

Catalytic Activity of Fibrous Complexites

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Abstract. The ecological situation in the world requires the solution of environmental problems associated with the processes of wastewater treatment, hydrogen sulfide pollution of various industrial facilities in order to remove such harmful impurities. The unpleasant odor that appears in industrial and adjacent waste processing areas is a constant concern for the protection of the environment. To solve these problems, it is advisable to use selective sorbents - complex. The introduction of metal ions into complex fibers due to the formation of a coordinated bond between the groups of the metal and the polymer ligand gives high molecular weight complex compounds. The operational and selective properties of the materials based on high-molecular-weight complex compounds can be determined by the type of metal ion introduced into macromolecules, the nature of the polymer chain, and thermodynamic stability. By changing the action of the above factors, complexites can be synthesized for use as highly selective catalysts for various chemical processes. The paper presents data on the catalytic activity of fibers with Cu^{2+} , Co^{2+} , Ni^{2+} ions with complexing fibers containing carboxyl, amidoxime, hydroxamic groups, using the example of the decomposition of hydrogen peroxide and oxidation of H_2S , Na_2S . An inhibitory effect on the oxidation of fiber functional groups in the pH range 5.5 - 12.5 was revealed. The operational and selective properties of materials based on complexites can be determined by the type of metal ion introduced into the macromolecules, by the nature of the polymer chain. By changing the action of the above factors, complexites can be synthesized to be used as highly selective catalysts for various chemical processes. It was found that the catalytic activity of the complex depends on pH, the stereochemistry of the coordination centers in the fiber matrix and significantly exceeds the activity of model low-molecular-weight complex compounds.

1 Introduction

The ecological situation in the world requires urgent solutions of environmental problems connected with the processes of wastewater treatment, hydrogen sulfide pollution of various industrial facilities in order to remove such harmful impurities [1, 2]. During their life, people have a multifaceted impact on natural objects, mostly negative [3, 4]. It may be the impact of industry and transport [5], agriculture [6, 7], housing and communal activities [8]. The unpleasant odor that appears in industrial and adjacent waste processing areas is a constant concern for environmental protection and especially for the quality of life [9, 10]. An important problem is also the deterioration of the ecological condition of surface sources of drinking water supply, especially for water bodies, as they are designed to accumulate water reserves [11]. To solve these problems, it is advisable to use selective sorbents-complexites, out of which fiber-based complexites have great advantages [12].

As a result of similar complexing properties of fibers with granular type complexites, they can be considered as a qualitatively new modification. Due to the peculiarities of the polymer chains morphology fibrous complexes have a number of advantages in comparison with granular objects. First of all, it is a high rate of processes with their participation, the availability of reactive groups in polymer chains, and rather high sorption characteristics.

A promising approach to solving the designing and directed synthesis problem of new polyfunctional materials with desired properties based on fibrous complexites is to study the physicochemical characteristics and spatial structure of complexites, which, due to the nature of the groups, usually exhibit the properties of polyelectrolytes. Therefore, the properties of complexites depend on the nature and groups reactivity as well as the polymer matrix [13].

The reactivity of the functional groups of polyelectrolytes is usually researched by studying protolytic equilibria. The material discussed above includes information relating to the catalysts based on granular complexites. The introduction of metal ions into complex-forming polymers results in the creation of materials that differ greatly in properties from the initial reagents [14]. This happens due to the implementation of the coordination bond between the metal ion and the ligand groups of the polymer matrix, leading to the formation of high-molecular complex compounds. The performance characteristics of the materials based on high molecular complex compounds (HMCC) are determined by the type of metal ion introduced into the macromolecule, the nature of the macromolecular chain, and the thermodynamic stability of HMCC [14]. By varying the influence of these factors, it is possible to purposefully synthesize HMCC in order to obtain highly selective catalysts for oxidation processes on their basis.

2 Main Part. Analysis of Literature Data and Problem Statement

Let us give some examples characterizing the catalytic activity of fibrous catalysts as well as modern methods that make it possible to remove H_2S in various ways. Thus, the authors [15] showed that it is impossible to bleach cellulose (wood pulp, paper) with hydrogen peroxide if some traces of heavy metal ions (Fe, Cu, Mn) are found in cellulose fibers. The latter form chelates compounds with the substances present in cellulose, for example, with lignin, and cause the catalytic decomposition of H_2O_2 .

Among the many options for removing substance traces that give the air an unpleasant odor, membrane methods or techniques are the preferred options [16]. Their advantages are in the easiness of installation and scalability, selectivity; in addition, the aromatics flows are direct, the automation is achieved with the available operating parameters (pH, temperature, ionic strength), and the operating costs are low. The article [16] presents the process of obtaining membranes from cellulose derivatives containing silver nanoparticles using available raw materials. The technique used for preparing membranes consisted in the reverse phase immersion precipitation of cellulose polymer solutions in methylene-chloride: methanol, volume 2:1. The resulting membranes were morphologically and structurally characterized by scanning electron microscopy (SEM) and high resolution SEM (HR SEM), energy dispersive X-ray analysis (EDAX), Fourier transform infrared spectrometry (FTIR), thermal analysis (TG, A). Then the membranes performance characteristics were determined (extraction efficiency and substance flow) using hydrogen sulfide (H_2S) and ethyl mercaptan (C_2H_5SH) as targets.

The authors [17] studied and developed a method for biotechnological destruction of hydrogen sulfide concentration, which, due to interaction with organic and inorganic compounds, causes great losses in industry. The authors developed efficient sulfide removal by studying sulfate-reducing metabolism and achieved effective elimination of hydrogen sulfide to prevent its negative impact on certain industrial and environmental problems.

So, modern research indicates the relevance of developing methods for the selective removal of hazardous hydrogen sulfide and sulfides. Thus, the authors [18] synthesized a polysulfone (PS) membrane from dimethylacetamide (DMAC) and dimethylformamide (DMF) as a solvent. The presence of hydrogen sulfide in natural gas can result in equipment corrosion, global warming, etc. Thus, the separation of hydrogen sulfide from nitrogen is important. SEM (scanning electron microscopy) images were used to compare the membranes obtained with DMF and DMAC. The authors [18] created an experimental facility and studied the effect of solvents, PS concentration, temperature, and pressure on the separation of H_2S from natural gas. The best results were achieved at 25 °C. The results showed that DMAC solvent was more suitable than DMF, and the degree of

selectivity of this type of membrane decreased with the increase in temperature and pressure. M3 membranes (20 % PS, DMAC solvent) were the best membranes.

The research the authors [19] paper contains the analysis of acts of natural and man-made emergency situations related to the deterioration of the water quality occurring in the world and in Ukraine. The method of the express analysis of aqueous solutions which involves measuring the conductivity of the investigated aqueous solutions and calculating the coefficient of identification is proposed. It is shown that the method is informative, simple, fast and environmentally safe.

The authors [20] investigated the H₂S removal efficiency applying four nano-fluids (NF) systems based on deep-penetration eutectic solvent (DES) and measured it carrying out a dynamic absorption experiment. The NF system containing Cu proved to be an excellent absorbent for H₂S removal with significantly improved desulfurization performance compared to the original DES solution. In addition, NF systems have a relatively high regeneration capacity. NF systems and Cu nanoparticles before and after the absorption as well as after the regeneration were characterized according to Fourier transform infrared spectra (FT-IR), scanning electron microscope (SEM), X-ray photoelectron spectroscopy (XPS), X-ray diffraction, transmission electron microscope (TEM) and energy dispersive spectrum (EDS). It was found that ethanolamine, choline cation, and sulfur accumulated on the surface of Cu nanoparticles after absorption, and the solid elements on the surface after regeneration were identified as Cu and S. The S²⁻ ion existed in the form of Cu₂S, and some sulfur was oxidized to zero valence sulfur after regeneration

Environmental pollution is a major problem, and actual recovery methods are limited. The authors [21] investigated a different kind of pollutant removal materials, namely, carbon nanomaterials made from biochar, activated carbon, carbon nanotubes, and graphene to adsorb toxic gases, remove pollutants from ecosystems, and improve anaerobic digestion. Carbon materials have been found to be effective in removing nitric oxide, hydrogen sulfide, heavy metals, dyes, pharmaceutical compounds and other pollutants from the environment with adsorption efficiency up to 80 % and decomposition efficiency up to 99 %. The addition of biochar [21] results in 60% increase in biogas production. Similarly, while composting, ammonia emissions are 60 % less when biochar is added. Biomass-based carbon materials appear to be economical, sustainable and environmentally friendly. The authors [22] the method for rapid detection of hazardous pollution of the atmosphere of cities, which is based on dynamic measures of recurrence (repeatability) of the states of the pollution concentration vector, was developed. The new scientific result is related to the use of the unconventional modification of the known measures of recurrence based on the dynamic window averaging the current recurrence of the states of atmospheric pollution concentration.

Taking into account the above mentioned studies and factors, fibrous structures – fibers of natural and synthetic origin – can be an effective and convenient matrix for HMCC used as catalysts. Such data in literature are very limited.

3 The Purpose and Objectives of the Study

The purpose of this work is to study the catalytic activity of Cu²⁺, Co²⁺, and Ni²⁺ HMCC with complexing CG and NAG fibers containing carboxyl (NAG), amidoxime, and hydroxam groups, using the decomposition reaction of hydrogen peroxide and oxidation reaction of sulfur compounds (H₂S, Na₂S) as an example.

4 Materials and Research Methods

4.1 Materials

Samples of fibrous complexite CG were used as objects of study. The fiber is a copolymer of cellulose and polyacrylonitrile with hydroxamic acid and amidoxime groups. The nature of the reaction centers, physicochemical properties - ion-exchange, solvation, protolytic complexite CG are given in [23]. Complexite NAG was received by chemical modification of the industrial fiber

nitron; its matrix contains functional groups of hydroxamic acid, amidoxime, and a small percentage of carboxyl groups. The polymer belongs to polyampholytes. The starting form of a polymer used in the experiments is mixed (hydrogen-hydrate-salt, H/OH, Cl): hydrogen by carboxylic, hydroxamic, oxyimino groups of amidoxime, and hydrogen-hydrate-salt – by amidoxime. Total exchange capacity, mmol/g of the complexite was calculated from solutions of 0.1 M NaOH и 0.1 M HCl in water. Physico-chemical properties, ion exchange, solvation, protolitic, of the complexite NAG are described in [23].

Weighed portions of metal salts were selected in such a way that the number of metal ions in them corresponded to their content in HMCC [23, 24]. Simultaneously, for comparison, some studies were carried out in the absence of a catalyst. The rate of the process was controlled using the volumetric method [23], and the concentration of H_2O_2 was controlled using the permanganometry method [23]. Kinetic curves $VO_2 - \tau$ (V is the volume of released oxygen in ml, τ is the process time in minutes) were studied at: constant catalyst weight (0.1 g), changing the initial concentration of H_2O_2 (0.06-0.3 mol/l); a constant concentration of H_2O_2 (0.3 mol/l) and various weighed portions of the catalyst (0.01-0.1 g); constant concentrations of H_2O_2 (0.3 mol/l), catalyst weights (0.1 g) and different pH values (from 5.5 to 12.5) created by adding NaOH solution (with the concentration of 0.5 mol/l). The discrepancies between the results of 2-3 simultaneous experiments did not exceed 3-4%.

4.2 The study results of kinetic research

The rate of the process was determined according to the linear segments of the kinetic curves, and according to the dependence of the decomposition rate on the concentration of H_2O_2 (in logarithmic coordinates), the weight portion of the catalyst, and the pH of the medium, the order of the reaction was found with respect to H_2O_2 , the catalyst, and the concentration of protons in the system. Similar research was carried out for the oxidation of H_2S and Na_2S compounds.

With the preliminary goal of selecting concentrations during the oxidation process, we carried out kinetic studies of H_2O_2 decomposition in the presence of HMCC. And only then some model experiments were carried out to purify gas mixtures from hydrogen sulfide.

5. Discussion of the Study Results of the Kinetic Activity of Complexite

5.1 Study of catalytic activity

The kinetic curves of gas emission indicate that the rate (W) of the decomposition reaction in the presence of HMCC and low molecular weight complex compounds (LMCC) increases with the rise in H_2O_2 concentration, the addition of a catalyst, while the order of the reaction with respect to peroxide and catalyst is equal to one. With an increase in pH, the rate increases, reaching a maximum value at pH 8.5, then decreases and increases again from pH 9.5 to 12.5. In the pH ranges of 5.5–8.5 and 9.5–12.5, an inverse dependence of W on the proton concentration in the system is observed. The reaction order n with respect to $[H^+]$ decreases from 0.3 (pH 5.5–8.5) to 0.1 (pH 9.5–12.5), and in the pH range 8.5–9.5 it acquires negative values. The data obtained are presented in Table 1.

Metal salts catalyze the decomposition reaction weaker than the corresponding LMCC [15]. The established experimental facts allow us to assume that the catalytic effect in the HMCC – H_2O_2 systems is achieved due to the formation of certain coordination compounds by metal ions in the complexite phase. The complex nature of the dependences of the reaction rate on pH can be explained taking into account the distribution of Cu^{2+} , Co^{2+} , Ni^{2+} not only over the complex forms of the $M - L$ type (where M is a metal ion, L is a functional group of the complexite), but also according to the forms of mixed hydroxo-, peroxomonomeric and dimerized complexes. To prove this, we obtained the IR spectra of the complexite and all HMCs, which resemble each other in appearance, but there is some difference in certain vibration frequencies.

Table 1. Catalytic activity of HMCC in the decomposition reaction of H_2O_2 with the content of metal ions in CG - $Cu^{2+} = 0.5$ mmol/g, Co^{2+} , $Ni^{2+} = 0.4$ mmol/g, in NAG - $Cu^{2+} = 0.6$ mmol/g, Co^{2+} , $Ni^{2+} = 0.5$ mmol/g

HMCC	$w \cdot 10^4$ [mol/(L·s·g)]			$k \cdot 10^1$ [L/(mol·c)]			lg K_{ins}
	[pH]			[pH]			
	6.3	8.5	12.2	6.3	8.5	12.2	
Without catalyst	0.22	0.45	0.58	-	-	-	
CG - Cu	1.19	3.27	7.22	14.9	24.4	73.6	7.8
CG - Co	0.89	2.68	0.67	2.62	4.45	-	6.8
CG - Ni	0.61	1.49	1.79	1.78	2.62	8.99	6.6
NAG - Cu	2.52	3.83	5.63	21.4	23.6	41.6	7.7
NAG - Co	2.43	3.12	2.14	4.56	6.32	-	6.9
NAG - Ni	1.93	2.94	2.95	3.58	5.33	9.45	6.6

The IR spectrum of the complexite has absorption bands at the level of $3600 - 3200$ and 2920 cm^{-1} , which characterize the tensile vibrations of the NH and OH groups in hydroxamic acids. The absorption in the range of 1680 and 1650 cm^{-1} is connected with the tensile vibrations of the C = O, NH_2 , and C = N bonds and is characteristic of monosubstituted amides. The band at $900 - 890 \text{ cm}^{-1}$ is specific for hydroxamic acids, which is connected with tensile vibrations in the NO bond. The absorption intensity at 1550 cm^{-1} depends on the pH medium. In an alkaline medium, it increases, and in an acidic medium it decreases, regardless of the solvent composition. At the same time, in an acid medium, the $1680 - 1650 \text{ cm}^{-1}$ band expands to the high-frequency region, and the shoulder appears at 1760 cm^{-1} . This is due to an increase in the concentration of dissociating hydroxamic groups in an alkaline medium and an increase in the protonation degree of amidoxime groups, accompanied by complex formation of cations, the deformation vibrations of which are found in the range of $1760 - 1750 \text{ cm}^{-1}$. Such changes are characteristic of the complexing formation of hydroxamic acids and indicate the coordination of copper(II), cobalt(II), and nickel(II) ions with the hydroxamic groups of the complexite [14].

5.2 The ability of reducing the concentration of hydrogen sulfide in the presence of complexites

Experimental data on the decrease in the concentration of hydrogen sulfide in the presence of complexites are presented in Table 2.

The scarcity of data about the influence of the pH environment on the composition of the coordination centers of the above mentioned fibrous catalysts and on the ratio of different complex forms of metal ions in the substrate makes it impossible to suggest a catalysis mechanism for the decomposition of H_2S , H_2O_2 in the studied systems. For the same reasons, it is difficult to estimate the rates of the reaction stages involving protonated and deprotonated forms of complexites, with the participation of HMCC and complex forms of metal ions in solution, as a result of which it is impossible to carry out a correct kinetic analysis of the processes.

According to our studies (Tables 1 and 2), the rate of H_2O_2 decomposition increases in the series $Ni < Co < Cu$. There are slight differences in the activity of identical HMCC with CG and NAG complexites. This may be due to the presence in the NAG matrix of a wider range of functional groups involved in the catalytic process, as well as due to the differences in the solvation characteristics of complexites [24]. A significant decrease in the activity of cobalt HMCC (pH 12.2) is probably due to some change in the redox potential of these complexes.

Table 2. Purification of gas mixtures from hydrogen sulfide using fibrous complexites

H ₂ S, [%] in gas mixture	HMCC	Catalyst, [g]	Metal in fiber, [mg-equ/L]	Alkali concentration [mol/L]	Temperature, [°C]	Gas velocity, [ml/min]	Flow time, [min]	Purification rate [%]
1.0	NAG-Ni	0.05	1.0	0.05	40	20	60	100
5.0	CG-Cu	0.05	1.8	0.05	40	20	60	100
10.0	NAG-Cu	0.10	2.0	0.10	30	10	45	100
10.0	CG- Ni	0.10	16.2	0.10	30	10	45	99
20.0	NAG-Cu	0.15	3.0	0.15	20	5	30	100
20.0	CG- Ni	0.15	22.5	0.15	20	20	30	95

Thus, the experimental studies performed indicate that the catalytic activity of HMCC in the decomposition reaction of H₂O₂ depends on pH, exceeds the activity of model low molecular weight complex compounds [23], and rises with the increase in the stability constants of polymer complexes (Table 1).

The introduction of metal ions into complex fibers as a result of some coordinated bond formation between the metal and polymer ligand groups produces high-molecular complex compounds. The performance and selective properties of materials based on HMCC can be determined by the metal ion type introduced into macromolecules, the nature of the polymer chain, and thermodynamic stability. By changing the effect of the above mentioned factors, HMCC can be synthesized to be used as highly selective catalysts for various chemical processes.

In conclusion, we should note that the actual experimental material already available and the research intensification in the direction of a detailed study of the factors determining the structure and properties of fiber-based HMCC will help to understand the nature of the catalytic action by determining the role of metal and polymer. This can significantly expand the range of catalysts activity and help predict the processes in which they will be catalytically active.

6 Conclusions

The paper presents some data on the catalytic activity of high-molecular complex compounds with Cu²⁺, Co²⁺, Ni²⁺ ions with complex-forming fibers containing carboxyl, amidoxime, hydroxamic groups, using the example of hydrogen peroxide decomposition reaction and oxidation reaction of H₂S, Na₂S compounds.

An inhibitory effect on the oxidation process of the fiber functional groups in the range of pH 5.5 - 12.5 was revealed.

It has been established that the catalytic activity of complexites depends on pH, the stereochemistry of the coordination centers in the fiber matrix and significantly exceeds the activity of model low molecular weight complex compounds. As the stability constants of complexites increase, their activity increases as well.

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