

DETERMINATION OF THE POSSIBILITY OF THE SYNTHESIS OF ZN-AL LAYERED DOUBLE HYDROXIDES, INTERCALATED WITH PEROXYANIONS, AS A PERSPECTIVE SOLID DISINFECTANT

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Infectious diseases in the modern world pose a significant threat to humanity in the form of epidemics and pandemics. To prevent and combat them, it is necessary to carry out antiseptic and disinfectant treatments of various environments, household and industrial surfaces, as well as wounds of various origins. Double-layer hydroxides intercalated with peroxyanions as active oxygen compounds are promising materials for this.

In order to determine the possibility of obtaining Zn-Al double-layer hydroxide intercalated with peroxy-lactic acid anions, samples were synthesized by the method of chemical co-precipitation in the presence of peroxy-lactic acid at controlled pH (8, 10) and $t=20$ °C. The properties of the synthesized samples were investigated. The content of active oxygen (in terms of H_2O_2) was determined by the method of iodometric titration with the calculation of the percentage of hydrogen peroxide that was intercalated in double layered hydroxides, remained in the mother solution or was lost. The crystal structure was studied by X-ray phase analysis, the yield of samples was determined gravimetrically, and sedimentation was determined by measuring and normalizing the thickness of the sediment layer.

It was found that the samples synthesized at pH=8 and 10 are biphasic and consist of an oxide phase and a double-layer hydroxide phase. The determined content of active oxygen (in terms of H_2O_2) in the samples synthesized at pH=8 (0.533 %) and at pH=10 (0.876 %) confirms the success of the synthesis of Zn-Al-peroxylactate double layered hydroxides. Synthesis at elevated pH is promising. A low percentage of H_2O_2 intercalation was revealed – 4.03–6.54 %, the majority of hydrogen peroxide (82.36–94.44 %) remains in the mother solution.

The yield of the synthesized samples was determined to be 61.9 % and 79.5 % at synthesis pH of 10 and 8, respectively. The sedimentation properties of the samples were studied and their improvement was shown when the pH of the synthesis was increased

Keywords: Zn–Al double-layer hydroxide, peroxy-lactic acid, solid disinfectant, intercalation, chemical coprecipitation

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1. Introduction

Peroxide compounds exhibit significant antiseptic and disinfectant properties [1], while their most important ad-

vantage over antibiotics is the absence of the effect of the formation of super-resistant strains of microorganisms. Due to the fact that H_2O_2 solutions have low stability, functional analogs are used instead – organic peroxyacids, such as per-

acetic and perlactic acids [2, 3]. The industry produces solutions of such peroxyacids, which consist of a stoichiometric mixture of free acid and hydrogen peroxide. However, such mixtures cannot be used as antiseptics due to their irritating effect. Anion-exchange materials containing peroxyacid anions, in particular layered double hydroxides, will not have this disadvantage [4, 5]. Another advantage of these materials is the prolonged release of active materials.

However, an in-depth analysis of a large number of publications showed that layered double hydroxides intercalated with peroxyanions have not been synthesized. Therefore, the production of such layered double hydroxide (LDH) as new types of disinfectants is relevant and promising.

2. Literature review and problem statement

The main obstacle to determining the effectiveness of peroxyanion-intercalated LDHs and the prospects for their production is that these substances have not been synthesized. For successful synthesis, it is necessary to take a critical approach to both the development of the LDH composition and the choice of synthesis method.

Layered double hydroxide is an α -modification of the "host" metal hydroxide, in the crystal lattice of which some of the "host" metal cations are replaced by "guest" metal cations. Because of this, an excess positive charge is formed in the crystal lattice, which can be compensated by the intercalation of additional anions into the interlayer space. Without the introduction of additional electrolytes, such anions are the anions of precursor salts. But to obtain functional materials, anions with special properties are purposefully intercalated into the LDH structure. Stabilizing [6] or activating anions [7] can be introduced into the LDH composition.

In general, LDH consists of three main components: "host" metal cations, "guest" metal cations and intercalated anions. The development of a functional material based on intercalated LDH consists of the stages of selecting the type of LDH (host and guest metal cations), selecting the anion for intercalation, and choosing the synthesis method and conditions. Since LDH intercalated with active oxygen compounds have not been synthesized, to obtain them it is necessary to justify the choice of both LDH components and synthesis parameters.

To synthesize materials based on LDH, divalent metal cations are used as host metal cations. However, the use of toxic cations, in particular Ni^{2+} , in solid disinfectants [8, 9] is not permissible. Mg^{2+} cations are low toxic [10], however, the synthesis of LDH based on magnesium hydroxide requires special conditions, which will complicate the development of production technology. The most promising is the use of Zn^{2+} as a "host" metal cation.

Cations of tri- and tetravalent metals are used as "guest" metal cations. LDHs with Fe^{3+} as a "guest" metal anion [11] are characterized by low toxicity, but have low stability and reproducibility of composition during synthesis. The most promising use is the most commonly used Al^{3+} . This is due to high structure-forming and stabilizing properties in relation to the α -modification of LDH [9].

To be used as a medical product, LDH as a basis must be safe for health ("health-friendly") [12]. A review article [13] shows the negligible toxicity of Zn-Al LDH.

The choice of a functional component for intercalation into the interlayer space of LDH is based on the nature of

the substance, which must be anionic in nature. At the same time, hydrogen peroxide is a non-ionic substance. Therefore, substances containing a peroxide group must be anions, in particular anions of peroxy-organic acids. A promising candidate for intercalation is the peroxy-lactic acid anion, which has significant disinfectant activity [14, 15].

The synthesis method and conditions directly determine the micro- and macrostructure of LDH particles. The preparation of hydroxides can be carried out by chemical precipitation using direct synthesis (adding an alkaline solution to a solution of a metal salt) [16], reverse synthesis (adding a solution of a metal salt to an alkali solution) [17], or the sol-gel method [18]. Homogeneous precipitation can also be used for production [19]. The method of cathode template synthesis is also used for production [20]. However, not all of the listed methods can be used for the synthesis of LDH intercalated with functional anions. The production of LDHs intercalated with peroxyanions using sol-gel synthesis, homogeneous precipitation and cathodic template synthesis is significantly complicated by the catalytic decomposition of peroxides by heavy metal compounds. To obtain such materials, the best one-stage method of chemical coprecipitation is direct and reverse synthesis, as well as synthesis at a constant pH. From the point of view of further development of the technology, the most promising method is the method of chemical coprecipitation at a constant pH.

It should be concluded that to increase the likelihood of successful preparation of peroxyanion-intercalated LDH, it is necessary to carry out the synthesis of Zn-Al LDH intercalated with peroxy-lactic acid anions by a one-step synthesis method at a constant pH.

3. The aim and objectives of the study

The aim of the study is to evaluate the fundamental possibility of synthesizing Zn-Al LDH samples intercalated with the peroxy-lactic acid anion as a source of active oxygen. This will make it possible to obtain new promising samples for use as solid disinfectants, and in the future to create a technology for their production.

To achieve the aim, the following objectives were set:

- synthesize samples of Zn-Al layered double hydroxide in the presence of peroxy-lactic acid at different pH values, study the structural characteristics of the samples, determine the content of active oxygen (in terms of H_2O_2), drawing up a partial material balance, and draw a conclusion about the success or failure of the synthesis;
- in case of successful synthesis, determine the yield and study the sedimentation properties of the obtained samples of Zn-Al-peroxy-lactate layered double hydroxide.

4. Materials and methods of the study

4.1. Object and hypothesis of the study

The object of study is the synthesis of layered double hydroxides intercalated with peroxy-lactate anion. The research hypothesis is that it is possible to intercalate peroxy-lactic acid anions into the interlayer space of a Zn-Al layered double hydroxide as a source of active oxygen.

To carry out the study, it was assumed that it is possible to intercalate the peroxy-lactate anion directly into the interlayer space of a layered double hydroxide using a one-step

synthesis method. The study adopted the simplification that the amount of active oxygen (in terms of H_2O_2), determined by the iodometric method, refers directly to the peroxy-lactic acid anion.

4. 2. Method of obtaining samples

To carry out the synthesis, salts of zinc and aluminum nitrates (in crystalline hydrate form) of grade (reagent grade), as well as NaOH in the form of granules of grade (reagent grade) were used.

Samples of Zn-Al-peroxylactate LDH were obtained by direct chemical synthesis at constant pH. During the synthesis of LDH samples, three solutions of solutions were fed independently by peristaltic pumps at the same speed: a solution of $Zn(NO_3)_2 + Al(NO_3)_3$ (with a molar ratio of Zn:Al=4:1), a solution of NaOH and a solution of peroxy-lactic acid (stoichiometric a mixture of lactic acid and 30 % H_2O_2). The reagent solutions were fed dropwise at a rate of 0.8 l/hour into a 2-liter reaction beaker, into which 150 ml of the original solution, the pH of which corresponded to the pH of the synthesis, had previously been added. The synthesis was carried out at pH=8 and pH=10.

Labeling of samples: Zn-Al-PerLA-20-8, where Zn-Al is the type of LDH, PerLA is the intercalated peroxy-lactic acid anion, 8 is the synthesis pH, 20 is the synthesis temperature ($^{\circ}C$). To carry out the experiment, the mass of NaOH was calculated as the sum of the mass of alkali for the LDH formation, the mass for neutralizing peroxy-lactic acid and the mass necessary to maintain the required pH. The synthesis was carried out with continuous stirring using a magnetic stirrer and maintaining a constant temperature of both the reagents and the reaction mixture. Upon completion of the addition of reagent solutions, the reaction mixture was stirred while maintaining the temperature for 30 minutes. After time, the reaction mixture was fed to filtration under vacuum to separate the precipitated LDH from the mother liquor. Next, the resulting precipitate was dried at a temperature of $50^{\circ}C$ (24 hours), crushed and soaked in distilled water for a day to dissolve the water-soluble impurities remaining after filtering. The soaked precipitate was filtered again and dried under the same conditions.

4. 3. Methods for studying the characteristics of samples

Determination of active oxygen content (in terms of H_2O_2) in LDH samples and solutions. The content of active oxygen (peroxide) in the starting and mother solutions was carried out by iodometric titration with acidification with 10 % sulfuric acid. To determine the content of active oxygen (peroxide) of the finished dried LDH samples, iodometric titration was also carried out with complete dissolution of the LDH sample. Based on the analysis data, the content of H_2O_2 (% mass), the fraction of H_2O_2 intercalated in LDH, remaining in the mother solution, and the fraction of losses were calculated. Iodometric titration for each analysis was carried out three times, and the difference in titrant volumes should not differ by more than 0.3 ml. The volume (or mass) for analysis was chosen such that at least 20 ml of titrant was used for titration.

Study of structural characteristics. The crystal structure of the materials was studied using X-ray phase analysis (XRD) on a DRON-3 diffractometer (Co-K α radiation, angle range $10-90^{\circ} 2\theta$, scanning speed $0.1^{\circ}/s$).

Determination of the yield of target synthesis products and the content of water-soluble impurities. The sample yield was determined by weighing the sample after the first and

second drying steps (before and after washing) and dividing by the theoretical mass of the sample, for which the masses of the reagents were calculated.

Study of sedimentation properties. The filterability of the resulting sediments is an important characteristic of the samples, which indicates the characteristics of the formation mechanism and which will be used in the development of the technology. For this reason, the study of sedimentation properties was carried out immediately after the completion of the sediment aging process [70] (after 30 minutes of stirring after dripping out the reagents) by visually measuring the thickness of the sediment in the reaction beaker for 30 minutes [52, 53]. To be able to compare data on sedimentation of different samples, the thickness of the sediment at each measured point is normalized – converted into a relative value by dividing by the thickness of the sediment at the initial time.

Since the aim of the study was to show the possibility of synthesis, and the characteristics of processes and samples are primary and require clarification, statistical processing of the results was not carried out.

5. Results of synthesis and study of parameters of Zn-Al-peroxylactate layered double hydroxide samples

5. 1. Results of sample synthesis, study of their structure and peroxide content

During the synthesis of Zn-Al LDH in the presence of peroxy-lactate, white samples were obtained, organoleptically similar to the samples of previously synthesized Zn-Al-nitrate LDH.

Fig. 2 shows diffraction patterns of samples synthesized at pH=8 and pH=10. The diffraction patterns of both samples (Zn-Al-PerLA-20-10 and Zn-Al-PerLA-20-8) revealed peaks corresponding to ZnO (2θ 37° , 40.3° , 42.3°). At the same time, the content of zinc oxide in the Zn-Al-PerLA-20-8 sample is higher than in the Zn-Al-PerLA-20-10 sample. The diffraction pattern of the Zn-Al-PerLA-20-10 sample shows a clear peak at $2\theta=13.4^{\circ}$, corresponding to both α -Zn(OH) $_2$ and Zn-layered double hydroxides. At the same time, there is no peak corresponding to Zn-LDH in the diffraction pattern of the Zn-Al-PerLA-20-8 sample.

Data on the H_2O_2 content, the proportions of intercalated, lost and remaining active oxygen in the mother solution (in terms of H_2O_2) for the synthesized Zn-Al-PerLA samples are shown in Fig. 2.

First of all, it should be noted that the content of active oxygen (in terms of H_2O_2) (Fig. 1, a) in the synthesized LDH samples is 0.876 % (synthesis at pH=10) and 0.533 % (synthesis at pH=8). This peroxide content is sufficient to use the samples as disinfectants.

Higher pH synthesis increases the active oxygen content. The proportion of intercalated peroxide also increases with increasing pH of the synthesis. At the same time, the degree of loss (decomposition) of peroxide during synthesis for the Zn-Al-PerLA-20-10 sample (pH=10) is 11.1 %, which is significantly higher than during synthesis with pH=8 (Zn-Al sample -PerLA-20-8) – 1.53 %. It is revealed that a significant part of peroxy compounds remains in the mother solution (Fig. 3, c) – 82.36 % during synthesis with pH=10 and 94.44 % during synthesis with pH=8.

In general, it should be concluded that the synthesis of Zn-Al-peroxylactate LDH samples is successful.

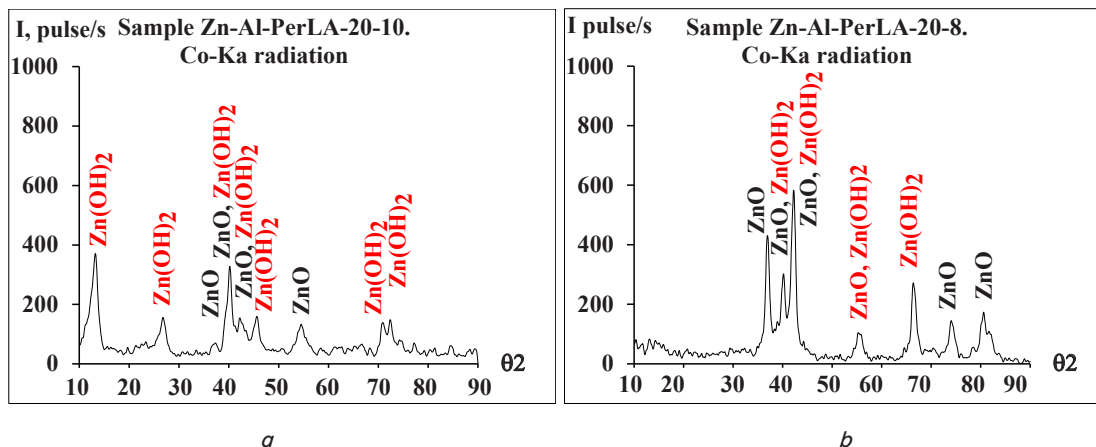


Fig. 1. Results of X-ray phase analysis for the synthesized samples: *a* – Zn-Al-PerLA-20-10; *b* – Zn-Al-PerLA-20-8

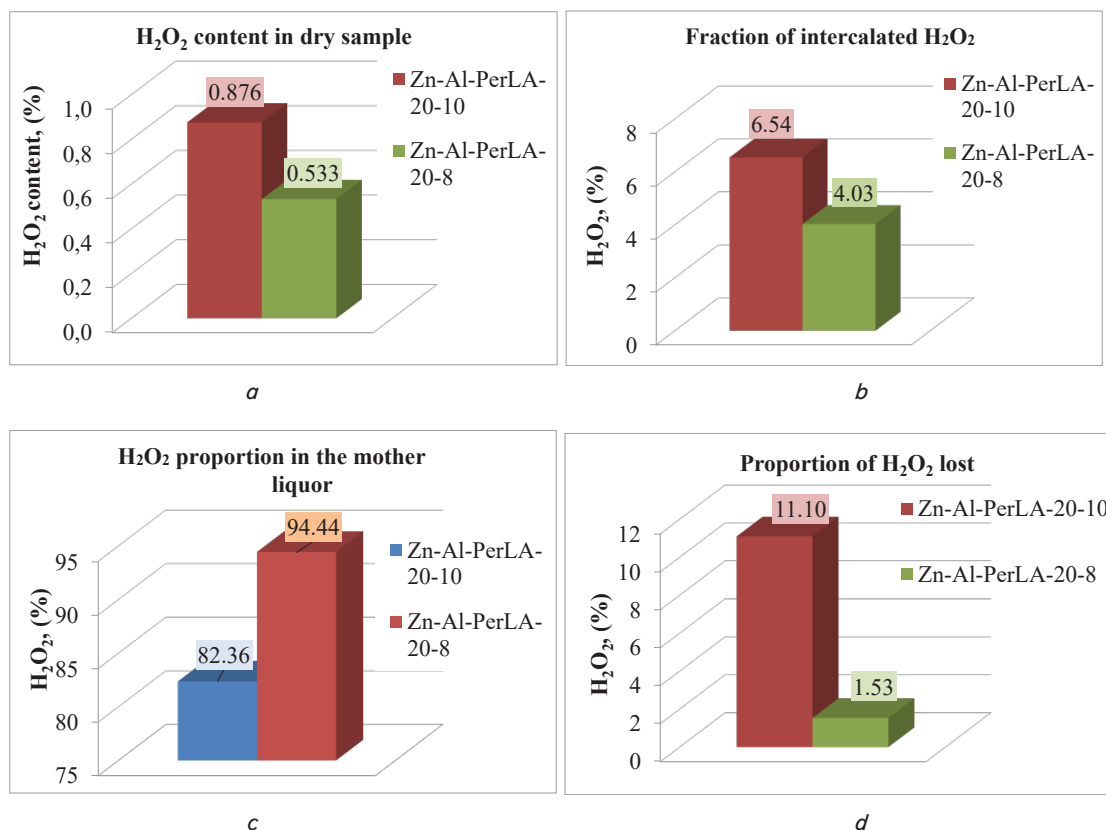


Fig. 2. Data on active oxygen (in terms of H₂O₂): *a* – content in a dry sample; *b* – fraction of intercalated in the dry sample; *c* – proportion remaining in the mother solution; *d* – proportion of losses during synthesis

5. 2. Results of determining the yield and content of water-soluble impurities, studying the sedimentation properties of samples

In connection with the conclusion that the synthesis of Zn-Al-peroxylactate LDH samples was successful, the yield and content of water-soluble impurities were determined (Fig. 3).

The product yield for both samples of synthesized LDH is quite high and amounts to 61.9–79.5 % of the theoretical value. With increasing pH, the product yield decreases, but

the content of water-soluble impurities, on the contrary, increases.

The results of studying the sedimentation of LDH samples after synthesis are shown in Fig. 4.

It should be noted that during the first 10 minutes the sedimentation of both samples did not differ. However, later the Zn-Al-PerLA-20-10 sample (synthesized at pH=10) sedimented faster, the relative layer thickness after 30 min of exposure was 78.5 % (compared to the relative layer thickness of 86 % for the Zn-Al-PerLA-20-8).

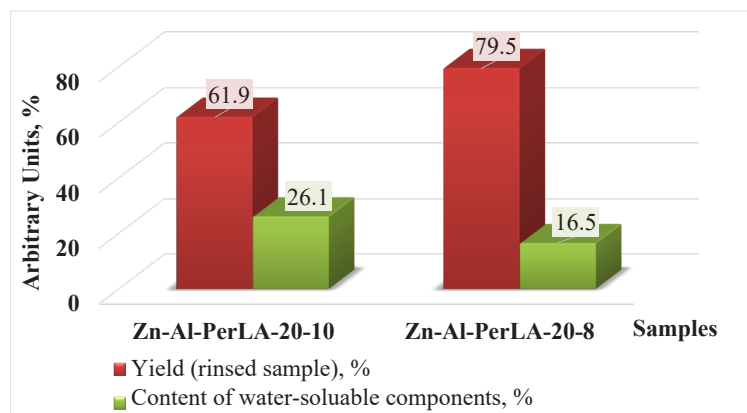


Fig. 3. Yield and content of water-soluble impurities for synthesized LDH samples

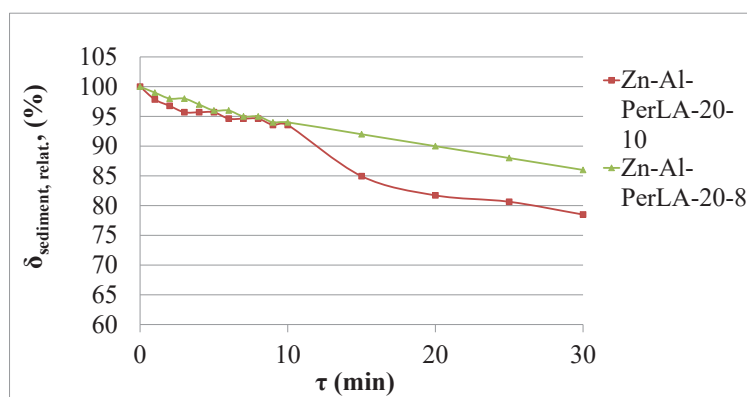


Fig. 4. Relative thickness of the sediment layer ($\delta_{\text{sediment, relat.}}$) during sedimentation for the synthesized samples (the trend shown in the figure is conditional)

6. Discussion of the results of synthesis and study of parameters of Zn-Al-peroxylactate layered double hydroxide samples

Research on the structure of LDH. Diffraction patterns of Zn-Al LDH samples synthesized in the presence of peroxylactate contain fairly clearly defined peaks of the ZnO-based oxide phase (Fig. 1). According to crystallographic parameters, this oxide phase corresponds to ZnO ($2\theta=37^\circ, 40.3^\circ, 42.3^\circ$). However, it should be noted that there are no peaks of aluminum hydroxide phases in the diffraction patterns. Therefore, it is possible to make a reasonable assumption that the oxide phase is a Zn-Al layered double oxide (LDO). The phenomenon of Zn-Al LDO formation was previously described for the synthesis of Zn-Al-nitrate LDH at various pH and temperatures [52]. The diffraction pattern of the Zn-Al-PerLA-20-10 sample (Fig. 1, a) reveals a clear peak at $2\theta=13.4^\circ$, corresponding to Zn-Al LDH [52]. Moreover, for this sample, the peaks of the oxide phase are broad and have a low value, which indicates low crystallinity of the oxide phase and its low amount. In the diffraction pattern of the Zn-Al-PerLA-20-8 sample (Fig. 1, b) at $2\theta=13.4^\circ$ there is a very weakly visible broad peak of Zn-Al LDH, which indicates its X-ray amorphism. Moreover, for this sample the oxide phase has higher crystallinity. It can be concluded that the synthesized samples are biphasic and consist of an oxide phase (Zn-Al LDO) and a Zn-Al LDH phase. In this case, synthesis at pH=10 is more promising.

Analysis of data on active oxygen content (in terms of H_2O_2). On the one hand, both dry samples of Zn-Al-PerLA have a hydrogen peroxide content (Fig. 2, a): 0.876 % by weight (sample Zn-Al-PerLA-20-10) and 0.533 % by weight (sample Zn-Al-PerLA-20-8). The higher peroxide content in the Zn-Al-PerLA-20-10 sample correlates with a higher amount of LDH phase with higher crystallinity. A material balance of the synthesis process for hydrogen peroxide was compiled (Fig. 2, b–d). It was revealed that the amount of intercalated peroxy-lactate anions is low: 6.54 % (sample Zn-Al-PerLA-20-10) and 5.03 % (sample Zn-Al-PerLA-20-8) of the introduced amount of active chlorine. This indicates competitive intercalation of nitrate ions.

In general, it is necessary to indicate that the resulting content of hydrogen peroxide is quite sufficient for the use of these LDHs as solid disinfectants for wound dressings, as well as for surface treatment. Synthesis at pH=10 seems more promising.

In addition, a significant part of the peroxy compounds (82.36 % for Zn-Al-PerLA-20-10 and 94.44 % for Zn-Al-PerLA-20-8) remains unreacted in the mother solution (Fig. 2, c).

Some of the peroxy compounds are lost during the synthesis process (Fig. 3, d). For the Zn-Al-PerLA-20-8 sample (synthesis pH 8) the loss was 1.53 %, while during synthesis with pH=10 (Zn-Al-PerLA-20-10) the loss sharply increased to 11.1 %. These losses indicate the proportion of peroxy compounds decomposed both during synthesis and during drying of the hydrophilic sediment.

Determination of LDH output. Analysis of the yield values of LDH intercalated with peroxy-lactate at different synthesis pH values (Fig. 3) showed fairly high values (61.9–79.5 %). Increasing the synthesis pH from 8 to 10 (Fig. 3) leads to a decrease in yield from 79.5 % to 61.9 %. However, this fact has no explanation yet, because in the sample synthesized at pH=10, the LDO amount is lower, and the intercalated amount of peroxide compounds is higher than in the sample synthesized at pH=8.

Study of sedimentation properties of synthesized LDHs. An analysis of the influence of synthesis pH on the sedimentation process of the samples (Fig. 4) showed that during the first 10 minutes there were no differences in the sedimentation process of both samples. Subsequently, the Zn-Al-PerLA-20-10 sample is deposited faster than the Zn-Al-PerLA-20-8 sample. This indicates a better filtration ability of the sediment of the Zn-Al-PerLA-20-10 sample.

In addition, it should be noted that both samples settle quite slightly – 78.5 % and 86 %. This indicates the structural features of the resulting sediments: the developed surface of the formed amorphous primary particles, the high saturation of the closed-cell structure of the LDH with the mother solution, which leads to an increase in its volume.

Limitations of this study include the synthesis carried out at only two pH values for a sufficiently accurate understanding of the properties of the samples. Also unclear was the form of the peroxy compound that intercalated into LDH. In the future, it is necessary to conduct additional studies to find conditions for increasing the amount of peroxyanions intercalated into the interlayer space of LDH.

7. Conclusions

1. Using the synthesis method at constant pH (8 and 10), previously unsynthesized Zn-Al LDH samples intercalated with peroxylic acid anions with an active oxygen content (in terms of H₂O₂) of 0.533 % and 0.876 %, respectively, were successfully obtained. It was revealed that the synthesized samples are biphasic systems consisting of the oxide phase of Zn-Al LDO and the hydroxide phase of Zn-Al LDH. A material balance for the synthesis of hydrogen peroxide has been compiled; it has been shown that the degree of intercalation of peroxy compounds is 4.03 % and 6.54 % for synthesis at pH 8 and 10, respectively. The most promising is the synthesis of Zn-Al-peroxylactate LDH at high pH values.

2. The yield of synthesized samples of Zn-Al-peroxylactate layered double hydroxide was determined to be 61.9 79.5 %. The sedimentation properties of sediment samples were studied by measuring the sediment thickness over time. It has been shown that the sample synthesized at pH=10 is more preferable in terms of sedimentation properties.

Conflict of interest

The authors declare that there are no conflicts of interest with respect to this article and the published results of

the study, including the financial aspects of conducting the study, obtaining and using its results, and any non-financial personal relationships.

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Data availability

The manuscript has no associated data. Any additional explanations and materials can be obtained upon request to the author for correspondence.

Use of artificial intelligence tools

The authors confirm that they did not use artificial intelligence technologies when creating the presented work.

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