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Wastewater Purification from Excess Phosphates Using Bentonite Activated by Microwave Radiation

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ABSTRACT
This article examines the possibilities of improving the ecological condition of small rivers on the example of the upper part of the Prut River in the vicinity of Yaremche (Ivano-Frankivsk region, Ukraine). The previously published data of the article authors and other researchers that the amount of pollutants in river water in this area has increased, in particular, phosphorus compounds, has been confirmed. It was shown that the incompletely purified wastewaters of Yaremche, which contains an excess of phosphate ions, has a significant impact on this fact. On the basis of their own previous research and current work, the authors propose removing these ions using a natural sorbent based on bentonite, activated by microwaves in various ways. Experiments have shown that microwave activation of this sorbent increases the coefficient of phosphate ions extraction from wastewater significantly higher compared to natural bentonite. An approximate technological scheme of wastewater purification from excess phosphates after the main standard purifying cycle was suggested.

Keywords: phosphates, adsorption, bentonite, microwaves.

INTRODUCTION

Some main directions of modern hydroecological studies of scientists in the western region of Ukraine are the observation, assessment and forecast of surface water in the Carpathian National Nature Park. The main waterway in this area is the Prut River, which is the left tributary of the Danube Raver.

It begins on the northern slopes of the Montenegrin ridge at an altitude of over 1750 m a.s.l. and flows between Mount Hoverla and Homul. The length of the Prut River is 967 km, whereas the catchment area is 23,540 km^2. The upper current of the river is located in Ukraine (Ivano-Frankivsk and Chernivtsi regions); the lower one is on the border of Moldova with Romania. The Prut River flows into the Danube River near the Gurguleșhti village (Moldova) [Nikolaev, 2014].

On the basis of a summary of scientific publications and production reports of companies that have researched the ecological condition of the Prut River in recent years, as well as relevant reports in periodicals, internet resources and other media, the authors found that its water quality in the upper current deteriorates markedly [Grytsku, 2014; Kirilyuk, 2013 et al.]

The average numerical values of physico-chemical parameters of the Prut River (pH, hardness, total mineralization, HSC), concentrations of major ions (bicarbonates, chlorides, sulfates, calcium, magnesium, sodium and potassium) and biogenic ions (ammonium, nitrites, nitrates, phosphates) for 2016–2018 are shown in Table 1.

This data was taken from the periodic reports of the hydrometeorological post in the Yaremche chemical laboratory (Ivano-Frankivsk region, Ukraine), which is located on the upper current of the River [Khilchevskyi, 2017; 2018].

The afore-mentioned laboratory carries out sampling and research of water quality in vicinities of this city once a quarter. The samples are taken at two observation points: 0.5 km above the city limits and in the city itself. The authors of the article got acquainted with the research protocols of these samples with the permission of hydrometeorological post authority in Yaremche.

According to the data of Table 1, owing to the good water aeration in the Prut River in the upper stream, all biological, physicochemical and biochemical processes are running very intensively. That is why the process of self-cleaning of this reservoir is fast. Therefore, the water quality of the Prut River in the studied area remains stable and close to the natural conditions [Prikhodko, 2014].

However, the increase of biogenic pollutants in the Prut River content near Yaremche is a matter of concern, especially an increase of nitrogen and phosphorus compounds in the river water content.

The authors of this article quarterly conducted ecological studies of the Prut River upper current water condition, as reported in their previous publications [Boichuk, 2019; a & b]. Physicochemical parameters of the water samples from several observation points in the vicinity of Yaremche and in the city itself were studied.

In these studies, the vast majority of the maximum pollution of water samples from the Prut River was found in Yaremche. For example, the content of phosphate ions in the Prut River within this city was 0.38 mg/dm³, which is several times higher than the results found in previous years (0.049… 0.181 mg/dm³) [Boichuk, 2019; a]. This fact indicates a significant increase of the anthropogenic component in the process of negative impact on river ecosystems in the region in recent years.

Since the maximum value of phosphate ions was found in the water sample from Yaremche, the question arises about the impact of urban waste waters (in particular, with a high content of detergents) on the pollution of the Prut River. The main role in solving the problem of the River protection from these runoffs belongs to the effective operation of municipal sewage purifying plants (SPP). A separate study conducted by the authors of this article was devoted to this issue [Boichuk, 2020].

In the afore-mentioned study, the hydrochemical parameters of the Prut River in the vicinity of Yaremche were re-studied, which took place near the place of purified wastewater output from the city SPPs into the river. The water samples from the Prut River were taken quarterly during 2020. The sampling site was selected 500 m downstream from the point of Yaremche SPP sewage pipe exit to the river. The effectiveness of SPP was evaluated based on the laboratory study of water samples at this point.

Among other physicochemical parameters of the river water at this point, the main attention was paid to the phosphate content. As a result, the data from previous studies were confirmed [Boichuk, 2019; a]. It was found that the average value of phosphate ions in 4 quarters of water samples from the Prut River was 0.32 mg/dm³.

In addition, with the permission of the “Yaremchevodokanal” authority, the authors got acquainted with the protocols of the purified waste waters study of this enterprise for 2017–2019. There is also an excess of phosphates in wastewater after the main purifying cycle, which indicates the need for additional treatment of these waters from phosphates. In particular, the average quarterly values of phosphate ions in

Table 1. The average hydrochemical water composition of the Prut River within Yaremche during 2016–2018

№	Index	0.5 km above the city			In Yaremche		
		Avg.	Max.	Min.	Avg.	Max.	Min.
1	pH	7.75	8.20	6.70	7.9	8.2	7.1
2	Hardness, mg-eq/dm³	2.74	3.66	2.24	2.68	3.79	2.10
3	Overall mineralization, mg/dm³	233	241	225	262	284	236
4	COD, mgO/dm³	7.6	9.1	6.1	8.4	10.3	6.2
5	Chlorides, mg/dm³	11.00	19.30	5.73	11.53	17.50	5.73
6	Sulfates, mg/dm³	19.75	37.70	10.20	23.85	46.10	13.30
7	Hydrocarbons, mg/dm³	167	228	131	143	181	109
8	Calcium, mg/dm³	48.6	68.3	35.6	46.8	70.0	31.6
9	Ammonium nitrogen, mg N/dm³	0.197	0.400	0.040	0.368	0.490	0.220
10	Nitrite nitrite, mg N/dm³	0.003	0.006	0.001	0.004	0.007	0.001
11	Nitrogen nitrate, mg N/dm³	0.465	0.490	0.400	0.508	0.550	0.420
12	Soluble phosphates, mg/dm³	0.24	0.43	0.16	0.30	0.59	0.16

purified wastewater of this enterprise for 2017–2019 range from 0.19 mg/dm³ to 0.26 mg/dm³.

The fact, that the content of phosphates in river water downstream from the point of Yaremche SPP sewage pipe exit to the river is higher than in the purified wastewater, due to the fact that phosphates are present in the Prut River and above the city. This was reported by the authors in their previous publication mentioned above [Boichuk, 2019; a].

Last but not least, this can be linked to the development of the tourism industry –the number of sanatoriums, rest homes and private buildings on the picturesque banks of the Prut River, among the Carpathian landscapes, is constantly growing. Most of these modern buildings have local or collective purifying facilities, but the dynamics of residential and recreational settlements outweigh the natural self-clean ability of the river. At the same time, private rural houses on the banks of the river, as a rule, do not have any purifying facilities, septic tanks or latrine pits are used instead.

The problem of phosphorus compounds removal from wastewater currently has no optimal solution and requires additional research. A special place in this direction is given to the use of natural clay materials (including bentonite) as cheap but effective sorbents.

Special physicochemical characteristics of bentonite clays are a large specific surface and high dispersion, which results in good adsorption rates. The general formula of these minerals can be represented as $x(Al_2O_3)\cdot y(SiO_2)\cdot z(OH)\cdot m(H_2O)\cdot n(MeO_k)$, where MeO_k are metals oxides (iron, alkali and alkaline earth metals, etc.). By the chemical nature and phase composition, bentonites are compositions of aluminosilicate minerals: montmorillonite, chlorite, hydromica, etc. In terms of crystal chemistry, they all have a negatively charged three-dimensional aluminosilicate framework with a strictly regular structure. Hydrated positive ions of alkali and alkaline earth metals are in the intervals of this framework which compensate the framework charge and water molecules. Only the molecules of substances which critical size is smaller than the effective size of the entrance window are sorbed into the adsorption cavities of these materials [Ravindra Reddy et al., 2017].

Clay sorbents are used both directly and after the chemical modification or activation. In recent decades, a promising direction of sorbents efficiency improving is the use of high-frequency electromagnetic radiation (microwave UFER, or microwaves) for their activation [Legras, 2013; Surendra, 2017; Foletto, 2013 et al.].

The authors of this article have already published the results of their work on studying the effect of microwave radiation on the sorption properties of bentonite. It was shown that bentonite irradiated with microwaves in various ways absorbs phosphate ions much better than untreated (native) sorbent. However, these studies were performed on model (pure) solutions of phosphate compounds [Konzur, 2016; 2018].

The aim of this work was to compare the effectiveness of different methods of bentonite irradiation with microwaves in the processes of real wastewater purification from excess phosphates.

MATERIALS AND METHODS

The objects of the study were the samples of wastewater from various institutions. The first sample was purified wastewater collected at the outlet of purifying plants in Yaremche; the second comprised purified waste water at the outlet of purifying facilities in Lviv (Ukraine).

To clarify the possibility of removing phosphates from untreated wastewater, the third sample was taken from untreated wastewater of Lviv state university of life safety (LSU LS, Lviv) from the general collector before discharging it into the city sewer. All these samples were taken in November 2020.

Preparation of the samples for research was carried out according to standard methods. In particular, after settling, the samples were filtered through membrane filters using a vacuum pump to obtain virtually clear solutions.

Adsorption purifying with bentonite of wastewater and additional samples was performed using the above-described method. They were obtained by stepwise dilution of native samples with tap water. For example, 10 ml of water was added to 100 ml of sample; then the degree of dilution is (100+10)/100 = 1.1 etc.

The purpose of this additional study was to determine how the dilution of wastewater with ordinary water under real conditions can affect the degree of phosphates extraction from them using bentonite. In addition, the dependence of the degree of phosphate extraction from wastewater on the medium acidity (pH) was studied.

Determination of the phosphate content in native (prepared) and additional (diluted) samples before and after sorption purification with bentonite was performed by photometric method [DSTU

7525:2014]. Concentration electrophotocolorimeter KFK-2, combined "molybdenum" reagent and ascorbic acid were used as a reducing agent.

Before studying the sorption capacity of bentonite, its purity and individual physicochemical parameters were checked. In particular, with the help of X-ray phase analysis (X-ray diffraction) its diffraction pattern was obtained, according to which the main mineralogical phases of the sample were identified. The diffraction pattern of the native bentonite sample (without special purifying) was taken by using the powder method on a DRON-3 diffractometer using copper filtered radiation. The qualitative phase composition of the sample was determined by indexing the *hkl* peaks of the corresponding minerals according to their tabular values *d/n;* quantitative phase composition – by integral intensities (areas) of the diffraction pattern corresponding peaks [Trushin, 2012].

Among the physicochemical parameters of bentonite, the acidity (pH) of its 5% aqueous suspension and the degree of swelling were measured. The pH was determined using a pH meter (pH-150M). To determine the degree of swelling, 20 cm^3 of dry sorbent was poured into the measuring cylinder, 50 cm^3 of distilled water was added, mixed and settled. After several stirring and settling, a new volume of sorbent was measured.

The sorption properties of bentonite, as in previous works [Konzur, 2016; 2018 et al.] were studied under static conditions. The sorbent was prepared in three ways (series): untreated bentonite (native or "nat"), washed with distilled water under irradiation ("stimulated" or "stim") and irradiated directly in the test solution (direct irradiation, or "DIR").

For this purpose, 1.0 g of bentonite was placed in 150 ml beakers. Next, in the series "nat" and "DIR" in the glasses with the sorbent was added 100 ml of appropriate wastewater samples, and in the series "steam" – 30 ml of distilled water.

In the "nat" series, the obtained suspensions were mixed and left to settle. In the "stim" series, flasks with bentonite suspensions in distilled water were irradiated with microwaves and defended. Then the wastewater was removed, the obtained "stimulated" sorbent breakdown of wastewater was poured (100 ml), stirred and left to settle. In the "DIR" series, a suspension of native bentonite in waste water was immediately irradiated with microwaves, mixed and left to settle.

In all three series, after settling of the suspensions, about 70–80 ml of the transparent part of the solution was taken from them and transferred to the next stage, i.e. determination of phosphate content.

Refinement of experimental data for plotting and calculating the degree of phosphate ions extraction was performed with the methods of mathematical statistics and regression analysis. The standard features of the well-known Microsoft Office suite were used.

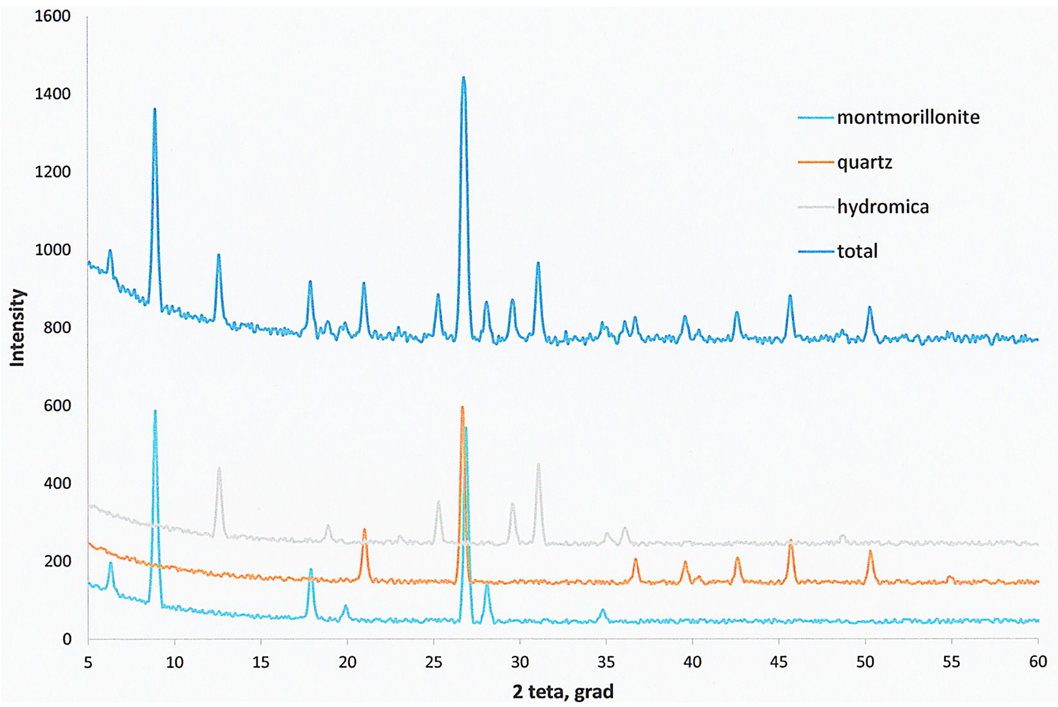

Figure 1. The results of X-ray phase analysis of the bentonite native sample (CuKα - ed.) diffraction pattern

The full range of experimental research within the framework of this work was conducted in the scientifically advanced laboratory of environmental safety of LSU LS.

RESULTS

Phase analysis

The X-ray phase analysis of the diffraction pattern of the bentonite native sample revealed that it contains 4 minerals in approximately equal amounts: hydromica (approx. 27% at.); montmorillonite (approx. 25% at.); quartz (approx. 22% at.) and chlorite (approx. 20% at.); impurities include calcite, sylvinite and other minerals. The diffraction pattern of the bentonite native sample (CuKα – ed.) after the corresponding digital processing is given in Figure 1.

Sorption research

As a result of the analytical phase of the research, a set of numerical values of phosphate concentrations in the wastewater samples of the

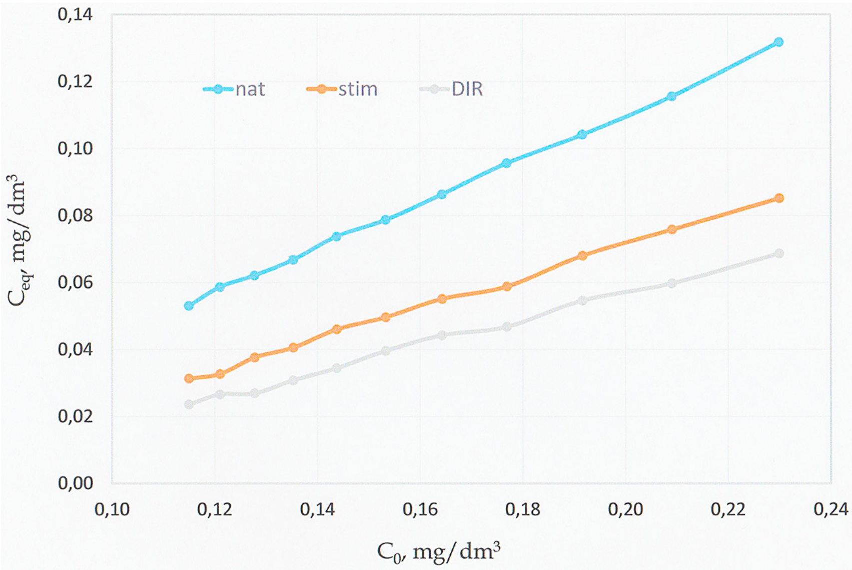

Figure 2. Change in the phosphate ions concentration in wastewater before (C_0) and after (C_{eq}) sorption cleaning with bentonite (sample from Yaremche)

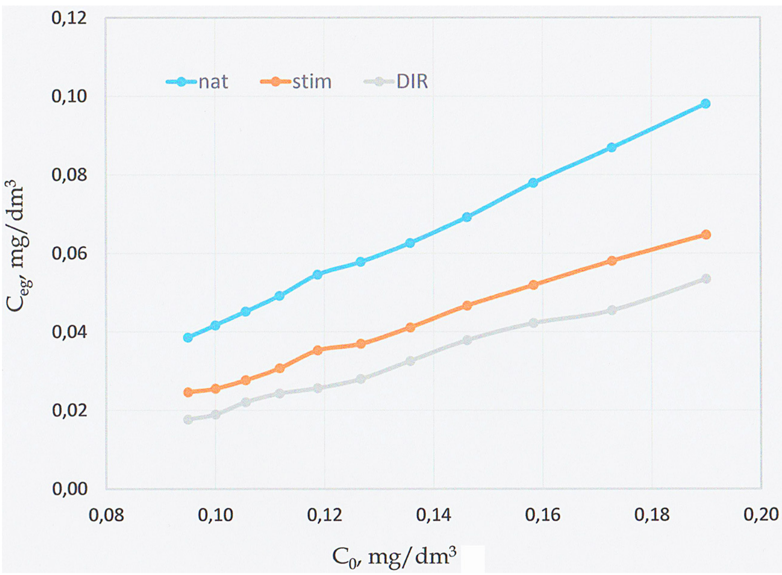

Figure 3. Change in the phosphate ions concentration in wastewater before (C_0) and after (C_{eq}) sorption cleaning with bentonite (sample from Lviv)

above-mentioned enterprises before and after their bentonite purifying of different series was obtained.

The initial concentrations of phosphate ions in the selected wastewater samples were: 0.23 mg/dm³ (the sample from the purifying plant in Yaremche); 0.19 mg/dm³ (the sample from sewage purifying plants in Lviv) and 6.85 mg/dm³ (sample from the sewage collector of LSU LS).

Figure 2 shows graphs of changes in phosphate ion concentrations in wastewater (native and dilute) after leaving the Yaremche purifying plant. The upper graph shows the concentration of phosphates in waste samples before their interaction with bentonite; in turn, lower graphs present phosphate concentrations after sorption extraction by bentonite (native and activated by microwaves in different ways).

Figure 3 shows similar graphs for purified wastewater samples after leaving the purifying plant in Lviv.

Figure 4 shows graphs of changes in phosphate ion concentrations in unpurified wastewater from LDU LS infrastructure objects before and after sorption purifying with bentonite.

On the basis of the numerical values of phosphate ions in the wastewater samples before and after sorption purifying with bentonite, the values of the extraction degrees of these ions (in %)

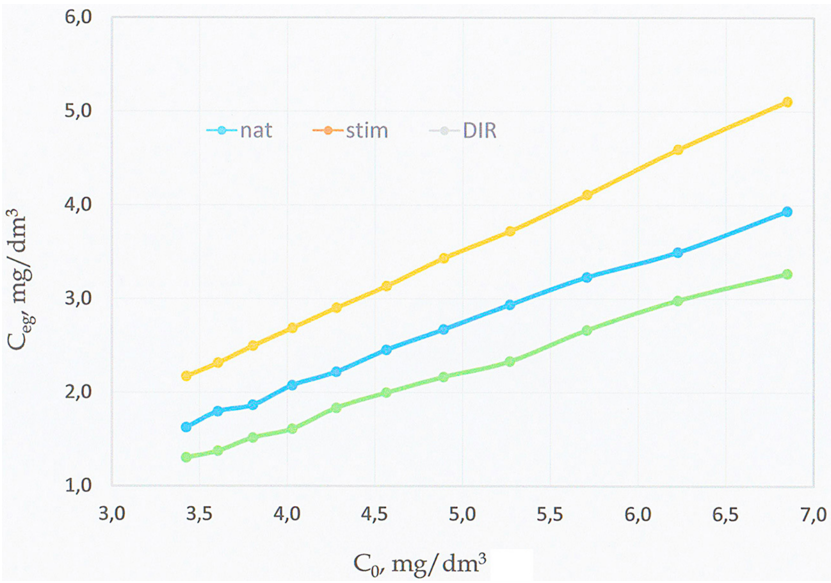

Figure 4. Change in the concentration of phosphate ions in wastewater before (C_0) and after (C_{eq}) sorption cleaning with bentonite (test with LDU LS)

Table 2. Levels of extraction of phosphate ions from waste water samples at different methods of microwave activation of the sorbent

№ of series	The method of processing the sorbent	Degree of dilution										
		1.0	1.1	1.2	1.3	1.4	1.5	1.6	1.7	1.8	1.9	2.0
Yaremche												
1	"nat"	0.435	0.439	0.441	0.463	0.474	0.484	0.487	0.494	0.502	0.528	0.531
2	"stim"	0.634	0.637	0.659	0.658	0.661	0.675	0.687	0.691	0.718	0.710	0.727
3	"DIR"	0.706	0.712	0.714	0.720	0.743	0.741	0.768	0.761	0.785	0.789	0.799
Lviv												
4	"nat"	0.481	0.500	0.514	0.516	0.534	0.537	0.554	0.556	0.575	0.589	0.593
5	"stim"	0.647	0.652	0.670	0.684	0.681	0.702	0.703	0.728	0.724	0.746	0.741
6	"DIR"	0.723	0.728	0.747	0.744	0.756	0.768	0.779	0.793	0.803	0.801	0.814
LSU LS												
7	"nat"	0.260	0.266	0.286	0.287	0.302	0.304	0.316	0.326	0.338	0.354	0.358
8	"stim"	0.415	0.436	0.444	0.455	0.459	0.476	0.477	0.483	0.504	0.507	0.517
9	"DIR"	0.527	0.538	0.542	0.549	0.554	0.577	0.586	0.588	0.591	0.609	0.613

from the studied samples were calculated. These numerical values of the degree for different options for bentonite pre-purifying (activation) are shown in Table 2.

Influence of acidity of the environment

The acidity of the 5% aqueous suspension of the studied bentonite sample was 7.2. This pH value is typical for bentonites of the (Na, K) type [Surendra et al, 2017]. The degree of swelling was approx. 2, which also corresponds to the mentioned type of bentonite clays.

The initial pH values of the undiluted wastewater samples from Yaremche, Lviv and LSU LS were 6.5, 6.9 and 7.4, respectively. After the addition of appropriate amounts of NaOH or HCl, new working samples with different pH values were obtained, from which phosphate ions were also removed using bentonite in the "nat", "stim" and DIR series. The result is the dependences shown in Figure 5.

DISCUSSION

As it can be seen from the results, "direct irradiation" of bentonite with microwaves in the process of sorption purifying of wastewater from phosphate ions increases the sorption characteristics of this material almost twice relative to the native sorbent. The method of "stimulation" gives slightly worse results, but is also much more effective than the method of purification with untreated bentonite.

The degree of the original wastewater samples dilution is also significant. As it can be seen from Table 2, the degree of phosphate ions extraction increases due to the dilution of the samples. Obviously, undiluted (more concentrated) wastewater contains a significant amount of impurities that were not removed in the pre-purifying process. These impurities can clog micropores on the bentonite surface and thus reduce the possibility of access to phosphate ions. This is especially noticeable in the case of untreated wastewater (samples from LSU LS), where the degree of these ions extraction is much lower than other studied samples. In diluted samples, the specific amount of impurities that can clog the micropores of the sorbent per unit area of its surface is reduced, which facilitates the access to phosphate ions.

In terms of the influence of the environment acidity, Fig. 5 shows that with increasing pH of the medium (slightly alkaline pH) the degree of phosphate ions extraction from wastewater decreases, and with decreasing pH (weakly acidic pH) it increases. In an acidic or strongly acidic environment (pH <5), the destruction of the sorbent begins. This is noticeable by the release of bubbles, which indicate the beginning of the carbonates decomposition.

This behavior of the studied bentonite sample is fully consistent with the general theory of the aluminosilicate framework chemical interaction of the main mineralogical phases

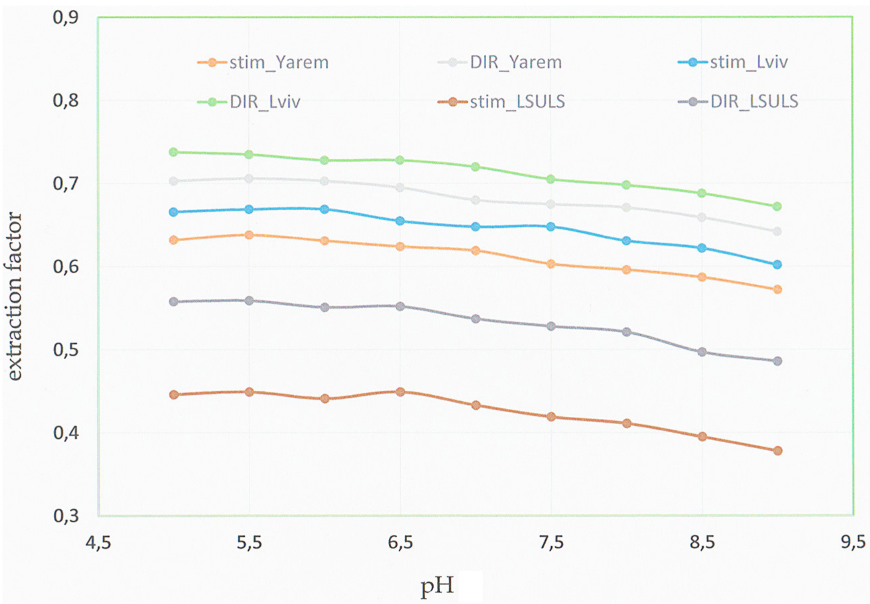

Figure 5. Dependencies of the phosphate ions level extraction from the acidity of the medium

(montmorillonite, chlorite, hydromica, zeolites) of this sorbent with acids and alkalis.

In particular, the examples of zeolite sorbents [Matsuska, 2014 et al.] found that phosphates are better sorbed in an acidic environment. At low pH, the chemical interaction of the proton with the zeolite framework, accompanied by the release of sodium into solution. In parallel, the destruction of the zeolite surface layer, the release of new sorption centers located in the volume of zeolite, increase the sorption capacity by opening dead ends and increasing the diameter of the micropores. It is likely that a stable and sparingly soluble aluminum phosphate AlPO is formed in the crystal lattice.

In an alkaline environment, in the case of sorption of two- and three-substituted potassium phosphate, potassium is replaced by sodium and calcium. Calcium interacts with hydrogen phosphate and phosphate ions, with the formation of stoichiometric and non-stoichiometric calcium hydroxyapatites.

As noted above, in the studied case, the process of the native sample irradiation already leads to the release of OH groups (increase in pH), so to quantify the dependence of the irradiated bentonite sorption parameters on the acidity of the solution is quite difficult.

Thus, the purification of wastewater with bentonite from excess phosphate ions after leaving the general purifying facilities could be most effectively carried out by using the method of "direct irradiation". However, it is difficult to apply this process on an industrial scale (on real purifying plants). In particular, it is a difficult engineering task to create such a mobile microwave installation that could move through a tank with wastewater and bentonite added to it.

At the same time, powerful stationary microwave units (in fact, large microwave ovens) have existed for a long time. Therefore, according to the authors, under the conditions of real purifying facilities it would be worthwhile to use the method of "stimulation".

The technological scheme of the process can be as follows. A certain amount of bentonite in the dielectric container is filled with distilled (desalinated) water and mixed in a large microwave unit. After several cycles of irradiation and stirring, the container is removed from the installation and left to settle. At this time, the next container can be loaded into the installation. The duration of the container irradiation should be at least 6 minutes (3 cycles of 2 minutes), as the authors reported in their previous publications [Konzur, 2016; 2018 et al.].

After settling, the wastewater is drained from the first container, and the bentonite "stimulated" in this way is ready for use. It can simply be loaded into a settling tank with pre-purified wastewater and mixed. After some time, the purified wastewater can be fed to the next stage of the process or discharged into the appropriate reservoir, and used bentonite is excellent as a filler in binders (for example, for paving slabs, building ceramics, mineral-polymer mixtures, etc.).

Regarding the optimal contact time of the sorbent and wastewater, the authors in their previous publications reported [Konzur et al., 2016; 2018] that almost complete deposition of phosphates and other pollutants occurs within 8 hours.

The ratio of the sorbent mass to the wastewater mass 1:100 allows completely precipitating the contaminant. Under real conditions, after conducting several practical studies, this ratio can be reduced, for example to 1:200 or 1:300, etc.

Thus, the authors believe that microwave activation of bentonite will significantly increase its role in waste water purifying processes from excess phosphate ions.

CONCLUSIONS

The authors proposed new ways to improve the quality of river water by reducing the content of phosphate ions in the wastewater of coastal settlements. The previously published own data and the results of other researchers have confirmed that the amount of pollutants, in particular phosphorus compounds, has increased in the upper part of the Prut River near Yaremche (Ivano-Frankivsk region, Ukraine). It was shown that the incompletely purified wastewater of Yaremche, which contains an excess of phosphate ions, has a significant impact on this fact. On the basis of their own previous and new research, the authors suggest removing these ions using the natural sorbent bentonite, activated by microwaves in different ways. As a result of current experiments, it was shown that microwave activation of this sorbent increases the coefficient of the phosphate ions extraction from wastewater several times compared to natural bentonite. An approximate technological scheme of wastewater purifying from excess phosphates after the main standard purifying cycle was proposed.

Acknowledgments

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