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Satbayev University

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ИЗВЕСТИЯ

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РЕСПУБЛИКИ КАЗАХСТАН
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TITANIUM OXIDE-OXIFLUORIDE SYNTHESIS AND STUDY OF ITS PHOTOCATALYTIC PROPERTIES

Abstract. The research article discusses a new method for the photocatalyst synthesis based on a mixture of titanium oxide - titanium oxyfluoride. The synthesis was performed by dissolving titanium metal in an acidified solution of ammonium bifluoride, followed by oxidation to the highest oxidation state and hydrolysis with ammonia to obtain metatitanic acid. The decomposition of metatitanic acid was carried out in a muffle furnace at a temperature of 380°C. It was found that during the precipitation of metatitanic acid, the titanium compound with the fluoride ion also passes into the precipitate, which, after calcination, transforms into titanium oxyfluoride. Photocatalytic activity was determined by the oxidation reaction of the methyl orange dye upon irradiation with ultraviolet radiation. The source of ultraviolet radiation was a DRT-125 lamp with a wavelength in the range of 200-400 nm. Suspensions containing 60 mg/L of the dye and various contents of the photocatalyst in the concentration range of 1-7 g/L were subjected to oxidation. The construction of the calibration graph and the determination of the concentrations were carried out on an SF-2000 spectrophotometer at a wavelength of 466.4 nm. The operating wavelength was determined by scanning the solution in the wavelength range of 200-800 nm. It was found that as a result of oxidation, the solution pH shifts to the acidic side to 3.5, which leads to a shift in the wavelength of the maximum absorption; in this regard, the solutions pH was adjusted to the same values.

Key words: Titanium oxide, titanium oxyfluoride, photocatalyst, ultraviolet radiation, oxidation.

Introduction. Highly dispersed titanium oxide in its pure form, as well as doped with various elements in the processes of heterogeneous oxidation of various organic compounds, exhibits high photocatalytic activity, and oxidation proceeds to carbon dioxide and water [1-4]. This phenomenon formed the basis of numerous developments aimed at cleaning air and water environments from various organic compounds [5]. Nanocrystalline titanium oxide also has bactericidal activity, which makes it possible to use it not only for photocatalytic purification, but also for simultaneous disinfection [6-8].

The mechanism of photocatalytic reactions proceeding on titanium oxide was proposed. Light absorption in TiO_2 occurs at wavelengths less than 387 nm (for anatase with a band gap of 3.2 eV), which leads to electron advancement from the valence band to the conduction band of the semiconductor. This excitation process creates an electron in the conduction band and an electron hole in the valence band. The electron-hole pairs formed in this way migrate to the surface, where they can initiate oxidation-reduction reactions with adsorbed organic molecules [9].

The most common method for titanium dioxide synthesis today is the hydrolysis of its compounds both in an aqueous medium, where its inorganic salts are precursors, and in an organic medium using titanium tetrabutoxide or tetraisopropoxide [10-13]. When precipitating in an aqueous medium with ammonia, alkalis and alkali metal carbonates, the minimum size of primary particles does not depend on precipitant nature, but varies by the synthesis conditions, while aggregates of various sizes are formed [14]. The sol-gel method makes it possible to synthesize titanium dioxide with various geometric shapes of primary particles - spherical, needlelike, filamentous and others [15-17]. Electrochemical anodizing of titanium foil in fluorine-containing ethylene glycol resulted in a self-assembled membrane of titanium dioxide nanotubes with a high ratio of length to diameter (about 1500) [18].

Materials and methods of research. In this research work, the precursor was synthesized by dissolving a titanium plate in an ammonium hydrogen fluoride solution with a concentration of 100 g/L. In the first hour, dissolution proceeds very intensively, then the rate decreases by half for each hour, while the plate surface was covered with a layer of insoluble violet-red precipitate. When the solution is acidified with hydrochloric acid, there was an intensification of the process. After complete dissolution, a green solution with a precipitate was obtained. By air flush and heating to 90°C, the precipitate was completely dissolved. Then the solution was electrochemically oxidized in the anode half-space until discoloration. Hydrolysis was carried out with an aqueous solution of ammonia with vigorous stirring for 4 hours. The precipitate that formed was washed and calcined at a temperature of 380°C.

Photocatalytic activity was determined by the oxidation reaction of the methyl orange dye upon irradiation with ultraviolet radiation. The source of ultraviolet radiation was a DRT-125 lamp with a wavelength in the range of 200-400 nm. Suspensions containing 60 mg/L of the dye and various contents of the photocatalyst in the concentration range of 1-7 g/L were subjected to oxidation. The construction of the calibration graph and the determination of the concentrations were carried out on an SF-2000 spectrophotometer at a wavelength of 466.4 nm. Operating wavelength was evaluated by scanning the solution in the wavelength range of 200-800 nm. It was found that as a result of oxidation, the pH of the solution shifts to the acidic side to 3.5, which leads to a shift in the wavelength of the maximum absorption; therefore, the pH of the solutions was adjusted to the same values. To compensate for the thermal energy released by the lamp, the oxidation process was carried out in a thermostatically controlled cell.

Result and discussion. According to figure 1, subsequent to the results of X-ray phase analysis, the obtained sample is a mixture of titanium dioxide in the anatase form and titanium oxyfluoride ($TiOF_2$). Such a composite material is a promising anode material in lithium-ion batteries [19].

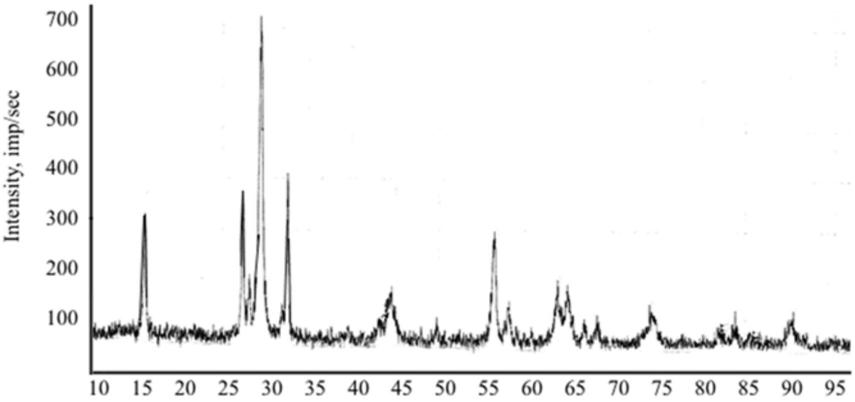

Figure 1 – X-ray diffraction pattern of a photocatalyst sample

In accordance with figure 2, electron microscopic studies have shown that powder aggregates consist of smaller particles; the average particle size calculated from the X-ray diffraction patterns using the Scherrer formula is 30-35 nm.

A spectrophotometric study of the prepared suspensions made it possible to estimate the adsorption degree of methyl orange on photocatalyst surface. The study was carried out with vigorous stirring without light access. In conformity with curve 1 in Figure 3a, in the first half hour the adsorption was 4.67%, followed by attenuation. Function selection for a given curve gives an equation of the form $y = -5.506\ln(x) + 99.722$. As follows from equation, after 10 hours the maximum adsorption will be about 18%. It ought to be noted that there is no visual change in the photocatalyst color. Conforming to curve 2 in figure 3a, intense oxidation of the dye is observed under ultraviolet irradiation. In addition, according to figure 3b, an oxidation intensity increases with an increase in the content of the catalyst in the suspension.

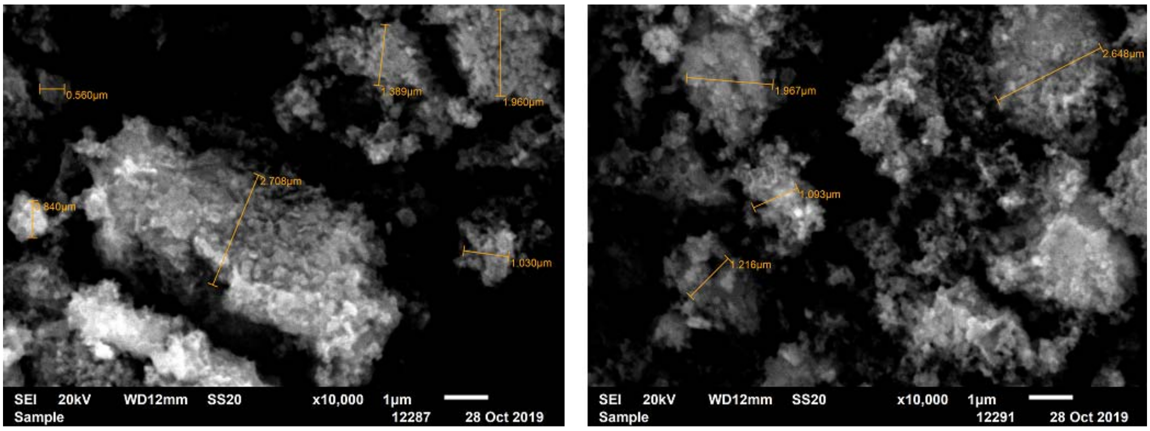

Figure 2 – Microphotography of a photocatalyst sample

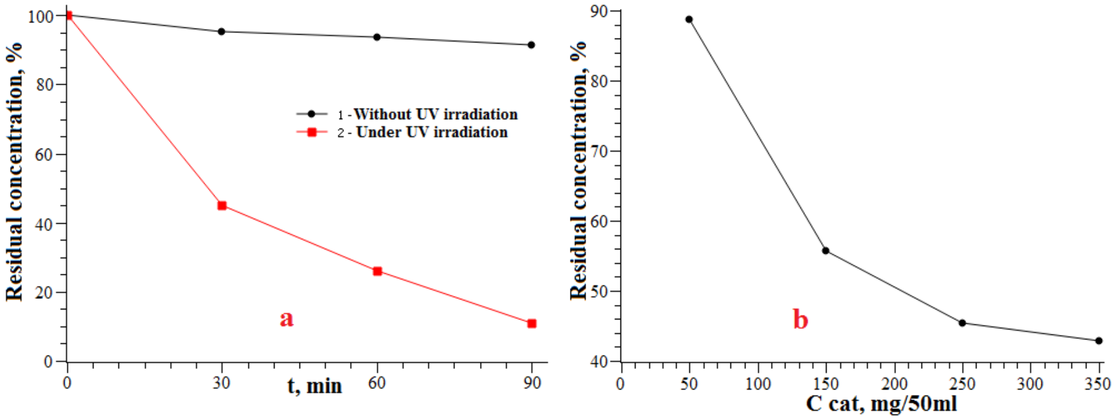

Figure 3 – Dye adsorption and oxidation dynamics

In the kinetics of homogeneous, fermentative and heterogeneous catalytic reactions, the Michaelis - Menten equation is often used [20]:

$$W_0 = \frac{kcat*[E]t*[A]}{A+Km}$$

where K_m and product of $k_{cat}[E]_t$ – parameters of the Michaelis - Menten equation: Michaelis constant and limiting speed; W_0 – initial reaction rate; [A] – initial reagent concentration (substrate). The Michaelis-Menten model assumes that substrate A initially forms a complex with catalyst E, which converts to product B much faster than in catalyst absence. The rate constant k_{cat} is much higher than the non-catalytic reaction constant k. The constant k_{cat} is also called the "number of rotations" because it corresponds to the number of substrate molecules converted into a product by one catalyst molecule in 1 s. The equation contains two parameters that do not depend on substrate [A] concentration, but characterize the properties of the catalyst: this is the product k_{cat} [E] t, which corresponds to the maximum reaction rate W_{max} at a high substrate concentration, and K_m, which characterizes the affinity of the catalyst to the substrate. The Michaelis constant is numerically equal to the substrate [A] concentration at which W_0 reaches half the maximum value W_{max}. The high affinity of the enzyme for the substrate is characterized by a low K_m value and vice versa.

To calculate W_{max} and K_m, it is necessary to find the values of W_0 at different initial concentrations of the dye and at a constant concentration of the catalyst. Figure 4a shows the kinetic curves. W_0 conforms to the slope of the corresponding curve. Water was taken as the resulting reaction product, as a final product in the oxidation of organic compounds. In obedience to figure 4b, plotting in coordinates $W_0 = f(W_0/[A])$ (Eadie-Hofstee coordinates) gives a straight line, the tangent of inclination angle is equal to "minus" K_m, and the segment cut off on the ordinate corresponds to W_{max}. When processing the results, W_{max} is 0.026 mmol/min, and K_m = 0.12 mmol.

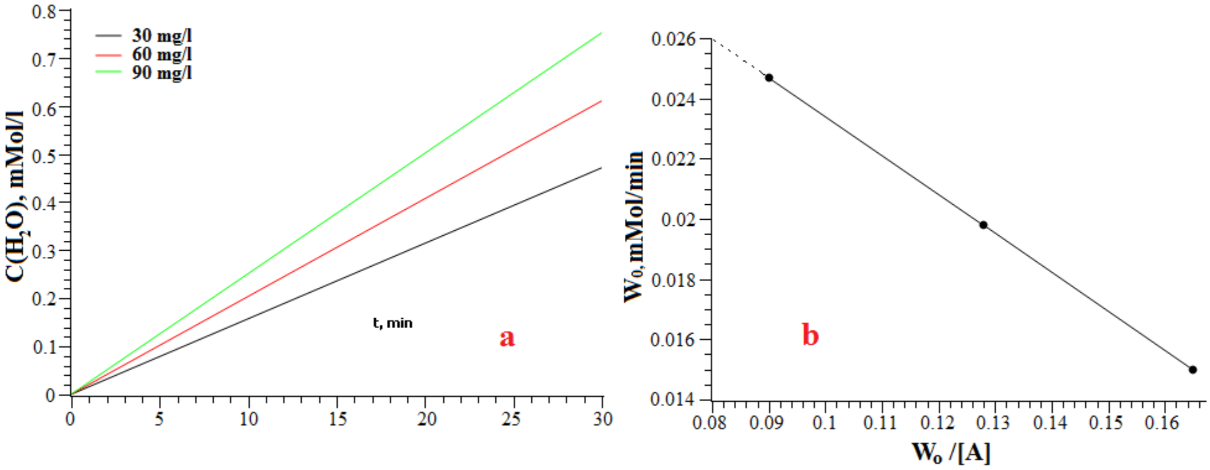

Figure 4 – Kinetic curves of methyl orange oxidation process

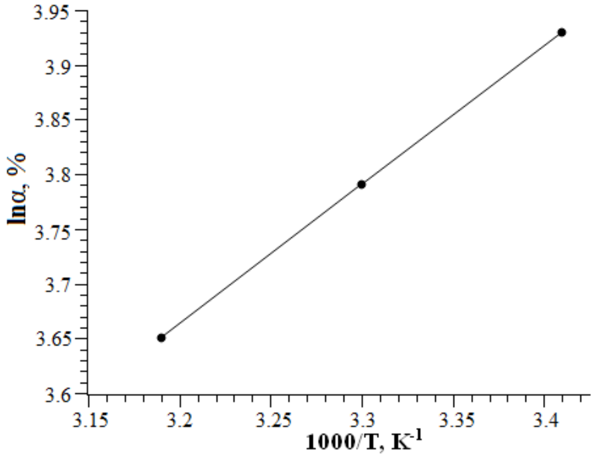

Figure 5 – The action of temperature on the oxidation state of methyl orange

The influence of temperature on the process under study was investigated. Plotting lnα/1000*T coordinates in accordance with figure 5 gives the slope tangent of 1.27. With these data, we can calculate the value of the effective activation energy: $E_{eff} = R*10^3a$, which will be 10.5 kJ/mol.

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ТИТАН ОКСИДІ-ОКСИФОРИДІНІҢ СИНТЕЗІ ЖӘНЕ ОНЫҢ ФОТОКАТАЛИТИКАЛЫҚ ҚАСИЕТТЕРІН ЗЕРТТЕУ

Аннотация. Мақалада титан оксиді – титан оксифторидінің қоспасы негізінде фотокатализаторды синтездеудің жаңа әдісі карастырылған. Синтез металл титанды аммоний бифторидінің қышқылданған ерітіндісінде еріту арқылы, одан кейін метатитан қышқылын алу үшін жоғары тотығу күйіне дейін және аммиак гидролизімен тотығу арқылы жүзеге асырылды. Метатитан қышқылының ыдырауы муфельді пеште 380°C температурада жүргізілді. Метатитан қышқылын тұндыру кезінде титан қосылысы фтор ионымен бірге тұнбаға өтетіндігі анықталды, ол кальциленгеннен кейін титан оксифторидіне айналады. Электронды микроскопиялық зерттеулер нәтиежесінде, алынған ұнтақтың агрегаттары ұсақ бөлшектерден тұрады, олардың орташа мөлшері Шеррер формуласын қолданып рентгендік дифракция үлгілері бойынша есептелген, 30-35 нм құрайды.

Фотокаталитикалық белсенділік ультракүлгін сәулесімен сәулелену кезінде метил қызғылт сары бояғышының тотығу реакциясы арқылы анықталды. Ультракүлгін сәулелену көзі ДРТ-125 шамы болды, оның толқын ұзындығы 200-400 нм аралығында болды. Концентрациясы 1-7 г/л аралығында 60 мг/л бояғыш пен фотокатализатордың әр түрлі мазмұны бар суспензиялар тотығуға ұшырады. Концентрациясы 466,4 нм толқын ұзындығында анықталды, ол 200-800 нм толқын ұзындығындағы ерітіндіні сканерлеу арқылы анықталды. Тотығу нәтижесінде ерітіндінің pH-ы қышқыл жағына қарай 3,5-ке ауысады, бұл максималды сіңірудің толқын ұзындығының ығысуына әкелетіндігі анықталды, осыған байланысты ерітінділердің pH мәні бірдей мәндерге келтірілді.

Түйін сөздер: Титан оксиді, титан оксифториді, фотокатализатор, ультрафиолет сәулеленуі, тотығу.

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СИНТЕЗ ОКСИДА-ОКСИФТОРИДА ТИТАНА И ИССЛЕДОВАНИЕ ЕГО ФОТОКАТАЛИТИЧЕСКИХ СВОЙСТВ

Аннотация. В статье рассматривается новый метод синтеза фотокатализатора на основе смеси оксид титана – оксифторид титана. Синтез осуществлялся путем растворения металлического титана в подкисленном растворе бифторида аммония с последующим окислением до высшей степени окисления и гидролизом аммиаком с получением метатитановой кислоты. Разложение метатитановой кислоты производилось в муфельной печи при температуре 380°C. Установлено, что при осаждении метатитановой кислоты в осадок переходит также соединение титана со фторид-ионом, которое после прокаливания переходит в оксифторид титана. Электронно-микроскопические исследования показали, что агрегаты полученного порошка состоят из более мелких частиц, средний размер которых рассчитан из рентгенограмм по формуле Шеррера 30-35 нм.

Фотокаталитическую активность определяли по реакции окисления красителя метилового оранжевого при облучении ультрафиолетовым излучением. Источником ультрафиолетового излучения служила лампа ДРТ -125 с длиной волны в диапазоне 200-400 нм. Окислению подвергались суспензии, содержащие 60 мг/л красителя и различные содержания фотокатализатора в диапазоне концентраций 1-7 г/л. Определение концентраций производилось при длине волны 466,4 нм, которая определялась путем сканирования раствора в диапазоне длин волн 200-800 нм. Установлено, что в результате окисления pH раствора смещается в кислую сторону до 3,5, что приводит к смещению длины волны максимального поглощения, в связи с этим производилась корректировка pH растворов до одинаковых значений.

Ключевые слова: оксид титана, оксифторид титана, фотокатализатор, ультрафиолетовое излучение, окисление.

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