phosphonic acid corresponding to GOST-1625-89 (W% 36.5–37.5) and TS 6-09-4023-75, respectively, are used, as well as chemically pure hydrochloric acid and chemically pure sodium hydroxide corresponding to GOST-3118-77 (W% 35–38) and GOST-4328-77 (W% 99), respectively. All reagents were purchased from REACHIM.

2.1.1. Adsorbent preparation

Phosphorylation as conducted in a 25% aqueous solution of phosphonic acid at different ratios of the polymer-base-modifying reagent at 333 K, 343 K, 353 K, 363 K, 373 K, and 383 K for 2, 4, 6, 8, and 10 h. With an increase in the concentration of the modifying reagent in the system to 55%, the product yield increases; with a further increase in the concentration, the yield of the final product remains constant.

2.2. Spectroscopic Studies of the Polyampholyte

Sample spectra were recorded on an IRTracer-100 SHIMADZU IR-Fourier spectrometer (Japan), equipped with a single ATR attachment with a diamond/ZnSe MIRacle 10 prism; this system was designed to analyze solid, liquid, pasty, and gel-like substances in the wavenumber range of 4600–600 cm^{-1}. SEM images were recorded on an EVO MA-10 scanning electron microscope (Carl Zeiss, Germany), which was equipped with a microanalytical system for energy-dispersive X-ray (EDX) microanalysis (Oxford Instruments, UK) that permits the detection of chemical elements starting from boron.

2.3. Procedure

Working solutions of copper(II) nitrate, indium(III) nitrate, and nickel(II) nitrate hexahydrate were prepared by dissolving $Cu(NO_3)_2$, $In(NO_3)_3$, and $Ni(NO_3)_2{\cdot}6H_2O$ samples in a certain amount of distilled water. The solution pH was adjusted using ammonium acetate buffer solution. Effects of time, temperature, and solution concentration on the adsorption of metal ions were investigated. The concentration of In(III) ions in solution was measured by a CAL-1M2 conductivity meter. The solution pH was measured using a universal ionomer EV-74 and pH-meter pH/mV/TEMP Meter P25 Eco Met. The change in concentrations of Cu(II) and Ni(II) ions in the solutions was measured by a spectrophotometric method. The change in the optical density of the solution was measured using an Enspire Perkin Elmer (USA) microplate reader.

The amount of metal ions absorbed on the sorbent is calculated by the following equation.

$$A = \frac{(C_0 - C_e)}{m} \times V \tag{1}$$

where, A-the amount of metal ions adsorbed on the ion-exchange resin, mg g^{-1}, C_0-initial concentration of metal ions, mg L^{-1}, C_e-equilibrium concentration of metal ions, mg L^{-1}; V-solution size, L, and m-dry sorbent mass, g (Belkhouche and Didi, 2010; Kusrini et al., 2018).

2.3.1. Learning the balance of adsorption

Adsorption isotherms constitute the most important method to analyze equilibrium processes. Langmuir models are the most widely models, which are convenient to describe equilibrium processes in liquid and solid systems.

2.4. *Langmuir Isotherm Model*

To estimate The Langmuir constant, the Langmuir equation is used as a linear equation, as shown below. A_{max} and K_L values are determined from C_e obtained by plotting C_e versus A (Hannachi et al., 2014).

$$\frac{C_e}{A} = \frac{1}{AK_L} + \frac{1}{A_{\max}} \times C_e \tag{2}$$

Hence, a conclusion about the relationship between the adsorbent and adsorbate by using the separation coefficient (R_L) can be drawn, which is an integral property of the Langmuir isotherm parameters (Hannachi et al., 2014).

$$R_L = \frac{1}{1 + K_L \cdot C_0} \tag{3}$$

According to Equation 3, the adsorption process $0 < R_L < 1$ is favorable, $R_L > 1$ is unfavorable, $R_L = 1$ is considered to be a linear adsorption isotherm, and $R_L = 0$ represents irreversible adsorption (Yu et al., 2009; Hannachi et al., 2014).

2.5. *Thermodynamic Analysis*

In this study, adsorption was investigated at 293 K, 303 K, and 313 K. Adsorption can be expressed by using the equilibrium constant:

$$K_e = A / C_e \tag{4}$$

to account for the change in the free energy of the process:

$$\Delta G^0 = -RT \ln K_e \tag{5}$$

In the linear plot of LnK_e versus 1/T, ΔH° and ΔS° values are estimated using the angular value of the intersection slope (Yu et al., 2009; Kusrini et al., 2018).

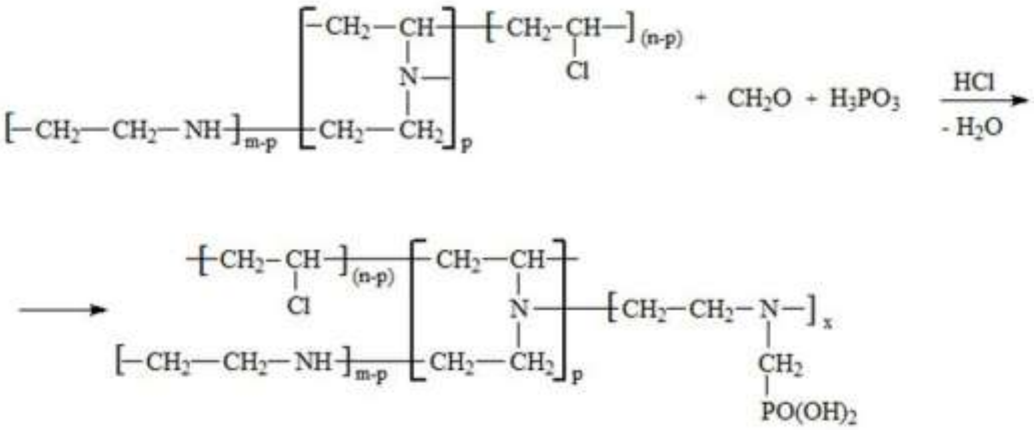
$$\Delta G^0 = \Delta H^0 - T\Delta S^0 \tag{6}$$

3. Results and Discussion

3.1. *Synthesis*

In a 250-mL flask, 10 g of PPE-1 was added, followed by the addition of 150 mL of a 55% aqueous solution of phosphite acid and 50 mL of 35–38% formaldehyde and heated in a water bath. The reaction was conducted at 363–373K for 8 h. The product was separated and dried under air. The SEC (static exchange capacity) was determined using NaOH, which was 5.78 mg-eqv/g.

The reaction scheme for the modification of the PPE-1 anion exchanger with phosphonic acid is shown below.



From the chemical formula of these ion-exchange materials (PPE-1-P), –NH and $–PO(OH)_2$ groups are present, which are known to exhibit both ion-exchange and complex-forming properties with ions of various metals.

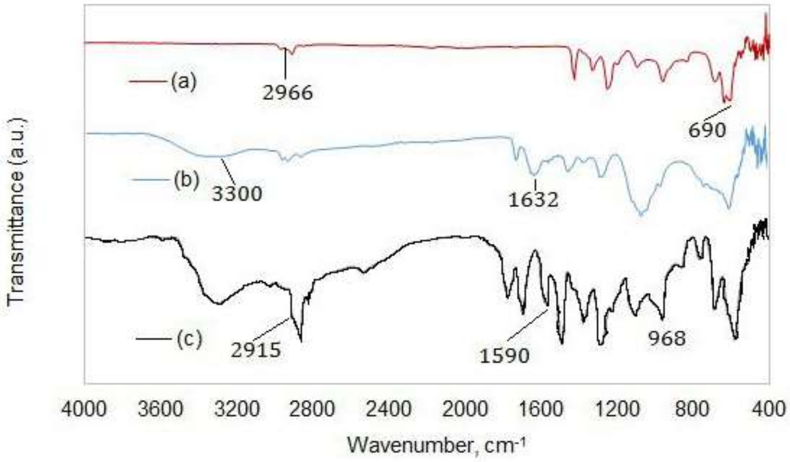
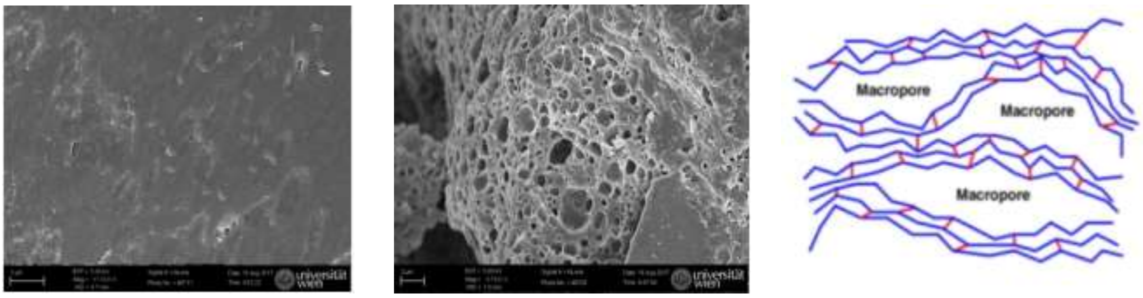


Figure 1 FTIR spectra of (a) PVC; (b) anion-exchange resin PPE-1; (c) polyampholyte PPE-1-P

FTIR spectra were recorded to identify the functional groups in the obtained polyampholyte. Figure 1 (a; b; c) shows the IR spectra of the initial and obtained ion exchangers.

FTIR spectral data confirm the presence of phosphonic and amine groups in the nitrogen–phosphorus-containing polyampholyte structure. The intensity of the absorption band observed at 609–690 cm^{-1}, corresponding to the stretching vibrations of C–Cl groups in PVC, is noticeably reduced, indicative of the decrease in the number of these groups in the macromolecule (Figure 1a). In addition, highly intense absorption bands are observed at 968 and 1099 cm^{-1}, characteristic of $P(O)(OH)_2$ bonds, as well as a band at 1170 cm^{-1}, characteristic of stretching vibrations. The band in the region of 1664 cm^{-1} can be band in the region of 1596 cm^{-1}, characteristic of the deformation vibrations of =N–H bonds. A wide absorption band is observed at 2915 cm^{-1}, characteristic of salts of secondary and primary amines; this result indicated that aminomethylene phosphonic groups are present in the polyampholyte as an internal salt (Ameer et al., 2003; Grachek et al., 2011).



(a) (b) (c)

Figure 2 SEM images of polymeric resin: (a) PVC; (b) phosphorylated resin PPE-1-P; and (c) porosity of PPE-1-P

SEM images were recorded to investigate the surface conditions of the original material and the perforated polymer before and after modification. The original morphology of the PVC structure is flat, and after modification, the polymer matrix changes to a macroporous structure (Figure 2).

Qualitative and quantitative elemental analyses of the polymer obtained by phosphorylating anion-exchange resin PPE-1 revealed that the polyampholyte contains 21.5% O, 8.1% N, and 3.6% P. This data confirm the presence of nitrogen- and phosphorus-containing groups in the obtained polymer.

Thus, based on the results obtained by IR spectroscopy, elemental analysis, and potentiometric titration, granular polyvinyl chloride is chemically modified with polyethylenepolyamine. Then, phosphorylation of the obtained anion-exchange resin (PPE-1) was conducted, and a new polyampholyte was synthesized with an SEC of 5.6–6.2 mg·eqv/g. By investigating the effect of temperature and reaction duration on the degree of conversion of the obtained modified material, the increase in the H_3PO_3 concentration, reaction duration, and temperature leads to an increase in the process.

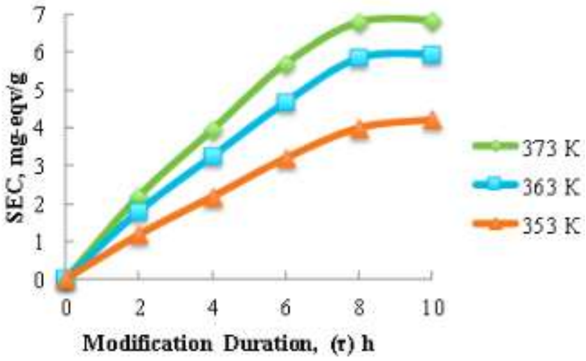


Figure 3 Effect of the temperature of the modification process and duration on the SEC value of the resulting polyampholyte

The obtained data reveal that the rate of phosphorylation reaction linearly depends on the phosphorous acid concentration, indicating that the ratio of this reaction to ordinary reactions is of the first degree. The order of the reaction was calculated by the value of the order of the reaction for phosphorous acid, which was determined to be 1.43; accordingly, the equation for the speed of this reaction can be expressed as follows:

$$V = K\,[H_3PO_3]^{1.43} \tag{7}$$

Based on the obtained results, the dependence of the logarithm of the phosphorylation reaction rate on the inverse temperature was plotted.

From the obtained data, the activation energy for the synthesized PPE-1-P was calculated, which was E = 47.8 kJ/mol. The activation energy values estimated for the phosphorylation reaction indicate the loyalty of the initial reagents to each other. The difference in certain values of the activation energies of the studied reactions is apparently explained by the difference in the electrophilicity of amino groups in the examined modifying reagents.

3.2. Sorption Properties

Polyampholytes contain amino and phosphonic groups. Therefore, the sorption of the examined metal ions occurs due to the ionic and coordination bonding of metal ions with these functional groups. In addition, the presence of an amino group in the polyampholyte promotes the binding of metal ions via coordination. The degree of ion sorption depends on the degree of ionization of the functional groups of the polyampholyte, which in turn depends on the pH of the metal ion solutions. Therefore, studies on the sorption of the examined metal ions are conducted in specific pH media.

The specific adsorption of metal ions is calculated by the difference in its solution concentrations:

$$A = \frac{(C_0 - C_e)}{m} \times V \tag{8}$$

where, *m* is the adsorbent mass (g), *V* is the volume of the solution taken for adsorption (l), equal to 50 mL, C_0 is the molar concentration of ions in the solution before adsorption, C_e is the molar concentration of ions in the solution after adsorption.

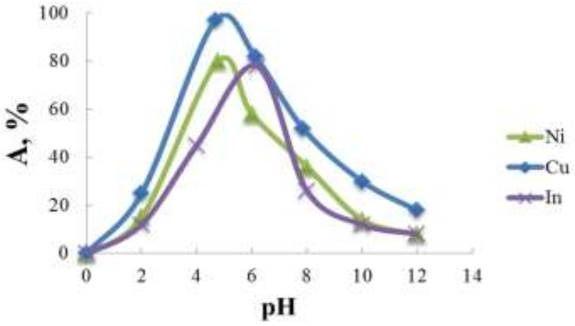


Figure 4 Dependence of the relative sorption of Ni^{2+} (1), Cu^{2+} (2), and In^{3+} (3) on pH

Figure 4 shows the dependence of the degree of sorption of copper, indium, and nickel ions on the solution pH. As can be observed from the graph, the maximum degree of sorption of metal ions is observed in a slightly acidic medium. The maximum sorption of Cu(II) and Ni(II) is observed at pH = 4.8 and of In(III) ions at pH = 6.28.

To investigate the sorption of metal ions in a polyampholyte, we used solutions of metal ions with pH determinations among. Based on the experimental results, an adsorption isotherm was constructed as a graph showing the dependence of the specific adsorption on the equilibrium concentration of the adsorbent at a constant temperature.

3.2.1. Langmuir isotherm

Figures 5–7 show the sorption isotherms of metal ions using the polyampholyte at various temperatures. With the increase in the metal-ion concentration and temperature of the initial solution, sorption increases. At the same time, the SEC values for synthetic solutions of Cu(II), Ni(II), and In(III) ions are not inferior to those of known ion-exchange materials.

It is known that, on the basis of the heterogeneous chemical processes of addition and the occurrence of substitution, including a fixed number of different functional groups, when considered on the basis of the law of mass equations of action, they are characterized by the presence of "Langmuir" properties (Belkhouche and Didi, 2010).

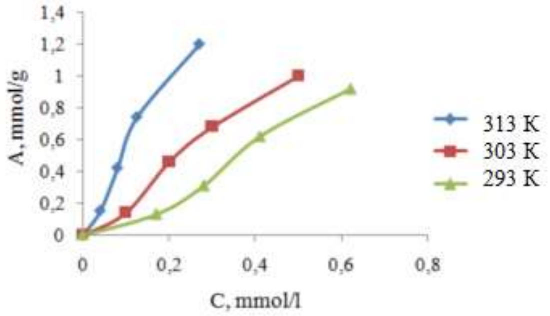


Figure 5 Sorption isotherms of Cu^{2+} with polyampholyte PPE-1-P at different temperatures

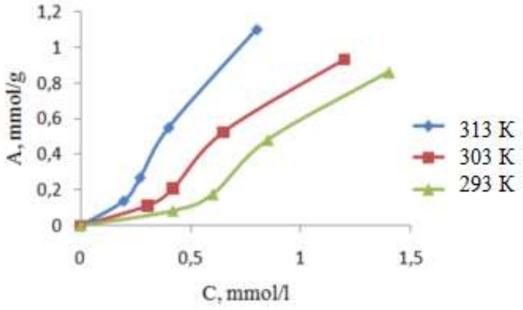


Figure 6 Sorption isotherms of Ni^{2+} with polyampholyte PPE-1-P at different temperatures

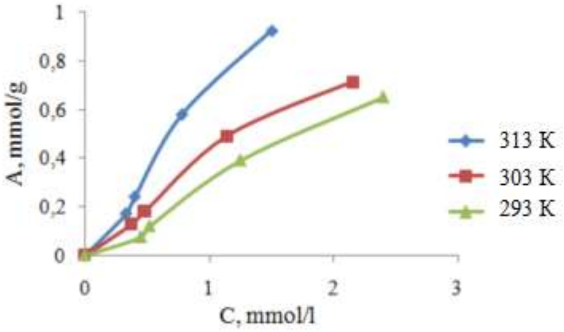


Figure 7 Sorption isotherms of In^{3+} with polyampholyte PPE-1-P at different temperatures

Table lists all of the calculated results for the sorption isotherm by the Langmuir equations. Correlation coefficient (R^2) values are 0.8575 for Cu^{2+}, 0.9851 for Ni^{2+}, and 0.9814 for In^{3+}. In addition, the change in concentration indicates that the adsorption conforms to the Langmuir monomolecular adsorption.

A Linear plot for the Langmuir isotherm is observed for the sorption of copper(II), nickel(II), and indium(III) ions on PPE-1-P. From the linear trend line on the 1/A versus 1/Ce graph, A_{max} and K_L can be obtained from the slope and intersection, respectively. Table lists the values of these calculated constants, together with the linear regression coefficients. From the experiments, the maximum retention capacity (A_{max}) values for copper(II), nickel(II), and indium(III) ions are 3.35 mmol/g, 2.74 mmol/g, and 2.36 mmol/g dry, respectively, gum, and the R_L values for the sorption of copper(II), nickel(II), and indium(III) are 0.91, 0.95, and 0.94, respectively.

Table 1 Sorption constants and basic thermodynamic characteristics for sorption of metals ionspolyampholyte

Metal ions	T, K	A_{max}, mmol/g	K_L, l/mmol	R_L	R^2	−ΔG, J/mol	−ΔH, J/mol	−ΔS, J/mol·K
Cu^{2+}	293	3.05	9.28	0.91	0.8575	4385	12500	13.8
	303	3.32	14.0			4892		28.2
	313	3.85	21.7			6038		29.7
Ni^{2+}	293	2.25	6.35	0.95	0.9851	3590	11500	13.0
	303	2.48	8.43			4310		25.0
	313	2.74	10.84			4597		25.8
In^{3+}	293	1.85	4.18	0.94	0.9814	2170	11200	11.8
	303	2.12	6.54			3127		21.6
	313	2.36	8.45			3670		23.1

Stability constants of the surface complexes formed during the sorption of metal ions on a polyampholyte containing amino and phosphorous groups reveal that copper(II) forms more stable complexes with functionalized sorbents compared to the other ions.

The change in the thermodynamic equilibrium parameters of the experimental results reveals negative ΔG values at all temperatures, indicative of the spontaneous sorption of metal ions. Negative ΔH values indicated that the ion exchange of copper(II), nickel(II), and indium(III) ions to the polyampholyte is an exothermic process. The decrease in entropy with increasing temperature during sorption indicates that the adsorption capacity of metal ions in solution in the polyampholyte increases with increasing temperature. These results reveal that the polyampholyte prepared using granular polyvinyl chloride can be used as an effective sorbent for the extraction of copper(II), nickel(II), and indium(III) ions, causing water hardness.

4. Conclusions

Under laboratory conditions for the obtained polyampholyte, the basic physicochemical properties established in the standard state were examined and compared with those of competitive ion exchangers. The as-obtained polyampholyte was not inferior to that used in the industry. From the kinetics and thermodynamic investigation of the extraction of Cu(II), Ni(II), and In(III) ions by the polyampholyte derived from granular polyvinyl chloride, the sorbent selectivity decreased in the order of Cu(II) > Ni(II) > In(III). This result was related to the strong coordination bonds of copper(II) ions with functional groups in the polyampholyte.

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