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## PHOTOCATALYTIC TREATMENT OF WATERS, POLLUTED WITH PHENOLS

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### Abstract

The analysis of the environmental and human health threats imposed by phenols was conducted to show the need for further improvement of methods of their destruction. Being toxic in their initial composition and precursor to toxic metabolites in human body, phenols should be controlled in natural water and waste waters. They are listed as priority pollutants in most national regulation around the world and are the initial compounds for the formation of persistent organic pollutants in the environment, polluted with other active radicals. A variety of physical and chemical methods were offered for the destructive or non-destructive removal of phenols and their derivatives from water. The comparative study of possible methods, described in research papers, was conducted in terms of their efficiency and complexity to define benefits and drawbacks. The analysis showed the need for development of low energy consuming method, which needs minimal equipment and can be run under industrial condition for phenol contaminated wastewaters. Among the possible methods which meet the mentioned criteria photocatalytic destruction of phenols was showed to be perspective. A series of experiments was conducted using a range of water solution of phenol and different dosage of catalysts. The catalysts used in experiments were made of 6 modification of titanium oxide and bismuth ferrite. The initial and residual concentration of phenol was controlled by the means of high-performance liquid chromatography. The duration of the exposure and the type of light were other independent variables. The results of the whole sequence of experiments demonstrated higher efficiency of rutile under visible light and one hour of exposure. The tested photocatalytic system is simple and therefore technically and economically feasible.

**Key words:** photocatalysis, pollution of water, phenol, wastewater, catalyst, titanium oxide.

### Introduction

The perspectives of photocatalysis application for the treatment of organic pollution in water

Under modern conditions of chemical synthesis development, the composition of environment pollution becomes increasingly complex. Moreover, there is a rising concerning about high and contains a wide variety of organic compounds not typically present in the environment and therefore impregnable to degradation under the influence of environmental factors. These concerns were formulated in the form of the Stockholm Convention on Persistent Organic Pollutants, putting a ban on production or limiting application of the most hazardous and toxic POPs since 2001. Currently this convention is considered one of successful multilateral environmental agreements, as it has managed to cut dramatically the input of the given substances into the environment [1]. The list of the POPs under control is not final yet, new substances are added to it as new evidence about environmentally and medically significant negative consequences of some chemicals are received and widely proved.

However, many of those substances included in the convention list have aromatic structure included and this fact makes pay more attention to the more common pollution of waters with simple aromatic substances, able to transform into the POPs in natural environment. Among the most significant precursor phenol and benzene are the most common and widespread. However, due to the low solubility of benzene in water, and, accordingly, low concentrations of such pollution,

the level of environmental issues raised by benzene in natural water is not urgent.

### Problem statement

Phenol and its chemical derivatives are essential for production of polycarbonates, epoxies, bakelite, nylon, detergents, herbicides, and numerous pharmaceutical drugs. The biological significance of phenol is usually considered in terms of its environmental impact. Phenol is one of the industrial pollutants. Sources of pollution of water bodies with phenols are wastewaters from the enterprises of oil refining, oil shale processing, wood-chemical, coke-chemical, aniline-paint industries [2]. The content of phenols in the natural waters affected by these discharges can reach over 40 µg/l with very diverse derivatives [3].

The discharge of phenolic waters into water bodies and watercourses sharply worsens their general sanitary condition, since, firstly, these compounds have a toxic effect, and secondly, they intensively absorb oxygen dissolved in water, which negatively affects the vital activity of organisms in water bodies [4]. It is also harmful to many microorganisms, therefore, industrial wastewater with high phenol content cannot be treated by biological methods efficiently [5].

Phenolic compounds are included into the list of pollutants of priority concern by the United States Environmental Protection Agency and the European Union [6]. In humans phenolic compounds exert endocrine disrupting effects, as they can easily penetrate the skin and the gastrointestinal tract. Next they are transformed by metabolic actions into intermediates

able to react with proteins and thus violate their functions [7–9]. Heart, kidneys, and liver damage may occur with exposure to high levels of phenol, and there are evidence of increased cancer risks due to exposure to its metabolites [9, 10].

Thus, there is an urgent need in finding ways to control the presence of phenolic compounds in waters, considering their easy distribution and high action potential at low concentrations.

**Analysis of the recent researches and publications.** The removal of phenols from water is thus a topic and still complicated problem, due to high diversity of its chemical forms and sources of formation, as well as the need for sophisticated equipment and reagents to be applied. There exists a range of potential methods, however, they are mostly presented in the form of innovation ideas, but not industrial level technologies of water treatment [11].

More typical methods being currently applied are distillation, liquid-liquid extraction, adsorption, solid-phase extraction, wet air oxidation, catalytic wet air oxidation, and biodegradation for removal of phenols. However, they all have certain drawbacks [9, 11–14]:

1. Distillation method is energy consuming, though some technologies give possibility to recover phenolic compounds.

2. Membrane technologies are reliable and economically feasible, but membrane fouling can radically bring the efficiency down. These methods include membrane bioreactors and fiber membranes, photocatalytic membrane reactors, high-pressure processes, including nanofiltration, reverse osmosis, and pervaporation; and membrane distillation.

3. Adsorption and extraction technologies demonstrate variable efficiency limited by the cost of using and recycling the required adsorbent, or extractant. Activated carbon is the most typical sorbent used, despite its high cost, and many research efforts are aimed at carbon activation by chemical modification, nanoparticles, or at its substitution with lowcost biosorbents, such as cellulose and chitin/chitosan, or their combination.

4. Chemical oxidation with ozone, chlorine compounds or permanganate destroys phenols with high efficiency and low cost, but works better when the concentrations are at the level of ppm and rises concerns about toxic byproducts.

Innovative methods employ combination of conventional techniques or novel equipment enabling specific treatment of carrying media: electrochemical oxidation, photo-oxidation, ozonation, UV/H<sub>2</sub>O<sub>2</sub>, Fenton reaction, membrane processes and enzymatic treatment. Most of these processes represent the forms of destructive treatment of aqueous phenols, with no reagent requirements and cost, but needs special equipment and consumes energy. The least expensive and technically feasible is photocatalytic destruction of phenols.

Photocatalytic technology has a significant advantage over the others, as it produces no waste, moreover, it completely degrades the pollutant instead of transforming it into possible toxic products [9]. The decomposition of pollution occurs to carbon dioxide and

water, that is, those compounds that are contained in the atmosphere. Of course, carbon dioxide is a greenhouse gas, and in this sense, it is pollution, but this type is not toxic for humans and other living organisms directly.

The research result from open-access materials demonstrate considerable efficiency of the photocatalysis for phenols destruction. However, some of them demonstrate the best results only under combination of very specific conditions, which are not applicable under real-industry conditions. So, there is a need to define the potential of the given method under the minimal manipulation of treated waters parameters and the simplest catalytic system.

The aim of this work is to determine the potential degree of phenol decomposition in water solutions by photocatalysis. The specific tasks of the research were:

- to determine the photocatalysts which provide higher efficiency of decomposition;

- to select the efficient working conditions, including limit concentration of the substance in water yielded by photocatalysis, amount of catalyst, and type of light;

- to customize the settings of the chromatograph for accurate determination of phenol and benzene in water.

#### **Methods and materials**

Photocatalysis is a change in the rate of a chemical reaction or its initiation under the influence of light (usually near UV, visible and, rarely, near IR) with the participation of a substance (photocatalyst), which, when it absorbs light quanta, is capable of causing chemical transformations of the reactants, repeatedly entering into intermediate chemical interactions with them and regenerating its chemical composition after each cycle of such interactions.

The most common structure of photo-oxidation system includes TiO<sub>2</sub> as a photocatalyst, but experiments show that clinoptilolite zeolite and FeO-based nanoparticles [15], TiO<sub>2</sub>/reduced graphene [16], ZnO [17], Fe<sub>2</sub>O<sub>3</sub> on carbon nanotubes [18] and CuO [19] can also demonstrate positive results.

To provide the highest efficiency it is necessary to pick up the combination of medium parameters, such as the catalyst dose, exposure time, solution pH and light intensity. In particular, Shahrezaei et al. obtained up to 90% degradation of phenols under temperature of 318 K, pH 3 and 100 mg/l catalyst concentration after 2 hours of application [20]. A combination of methods is also able to improve efficiency and even provide 100% removal, as in the case of O<sub>3</sub>, UV, and TiO<sub>2</sub> combination [21].

Since the aim of the research is to evaluate the efficiency of the most feasible parameters of water treatment, which could be applied in bulk to wastewaters at the enterprises of interest, high phenol concentrations were investigated with application of the most common and available catalysts.

A series of aqueous solution of phenol were used in the experiment: 4 g/l; 1 g/l; 0.5 g/l; 0.25 g/l; 0.16 g/l; 0.8 g/l. Titanium dioxide (IV) and its derivatives were used as the catalysts (see Table 1). Titanium dioxide was annealed for use in all experiments and synthesis. Annealing was carried out in the furnace at 400 °C for 4 hours. Then, the obtained powder was air dried and

applied in the amount depending on the target substance concentration and experimental conditions: 5 mg, 10 mg, 15 mg, 50 mg, 75 mg, 100 mg of catalyst powder.

No additional oxygen was supplied to the model system, as the efficiency of the process doesn't change much depending on oxygen flow rate manipulations [22].

Table 1 – Description of the catalysts used

Catalysts	Composition
TiO <sub>2</sub>	Annealed titanium dioxide
CNTs (TiO <sub>2</sub> -coated)	TiO <sub>2</sub> -coated carbon nanotubes obtained in the laboratory
TiO <sub>2</sub> (rutile)	Rutile-structured titanium dioxide
TiO <sub>2</sub> (Ni-coated)	Nickel-coated titanium dioxide
TiO <sub>2</sub> (Pt-coated)	Platinum coated titanium dioxide
TiO <sub>2</sub> / CNTs (commercial)	Titanium dioxide composite with commercial carbon nanotubes
TiO <sub>2</sub> / CNTs (obtained)	Titanium dioxide composite with carbon nanotubes obtained in the laboratory
BiFe <sub>0.8</sub> Mn <sub>0.2</sub> O <sub>3</sub>	Bismuth ferrite obtained in the laboratory

In the course of experiment 30 mL of aqueous solution of the substance used and 5 - 100 mg of the catalyst powder were placed in the reactor. The reaction mixture was stirred under the UV or visible light for 0.5, 1, 2, 4 or 8 h. Then, obtained suspension was filtered to obtain a pure solution without dispersion. Afterwards, 5-20 µL of the sample were loaded into a vial and examined by HPLC, using the chromatograph "Perkin Elmer HPLC Series 200". Parameters of the research are showed below:

- Detector: DAD (detectors A and B);
- Wavelength: 205 nm – detector A, 254 nm – detector B;
- Temperature: 10...30 °C;
- Pressure range: 145...195 MPa;
- Chromatograph phases: A and D – acetonitrile or methanol, B and C – water;
- Flow rate: 0.3...1;
- Injection volume: 5...20 µl;
- Detector time: 15...40 min;
- Injection Source: Autosampler;
- Loop size: 200 µl;
- Excess volume: 10 µl;
- Air cushion: 10 µl;
- Sample syringe size: 250 µl;
- Needle level: 10%;
- Flush volume: 1000 µl;
- Flush cycles: 2;
- Post-injection cycles: 1;
- Detectors bandwidth: 20 nm;
- Detectors reference wavelength: 360 nm.

The sample is moved to the detector by the acetonitrile or methanol (A and D), and water flow (B

and C). The study was carried out at 10...30 °C and 145...195 MPa for 15 or 40 min.

The obtained peaks of water and phenol were studied with the HPLC software. Also, clear solution of substance used are studied. The resulted efficiency was defined by comparing peak areas before and after the reaction. The measurement error was ±10%. The materials and equipment used for experiments are given at Fig. 1 and Fig. 2.



Figure 1 – Equipment for experiments



Figure 2 – Chromatograph and PC for results analyzing

**Results and discussions**

The first experiment was carried out under the following conditions: lamp with visible light, 50/50 acetonitrile and water phases, 0.8 g/l of phenol in water, and 5 mg of titanium dioxide with a rutile structure, time 5, 10, 20, 30 minutes. The results were compared with data, obtained from pure sample.

With the help of the TotalChromNavigator software and its GraphicEdit component, the amount of phenol that has undergone photocatalysis was determined. The conversion of phenol after 5 minutes reached 35.19%; after 10 minutes it reached 25.77%; after 20 minutes it reached 24.32%; after 30 minutes it reached 25.96%. Such a strong difference with the results at the fifth minute is due to the shift in the baseline.

In the next experiment the platinum coated titanium dioxide was used as a catalyst in the amount of 10 mg. The conversion of phenol after 30 minutes reached 24.56%; after 60 minutes it reached 12.99%; after 120 minutes it reached 19.33%.

Then we selected the optimal amount of titanium dioxide with the rutile structure to increase the efficiency of the experiment. We took 50-75-100 mg of the catalyst and compared their effectiveness when under the lamp for 30 minutes. The conversion at 50 mg was 20.57%, at 75 mg it was 29.42%, at 100 mg it was 20.74%.

Next, the efficiency of 100 mg of TiO<sub>2</sub> in relation to 100 mg of nickel-covered TiO<sub>2</sub> was studied under the application of mercury lamp with ultraviolet and visible light. The concentration of the working solution was reduced to 0.16 g/l. After 30 minutes with TiO<sub>2</sub> Ni-coated catalyst the conversion efficiency was 48.14% and with TiO<sub>2</sub> catalyst it reached 63.33%, which confirms its higher efficiency.

Further experiments with this catalyst showed very promising results: after 60 minutes the conversion was 70.62% and after 120 minutes 75.9% was finally decomposed. As it is seen from the dynamics, no further increase of exposure duration is necessary, as the efficiency would not change dramatically.

We also considered the possibility of working with solution with the concentration of 4 g/l and 100 mg of TiO<sub>2</sub>, but the conversion was low: after 30 minutes only

11.37% were converted, after 60 minutes it was 11.43%; after 120 minutes it was 5.89%, which showed that for these conditions, the concentration of 4 g/l is high and weakly amenable to photocatalysis. Concentrations of 0.5 mg/l and 0.25 mg/l were also too strong for these conditions.

Thus, we continued with the main working concentration of 0.16 g/l and tried TiO<sub>2</sub> Pt-coated as a catalyst in the amount of 10 mg, and the periods of exposure were 15, 30, 60, 120, 240 minutes, 3 samples were also taken at each time interval. In this course of experiments the conversion after 15 minutes was 5.86%; after 30 minutes it was 6.03%; after 60 minutes it was 8.37%; after 120 minutes it was 29.33%; after 240 minutes it was 28.35%, which shows the greater efficiency of TiO<sub>2</sub> Pt-coated over long periods of time, but the value of the conversion was not considerable.

The experiment with BiFe<sub>0.8</sub>Mn<sub>0.2</sub>O<sub>3</sub> as a catalyst in the amount of 15 mg under the same conditions and timing demonstrated similar efficiency: the conversion after 15 minutes was 5.94%; after 30 minutes it was 8.87%; after 60 minutes it was 17.45%; after 120 minutes it was 26.46%; after 240 minutes it was 28.08%.

The last experiment was conducted using the same experiment framework with 10 mg TiO<sub>2</sub>/CNTs catalyst. The conversion after 15 minutes was 10.25%; after 30 minutes it was 9.34%; after 60 minutes it was 17.84%; after 120 minutes it was 19.09%; after 240 minutes it was 19.78%, which shows average efficiency for both small and long time intervals.

Combining the results obtained we can conclude that the most perspective catalyst for the treatment of water to reduce the phenol concentration is titanium dioxide with the rutile structure, the corresponding chromatograms are shown below (Fig. 3–6).

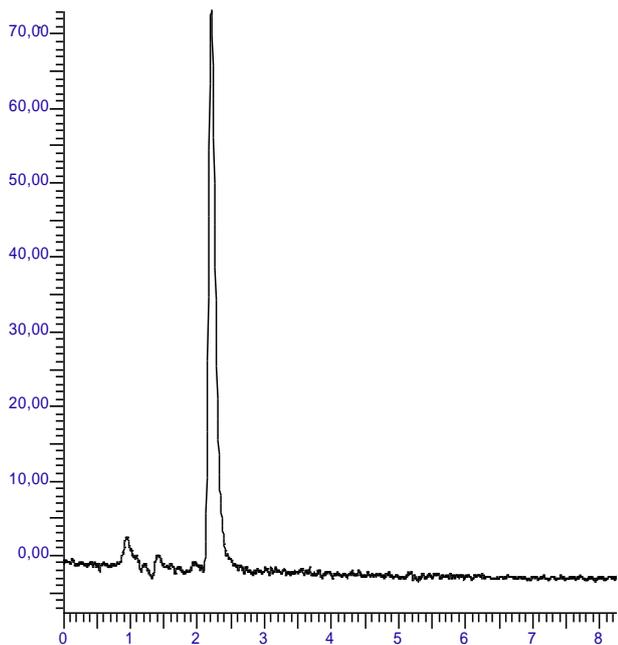


Figure 3 – The solution of phenol before photocatalysis

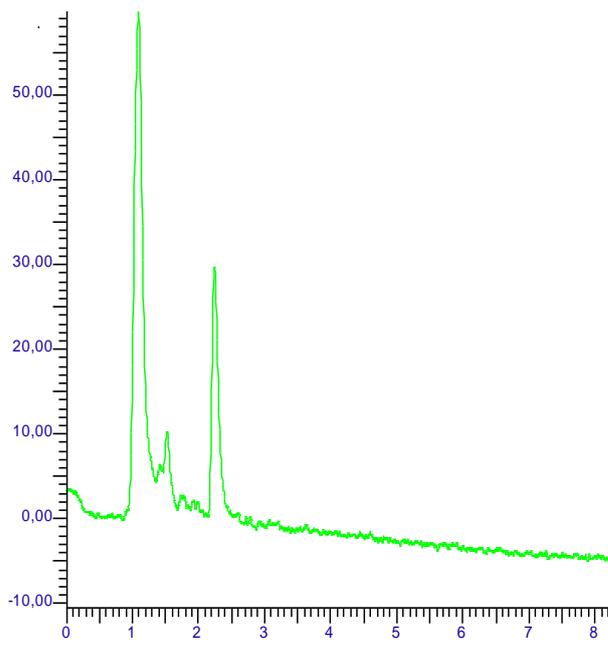


Figure 4 – The solution of phenol photocatalyzed for 30 minutes with 100 mg TiO<sub>2</sub> (rutile)

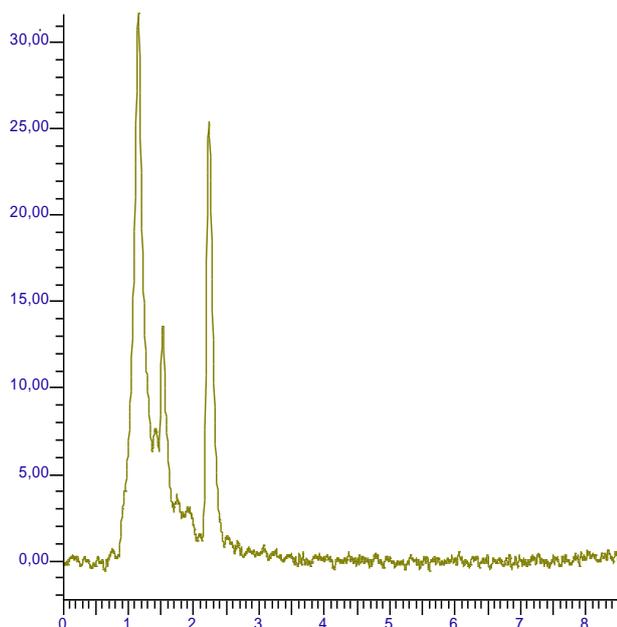


Figure 5 – A solution of phenol photocatalyzed for 60 minutes with 100 mg TiO<sub>2</sub> (rutile)

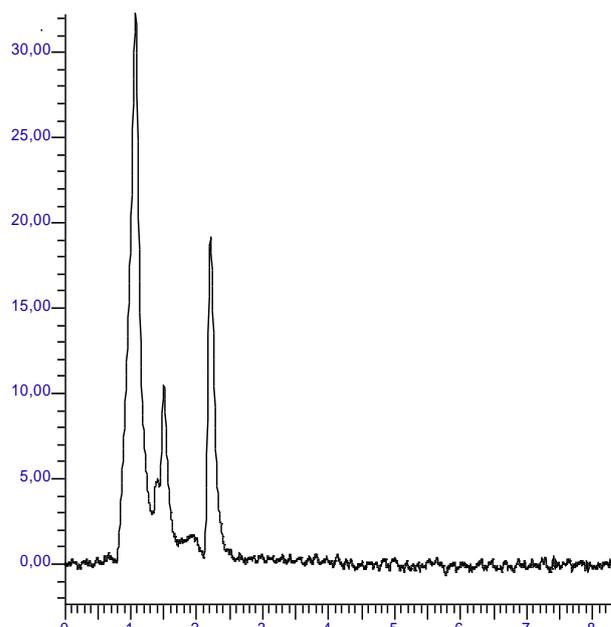


Figure 6 – A solution of phenol photocatalyzed for 120 minutes with 100 mg TiO<sub>2</sub> (rutile)

Recent studies of photocatalytic degradation of phenol also try to target the most natural conditions, with natural pH and minimal dosage of catalyst. In particular, the research by Norouzi et al. demonstrates up to 92.91% degradation with application of Ag/TiO<sub>2</sub> nanofibers, which are less common and more expensive [23]. Still, the perspectives of the given method are wide and it worth further research efforts.

### Conclusions

1. The level of environment pollution with organic substances is growing higher in terms of both quantity and variety of compounds. Many of these substances have proved to be toxic for humans and other organisms, leading to carcinogenic and other systemic effects. Such substances production and application is now limited by the Stockholm Convention on Persistent Organic Pollutants.

2. There are numerous conventional and innovative methods for the treatment of waters from phenol, but they are mostly under development and discussion and the industrial installations rely on treated using ozone (O<sub>3</sub>), activated carbon or reverse osmosis. The possibility of full-scale treatment of wastewaters under industrial conditions is limited by the need to create specific conditions and apply complex equipment.

3. Photocatalysis is a developing area that has prospects in the future, since pollution with organic pollutants occurs every day, and there are not so many efficient and safe methods for dealing with such

pollutants. Photocatalytic technology has a significant advantage over the others, due to absence of toxic waste products.

4. Phenol is a precursor to the formation of many most toxic organic pollutants of the environment, exerting threats to the health of various groups of organisms, including humans. It is listed as a highly hazardous pollutant in many hygienic standards, as it is a common component of wastewaters at various industries.

5. A series of experiments was conducted using model solutions of phenol of various concentrations (0.08-4 g/l) and dosage of catalyst (5 - 100 mg of the catalyst powder). The duration of treatment varied from 30 to 120 minutes. The different types of light (UV and visible range) and composition/structure of catalyst were tested, including BiFe<sub>0.8</sub>Mn<sub>0.2</sub>O<sub>3</sub>, TiO<sub>2</sub>/CNTs, Ti oxide with the rutile structure, Ti oxide covered with Ni or Pt.

6. After analyzing the obtained experimental results, we can say that TiO<sub>2</sub> with the rutile structure, which was obtained in a furnace by firing at high temperatures, plays the best role of a catalyst. The resulted efficiency was over 60% and it was also established that the sufficient duration of treatment is 60 minutes, as further prolongation of exposure doesn't give noticeable improvement of results.

The obtained data may be used for the development of industrial technologies of wastewaters treatment with minimal application of complex equipment and catalyst.

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#### ФОТОКАТАЛІТИЧНЕ ОЧИЩЕННЯ ВОД, ЗАБРУДНЕНИХ ФЕНОЛАМИ

Проведено аналіз загроз довкіллю та здоров'ю людей, зумовлених присутністю фенолів у природних водах та показано необхідність подальшого вдосконалення методів їх знищення. Оскільки феноли є токсичними речовинами у незмінній формі і є попередниками токсичних метаболітів в організмі людини, їх слід контролювати в природних і стічних водах. Вони включені до переліків пріоритетних поллютантів у національних нормативних актів у всьому світі, оскільки сприяють утворенню стійких органічних забруднювачів довкілля в присутності інших активних радикалів. Для деструкції фенолів та їх похідних у воді без утворення залишкового забруднення слід надавати перевагу фізико-хімічним методам. Проведено порівняльне вивчення можливих методів, описаних у наукових роботах, з точки зору їх ефективності та технічної складності. Аналіз показав необхідність розробки малоенергоємного методу, який потребує мінімального обладнання та буде дієвим при застосуванні у промислових умовах для очищення стічних вод, забруднених фенолом. Серед можливих методів, що відповідають зазначеним критеріям, перспективним виявилось фотокаталітичне руйнування фенолів. Була проведена серія експериментів з використанням ряду водних розчинів фенолу та різних доз каталізаторів. Каталізатори, використані в експериментах, включали 6 модифікацій оксиду титану та ферит вісмуту. Початкову та залишкову концентрацію фенолу контролювали за допомогою високоефективної рідинної хроматографії. Іншими незалежними змінними були тривалість експозиції та тип світла. Результати всієї послідовності експериментів продемонстрували більш високу ефективність рутиду при видимому світлі та протягом години експозиції. Перевірена фотокаталітична система є простою і тому технічно та економічно доцільною.

**Ключові слова:** фотокаталіз, забруднення води, феноли, стічні води, каталізатор, оксид титану.

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