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Adsorption and electrokinetic studies of sodalite/lithium/poly(acrylic acid) aqueous system

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Abstract: The synthetic zeolite-sodalite obtained by the hydrothermal conversion of fly ash with aqueous sodium hydroxide was used in the experiments. Its adsorption properties in relation to lithium ions were examined. The effects of: solution pH, presence of polymeric substance – poly(acrylic acid) and order of individual adsorbates addition were determined. To specify the binding mechanism of lithium ions on the sodalite surface, besides adsorption experiments, the measurements leading to the solid surface charge density and zeta potential determination, were performed. As a result, the structure of mixed adsorption layer composed of polymer+metal complexes was characterized. The presented study concerns two important issues: management of environmentally harmful wastes such as coal combustion products as well as searching for new sources of lithium and effective methods of its acquisition.

Keywords: sodalite, zeolite from fly ash, lithium acquisition, zeta potential, solid surface charge density, poly(acrylic acid) adsorption, mixed adsorbates

1. Introduction

Zeolites are a group of microporous aluminosilicates of alkali metals (from I or II group) in the hydrated form, having a crystalline structure - the water molecules are present in the crystal lattice of the zeolite particles (Maesen and Marcus, 2001). The elemental unit of their skeleton are AlO_4 and SiO_4 tetrahedrons. They are connected with oxygen and create three-dimensional structures called polyhedra with a total negative charge (McCusker and Baerlocher, 2001). Polyhedra charge is compensated by metal cations of the first or second group tetrahedrally arranged in the network, having a certain freedom of movement. Depending on the number of tetrahedrons connected together, different zeolite crystallographic structures are formed. A characteristic feature of this group of aluminosilicate minerals is the presence of a network of channels and pores forming spaces filled with water. This is called "zeolite water" whose molecules are attached to the zeolite skeleton through a dipole interaction. In the free spaces, in addition to water molecules, the alkali metal cations, exhibiting high mobility and being easily replaced by other ions, can be collected. As a result, zeolites have ion exchange capabilities, i.e. they adsorb specific ions and molecules selectively. Moreover, the highly developed specific surface of

zeolites makes that these solids are characterized by excellent sorption properties and are used as molecular sieves (Flanigen, 2001; Franus and Wdowin, 2010).

Taking into account the origin, there are natural and synthetic zeolites. Natural microporous crystalline aluminosilicates occur in the form of minerals formed from volcanic ash or as a result of magma crystallization (magmatic zeolite). Volcanic ash, which is an acidic volcanic glaze, creates zeolite deposits by settling in the areas of alkaline lakes, where they react with the present salts. The resulting mineral deposits settle in the crevices and spaces of the rocks or are part of the tuffs (pyroclastic rocks formed from the material of volcanic origin - ashes, dusts and sands, which are connected with each other by means of clay or silica binder). In such a way zeolite tuffs containing clinoptilolite are formed. Synthetic zeolites are obtained as a result of synthesis processes using fly ash as a substrate. They are most often obtained applying the hydrothermal method. It involves the synthesis of fly ash in an alkaline solution, under the high temperature conditions over 100 °C, at the atmospheric or increased pressure. Fly ash reacts in the aqueous solution with NaOH, which is designed to dissolve the aluminosilicate enamel contained in the ash. Then crystallization and separation of aluminosilicate gel occurs on the ash surface. Depending on the parameters such as temperature, pressure, synthesis time, base concentration, zeolites with different structures are obtained as the final product – for example sodalite, Na-A, Na-P1 and Na-X type zeolites (Wdowin et al., 2014; Franus et al., 2014).

The growing demand for lithium is observed nowadays. For this reason the search for new sources of lithium is very important, particularly in the context of the production of lithium ion batteries characterized by high energy density and low weight (Languang et al., 2013). Besides the lithium recovery process from ores (lepidolite, spodumene, petalite, amblygonite) and sea water (Swain, 2017; Suzuki et al., 2017), the geothermal water has been recently considered as a potential lithium source (Tomaszewska et al., 2017). It was determined that the concentration of lithium ions in the geothermal sources reaches 16 mg/dm³ and is higher than that in the sea water (Siekierka et al. 2018). Thus, the studies of the mechanism of adsorptive lithium binding on the surface of various adsorbents (also those modified e.g. with polymer) may contribute to the development of effective separation methods of this element which is considered as “a chemical source of electricity”.

Taking the above into consideration, the main aim of the present study was determination of lithium ions uptake from aqueous solution using synthetic zeolite – sodalite. The impact of pol(acrylic acid) – PAA polymer presence on this process was also studied. The adsorption and electrokinetic properties of in relation to the system containing mixed adsorbates, namely PAA polymer and inorganic lithium ions, were determined. The effect of the order of individual adsorbates addition was examined. Moreover, the solution pH influence was also studied. The applied sodalite was obtained from fly ash, originating from conventional coal combustion. In such a way, the hazardous waste material has been transformed into an effective sorbent which can be reused e.g. for lithium ions accumulation from geothermal water. Our previous investigations proved the effective applicability of other zeolites – natural clinoptilolite and synthetic Na-X zeolite for this purpose (Wiśniewska et al., 2018).

2. Materials and methods

2.1. Sodalite, polymer and lithium characteristics

Sodalite was prepared on a quarter-technical scale using a prototype installation for the synthesis of zeolites from fly ash located at the Lublin University of Technology. This solid was obtained by the hydrothermal conversion of fly ash with aqueous sodium hydroxide (Wdowin et al., 2014). Fly ash originated from conventional coal combustion. Fly ash came from the Jaworzno Power Plant (Poland). The textural parameters of the sodalite were determined by the BET method (ASAP 2020 analyzer, Micromeritics Instrument Corporation). The adsorption/desorption isotherms of nitrogen vapour were measured at the temperature of liquid nitrogen (77 K) for the relative pressures p/p_0 changing from $1.7 \cdot 10^{-7}$ to 0.991. The textural parameters of synthetic zeolite are as follows: BET surface area – 53.6 m²/g; micropore area – 28.7 m²/g; pore volume – 0.23 cm³/g; micropore volume - 0.013 cm³/g; mean pore diameter – 17.5 nm and nanoparticle size – 11.2 nm. The elemental composition of zeolites was determined using the XRF technique (Panalytical ED-XRF type Epsilon 3 spectrometer). These studies were performed in the Na-Am range using the apparatus equipped with the RTG tube Rh 9W (X-ray energy 50 kV). The obtained data are presented in Fig. 1.

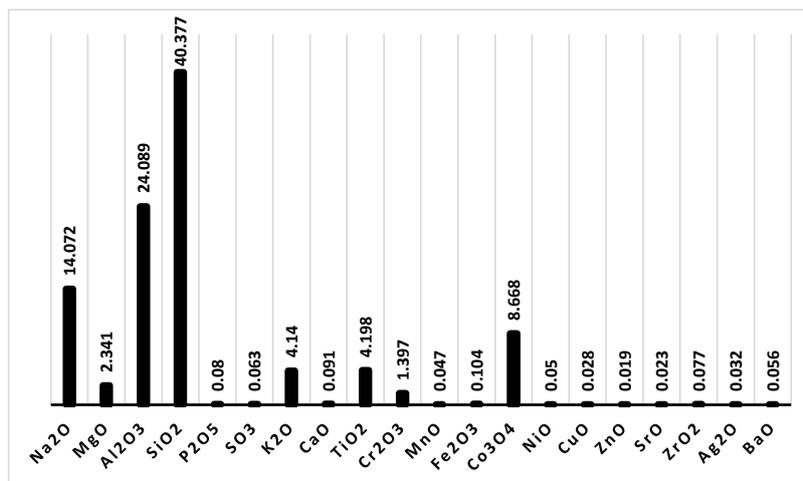


Fig. 1. Elemental composition [%] of sodalite

Poly(acrylic acid) – PAA with the weight average molecular weight 240 kDa was supplied by Sigma-Aldrich. This is an anionic polymer containing the carboxyl groups -COOH which dissociate in aqueous solutions. PAA is a weak acid with $pK_a = 4.5$ (Chibowski et al., 2003) which means that at pH 4.5 half of the functional groups is dissociated (dissociation degree is equal to 0.5). The following equation allows to determine the degree of the carboxyl groups dissociation (α):

$$pH - pK_a = \log \frac{\alpha}{1-\alpha} \quad (1)$$

Fig. 2 shows the dependence of PAA dissociation degree on the solution pH. As the pH increases, the dissociation of the polymer increases and more numerous carboxyl groups are in the ionized form (at pH 6 $\alpha = 0.97$ and at pH 9 $\alpha = 0.99$). Only at pH 3 dissociation is minimal and $\alpha = 0.33$. During the measurements there was also used the LiCl solution (Sigma-Aldrich) with the initial concentration of 1000 mg/dm³.

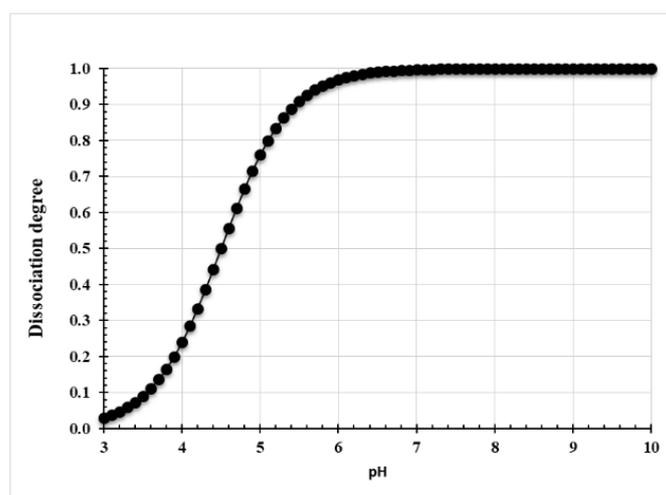


Fig. 2. Dissociation degree of PAA as a function of solution pH

2.2. Adsorption measurements

The adsorption of lithium ions and poly(acrylic acid) on the surface of sodalite was performed at pH 3, 6, 4.5 and 9 ± 0.1 , which are equilibrium values, at 25 °C by the means of the static method. Based on the difference between the adsorbate concentrations in the solution before and after the adsorption process its adsorbed amount was calculated from the following formula:

$$\Gamma = \frac{c_{ads} \cdot V}{m} \quad (2)$$

where: c_{ads} is the difference in the adsorbate concentration before and after the adsorption process, V is the volume of suspension, m is the mass of zeolite.

The HCl (Chempur) and the NaOH (POCh) solutions in the concentration range from 0.1 to 1 mol/dm³ were used for pH adjustment (pH - Φ360 pH/temperature/mV Meter, Beckman). The analysis of lithium ions adsorbed on the zeolite surface was made using an ICP-OES spectrometer. The adsorbed amount of poly(acrylic acid) on the zeolite surface was determined according to the method proposed by Crummet and Hummel (1963). It is based on the spectrophotometric measurements of turbidity coming from the reaction of PAA with hyamine 1622 solution with 1% concentration (BDH Chemicals Ltd). The zeolite weight in each examined system was 1 g per 10 cm³ of the solution. The prepared suspensions were shaken in the water bath (OLS 200 Grant) for 24 hours. Then the solids were centrifuged with the use of a micro-centrifuge (type MPW-223e, MPW Med Instruments) and the clear polymer solution was collected for the reaction with hyamine. The resulting turbidity was measured after 15 minutes using the UV-VIS spectrophotometer (Carry 100, Varian) at 500 nm. At the very beginning the calibration curve presenting absorbance changes versus PAA concentration (varying in the range 10-200 ppm was obtained).

The examined initial concentrations were: 100 ppm for PAA and 50 ppm for Li ions. The effect of the order of both adsorbates addition (PAA and Li ions) was also studied – these adsorbates were added at the same time (simultaneously) and 1 hour after the other.

2.3. Potentiometric titrations

The surface charge density of sodalite (without and with adsorbates) was determined at 25 °C using the potentiometric titration method (Janusz 1994). The measuring set consisting of a thermostated Teflon vessel, a thermostat RE 204 (Lauda), calomel and glass electrodes (Beckman Instruments), a pH-meter PHM 240 (Radiometer), laboratory stirrers and an automatic micro-burette (765 Dosimat, Methrom) as well as the special computer program Titr_v3 (author W. Janusz) was applied. The NaOH solution of 0.1 mol/dm³ concentration was used as the titrant. The obtained results enabled determination of pH_{pzc} (pzc – point of zero charge) values for the examined systems. The aqueous suspension containing 0.41 g of the solid in 50 cm³ of appropriate solution was prepared. The solid surface charge density σ_0 (μC/cm²) was calculated by the computer programme based on the following equation:

$$\sigma_0 = \frac{\Delta V \cdot c_b \cdot F}{m \cdot S_w} \quad (3)$$

where: c_b is the base (NaOH) concentration (mol/dm³), F is the Faraday constant (C/mol), m is the solid mass in the suspension (g), S is the specific surface area of the solid (m²/g), ΔV is the difference in the volume of base which must be added to bring the pH of suspension and reference solution (water) to the specified value (dm³).

To determine the ΔV value the titration curve of the basic electrolyte must be obtained at the beginning. Next, the appropriate suspension titration curves were prepared.

The examined initial concentrations were: 100 ppm for PAA, whereas for Li ions - 10 and 50 ppm (both adsorbates were added simultaneously).

2.4. Electrophoretic mobility measurements

The electrophoretic mobility U_e (cm²/Vs) of sodalite particles (without and with adsorbates) was measured at 25 °C using the Doppler laser electrophoresis technique applying Zetasizer Nano ZS (Malvern Instruments) containing a universal dip cell. The zeta potential ζ (mV) was calculated with the special computer program coupled with the zetameter using the Henry equation (Hunter 1988):

$$U_e = \frac{2\varepsilon_0\varepsilon\zeta}{3\eta} f(\kappa a) \quad (4)$$

where: ε is the dielectric constant, ε_0 is the electric permeability of vacuum (F/m), η is the viscosity of liquid medium (Pa·s), $f(\kappa a)$ is the Henry function.

The determination of pH_{iep} value of the examined system (iep – isoelectric point) was also possible. The suspensions were prepared by the addition of 0.03 g of the zeolite to 100 cm³ of the appropriate solution. After the 3-minute sonification (XL 2020 sonificator, Labcaire System Limited) the obtained

suspension was divided into 7 parts. In each of them the appropriate solution pH value was adjusted, namely: 3, 4, 5, 6, 7, 8, and 9 (with the accuracy of 0.1). In the case of suspensions containing the polymer, it was introduced to the system immediately after sonification process.

In the systems containing both adsorbates, they were added simultaneously (the initial concentrations were: 100 ppm for PAA, whereas for Li ions - 10 and 50 ppm).

3. Results and discussion

Fig. 3 presents the adsorbed amount of lithium cation on the sodalite surface at four examined pH values (3; 4.5; 6 and 9) of the solution in the systems not containing and containing poly(acrylic acid). As can be seen adsorption of Li^+ increases with the pH rise and at pH 9 it reaches a maximum level, corresponding with 40% of lithium removal from the aqueous solution. The possibilities of lithium cation binding with the zeolite surface depend mainly on the sign and magnitude of its charge. The potentiometric titration results presented in Fig. 4 indicate that the pH_{pzc} value for sodalite is about 9.9. The positive charge of zeolite surface up to pH approx. 10 results from compensation of negative charge of three-dimensional structure of AlO_4 and SiO_4 tetrahedrons by metal cations of the first or second group.

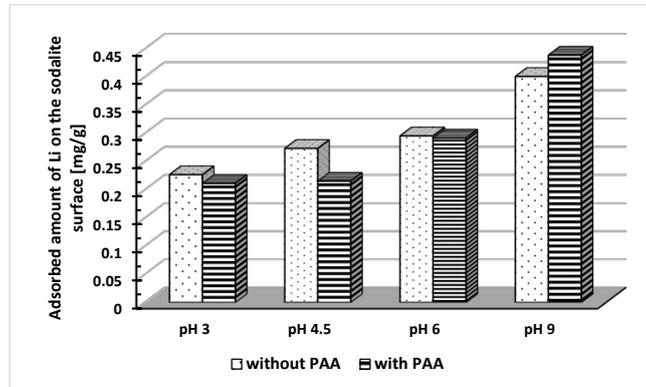


Fig. 3. pH dependence of adsorbed amount (Γ) of lithium ions on the sodalite surface without and with PAA; C_{Li} 50 ppm, C_{PAA} 100 ppm; both adsorbates were added simultaneously

This suggests that within the studied adsorption pH range (3-9) there is the electrostatic repulsion between the positively charged solid surface and the lithium cations. As the pH increases this repulsion is slighter and the adsorption of inorganic ions increases.

In the case of sodalite, the other important mechanism of adsorption is the ion exchange of Li^+ with cations such as Na^+ , Mg^{2+} , Ca^{2+} . The decreasing charge of zeolite with the increase of pH causes that lithium ions can penetrate into the solid pores and chambers more easily, which favours the process of effective ion exchange and the increase of Li adsorption.

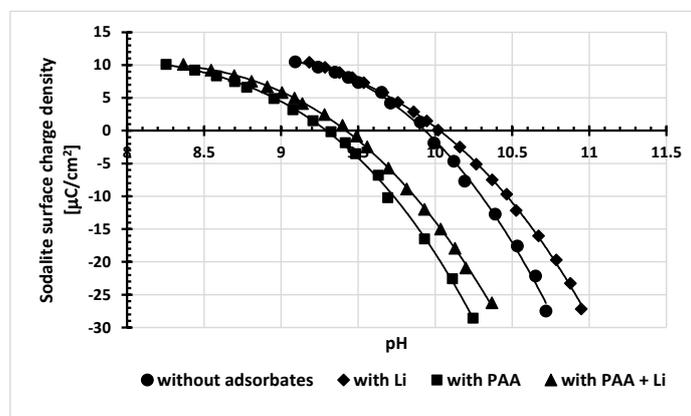


Fig. 4. pH dependence of sodalite surface charge density (σ_0) without and with Li ions or/and PAA; C_{Li} 50 ppm, C_{PAA} 100 ppm; both adsorbates were added simultaneously

The slight increase of the sodalite surface charge density in the lithium ions presence (Fig. 4) indicates the accumulation of Li cations in the by-surface layer of the solution, resulting from their adsorption at the solid/liquid interface. This effect is more visible at pH higher than pH_{pzc} due to the electrostatic attraction between the negatively charged zeolite surface and the lithium cations.

The addition of poly(acrylic acid) with anionic character causes inconsiderable modification of the zeolite surface and influences the Li^+ adsorbed amount slightly (Fig. 3). At pH 3 practically neutral macromolecules of PAA (minimal dissociation of their carboxyl groups), undergo adsorption on the zeolite surface in the form of polymeric coils mainly through hydrogen bonds (Wiśniewska and Nowicki, 2019; 2020). Such structure of the adsorbed polymer confines the Li ions access to the zeolite pores and the lithium adsorption increases slightly. The similar effect is observed at pH 4.5, at which the conformation of PAA chains is looser (dissociation degree equal to 0.5), but the polymeric molecules have still the form of coils. With the pH increase, the conformation of poly(acrylic acid) becomes more and more developed due to the total ionization of polyacid chains. As a consequence, significant development of the adsorbed macromolecules is observed (especially at pH 9, at which the electrostatic attraction between the PAA chains and the zeolite surface is the slightest). Such structure of polymeric layer allows Li^+ cations to enter the zeolite channels. Additionally, the negatively charged polymer chains can bind metal cations effectively forming intra- and inter-molecular complexes (Fig. 5). As a result, at pH 9 an increase in the amount of adsorbed lithium ions in the presence of polymer compared to the system without PAA is observed.

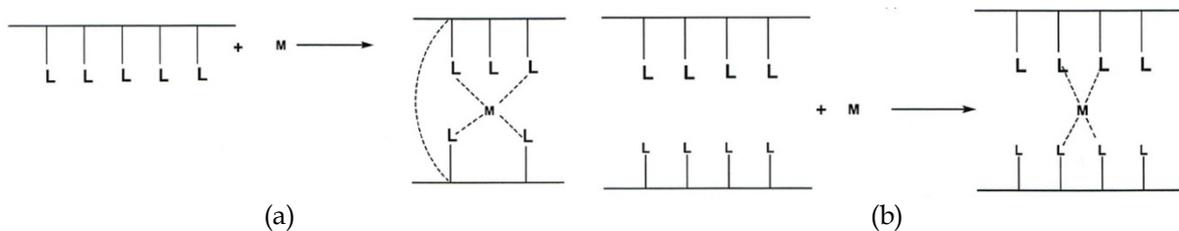


Fig. 5. Structure of polymer-metal complexes: (a) intra-molecular, (b) inter-molecular (El-Sonbati et al., 2012)

In the zeolite suspensions containing PAA or mixed adsorbates (PAA+Li) the considerable decrease of the solid surface charge density (in comparison to the system without adsorbates) in the whole examined pH range is observed (Fig. 4). The main reason for such behaviour is the presence of dissociated carboxyl groups of the adsorbed polymer at the solid-liquid interface. They belong to the segments located in the tail and loop structures, which are not directly bound with the zeolite surface (Wiśniewska et al., 2016). This effect becomes more and more visible when pH rises because in such a situation the number of ionized PAA groups increases. In the systems containing PAA and Li^+ mixed adsorbates the zeolite surface charge density is minimally higher than that of the polymer containing system. Thus, the polymer-metal complexes present in the interfacial layer assume such a spatial arrangement that the lithium ions cause insignificant changes in the surface charge density of the examined aluminosilicate.

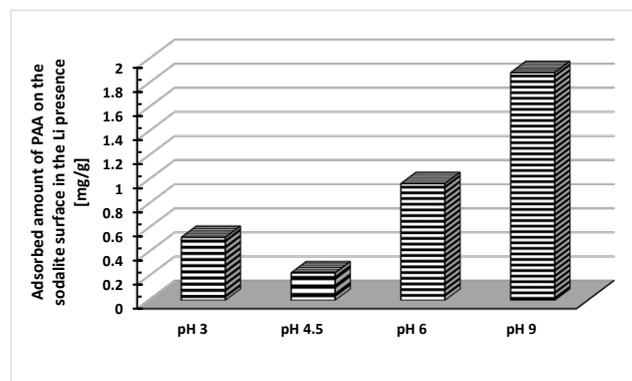


Fig. 6. pH dependence of adsorbed amount (Γ) of PAA on the sodalite surface in the Li ions presence; C_{Li} 50 ppm, C_{PAA} 100 ppm; both adsorbates were added simultaneously

The analysis of the data presented in Fig. 6 leads to the conclusion that in the system of mixed adsorbates the PAA adsorbed amount in the lithium ions presence is the highest at 9 (above 90%). This proves that the PAA-Li complexes formation is the most effective at pH 9 which is consistent with the adsorption data obtained for lithium ions in the polymer presence.

The dependence of Li^+ adsorption on the order of individual components addition (at pH 3 and 4.5) is presented in Fig. 7. The amount of adsorbed lithium ions is the smallest in the case when Li^+ and PAA were added to the system together. This is caused mainly by the fact that lithium and polymer form complexes in the solution even before their adsorption on the zeolite surface.

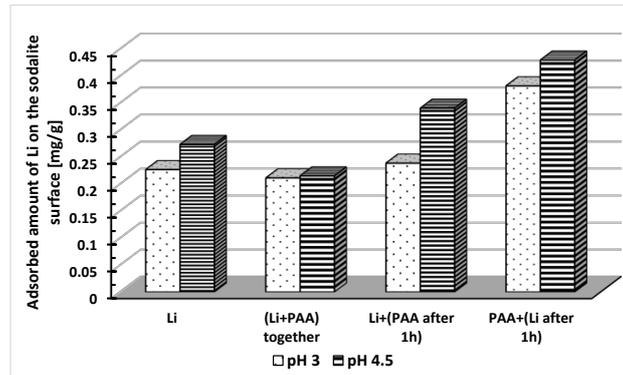


Fig. 7. Effect of the order of adsorbates addition on the adsorbed amount (Γ) of Li ions on the sodalite surface at pH 3 and 4.5; C_{Li} 50 ppm, C_{PAA} 100 ppm

The greatest increase of Li ions adsorption was obtained in the case of the lithium addition after 1 hour after the polymer introduction (at pH 4.5). As it was mentioned above, when both adsorbates are added simultaneously the adsorbing polymer coils with more or less loose structure can block entrances to the zeolite pores and lithium ions can not freely penetrate into their interior. When the polymer is added as first, their coiled chains “have time” to their reformation for an hour. In such a situation the structure of the adsorbed polymer undergoes some development due to the interactions of partially dissociated macromolecules with the positively charged solid surface. As a result, more PAA carboxyl groups are available for creation of complexes with Li ions, which are added with an hour delay. Moreover, the entrances to the sodalite pores are more accessible to the lithium ion penetration.

The presence of adsorption layers of PAA and Li, as well as mixed PAA+Li ones influences also the zeta potential of zeolite particles (Fig. 8). This parameter characterizes the total charge accumulated in the area of slipping plane formed around the solid particle. The isoelectric point (iep) of sodalite without adsorbates is placed at pH 5 (the zeta potential assumes the zero value).

The greatest changes in the electrokinetic properties of solid particles are observed when they are dispersed in the mixed solution of both adsorbates. In such a case a considerable decrease of zeta poten-

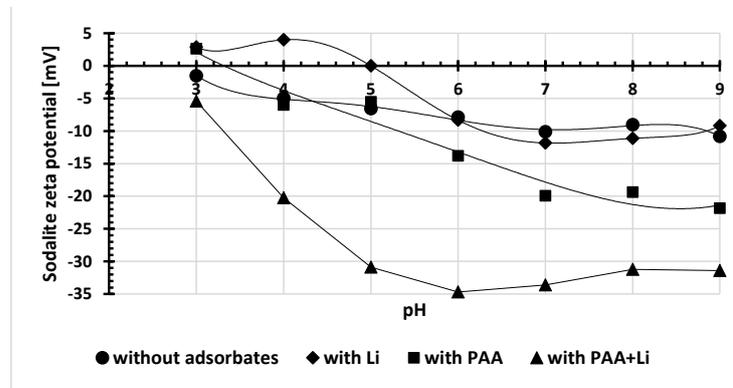


Fig. 8. pH dependence of sodalite zeta potential (ζ) without and with Li ions or/and PAA; C_{Li} 50 ppm, C_{PAA} 100 ppm; both adsorbates were added simultaneously

tial in the whole range of examined pH takes place. The main reason for this behaviour is a significant shift of the slipping plane from the zeolite surface, resulting from formation of thick adsorption layers of both adsorbates (M'Pandou and Siffert, 1987). The polymer-metal complexes participate in this process to a large extent. The other important factor can be the presence of negatively charged functional groups of adsorbed poly(acrylic acid) macromolecules in the slipping plane area (Wiśniewska et al., 2015). They belong to the polymeric chains adsorbed both separately and in the form of PAA+Li complexes.

4. Conclusions

The lithium ions adsorbed amount on the surface of synthetic zeolite - sodalite depends on the solid textual properties, the solution pH value, the presence of anionic poly(acrylic acid) and the order of individual adsorbates addition. The increase in the solution pH results in an increase of lithium cations adsorption (due to the electrostatic repulsion decrease, Li ions exchange with other cations present in the zeolite structure is facilitated). In addition, the pH rise results in the increase of dissociation degree of the polymer functional groups, which leads to the development of PAA chains. This favours more effective formation of polymer-metal complexes and lithium ions acquisition. Moreover, the simultaneous addition of metal ions and poly(acrylic acid) macromolecules results in the decreasing lithium removal from the liquid medium compared to the system in which PAA or Li⁺ was added after an hour. The adsorption layers of single or mixed adsorbates influence the electrokinetic properties of sodalite particles. This is manifested by distinct changes in the values of both solid surface charge density and its zeta potential. It was proved that synthetic zeolite obtained from the waste material shows good adsorption properties for acquisition of lithium ions from the aqueous solution under specific conditions and it can be successfully used for potential extraction of lithium ions from the geothermal water.

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