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OBTAINING BENZENE BY PHOTOCATALYTIC OXIDATION OF TOLUENE

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Abstract. Work revealed the mechanistic features of the process by studying the intermediate products formed during the photocatalytic oxidation of toluene, the reaction mechanism, and the pathways for the formation of benzene. The photocatalytic oxidation of toluene was carried out, and benzene, benzaldehyde, benzoic acid, and benzyl alcohol were obtained as reaction products. In addition, the role of intermediate compounds in the formation of benzene was evaluated, and based on the experimental data obtained, the mechanism of the oxidation of toluene to benzene was proposed. The toxic and carcinogenic properties of benzene formed during photocatalytic processes and its environmental hazard to the environment were considered.

Main factors affecting the photocatalysis process were considered during the study - light source, catalyst type, reaction time and reaction medium. As a result of the



experiments, the formation of benzene was recorded using the relative concentration method and its intermediate stages were clarified.

Results obtained allow a deeper understanding of the laws of benzene formation during the photocatalytic oxidation of toluene and can serve as a basis for the development of effective and environmentally safe technologies for the purification of volatile organic compounds in the air. The study provides additional insight into the role of benzaldehyde as a key intermediate in benzene formation and contributes to a better understanding of the formation of hazardous by-products during photocatalytic treatment of volatile organic compounds.

Keywords: photocatalysis, toluene, photocatalysts, benzene, benzaldehyde, carcinogens

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ТОЛУОЛДЫҢ ФОТОКАТАЛИТИКАЛЫҚ ТОТЫҒУЫ АРҚЫЛЫ БЕНЗОЛ АЛУ

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Аннотация. Бұл мақалада толуолдың фотокаталитикалық тотығуы кезінде түзілетін аралық өнімдерді алу процесі, реакция механизмі және бензолдың түзілу жолдары қарастырылады. Толуолдың фотокаталитикалық тотығуы жүргізілді,

нәтижесінде бензол, бензальдегид, бензой қышқылы және бензил спирті реакция өнімдері ретінде алынды. Сонымен қатар, бензолдың түзілуіндегі аралық өнімдердің рөлі бағаланды және алынған тәжірибелік деректерге негізделген толуолдың бензолға тотығу механизмі ұсынылды.

Фотокаталитикалық процестер кезінде түзілетін бензолдың уытты және канцерогендік қасиеттері, сондай-ақ оның қоршаған ортаға және адам денсаулығына тигізетін қауіптері талқыланады, өйткені бензол ұзақ уақыт әсер еткен жағдайда ауыр аурулардың, соның ішінде қатерлі ісік түрлерінің даму қаупін арттыратын қауіпті ластаушы заттардың бірі болып табылады.

Зерттеуде фотокаталитикалық процеске әсер ететін негізгі факторлар қарастырылды: жарық көзі, катализатор түрі мен қасиеттері, реакция ұзақтығы, реакция ортасының құрамы және бастапқы материал концентрациясы. Олардың толуолдың фотокаталитикалық тотығу жылдамдығына, оның түрлену дәрежесіне және аралық және соңғы өнімдердің түзілу селективтілігіне әсері талданды. Салыстырмалы концентрация әдісін қолданатын тәжірибелер бензолдың түзілуін көрсетті, және оның түзілуінің бірнеше аралық сатылары анықталып, нақтыланды. Алынған деректер бізге процесс жағдайлары мен бензол шығымы арасындағы байланысты орнатуға мүмкіндік берді, бұл фотокаталитикалық технологиялардың қауіпсіздігін бағалау үшін маңызды.

Бұл нәтижелер фотокаталитикалық толуол тотығуы кезінде бензолдың түзілуінің негізгі механизмдерін тереңірек түсінуге ықпал етеді, реакция механизмінің ерекшеліктерін және соңғы өнімдердің түзілуіндегі аралық өнімдердің рөлін ашады. Бұл зерттеу нәтижелерін фотокаталитикалық тазарту жағдайларын оңтайландыру, улы қосалқы өнімдердің түзілуін азайту және ауа мен су ортасынан ұшпа органикалық қосылыстарды кетірудің тиімдірек, экологиялық таза технологияларын әзірлеу үшін пайдалануға болады.

Түйін сөздер: фотокатализ, толуол, фотокаталитикалық тотығу, бензол, бензальдегид, канцерогенді қосылыстар

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ПОЛУЧЕНИЕ БЕНЗОЛА МЕТОДОМ ФОТОКАТАЛИТИЧЕСКОГО ОКИСЛЕНИЯ ТОЛУОЛА

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Аннотация. В работе исследованы промежуточные продукты, образующиеся при фотокаталитическом окислении толуола, а также рассмотрены механизм протекания реакции и пути образования бензола. В результате фотокаталитического окисления толуола в качестве продуктов реакции были идентифицированы бензол, бензальдегид, бензойная кислота и бензиловый спирт. На основе экспериментальных данных оценена роль промежуточных соединений в формировании бензола и предложена схема механизма его образования. Особое внимание уделено токсическим и канцерогенным свойствам бензола, образующегося в ходе фотокаталитических процессов, а также потенциальным экологическим рискам, связанным с его присутствием. В ходе исследования проанализированы основные факторы, влияющие на эффективность фотокаталитического процесса, включая тип и свойства катализатора, характеристики источника света, продолжительность реакции, состав реакционной среды и концентрацию исходного вещества. Проведена оценка их влияния на скорость фотокаталитического окисления толуола, степень его превращения и селективность образования промежуточных и конечных продуктов. В результате экспериментов методом относительной концентрации было подтверждено образование бензола и выявлены отдельные промежуточные стадии его формирования. Полученные данные позволили установить взаимосвязь между условиями проведения процесса и выходом бензола, что имеет важное значение для оценки безопасности фотокаталитических технологий. Результаты исследования способствуют более глубокому пониманию закономерностей образования бензола при фотокаталитическом окислении толуола, раскрывают особенности механизма реакции и роль промежуточных соединений в формировании конечных продуктов. Полученные результаты могут быть использованы для оптимизации процессов фотокаталитической очистки, снижения образования токсичных побочных продуктов и разработки более эффективных и экологически безопасных технологий удаления летучих органических соединений из воздушной и водной среды.

Ключевые слова: фотокатализ, толуол, фотокаталитическое окисление, бензол, бензальдегид, канцерогенные соединения

Introduction. Volatile organic compounds (VOCs) are organic chemical compounds found in a variety of products that readily evaporate and are distributed to the environment under normal conditions. VOCs are highly volatile, mobile, and resistant to degradation, and can be transported long distances in the environment (Faroon et al.,

2005). The most common VOCs are aromatic hydrocarbons (e.g., benzene, toluene, xylene, and ethylbenzene) and halogenated hydrocarbons (e.g., chloroethylene and trichloroethylene).

A special type of VOC is carcinogenic volatile organic compounds, which can cause cancer in humans. The main routes of VOC entry into the body from contaminated water are through drinking, bathing, eating, swimming and washing clothes (Chowdhury et al., 2009). The sources of VOC can be natural and anthropogenic. Natural sources include emissions from factories, natural forest fires and anaerobic processes in wetlands. Anthropogenic sources include domestic and industrial processes: food production, use of fertilizers and pesticides, septic systems, chlorination, transportation, combustion of hydrocarbon fuels, oil storage and distribution, textile cleaning, printing, pharmaceutical industry, etc. (Pandey et al., 2018).

The growth of agricultural activities worldwide (fuel use, burning of agricultural waste, and use of VOCs as inert components of pesticides) has led to the widespread release of VOCs into the environment. In addition, many studies have shown that VOCs are indoor air pollutants, with the main sources being tobacco smoke, chlorinated water, perfumes, paint strippers, adhesives, new clothing, and heaters that use plastic or kerosene. Some researchers have detected VOCs in bottled water, while others have found microbial volatile organic compounds in the air, which may originate from microbial metabolites or airborne mold spores (Fischer et al., 2003, Murrells et al., 2007, Guerra et al., 2018).

VOC has direct and indirect effects on humans and the environment, and the main problems they pose include: harm to human health and the environment through toxicity; carcinogenicity and other adverse effects; degradation of materials; formation of tropospheric photochemical oxidants; depletion of stratospheric ozone; global climate change; and the emission of unpleasant odors. In addition, VOC and nitrogen oxides react in the presence of sunlight to form photochemical oxidants (ozone, peroxyacyl nitrates, peroxides, etc.) (Williams et al., 2007). These compounds are harmful to human health and have a negative impact on the environment, as they oxidize NO gas, converting it into the even more dangerous NO₂.

Although the photocatalytic oxidation of toluene over TiO₂-based photocatalysts has been extensively investigated, the formation pathways of benzene as a toxic and carcinogenic intermediate remain insufficiently clarified. Most previous studies have focused primarily on the degradation efficiency of toluene and mineralization processes, while limited attention has been paid to the quantitative relationship between benzaldehyde, benzyl alcohol, benzoic acid, and benzene during photocatalytic transformation.

The novelty of the present study lies not only in the identification of intermediate products formed during photocatalytic oxidation of toluene, but also in the comparative assessment of their relative abundance under identical experimental conditions and in evaluating the contribution of benzaldehyde to benzene formation. Unlike previous studies primarily focused on toluene degradation efficiency and mineralization, this work provides experimental evidence regarding the relative distribution of benzaldehyde,

benzyl alcohol, benzoic acid and benzene, allowing a more detailed interpretation of the pathways leading to the formation of hazardous by-products during photocatalytic treatment.

Materials and methods. Photocatalytic oxidation of gaseous organic compounds involves several processes, the most important of which are: the generation and recombination of electrons and holes, adsorption of pollutants, chemical reactions, and desorption of products (Ajmal et al., 2022). The initial stage of photocatalytic oxidation is the formation of electron-hole pairs under the influence of light. They can migrate to the surface of the catalyst and be trapped in various regions. Photogenerated electrons can reduce O_2 molecules or organic molecules adsorbed on the catalyst surface. On the other hand, photogenerated holes can oxidize water molecules, forming hydroxyl radicals ($OH\cdot$) and hydrogen peroxide, which can oxidize organic pollutants.

Electron-hole pairs can recombine instead of participating in surface reactions, which reduces the efficiency of the photocatalytic reaction. To prevent recombination, it is important to improve the separation of electrons and holes. This can be achieved by alloying, deposition of co-catalysts, or the use of heterostructures. The use of nanomaterials improves the access of electrons to the catalyst surface and reduces the interaction distance with electron acceptors (Yamada, et al., 2012, Yli-Juuti, et al., 2020).

The mechanism of photocatalytic oxidation of gaseous organic substances is shown in Figure 2 below, and the reaction stages are described as follows:

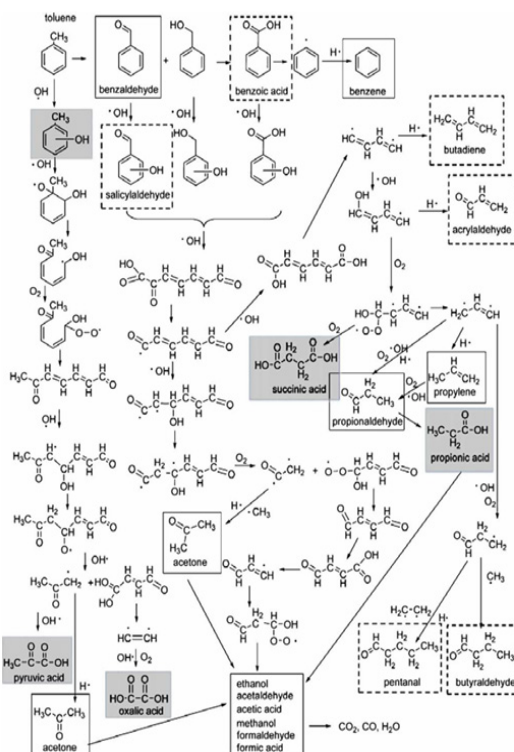
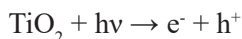


Figure 1. Mechanism of photo catalytic oxidation of toluene.

The figure shows the multi-step mechanism of the photocatalytic oxidation of toluene molecule (Mo, et al., 2009). This process usually occurs on a semiconductor photocatalyst (e.g., titanium(IV) oxide) under the influence of light. The basic idea is that when light is applied, active species ($\bullet\text{OH}$, $\text{O}_2\bullet^-$, h^+) are formed, which gradually oxidize toluene and finally mineralize it to CO_2 and H_2O .

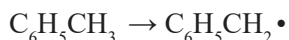
Excitation of the photocatalyst under the influence of light:



where:

- h^+ is a "hole" in the valence band;
- e^- is an electron in the conduction band.

When water reacts with oxygen, hydroxyl radicals ($\bullet\text{OH}$) and superoxide radicals ($\text{O}_2\bullet^-$) are formed. The methyl group in toluene, $\text{C}_6\text{H}_5\text{CH}_3$, is the most reactive. The $\bullet\text{OH}$ radical abstracts a hydrogen, forming a benzyl radical:



Further, oxygen is added to form benzyl alcohol, benzaldehyde, and benzoic acid. Finally, all organic intermediates are completely oxidized to form $\text{CO}_2 + \text{H}_2\text{O}$.

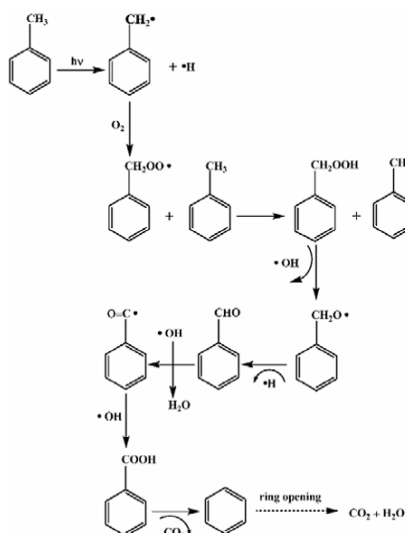


Figure 2. The mechanism of oxidation of toluene to benzene.

Toluene, under the influence of light, first forms a benzyl radical, which reacts with oxygen and turns into peroxide intermediates; then gradually benzyl alcohol, benzaldehyde and benzoic acid are formed, and decarboxylation of benzoic acid results in benzene (Sun et al., 2010, Malayeri et al., 2022, Frank et al., 2020).

Methods of preparing the photocatalyst. Titanium dioxide (TiO_2 (anatase) HTA301 –

pigment), which plays a key role in the photocatalysis process, is applied to the surface of the catalyst in the form of a suspension using a solution of methanol (CH_3OH). For this purpose, pre-cleaned chromatographic glass vials with a volume of 20 ml are used. TiO_2 is placed in each chromatographic glass vial at the rate of 0.2 g.

0.5 ml of methanol is added to it and a homogeneous suspension is obtained. The suspension is applied evenly to the inner wall of the chromatographic glass vial. The Applied area is on average 12 cm^2 . This suspension is blown and dried by compressed air. Due to the high volatility of methanol, the catalyst surface quickly and evenly becomes a solid layer. The inside of the chromatographic glass vial is blown under compressed air for 30 seconds so that it is completely filled with compressed air. Immediately after blowing, the mouth of the chromatographic glass bottle is hermetically sealed with a special SEPTA sealing device consisting of aluminum and silicone.

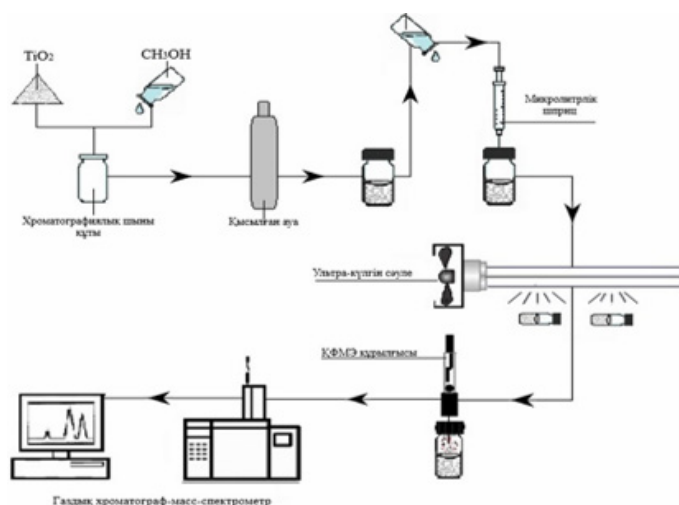


Figure 3. Diagram of photocatalytic oxidation of toluene.

A low-pressure fluorescent lamp of the Philips PL-L 36W/10/4P SECURA model was used as a source of ultraviolet radiation to carry out the photocatalytic oxidation process. This lamp steadily emitted light at a wavelength of 365 Nm, providing the optimal spectral range for activation by the titanium dioxide (TiO_2) photocatalyst. During the experiment, chromatographic glass vials prepared according to each organic compound were alternately placed in the radiation source.

The vials were placed in the very center of the two fluorescent tubes of the Philips lamp and exposed to ultraviolet radiation for 30 minutes at a distance of 1.5 cm from the radiation source. These conditions made it possible for the photocatalysis process to proceed effectively and for the active formation of electron-pore pairs (e^-/h^+) that occur at surface intervals. During irradiation, active oxygen species such as hydrogen peroxide (H_2O_2), hydroxyl radicals ($\bullet\text{OH}$) and superoxide-anion radicals ($\text{O}_2^-\bullet$) were formed on the surface of the TiO_2 photocatalyst, which caused the oxidation of the introduced organic compounds.

Results and discussion. To determine the formed volatile intermediates, the method of gas chromatography-mass spectrometry (GC–MS) was used. The exact composition of the extracted products was determined by the method. As a result, all compounds were effectively detected and fully registered with a gas chromatograph. The chromatogram of intermediates during photocatalytic oxidation of toluene is shown in Figure 4.

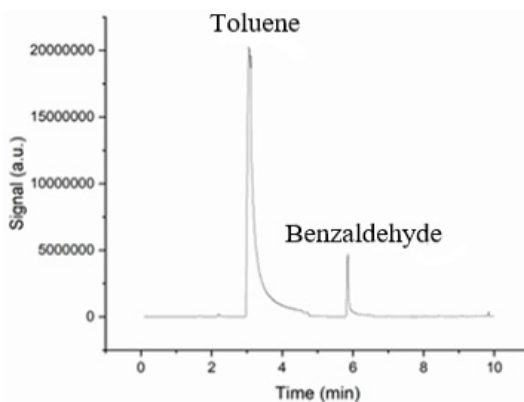


Figure 4. Chromatogram of intermediates during photocatalytic oxidation of toluene.

On the chromatogram, a very high intensity peak is observed in the area of about 3 minutes, which corresponds to toluene. This indicates that the amount of toluene predominates in the reaction mixture. In addition, a second peak characteristic of benzaldehyde was recorded in the period of about 5.8–6.0 minutes. The formation of benzaldehyde proves that intermediates are formed during photocatalytic oxidation of toluene.

The results obtained indicate that the oxidation process of toluene occurs in stages and benzaldehyde is an important intermediate compound in the reaction mechanism.

In chromatograms obtained after photocatalytic oxidation of benzaldehyde, the formation of benzene from benzaldehyde was clearly observed. The chromatogram of intermediates during photocatalytic oxidation of benzaldehyde is presented in Figure 5.

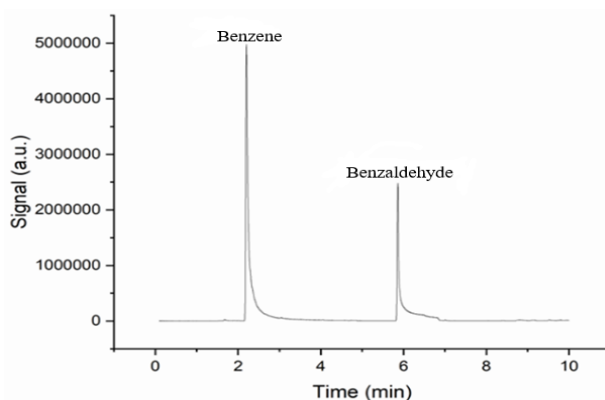


Figure 5. Chromatogram of intermediates during photocatalytic oxidation of benzaldehyde.

As you can see from the image, the result proves that benzaldehyde is further converted to benzene by oxidation or decarbonylation, that is, the role of benzaldehyde as one of the ways of benzene formation has been established.

Table 1. The ratio of the common values of the intermediates formed by toluene.

Benzaldehyde / Toluene	0,069:1
Benzyl alcohol / Toluene	0,004:1
Benzene / Toluene	0,002:1
Acetone / Toluene	0,0004:1

The relative quantitative ratios of the total values of the intermediates formed by the photocatalytic oxidation of toluene are investigated. This was important for studying in which direction and through which intermediate stages toluene is oxidized.

The benzaldehyde / toluene ratio was 0.069:1. This result showed that benzaldehyde is the main intermediate in the pathway of photocatalytic oxidation of toluene. That is, as a result of the first oxidation of toluene, mainly benzaldehyde is formed.

The benzyl alcohol / toluene ratio was 0.004:1. This indicator means that a small amount of benzyl alcohol is formed from toluene. The benzyl alcohol can further oxidize to benzaldehyde. Thus, benzyl alcohol also plays a role as an intermediate product, but the amount is significantly smaller.

The benzene / toluene ratio was recorded at around 0.002:1. This means that a small amount of benzene is formed directly from toluene or through benzaldehyde. This situation indicates that benzene is not the main end product, the path of its formation in the reaction is limited. However, this does not exclude the way benzene is formed as an intermediate product.

The acetone / toluene ratio was at 0.0004:1. This very small concentration means that toluene forms acetone indirectly or through secondary processes. It is observed that acetone appeared only as an additional by-product, and not in the main reaction pathway.

In general, it was found that after the photocatalytic oxidation of toluene, benzaldehyde was formed as the most dominant intermediate. Its amount is quite high in comparison with other products. This proves that the main oxidation pathway of toluene is through benzaldehyde. Although benzyl alcohol and benzene are also formed as intermediates, their proportion is low. Acetone, on the other hand, is a possible secondary or by-product formed as a result of combustion.

The ratio of the common values of the intermediates formed by benzaldehyde is given in Table 4.

Table 2. The ratio of the common values of the intermediates formed by toluene.

Benzene / Benzaldehyde	0,371:1
Benzyl alcohol / benzaldehyde	0,02:1
Benzoic acid / benzaldehyde	0,017:1
Acetone / Benzaldehyde	0,0025:1

As a result of a comparative quantitative analysis, the proportions of intermediate products formed in comparison with benzaldehyde were determined. This analysis was carried out to assess which compounds can be further formed from benzaldehyde.

The benzene / benzaldehyde ratio was 0.371:1. This result indicates that a significant amount of benzaldehyde is further converted to benzene. That is, benzaldehyde is an important intermediate product in the benzene formation pathway.

However, the formation of benzene from benzaldehyde is considered a tentative pathway under photocatalytic conditions and may not represent the dominant reaction route. In conventional TiO₂-mediated oxidation systems, benzaldehyde is typically further oxidized to benzoic acid and ultimately mineralized to CO₂ and H₂O. The observed benzene formation may be associated with partial deoxygenation or decarbonylation reactions occurring under specific surface properties and reactive oxygen species conditions. Therefore, this pathway should be interpreted as a secondary transformation rather than a primary reaction mechanism. Additional confirmation through in situ spectroscopic techniques or isotopic labeling experiments would be required to fully validate this pathway.

The benzyl alcohol / benzaldehyde ratio was around 0.02:1. This means that benzyl alcohol is oxidized to benzaldehyde, but its concentration is low. That is, benzaldehyde is considered as a fact confirming that it is formed from benzyl alcohol, but the proportion of this pathway is limited.

The benzoic acid / benzaldehyde ratio was recorded at 0.017:1. This indicator means that benzoic acid is formed in small quantities by oxidation from benzaldehyde. This also proves the ability of benzaldehyde to oxidize further, but shows that it is not the main pathway.

The acetone / benzaldehyde ratio was only 0.0025:1. This indicates the formation of a very small amount of acetone, perhaps indirectly or as a product of the combustion process. The probability of its formation from benzaldehyde is low, but indirect processes occurring in the course of the reaction cannot be ruled out.

Based on these relative values, it was found that benzaldehyde becomes benzene as the main intermediate product, and the proportion of paths with the participation of benzoic acid and benzyl alcohol is significantly less. That is, the formation of benzene is the main transformation pathway of benzaldehyde.

Conclusion. The paper investigated the process of photocatalytic oxidation of toluene. It was found that under the influence of a photocatalyst based on titanium(IV) oxide and ultraviolet radiation, multi-stage radical oxidation of toluene occurs. It was shown that the main active particles in the reaction are •OH, O₂•⁻ radicals and electron-hole pairs.

According to the results of gas chromatography-mass spectrometry, benzaldehyde was identified as the main intermediate product (benzaldehyde/toluene=0.069:1). It has also been observed that small amounts of benzyl alcohol, benzene and acetone are formed.

A study of the photocatalytic oxidation of benzaldehyde showed that it is the main intermediate compound in the formation of benzene. The presence of a benzene/

benzaldehyde ratio of 0.371:1 proved that benzaldehyde is converted to benzene by photochemical transformation or decarbonylation.

The results of the study showed that the photocatalytic oxidation of toluene is a complex and multi-stage process. During the reaction, partial oxidation of the aromatic ring and the formation of intermediates occurs, and at the final stage, organic compounds are completely mineralized and decomposed to CO₂ and H₂O. It was also found that the photocatalytic system has the ability to form toxic and carcinogenic intermediates, including benzene. This indicates the importance of controlling the formation of intermediate products when improving photocatalytic cleaning technologies.

In general, the results obtained allow us to deeply understand the mechanisms of neutralization of volatile organic compounds by the photocatalytic method and can serve as a scientific basis for the development of environmentally safe and effective photocatalytic technologies.

The scientific contribution of this work consists in establishing the relative distribution of major intermediate products formed during photocatalytic oxidation of toluene and demonstrating the potential involvement of benzaldehyde in benzene formation under TiO₂/UV conditions. These findings contribute to a better understanding of benzene formation during photocatalytic oxidation of toluene and may assist in the development of photocatalytic systems that minimize the generation of toxic intermediate products.

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