

UDC 628.349

DOI: 10.15372/CSD20180404

Sorption of Heavy Metals with Urea-Containing Sorbent Made of Aspen Bark

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(Received April 20, 2018; revised July 13, 2018)

Abstract

The sorption activity of urea-containing sorbent made of aspen bark was explored against nickel, zinc, and lead from aqueous solutions with different concentrations of metals was examined. The effect of urea content in aspen bark sorbent on its sorption activity against the impurities under examination was revealed. It was demonstrated that the adsorptive coating of 10.7 mass % of urea onto the aspen bark carrier led to an increase in sorbent capacity by 1.9–3.3 times depending on metal nature. It was determined that the produced aspen bark sorbent was characterized by high sorption capacity. Maximum sorption of lead, zinc, and nickel is 91.17, 25.78, and 23.34 mg/g, respectively. Metal sorption kinetics data and its analysis using pseudo-first and pseudo-second order models are given. The pH values of aqueous solutions ensuring efficient sorption of the examined metals (pH 5.5–6.0 and pH 5.5–7.0 for zinc, and also lead and nickel solutions, respectively) were determined. The efficiency of use of urea-containing sorbent made of aspen bark to purify low concentration metal solutions was shown. For solutions containing 10.5 and 1.0 mg/L of impurities, the degree of purification is not less than 66.7 and 89.9 %, correspondingly. When processes of nickel, zinc, and lead sorption were explored, the resistance of the developed urea-containing sorbent towards the washing-out of urea was revealed. When combined with high sorption capacity; this defines the prospects of its use for additional purification of water for technical purposes from metals impurities.

Keywords: aspen bark, urea-containing sorbent, nickel, zinc, lead, sorption

INTRODUCTION

Environmental pollution with heavy metals represents a danger for the stable functioning of various objects of the biosphere. The main source of their ingress is run-offs from different productions, runoffs from waste storages of industrial wastes, *etc.* Timely and efficient wastewater treatment prevents environmental pollution. Sorption methods occupy an important place in modern technologies of complex removal of heavy metals from aqueous media [1]. Moreover, inexpensive and available materials, such as wastes from processing of agricultural complex, wood conversion, and plant biomass waste, may be used as sorbents [2–5].

As demonstrated by a literature review, the use of wood conversion waste, including bark wastes in order to make new sorbents to remove heavy metals remains, remains a promising area of their utilization. It is worth noting that the chemical modification of lignocellulosic materials facilitates a significantly increased sorption capacity of sorbents against different metals [9, 10].

Paper [11] demonstrates that treatment of cellulose waste (sawdust, flax, *etc.*) with a mixture of urea and phosphoric acid allows producing sorbents capable of absorbing copper, zinc, cadmium, nickel, and other metals from aqueous solutions. Such nitrogen-containing sorbents are efficient in air purification from formaldehyde and

ammonia impurities. It was determined that treatment of aspen bark with an aqueous solution of urea improved its sorption properties during treatment of water solutions from copper, nickel, zinc, and lead [12]. Furthermore, the content of polyphenolic water-soluble compounds in the modified bark is decreased, which enhances sorbent use safety. Research results [13] proved the high efficiency of urea sorbents to remove copper and lead from aqueous media. Literature data point to the potential of urea as a modifying substance to produce efficient biomass waste-based sorbents. Multi-tonnage aspen bark wastes are inexpensive and affordable raw material resources to make sorption materials for various purposes. There have been suggested a method to obtain porous aspen bark substrates, based on which various biocomposite materials may be designed [14]. The former have strong texture due to the removal of polyphenolic substances of the substrate, which allows their use as sorbents.

The purpose of the research was to examine sorption of nickel, zinc, and lead from aqueous solutions with aspen bark-derived urea-containing sorbent.

EXPERIMENTAL

Air-dry aspen bark was the initial raw materials to produce the sorbent. The mass ratio of peel and bast therein was 60.6 and 39.4 mass %, respectively. The bark was milled using a Nossen desintegrator (Germany); a 0.5–1.0 mm fraction was selected and treated with a 1 % aqueous solution according to the technique described in [14]. The hydromodulus value in the first step of the alkaline treatment was 15 and in other steps – 10. The resulting sample was dried until the air-dry state at (85 ± 5) °C and used for urea coating by the sorption method. In order to coat 5.0, 7.5, and 10.7 mass % of urea, its aqueous solutions with concentrations of 1.5, 3.0, and 5.0 % were used. Urea solutions contained a background electrolyte, *i.e.* 1.5 mass % NaCl.

The sorption coating of urea was carried out as follows: 2 g of the bark sample was introduced into 100 cm³ of the prepared solution; process duration was 24 h under room conditions and with periodic stirring. Upon completion of the process, urea samples were filtered off and treated with 0.25 % Na₂SO₄ solution (hydromodulus 3, processing time of 30 min). The salt residues were rinsed with distilled water under the following condi-

tions: double washing with hydromodulus 10, each washing duration of 45 min, room temperature, and stirring. After separation from the wash water, urea-containing sorbents were dried until the air-dry state at (85 ± 5) °C.

The urea content in the resulting sorbents was determined according to its concentration change in initial solutions, considering losses in treatment steps with Na₂SO₄ solution and sorbent aqueous washing. Quantitative analysis of aqueous solutions to determine urea nitrogen (according to biuret content) was carried by the photometric method according to GOST 32555–2013. The amount of urea in the sorbent was computed by the formula:

$$Q_r = 100 (Q_{ur}^1 - Q_{ur}^2)/m \quad (1)$$

where Q_{ur}^1 is the amount of sorbed urea, mg; Q_{ur}^2 is the total amount of urea in the wash water, mg; m is the sample mass, mg.

The nitrogen content in urea-containing sorbents was additionally determined using the FLASH TFTM-1112 analyser (Italy).

Sorption of Zn²⁺, Ni²⁺, and Pb²⁺ was carried out from solutions of the corresponding nitrates under static conditions at room temperature and with continuous stirring. The ratio of the sorbent and metal salt solution was 0.2 g to 50 mL. The metal sorption kinetics was explored under experimental conditions with a 750.0 mg/L initial concentration of metals in solutions; process duration was varied from 5 to 120 min. The latter was 60 min in the remaining experiments. The effect of pH values of solutions for metals sorption was investigated in the pH 3.5–7.0 range. The solution acidity was pH 6.0 in other experiments. Sorption isotherms were obtained using model solutions (pH 6.0) with an 11.5–750.0 mg/L initial concentration of metals.

Changes in Zn²⁺, Ni²⁺, and Pb²⁺ concentrations in model solutions during sorption were determined by the atomic absorption method using the Analyst-400 spectrometer. Their sorption was calculated according to the former. The above technique was also used to determine the urea content after sorption.

The degree of filling (F) of the sorbent surface was determined by the ratio of sorption (A_t) at the time (t) to the equilibrium sorption capacity (A_{eq}) [15]:

$$F = A_t/A_{eq} \quad (2)$$

In order to process data for sorption kinetics of Zn²⁺, Ni²⁺, and Pb²⁺, the models of the pseudo-first

$$A_t = A_{eq} (1 - e^{-k_1 t}) \quad (3)$$

and pseudo-second orders were used:

$$t/A_t = (k_2 A_{\text{eq}}^2)^{-1} + t/A_{\text{eq}} \quad (4)$$

where A_t is sorption at time t , mg/g; A_{eq} is equilibrium sorption, mg/g; t is time, min; k_1 and k_2 are sorption rate constants of the model of the pseudo-first (in min^{-1}) and pseudo-second orders (in $\text{g}/(\text{mg} \cdot \text{min})$), respectively.

According to the Langmuir sorption isotherm equation given the linear form, the corresponding coefficients for sorption processes of the investigated metals were computed by the least squares method [12]:

$$A^{-1} = (A_{\infty} K_L)^{-1} C_{\text{eq}}^{-1} + A_{\infty}^{-1} \quad (5)$$

where A is sorption, mg/g; A_{∞} is limit sorption, mg/g; C_{eq} is equilibrium concentrations of Zn^{2+} (Ni^{2+} and Pb^{2+}) mg/L; K_L is Langmuir equation constant, L/mg.

RESULTS AND DISCUSSION

It was determined that increasing sorption of the investigated metals with urea-containing sorbent made of aspen bark was dependent on the urea content therein and the nature of the investigated metal ion (Table 1).

When increasing the urea content in the sorbent as high as 7.5 mass %, sorption of zinc and lead increases by 1.7 times, and that of nickel – by 1.26 times. The maximum amount of urea that was possible to coat by the adsorption method was 10.7 mass %. Herewith, sorbent sorption capacity against nickel, zinc, and lead increased by 1.9, 2.2, and 3.3 times, respectively (see Table 1).

An increase in sorption of metals with a sorbent based on urea is driven by its ability to form complexes with the investigated metals. For example, the literature describes zinc complexes containing from 1 to 2 urea molecules [16, 17]. Nickel complexes may contain till ten urea molecules in the coordination sphere [18]. Apparently, this is the difference in the composition of complexes that determines different sorption capacities against Ni^{2+} and Zn^{2+} .

As shown by the analysis of data in Fig. 1, the equilibrium for nickel and zinc sorption with the urea fragment (10.7 mass % of urea) is set faster (for 40 min) compared to the lead sorption process. In the case of Pb^{2+} sorption, equilibrium is reached for 60 min. The half-sorption time of nickel, lead, and zinc is 8 min, 9.5, and 11 min, respectively.

As demonstrated by the presentation of the findings in $F - t^{1/2}$ coordinates, nickel sorption is

TABLE 1

Sorption capacity of aspen bark sorbent with different urea content

| Urea content in sorbent, mass % | Sorption ⁺ , mg/g | | |
|---------------------------------|------------------------------|------------------|------------------|
| | Ni ²⁺ | Zn ²⁺ | Pb ²⁺ |
| 0 | 9.74 | 10.37 | 14.47 |
| 5.0 | 9.84 | 11.08 | 15.44 |
| 7.5 | 12.28 | 17.65 | 24.31 |
| 10.7 | 18.25 | 22.43 | 47.55 |

Note. Sorption duration is 60 min; pH 6.0; initial concentrations of metals are 750.0 mg/L.

characterised by the maximum degree of filling the sorbent surface during time till 900 s (Fig. 2).

The linear nature of the initial sections of $F - t^{1/2}$ dependencies points to the significant contribution of intradiffusion processes on sorption of the investigated metals with urea-containing sorbent made of aspen bark.

The subsequent curvature and appearance of linear sections with a smaller inclination angle testify that diffusion in the sorbent pores limits the overall rate of metal sorption processes [15, 19]. Obviously, this effect is more significant in case of lead sorption, as indicated by a pronounced flat area depending on the degree of filling of the sorbent surface F from $t^{1/2}$ (see Fig. 2, curve 3). The revealed effect of diffusion processes on Pb^{2+} sorption is due to its ion radius equal to 1.21 Å (according to Pauling) and exceeding the ion radii of Ni^{2+} and Zn^{2+} (0.69 and 0.74 Å, respectively) by an average of 1.7 times. It is obvious that the size of lead ions complicates their transport to adsorption centres in sorbent pores to a greater extent,

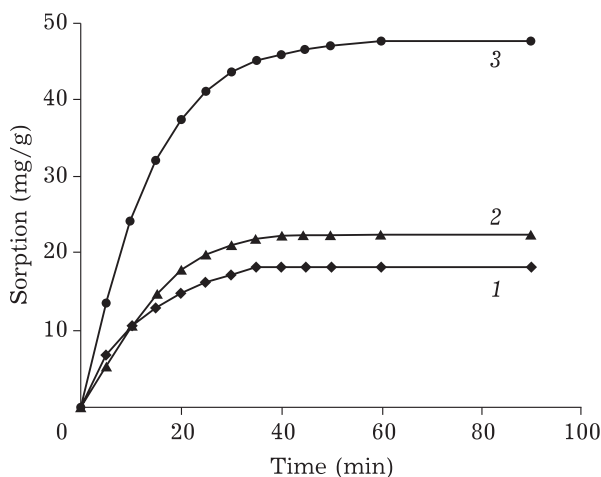


Fig. 1. Sorption kinetics of metals with urea-containing sorbent made of aspen bark: 1 – nickel, 2 – zinc, 3 – lead.

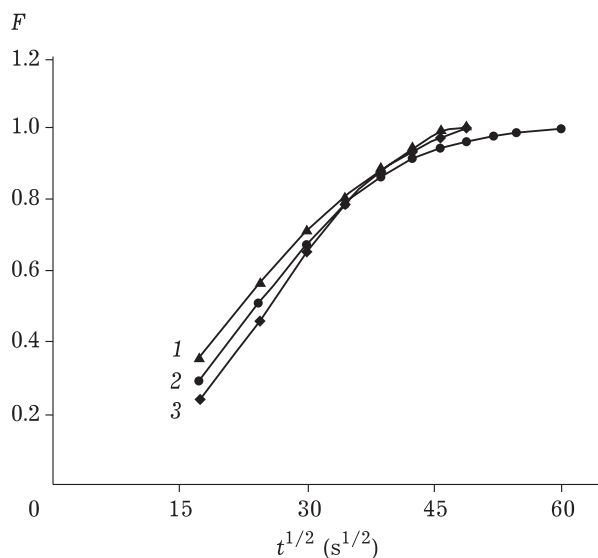


Fig. 2. Curves of the degree of surface filling (F) of urea-containing sorbent made of aspen bark versus metal half-sorption time ($t^{1/2}$): 1 – nickel, 2 – zinc, 3 – lead.

therefore in order to reach sorption equilibrium, by 1.2 longer time is required compared to nickel and zinc sorption.

It is worth noting that the resulting degree of surface filling *versus* $t^{1/2}$ for nickel and zinc stay in the origin (see Fig. 2). Time intervals cut off in the axis of abscissa under the condition $F = 0$ and equal to 12, 83 and 36 s for Ni^{2+} , Zn^{2+} , and Pb^{2+} , respectively, may be due to the transport of metal ions through the diffusion layer on the sorbent surface. Thus, a set of external and internal diffusion processes has an effect on investigated metals sorption [15, 19].

The urea present in the sorbent surface is able to form complexes with the investigated metals, which may also have an effect on the rate of sorption processes. In order to determine the rate-limiting step, the graphic-analytic method of experimental data processing for sorption kinetics of the investigated metals using the pseudo-first (equa-

tion 3) and pseudo-second (equation 4) order models. According to the pseudo-first order model, the limiting step is diffusion processes. The pseudo-second order model assumes that the chemical interaction limits the sorption process. The linear nature of the dependencies in the coordinates $\lg(A_{\text{eq}} - A_t) - t$ or $t/A_t - t$ indicates an opportunity to apply pseudo-first or pseudo-second order models to interpret the resulting kinetic data. It should be noted that these models are most commonly used to describe metal sorption processes on various sorbents [15, 19, and 20].

Table 2 gives approximation results of experimental data for the kinetics of Ni^{2+} , Zn^{2+} , and Pb^{2+} sorption by the selected models.

As demonstrated by the comparison of coefficients of determination (R^2) and computed values of equilibrium sorption (A_{eq}), the pseudo-first order model reliably describes zinc and lead sorption kinetics (see Table 2). Coupled with the data in Fig. 2, this proves that their sorption is monitored by diffusion processes. Nickel sorption kinetics corresponds to the pseudo-second order model ($R^2 = 0.998$), which indicates the effect of their chemical interaction with the sorbent on the total rate of the process. Considering the nature of the graph of $F - t^{1/2}$ determined for Ni^{2+} , one may assume that its sorption with urea-containing sorbent made of aspen bark proceeds in a mixed mode.

It was determined that the pseudo-first order model was more suitable to describe nickel sorption kinetics from solutions with lower concentrations. Higher coefficients of determination were acquired for kinetic data approximation by the pseudo-second order model, but there was a greater discrepancy between computed and experimental equilibrium sorption values (Table 3). With decreasing the content of nickel, the effect of diffusion processes on its sorption is likely to increase.

TABLE 2

Parameters of kinetic equations for sorption of nickel, zinc, and lead by urea-containing sorbent made of aspen bark (initial concentration of metals in the solution of 750.0 mg/L)

| Metal | $A_{\text{eq}}^{\text{exp}}$, mg/g | Pseudo-first order | | | Pseudo-second order | | |
|--------|-------------------------------------|------------------------|-------------------------|-------|------------------------|---|-------|
| | | A_{eq} , mg/g | K , min^{-1} | R^2 | A_{eq} , mg/g | K , $\text{g}/(\text{mg} \cdot \text{min})$ | R^2 |
| Nickel | 18.25 | 40.54 | 0.1566 | 0.816 | 25.06 | 0.0029 | 0.998 |
| Zinc | 22.43 | 28.93 | 0.0923 | 0.994 | 38.61 | 0.0009 | 0.956 |
| Lead | 47.55 | 55.49 | 0.0875 | 0.999 | 60.61 | 0.0012 | 0.989 |

Note. Here and in Table 3: $A_{\text{eq}}^{\text{exp}}$ and A_{eq} are experimental and calculated values of equilibrium sorption, respectively; R^2 is the coefficient of determination.

TABLE 3

Parameters of kinetic equations of nickel sorption from solutions with different initial concentrations of urea-containing sorbent made of aspen bark

| Initial concentration Ni ²⁺ , mg/L | A _{eq} ^{exp} , mg/g | Pseudo-first order | | | Pseudo-second order | | |
|--|---------------------------------------|------------------------|----------------------|----------------|------------------------|-----------------|----------------|
| | | A _{eq} , mg/g | K, min ⁻¹ | R ² | A _{eq} , mg/g | K, g/(mg · min) | R ² |
| 200.0 | 11.75 | 12.78 | 0.0894 | 0.995 | 18.98 | 0.0028 | 0.989 |
| 400.0 | 16.22 | 18.28 | 0.0914 | 0.997 | 24.15 | 0.0024 | 0.991 |

Note. See symbols in Table 2.

Absorption processes of Ni²⁺, Zn²⁺, and Pb²⁺ from solutions at a 11.5–750.0 mg/L initial concentration of metals (pH 6.0) are described by type I isotherms according to the Brunauer–Emmett–Teller (BET) classification, which points out multi-layered sorption of these ions (Fig. 3).

As demonstrated by the data of Table 4, the resulting isotherms are in agreement with the Langmuir equation to a high degree of approximation.

Experimental values of maximum sorption (A_{\max}^{exp}) are close to computed magnitudes of maximum sorption (A_{∞}) in case of nickel and zinc sorption. Their ratio is 0.78 and 0.87, which testifies a high degree of sorption layer filling under examined conditions. Comparison of equilibrium constants (K_L) makes it possible suggests that zinc absorption with aspen bark sorbent is characterized by the higher heat of sorption compared to other metals [2].

It was determined that the effect of the pH of solutions on sorption of the investigated metals on urea-containing sorbents made of aspen bark did not depend on their nature (Fig. 4).

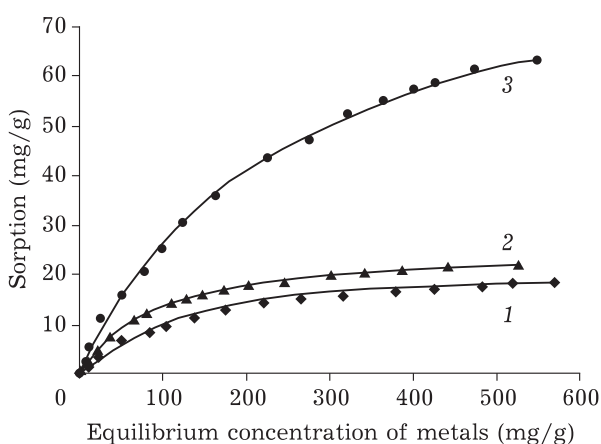


Fig. 3. Metal sorption isotherms from aqueous solutions of urea-containing sorbent made of aspen bark: 1 – nickel, 2 – zinc, 3 – lead; points indicate experimental data and lines are calculation.

There is a significant decrease in nickel, zinc, and lead sorption upon decreasing pH values from 5.0 to 3.5. Already with changing the pH of solutions from 5.5 to 5.0, sorption of nickel decreases by 1.8 times, while those of lead and zinc – only by 1.3 and 1.4 times, respectively. There is also this difference with a pH of 4.5. This is in a good agreement with values of ion indexes of the investigated metals that reflect the degree of involvement of ions into electrostatic interactions [12]. Nickel is characterised by the maximum value of this parameter compared to other metals. It is obvious that Ni²⁺ interaction effects with the sorbent surface, the degree of protonation of which would increase with increasing the acidity of solutions, complicate its sorption to a greater extent.

The degree of protonation of the urea present in the surface of aspen bark sorbent also increases with increasing the acidity of solutions. This stops the addition of metal ions to the urea oxygen atom, which also has a negative effect on their sorption with the developed sorbent.

Urea-containing sorbent made of aspen bark is characterized by a very low sorption capacity

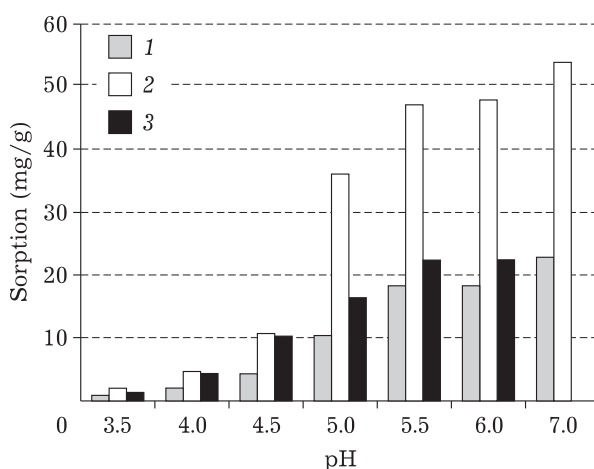


Fig. 4. Metal sorption with urea-containing sorbent made of aspen bark versus pH of model solutions: 1 – nickel, 2 – zinc, 3 – lead.

TABLE 4
Langmuir isotherm parameters for sorption of nickel, zinc, and lead with urea-containing sorbent made of aspen bark

| Sorbed metals | A_{\max}^{exp} , mg/g | Isotherm parameters | | |
|---------------|--------------------------------|---------------------|---------------------|-------|
| | | A_{∞} , mg/g | $K_L \cdot 10^{-2}$ | R^2 |
| Nickel | 18.25 | 23.34 | 0.736 | 0.979 |
| Zinc | 22.43 | 25.78 | 1.14 | 0.993 |
| Lead | 47.55 | 91.17 | 0.41 | 0.995 |

Note. A_{\max}^{exp} is the experimental maximum sorption and R^2 is the coefficient of determination.

for the investigated metals at pH 3.5, not more than 1.8 mg/g (see Fig. 4), therefore their use for acidic wastewater treatment is impractical. In order to efficiently remove nickel and lead from aqueous solutions, one may recommend the pH 5.5–7.0 value range, and for zinc – pH 5.5–6.0. It is worth noting that acidity reduction for zinc is probable only until pH 6.3. Thereafter, hydroxide precipitation begins, which leads to distorting the results of sorption capacity determination, as the metal concentration in the solution changed due precipitation and in fact, sorption.

Due to the inability to separate the precipitate from sorbent particles, it was impossible to separate the contribution of these processes to changing zinc concentration in solutions with pH > 6.3, which is necessary to determine the sorption capacity of the investigated sorbent. It is inadvisable to reduce the acidity of solutions at pH greater than 7.4 in case of nickel and lead for the same reason.

It is known that the use of sorbents based on lignocellulosic raw materials is most efficient to purify solutions with low concentrations of toxic metals (1.0–10.5 mg/L) [21, 22]. These solutions simulate effluences of some technological processes, such as washing water of metal-containing secondary raw materials processing, galvanic production, etc.

TABLE 5
Results of purification of low concentration model solutions from nickel, zinc, and lead with urea-containing sorbent made of aspen bark

| Metal | Purification degree, % | | |
|--------|--|------|--------------|
| | Initial concentrations (C_0), mg/L | | |
| | 10.5 | 5.5 | 1.0 |
| Nickel | 66.7 | 73.6 | 89.9 |
| Zinc | 76.2 | 85.7 | 91.2 (63.8)* |
| Lead | 90.4 | 91.4 | 92.6 (86.6)* |

*Purification degree values for AC_{ox} are given in [21].

It has been determined that the degree of purification of solutions from nickel and zinc increases with decreasing their initial content, and the concentration of solutions has little effect on purifying from lead (Table 5). The data therein have been acquired for solutions of nickel and lead at pH 7.0, and zinc – with pH 6.0.

The use of urea-containing sorbent made of aspen bark is most efficient for purification of model solutions with a 1.0 mg/L concentration of metals. Herewith, the degree of purification for different metals is about the same. Note that in this case, according to the ability to remove zinc and lead, the developed sorbent is superior to oxidized active carbon (AC_{ox}), properties of which are given in [21].

It is worth noting that regardless of experimental conditions of metals sorption (metal concentrations, duration and pH values), urea does not pass into solution, and this bears evidence of its durable fixation in aspen bark sorbent surface. Combined with high sorption capacity, this defines the potential of this sorbent for additional purification of water from nickel, zinc, and lead impurities.

CONCLUSIONS

It has been demonstrated that the adsorptive coating of urea onto aspen bark carrier allows producing a sorbent able to absorb nickel, zinc, and lead from aqueous solutions with different metal concentrations. Introducing 10.7 mass % of urea into the sorbent enhances the sorption capacity thereof against nickel, zinc, and lead by 1.9, 2.2, and 3.3 times, respectively. According to the ability to be sorbed on the resulting sorbent, metals generate the following series: $Pb^{2+} > Zn^{2+} > Ni^{2+}$. As demonstrated by research, the sorption equilibrium of nickel and zinc can be reached in 40 min, and that of lead – in 60 min.

It has been determined that efficient sorption of lead and nickel proceeded with aqueous solutions acidity of pH 5.5–7.0, and that of zinc – near pH 5.5–6.0. It has been shown that urea-containing sorbent made of aspen bark was efficient to purify low concentration metal solutions. The degree of purification for solutions containing 10.5 mg/L of metal impurities is not less than 66.7 % and at least 89.9 % for those with concentrations of 1.0 mg/L.

The resistance of the sorbent to the washing-out of urea combined with high sorption capacity defines the prospects of its use to additionally purify water for technical purposes from nickel, zinc, and lead impurities.

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