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Development of Adsorbents for the Removal of Phenol from Aquatic Environments

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ABSTRACT: The results of a research study of the catalytic activity of pillar aluminosilicates in the removal of phenol from wastewater are presented. The system of heterogeneous photo-Fenton was studied as an oxidizing system. For this, first, the processes of phenol adsorption on aluminosilicates developed by pillar are analyzed. It turned out that all the obtained adsorption dependences are described by the linear form of the Langmuir equation at low concentrations of equilibrium concentrations. The adsorption capacity of intercalated aluminosilicates for phenol exceeds those for the anionic dye Congo red, which is associated with the smaller size of phenol molecules and its greater activity with respect to the surface of adsorbents. The adsorption capacity decreases in the following order: 10-Fe-KR-1> 10-Fe-NAB-1> V4-Fe> Fe-EK and is: 0.221; 0.199; 0.187 and 0.123, respectively. It was found that Fe- and Fe/Cu-aluminosilicates are active catalysts for the oxidation of phenol with hydrogen peroxide (HP) in aqueous solutions and lead to its complete removal. The use of ultraviolet radiation, direct sunlight, reduces the time of 100% conversion of phenol. The activity of Fe/Cu-aluminosilicates is higher than their Fe-forms at the beginning of the reaction, but the time of complete oxidation of phenol for these catalytic systems has similar values.

KEYWORDS: adsorption, photo-Fenton system, catalysis, stability, hydrogen peroxide, ultraviolet radiation.

I. INTRODUCTION

Recently, numerous scientific works on the creation of new materials based on intercalation processes of layered aluminosilicates have appeared in foreign literature. It should be noted that the use of intercalated aluminosilicates as adsorbents and catalysts for deep purification of industrial wastewater is becoming more and more popular due to the increase in the amount of industrial waste [1].

The problems of creating intercalated layered aluminosilicates - active solid materials for photocatalysis, catalytic oxidation of peroxide compounds, the Fenton process, and catalytic oxidation of stubborn organic compounds in water bodies with moist air - are topical. The development of a columnar clay catalyst to reduce the cost of the oxidation reaction with an emphasis on low cost, stability, recyclability, and safety of the catalysts used are key issues discussed by the authors of this paper.

Scientific advances in green chemistry and engineering are aimed at solving the problem of neutralizing toxic substances in the environment. In recent years, there has been an increasing interest in the use of columnar clays as non-toxic adsorbents and heterogeneous catalysts in the treatment of wastewater from chemical, petrochemical, textile, construction, pharmaceutical and other industries [2-4].

The research and application of clay minerals in heterogeneous catalysis is becoming more and more interesting among scientists and specialists [5]. However, clay minerals inherently have a disadvantage in the form of inaccessibility of their active centers, which reduces their efficiency in catalytic reactions. Nevertheless, the modification of natural clay minerals with pillaring of giant particles has become a promising technology, due to the expansion of clay layers, providing thermal stability, increased porosity, increased values of surface area and basal distance, as well as other physicochemical characteristics [6]. Polyhydroxocations of metals such as Al, Fe, Zr, Cr, Ni and Ti are types of inorganic pillar agents that have been fairly well studied and have been discussed in detail in numerous studies [6, 7].

As noted in early works, the process of preparing pillar clays consists of important stages: preparing a pilling agent from a metal salt in a solution and mixing it with a suspension of clay in water [8]. However, it should be noted that this method requires long periods of time and a large amount of water for production, which is a major limitation for scaling up the intercalation method to industry [8, 9].

To reduce the volume of water, the intercalation method is currently focused on the use of concentrated clay suspension, dry clay and concentrated intercalation solution [10]. Research is being carried out to reduce the duration of the process, due to the direct addition of dry clay to intercalating solutions and the use in the technological process of such methods as ultrasonic [10] and microwave treatment, as well as one-stage high-temperature synthesis [11]. Several studies have reported that when using the conventional method, the aging time can vary from hours or days (1-30 days) depending on the structural characteristics of the laminates as well as the pillar agents [12]. When using ultrasonic, microwave, and high-temperature synthesis methods, the aging process and ion exchange occurs in minutes (5-30 minutes), which facilitates its distribution on an industrial scale.

The industrial production of catalysts and adsorbents based on pillar materials is influenced not only by the optimization of preparation parameters, but also by the ability to form powder materials into granules, monoliths, agglomerates, etc. In this case, the prepared solid catalytic materials must retain their physicochemical characteristics and properties of catalytic activity and stability during use.

Recently, in order to improve heterogeneous processes with the use of catalysts, since Supported iron and others are studying clay materials as a carrier [13, 14], which is associated with their low cost, availability and ease of the process of pillar formation in comparison with other methods of modification and preparation of adsorbents and catalysts [15]. Such columnar clay catalysts have been used to remove organic contaminants because phenol and its derivatives, organic dyes, toluene [16, 17], tyrosol and other stable compounds [18].

The current time is an acute issue of wastewater disposal from toxic organic compounds. Phenols and their derivatives are widely used reagents in many branches of modern industry; production of drugs, polymeric materials, pesticides, surfactants, paints and dyes, etc. Therefore, these industries, as well as others involved in the production of phenols, have emissions as waste water with a large set of polluting toxic substances, since phenols, nitrophenols, chlorophenols, etc. From the analysis of the literature, it is known that an effective and economical method of neutralizing phenol-containing wastewater is catalytic oxidation in the aqueous phase, which allows to significantly reduce other costs in the form of lowering the temperature and pressure of the oxidation processes of phenols and other organic compounds (dyes, fat and oil substances, surfactants).

Hydrogen peroxide (H_2O_2) is also an oxidizing agent in wastewater treatment. The main advantage of hydrogen peroxide over other oxidants (chlorine is usually used) is its ecological purity, since after its use there is no possible secondary pollution of various waters. This oxidizing agent is highly soluble in water and its solutions can be stored for quite a long time. Consequently, H_2O_2 is an environmentally friendly and promising oxidant in wastewater treatment from various pollutants. Modern industrial practice mainly uses the homogeneous oxidizing system of Fenton and Raff (Fe^{2+}/Fe^{3+}-H_2O_2), despite a number of significant disadvantages. It is to eliminate these shortcomings that the research results are directed, which allow the use of heterogeneous catalysts with relatively high activity and stability. The mechanism of the oxidation process was established according to HPLC (Shimadzu Prominence 20 HPLC UFLC Syst) and spectral analysis by scanning spectrophotometry in the 190-700 nm range using a UV / V-5100 spectrophotometer (Shanghai Metash Instruments Co.).

II. SIGNIFICANCE OF THE SYSTEM

The results of a research study of the catalytic activity of pillar aluminosilicates in the removal of phenol from wastewater are presented. The study of methodology is explained in section III, section IV covers the experimental results of the study, and section V discusses the future study and conclusion.

III. METHODOLOGY

As objects of research, synthesized samples of pillar materials based on Navbakhor (10-Fe-NAB) and Krantau (10-Fe-KR) bentonite, Pakhtachi enriched kaolin (Fe-EK) and Tebinbulak vermiculite (V4-Fe) were selected, data about which are given in the early works of the authors [19, 26].

Identification at the qualitative level of the products of destruction of phenol and its derivatives was carried out by the scanning method. The influence of temperature was studied in the range from 25 to 60°C, and the pH values of the medium from 3 to 9.

In the experiments, working solutions of phenol and its derivatives were prepared by successive dilution of a standard solution with an initial concentration of 1 g/dm^3. The resulting solution was standardized by iodometric titration. A solution of potassium hexacyanoferrate with a concentration of 1 g/L was used as working solutions. A borate buffer solution was prepared by dissolving 20 g of sodium tetroborate in 1 L of bidistilled water. The pH of the buffer solution was adjusted using HCl and NaOH solutions with concentrations of 1 M. Filtration of the investigated solutions and water samples was carried out using an oil-free anticorrosive diaphragm vacuum pump LH-95D/C, with a capacity of 30 l/min, which was connected to the column at the outlet. The study of stability was carried out with the determination of the concentration of metals in solution after catalysis using an atomic adsorption spectrophotometer.

IV. EXPERIMENTAL RESULTS

To establish the possibility of sorption of phenols on intercalated materials, the sorption isotherm was obtained firstly, shown in Fig. 1.

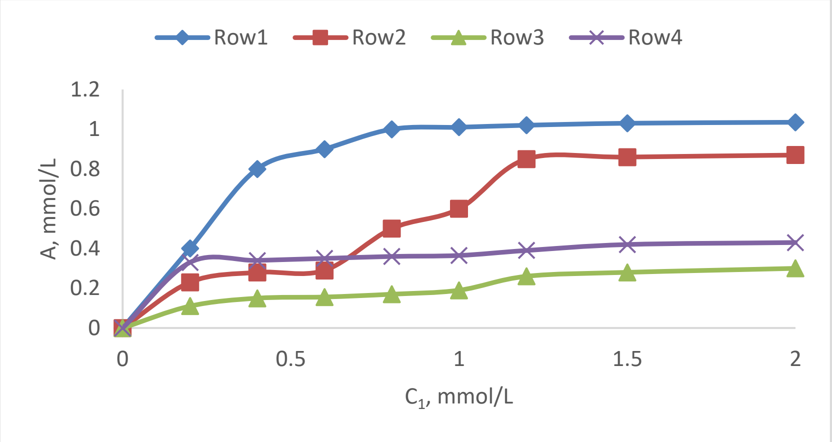

Fig. 1. Isotherms of phenol adsorption on: 1) 10-Fe-KR-1; 2) 10-Fe-NAB-1; 3) Fe-EK; 4) V4-Fe.

All obtained dependences are described by the linear form of the Langmuir equation at low concentrations of equilibrium concentrations. It turned out that the adsorption capacity of intercalated aluminosilicates for phenol exceeds those for the anionic dye Congo red [22, 23]. Probably, this is due to the smaller size of phenol molecules and its greater activity with respect to the surface of adsorbents. The adsorption capacity decreases in the following order: 10-Fe-KR-1> 10-Fe-NAB-1> V4-Fe> Fe-EK and is: 0.221; 0.199; 0.187 and 0.123, respectively. At the same time, the activities of polynuclear polycations were investigated. It turned out that the amounts of the limiting adsorption of Fe and Fe/Cu intercalated aluminosilicates differ insignificantly, which indicates a similar activity with respect to phenols.

It is known that phenol, due to mesomeric effects, has a nucleophilic center in the para position to the hydroxyl group and oxidation begins at this active center with the formation of hydroquinone and p-benzoquinone.

The kinetics of phenol oxidation by hydrogen peroxide was studied with the participation of intercalated aluminosilicates and under the influence of ultraviolet radiation (UV lamp UL-00007276 Uniel G13 18W) at temperatures from 293 to 353K and at a pH of the aqueous phase of 2-11, the data obtained are shown below (Fig. 2- 5 and Table 1).

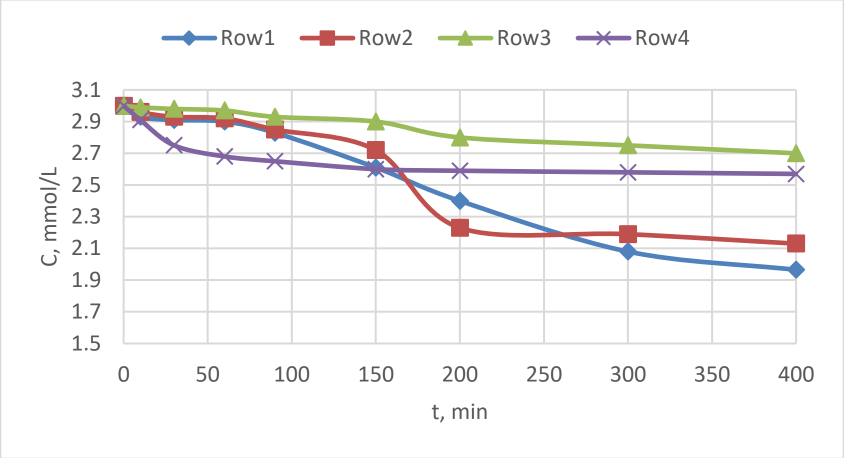

Fig. 2. Kinetics of phenol adsorption on: 1) 10-Fe-KR-1; 2) 10-Fe-NAB-1; 3) Fe-EK; 4) B4-Fe. Process conditions: 25 ± 1 ° C, pH = 7 ± 0.5, adsorbent consumption 2 g / l.

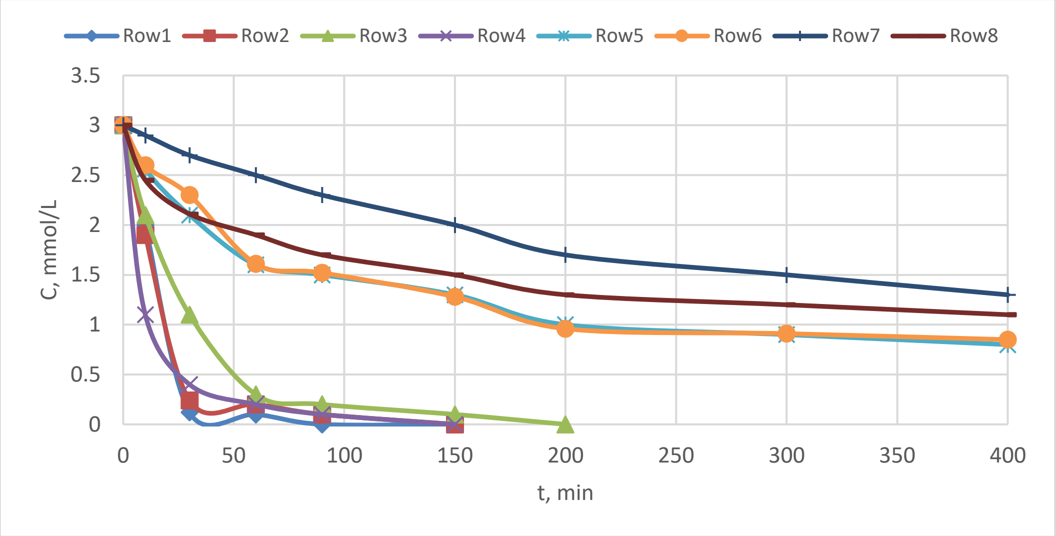

Fig. 3. Kinetics of phenol oxidative destruction in the system: 1) 10-Fe-KR-1; 2) 10-Fe-NAB-1; 3) Fe-EK; 4) V4-Fe + H_2O_2 + UV and in the system: 5) 10-Fe-KR-1; 6) 10-Fe-NAB-1; 7) Fe-EK; 8) V4-Fe + H_2O_2. Reaction conditions: 25 ± 1 ° C, pH = 7 ± 0.5, catalyst consumption 1 g / l.

It was found that Fe- and Fe/Cu-aluminosilicates are active catalysts for the oxidation of phenol with hydrogen peroxide (HP) in aqueous solutions and lead to its complete removal. Probably, the oxidation process begins with phenol molecules, which are already adsorbed on the surface of intercalated aluminosilicates. Therefore, one should expect a change in the concentration of phenol in accordance with the adsorption activity of these materials. At the same time, their activities depend on the nature of the intercalated cation.

The use of ultraviolet radiation, direct sunlight, reduces the time of 100% conversion of phenol. The activity of Fe/Cu-aluminosilicates is higher than their Fe-forms at the beginning of the reaction, but the time of complete oxidation of phenol for these catalytic systems has similar values.

UV irradiation promotes the reduction of valence iron (III) ions to Fe^{2+}. In this case, the concentration of hydroxyl radicals increases, and the rate of oxidation of phenol and its derivatives increases.

However, what is the question of the stability of the catalysts. It turned out that the stability of systems obtained by introducing polynuclear mixed Fe/Cu cations is greater than that of systems with monocations. The leaching of Me^{n+}

ions during the oxidation reaction of phenol and its homologues for the first use of intercalated montmorillonites and kaolin is less than 0.23%, while for the V4-Fe and V4-Fe/Cu samples it is 2.1 and 1.9%, respectively. It is likely that it is associated with the instability of the pillar vermiculite as a result of the separation of layers due to the low energy of bonds between the intercalated cation and the negatively charged surface of the vermiculite.

When reusing catalysts (more than 10 times) 10-Fe-KR-1 and 10-Fe-NAB-1, the loss of Fe^{3+} does not exceed 5 ± 1%, while for samples 10-Fe/Cu-KR-1 and 10 -Fe/Cu-NAB-1 losses are no more than 3%. First of all, the differences in the stability of intercalated montmorillonites are a consequence of the stability of the cations themselves and their ability to dissolve in an aqueous medium. The catalytic activity of samples V4-Fe and V4-Fe/Cu decreases with repeated use, and Fe/Cu-EK does not lose activity when used again without regeneration for 2 cycles (20 hours).

In the reaction of phenol oxidation, the catalyst 10-Fe-KR-1 is more active than 10-Fe-NAB-1, but the amount of Fe^{3+} ions in it is less. Probably, the higher activity of the former can be explained by the high dispersion of iron particles in it. Previously, the authors have shown [19-21] that the activity and stability of Fe-intercalated clays largely depend on the state of iron in them.

It was found that the process proceeds through the formation of intermediate products (hydroquinone, p-benzoquinone, the corresponding carboxylic acids with a smaller number of atoms), which undergo further complete oxidation to CO_2 and H_2O.

As it turned out, temperature is the main factor affecting the entire process. In the temperature range from 20 to 45°C, the obtained dependences are satisfactorily described by the kinetic equations of the second order reaction. Linear dependences of the reciprocal values of the phenol concentration on the process time are shown in Fig. 4, on the basis of the dependence, approximating equations and rate constants are obtained. From the temperature dependences of the logarithm of the reaction rate constants shown in Fig. 5, the values of the activation energy E_a are determined.

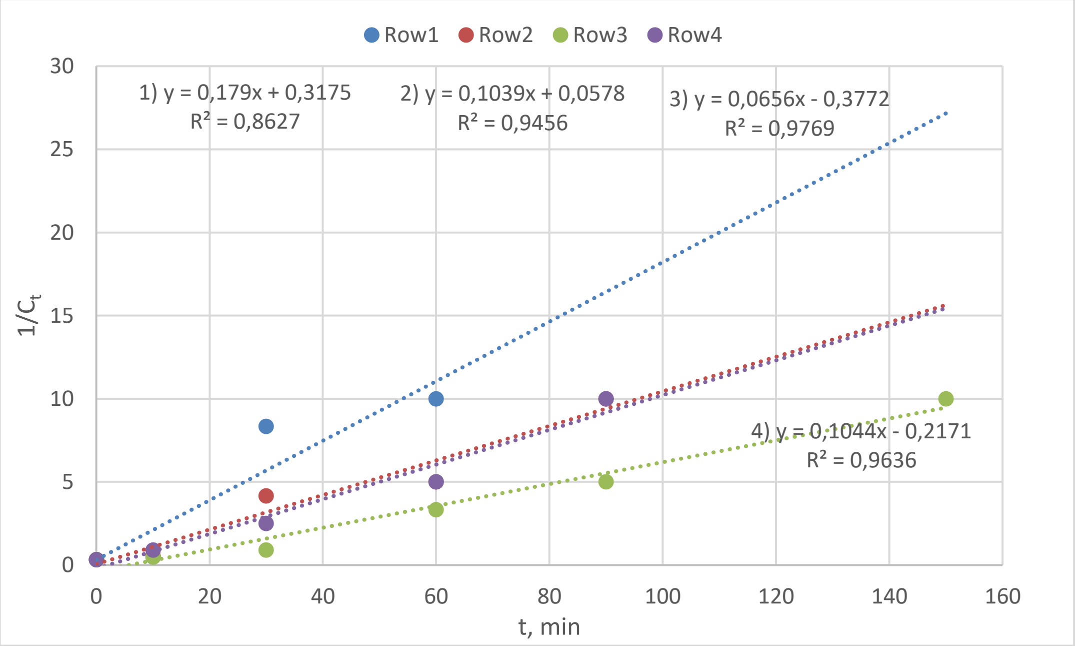

Fig. 4. The linear form of the kinetic dependences of $1/C_t$ on the time of the oxidation of phenol with an initial concentration of 5 mmol/L, at a catalyst flow rate of 1 g/L, peroxide consumption of 10 mmol/L at 25 ° C: 1) 10-Fe-KR-1; 2) 10-Fe-NAB-1; 3) Fe-EK; 4) V4-Fe.

It turned out that an increase in temperature over 45 degrees causes changes in the nature of the process, i.e. changes in the limiting stage of the process.

Table 1
Parameters of phenol oxidation process

Sample	T, °C	Approximating equation	Correlation coefficient, R²	Rate constant, k	* Ea, kJ/mol
10-Fe-KR-1	25	y=0,179x+0,3175	0,8627	0,179	48,6
	35	y=0,361x+0,2233	0,8924	0,361	
	45	y=0,786x+0,2121	0,9271	0,786	
	55	y=0,0056x	0,9828	0,0056	
	65	y=0,0061x	0,9811	0,0061	
10-Fe-NAB-1	25	y=0,1039x+0,0578	0,9456	0,1039	51,2
	35	y=0,2134x+0,0118	0,9896	0,2134	
	45	y=0,8318x+0,012	0,9891	0,8318	
	55	y=0,00511x	0,9981	0,00511	
	65	y=0,0064x	0,9986	0,0064	
Fe-EK	25	y=0,056x-0,3772	0,9769	0,056	67,3
	35	y=0,121x-0,2882	0,9898	0,121	
	45	y=0,256x-0,101	0,9689	0,256	
	55	y=0,0062x-0,116	0,9615	0,0062	
	65	y=0,0071x+0,109	0,9911	0,0071	
V4-Fe	25	y=0,1044x-0,2171	0,9636	0,1044	45,5
	35	y=0,2256x+0,266	0,9112	0,2256	
	45	y=0,468x+0,211	0,8912	0,468	
	55	y=0,0064	0,9555	0,0064	
	65	y=0,0075	0,9622	0,0075	

** -Values of activation energies, calculated by the slopes of the approximating equations based on the reaction constants in the temperature range from 25 to 45°C.*

The dependences lnk = *f*(1/T) shown in Fig.5 are also described by linear equations.

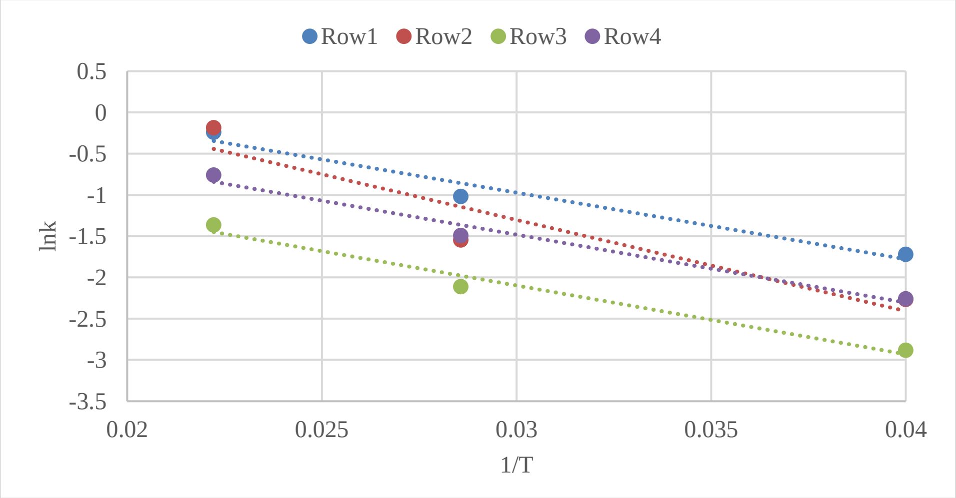

Fig. 5. Dependence lnk = *f*(1/T) in the temperature range from 25 to 45°C: 1) 10-Fe-KR-1; 2) 10-Fe-NAB-1; 3) Fe-EK; 4) B4-Fe.

Thus, the induction period decreases with increasing temperature, as well as increasing the concentration of hydrogen peroxide and decreasing the pH of the medium to 3.5. Consequently, the catalytic activity of Fe and Fe/Cu intercalated aluminosilicates in phenol oxidation reactions at pH> 4 can be related to their surface acidity.

Analysis of the experimental data obtained allows us to conclude that the oxidation of phenol by hydrogen peroxide in the presence of Fe-, Fe/Cu intercalated aluminosilicates proceeds in two different ways, mainly by the radical-chain mechanism. At the initial moment of the reaction, the interaction of PV and water molecules occurs with the formation of compounds with Fe^{3+} (Fe^{3+}/Cu^{2+}) ions:

$$> Fe^{2+} + H_2O_2 \rightarrow Fe^{3+} + OH^- + HO^{\circ};$$
$$> Fe^{2+} + HO^{\cdot} \rightarrow > Fe^{3+} + OH^-;$$
$$HO^{\cdot} + H_2O_2 \rightarrow HO_2^{\cdot} + H_2O;$$
$$HO_2^{\cdot} + > Fe^{3+} \rightarrow H^+ + O_2 + > Fe^{2+};$$
$$C_6H_5OH \rightarrow (OH)(H)C^{\cdot}(CH)_4CHOH \rightarrow C_6H_4(OH)_2 + HO^{\cdot} \rightarrow O(C_6H_4)O^- \rightarrow CO_2$$

The disappearance of the induction period on the kinetic curves upon the incorporation of copper ions into the composition of polyoxocations is explained by an increase in the rate of formation of active intermediate complexes. The data show that the rate and degree of removal of phenol and its homologues correlate with the amount of active ions in the composition of the catalysts. Therefore, for these purposes, it is advisable to use intercalated montmorillonites.

V. CONCLUSION AND FUTURE WORK

Consequently, the use of the Photo-Fenton system for the removal of phenol residues and its derivatives has its advantages in the form of high yields of the process, due to the adsorption of both adsorbates and hydrogen peroxide molecules, as well as the occurrence of oxidative processes in which active ions Fe^{3+} and OH^- participate. Thus, the heterogeneous photo-Fenton process is not only more efficient, but also environmentally friendly and economical, due to the intensification of the process by sunlight.

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