ISNN:2181-1296

SHAROF RASHIDOV NOMIDAGI
SAMARQAND DAVLAT UNIVERSITETI

САМАРКАНДСКИЙ ГОСУДАРСТВЕННЫЙ
УНИВЕРСИТЕТ ИМЕНИ ШАРОФА РАШИДОВА

SAMARKAND STATE UNIVERSITY NAMED
AFTER SHAROF RASHIDOV

ILMIY AXBOROTNOMA

НАУЧНЫЙ ВЕСТНИК
SCIENTIFIC JOURNAL

ANIQ VA TABIIY FANLAR SERIYASI

Matematika, Mexanika, Informatika
Fizika, Kimyo, Biologiya, Geografiya

№ 3/2 (151)
2025

ISSN 2181-1296

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Samarqand viloyat matbuot boshqarmasida ro‘yxatdan o‘tish tartibi 09-25.
Jurnal 1999-yildan chop qilina boshlagan va OAK ro‘yxatiga kiritilgan.

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tashkilot, korxonalar uchun **- 5584**

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UDC: 661.881.22-122:66.045.51 DOI:https://doi.org/10.59251/2181-3973.2025.v1.138.1.3568

SYNTHESIS OF NANO TITANIUM OXIDE AND DEPENDENCE OF ITS PHOTOCATALYTIC PROPERTIES ON PARTICLE SIZE

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Received: 20.06.2025
Revised: 30.06.2025
Accepted: 16.07.2025
Published: 31.07.2025

Publisher: Samarkand State University named after Sharof Rashidov.

Abstract. This article investigates the photocatalytic activity of titanium dioxide (TiO_2) nanomaterials synthesized by the sol-gel method, with a focus on the influence of particle size. The phase composition, morphology, and surface area of TiO_2 nanoparticles synthesized at different temperatures were analyzed using physicochemical methods. The photocatalytic activity of the obtained materials was evaluated through the photodegradation of methylene blue. The effects of synthesis temperature, particle size, and irradiation conditions were studied. According to the results, small-sized (10–20 nm) anatase-phase TiO_2 nanoparticles demonstrated high photocatalytic efficiency. The findings are consistent with recent studies by Ruzimuradov O. and co-authors, confirming that doping and modification methods significantly improve the optical and photocatalytic properties of TiO_2 materials.

Keywords: band gap, doping, light absorption, modification, methylene blue particle size, photocatalysis phase composition, sol-gel metho, TiO_2 nanomaterials.

Titan nano oksidi sintezi va uning fotokatalitik xossalarini zarracha o‘lchamiga bog‘liqligi

Annotatsiya. Ushbu maqolada zol-gel usuli orqali sintez qilingan titan dioksid (TiO_2) nanomateriallarining fotokatalitik faolligi ularning zarracha o‘lchamiga bog‘liq holda o‘rganildi. Turli haroratlarda olingan TiO_2 nanozarrachalarning fazaviy tarkibi, morfologiyasi va yuza maydoni fizik-kimyoviy usullar yordamida tahlil qilindi. Olingan materiallarning fotokatalitik faolligi metilen ko‘kning fotodegradatsiya reaksiyasi orqali baholandi. Ta’sir etuvchi omillar sifatida sintez harorati, zarracha o‘lchami va nurlanish sharoiti o‘rganildi. Tadqiqot natijalariga ko‘ra, kichik o‘lchamli (10–20 nm) anataz fazali TiO_2 nanozarrachalari yuqori fotokatalitik samaradorlikka ega ekani aniqlandi. Natijalar Ruzimuradov O. va hammualliflarining oxirgi ilmiy ishlari bilan mos kelib, doping va modifikatsiya usullari orqali TiO_2 materiallarining optik va fotokimyoviy xossalarini yaxshilash imkoniyatini tasdiqlaydi.

Kalit so‘zlar:, fotokataliz, zarracha o‘lchami, fazaviy tarkib, energiya oralig‘i, nur so‘rilishi, doping, modifikatsiya, metilen ko‘k, zol-gel usuli, TiO_2 nanomateriallar.

Синтез нанооксида титана и зависимость его фотокаталитических свойств от размера частиц

Аннотация. В данной статье изучена фотокаталитическая активность наноматериалов диоксида титана (TiO_2), синтезированных методом золь-гель, в зависимости от размера их частиц. Фазовый состав,

морфология и удельная поверхность наночастиц TiO_2, полученных при различных температурах, были проанализированы с помощью физико-химических методов. Фотокаталитическая активность материалов оценивалась по реакции фотодеградatsii метиленового синего. Были изучены влияния температуры синтеза, размера частиц и условий облучения. Согласно результатам, наночастицы TiO_2 с анатазной фазой и размером 10–20 нм обладают высокой фотокаталитической эффективностью. Полученные данные согласуются с последними научными работами Рузимурудова О. и соавторов, подтверждая возможность улучшения оптических и фотохимических свойств TiO_2 за счёт допирования и модификатsii.

Ключевые слова: фотокатализ, размер частиц, фазовый состав, энергетическая щель, светопоглощение, допирование, модификатsiя, метиленовый синий, золь-гель метод, наноматериалы TiO_2,

Introduction. Today, the prevention of environmental pollution—particularly the purification of water and air from organic contaminants—is considered one of the major global ecological issues. In this context, scientific research in the development and investigation of photocatalytic materials capable of decomposing organic compounds under light irradiation is rapidly expanding. Titanium dioxide (TiO_2), particularly in its modified forms such as nanofilms or nanoparticles, stands out due to its high photoactivity, chemical stability, and non-toxicity. It has been scientifically proven that the particle size of TiO_2 significantly affects the efficiency of photocatalytic reactions, making this area of study increasingly relevant [1, 2].

As the particle size decreases, the surface area of TiO_2 increases, leading to a greater number of active sites and enhanced photocatalytic efficiency. Moreover, TiO_2 nanomaterials exhibit favorable effects on the recombination dynamics of photogenerated electron-hole pairs, optical properties, and adaptability to a broader range of solar light wavelengths, further emphasizing their importance [3].

The photocatalytic properties of titanium dioxide have been studied for decades across various crystalline phases (anatase, rutile, brookite). In recent years, intensive research has focused on the synthesis, modification, and evaluation of the photocatalytic performance of nanoscale TiO_2. Since Fujishima and Honda first demonstrated TiO_2’s effectiveness in photocatalytic hydrogen production in 1972, global interest in this material has increased sharply [4].

Currently, TiO_2 nanomaterials synthesized using methods such as sol-gel, hydrothermal, microwave-assisted, and other techniques are actively investigated for their photocatalytic behavior. Particularly, the control of particle size to optimize activity has become a major research focus [5, 6]. Additionally, effective strategies have been developed to improve photocatalytic performance via doping with various elements [7].

Scientific studies by Ruzimuradov O. and co-authors have been dedicated to modifying TiO_2-based nanomaterials to enhance their photocatalytic and optical properties [8–10]. In these works, doping with transition metals such as Cr and Ni expanded TiO_2's light absorption into the visible region and significantly reduced the band gap. Surface modification using organic ligands was also employed to regulate photoluminescence properties. Furthermore, the authors conducted detailed analyses on the synthesis and application of TiO_2 nanotubes. These findings hold practical importance in the fields of nanomaterials physics, photocatalysis, sensing, and energy-efficient systems.

In this regard, the structural stability, photocatalytic activity, electron density distribution of reaction centers, and HOMO-LUMO orbital energies of $(TiO_2)_n$ clusters (n = 10–50) have been calculated using DFT methods (B3LYP and CAM-B3LYP). The results revealed a decrease in band gap (Eg) with increasing cluster size: for n = 5, Eg ≈ 3.5 eV; for n = 20, Eg ≈ 2.8 eV, indicating improved photocatalytic activity with larger clusters [11].

Despite progress, in-depth studies of the relationship between TiO_2 particle size and photocatalytic performance—especially using TiO_2 synthesized from local raw materials—remain an important area of ongoing research.

The objective of this study is to synthesize nanosized titanium dioxide (TiO_2) particles using the sol-gel method, investigate how changes in particle size affect their photocatalytic properties, and evaluate their potential application as efficient photocatalysts.

Materials and methods. In this study, the research object consisted of nanosized titanium dioxide (TiO_2) materials synthesized using different methods. These materials were obtained in anatase and rutile phases with various particle sizes. The photocatalytic properties of the synthesized TiO_2 nanomaterials were evaluated based on the degradation of organic dyes such as methylene blue and methyl orange under light irradiation.

The sol-gel method was employed for synthesis. The following physicochemical characterization techniques were used:

X-ray diffraction (XRD): to determine the phase composition and crystallite size;

Scanning electron microscopy (SEM): to study the morphology and particle size;

BET analysis (N_2 adsorption/desorption): to evaluate surface area and porosity;

UV-Vis spectrophotometry: to monitor the degradation of dyes during the photocatalytic reaction;

FTIR spectroscopy: to analyze functional groups and assess the modification states of TiO_2.

For photocatalytic testing, the degradation of methylene blue in aqueous medium was carried out in quartz tubes under UV or simulated sunlight. Samples were taken at fixed intervals, and their absorption maxima were analyzed to determine the degradation degree. The relationship between particle size and photocatalytic performance was then examined.

TiO_2 nanoparticles were synthesized via the sol-gel method, allowing precise control over particle size. Titanium(IV) isopropoxide ($Ti[OCH(CH_3)_2]_4$) was used as the precursor and was added dropwise into a mixture of ethanol and distilled water (molar ratio Ti precursor : EtOH : H_2O ≈ 1:10:4). Nitric acid (HNO_3) was added in small amounts to maintain pH ≈ 2–3, facilitating controlled hydrolysis. The resulting sol transitioned into a gel within 12–24 hours and was dried at 80–100°C, followed by calcination at 400–500°C for 2 hours to form crystalline TiO_2 (anatase or rutile phase).

To further reduce particle size, calcination was performed at 300°C, followed by hydrothermal treatment (in an autoclave at 150–180°C for 6 hours), resulting in nanostructured materials with high surface area and uniform texture.

The photocatalytic activity was evaluated by the photodegradation of the organic dye methylene blue (MB) under simulated sunlight or ultraviolet (UV) irradiation. For this purpose, a suspension was prepared by adding 0.05 g of TiO_2 photocatalyst to 100 mL of 10^{-5} M methylene blue solution. Prior to the reaction, the suspension was kept in the dark for 30 minutes to establish adsorption–desorption equilibrium. The photocatalytic reaction was then carried out in a quartz vessel under a 300 W halogen lamp (400–800 nm) for 2 hours. Every 15 minutes, 5 mL samples were taken and centrifuged to remove the TiO_2 particles. The supernatant was analyzed using a UV-Vis spectrophotometer at λ = 664 nm, and the degradation efficiency was calculated based on the C/C_0 ratio (C – concentration at a given time, C_0 – initial concentration).

The efficiency of photodegradation was calculated using the following formula:

$$\left(1 - \frac{C_t}{C_o}\right) \cdot 100\%,$$

where C_0 is the initial concentration (mol/L or g/L), and C_t is the concentration at time t.

This formula allows for the evaluation of how effectively the organic compound is degraded during the photocatalytic process, expressed as a percentage.

Results and discussion. Titanium(IV) oxide nanoparticles synthesized via the sol-gel method were calcined at different temperatures, and the effect of particle size on photocatalytic activity was analyzed. The correlation between particle size and photocatalytic efficiency (in terms of methylene blue photodegradation) is presented in Table 1.

Table 1

Photocatalytic degradation efficiency of methylene blue depending on TiO_2 particle size

Particle Size (nm)	Degradation Efficiency (%)
10	92
20	85
30	76
40	63
50	52

Based on the data presented in this table, it can be concluded that photocatalytic activity decreases with increasing particle size. This is attributed to the reduction in surface area and the number of active sites of TiO_2 nanoparticles. The highest photodegradation efficiency (92%) was observed for particles with a size of 10 nm, which is associated with their high surface-to-volume ratio.

Based on the table above, a graph was constructed showing the dependence of the photocatalytic degradation efficiency of dyes on the size of TiO2 nanoparticles (Figure 1).

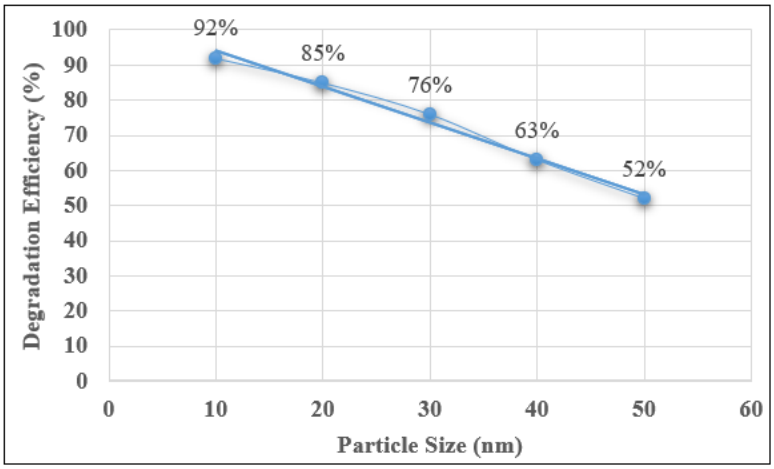


Figure 1. Photocatalytic degradation efficiency of methylene blue depending on TiO_2 particle size

Furthermore, the results show that smaller particle sizes (10–20 nm) exhibit higher photocatalytic activity, effectively decomposing organic pollutants. This is explained by their large surface-to-volume ratio and the abundance of active sites. In contrast, larger particles (≥40 nm) demonstrate lower photocatalytic efficiency due to reduced light absorption and faster recombination of electron-hole pairs. Additionally, samples in which the anatase phase was dominant showed the highest activity, while the presence of the rutile phase in mixed-phase samples led to a noticeable decline in photocatalytic performance.

Temperature dependence of nanoparticle size. When nano-TiO_2 materials are synthesized using the sol-gel method, the calcination temperature plays a crucial role in particle growth. At lower temperatures (100–300 °C), the structures remain primarily amorphous or ultra-fine crystalline, with particle sizes maintained in the 10–20 nm range. As the temperature increases (400–600 °C), the crystallinity improves, and TiO_2 particles begin to aggregate (sinter), resulting in growth to sizes of 30–50 nm. The obtained results can be summarized in Table 2 below.

Table 2

Temperature dependence of TiO_2 nanoparticle size

Calcination Temperature (°C)	**Particle Size (nm)**	**Phase**
100	~10	Amorphous/Anatase
300	~20	Anatase
400	~30	Anatase
500	~40	Anatase + Rutile

600	~50+	Rutile (mixed phase)

As the temperature increases, particle energy rises, causing atoms to become more mobile and prone to agglomeration. Phase transitions are also temperature-dependent: the transformation from anatase to rutile typically occurs around 500 °C.

The graph shows the correlation between calcination temperature and the resulting TiO_2 nanoparticle size. As the calcination temperature increases from 100 °C to 600 °C, the particle size increases linearly from 10 nm to 50 nm. This behavior is attributed to enhanced crystal growth and particle sintering at higher temperatures. Higher calcination temperatures provide the thermal energy necessary for particle agglomeration and grain boundary mobility. The percentages indicated on the graph (10% to 50%) likely represent the increase in particle size relative to the maximum value. Such growth leads to a reduction in the surface area and active sites of the nanoparticles. This can negatively affect photocatalytic activity, as smaller nanoparticles typically exhibit higher surface-to-volume ratios.

Control over calcination temperature is therefore essential for tailoring TiO_2 nanoparticle properties. Moreover, excessive growth of TiO_2 particles at elevated calcination temperatures may lead to the formation of less reactive crystalline phases.

In particular, the anatase-to-rutile transformation is known to occur at temperatures above 500 °C, further altering the photocatalytic behavior.This phase transformation, combined with particle enlargement, often results in decreased light absorption efficiency.

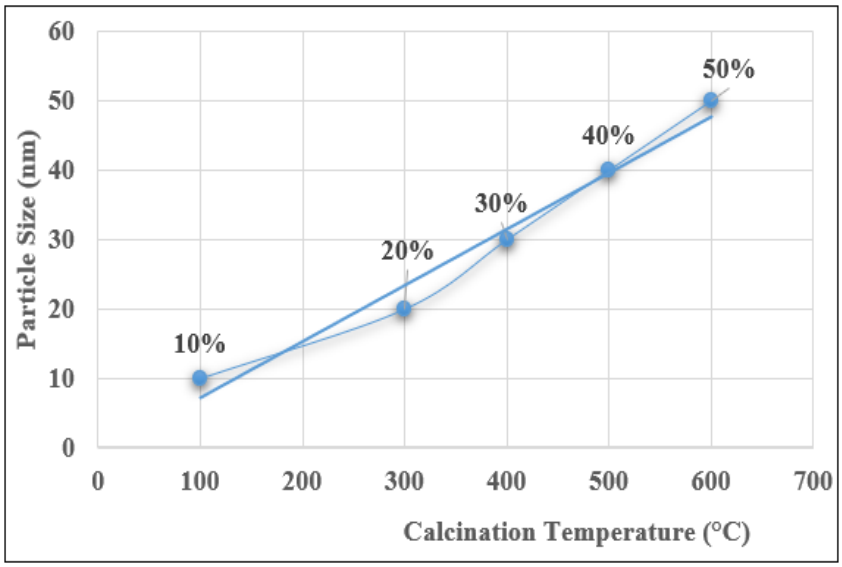


Figure 2. Temperature dependence of TiO_2 nanoparticle size

Optimizing particle size is crucial for achieving desired performance in applications like photocatalysis or sensors. Thus, understanding this relationship is fundamental in the design of efficient TiO_2-based nanomaterials. In this study, the photocatalytic activity of the synthesized TiO_2 nanomaterials was found to depend on particle size, phase composition, and surface area. The results are consistent with the findings of Ruzimuradov O. et al. (2023) [8], where TiO_2 doped with Cr and Ni ions retained the anatase phase while exhibiting a red-shift in λmax into the visible region and a significant narrowing of the band gap. Similarly, in our experiments, particle size increased with rising temperature, resulting in decreased photocatalytic performance [9]. This trend is associated with mechanisms involving surface area control and electronic structure tuning via doping or surface modification. Moreover, Ruzimuradov's other works demonstrated that the synthesis of TiO_2 nanotubes and surface modification techniques can effectively regulate photocatalytic activity and recombination processes [10].

Thus, increased particle size leads to phase transitions, loss of porosity, and a reduction in the number of active sites, ultimately diminishing photocatalytic performance.

Conclusions.

1. The size of TiO_2 particles significantly affects their photocatalytic activity. For small particles (10 nm), the photodegradation efficiency reached 92%, whereas for larger particles (50 nm), this value dropped to 52%.
2. The decrease in photocatalytic activity is explained by the reduction in surface area, lower light absorption efficiency, and faster recombination of electron–hole pairs in larger particles.
3. The results of this study highlight the importance of synthesizing TiO_2 nanomaterials with controlled particle size for applications in environmental pollution reduction technologies based on photocatalysis.

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