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SYNTHESIS AND PHOTOCATALYTIC ACTIVITY OF HIERARCHICAL FLOWER-LIKE ZNO FOR METHYLENE BLUE DEGRADATION UNDER UV LIGHT

Abstract

In this study, a porous, hierarchical flower-like zinc oxide (ZnO) structure was successfully synthesized via a hydrothermal method using urea and CTAB as shape-directing agents to optimize photocatalytic efficiency for environmental remediation. Structural characterization via scanning electron microscopy (SEM) and X-ray diffraction (XRD) confirmed the formation of a highly crystalline, pure hexagonal wurtzite phase assembled from ultrathin nanosheet building blocks. The photocatalytic performance of the synthesized ZnO was evaluated through the degradation of methylene blue (MB) dye under UV light illumination (365 nm). The catalyst achieved a (98.7 ± 0.5)% degradation efficiency within 40 minutes, following a pseudo-first-order kinetic model with a reaction rate constant of 0.109 min^{-1}. Optimization studies identified a catalyst dosage of 0.2 g/L as near-optimal, while coexistence tests demonstrated that the system maintains high stability and excellent removal efficiency in the presence of various competing environmental anions (Cl^-, NO_3^-, HCO_3^-, CO_3^{2-}). Radical scavenger investigations revealed a clear inhibition hierarchy, establishing that the degradation mechanism is predominantly driven by hydroxyl radical-mediated oxidation alongside secondary contributions from photogenerated holes. These findings highlight the prepared porous ZnO architecture as an efficient, stable, and sustainable candidate for the degradation of persistent organic pollutants in municipal wastewater.

Keywords: zinc oxide, hydrothermal synthesis, methylene blue, photocatalytic degradation, hierarchical nanostructures.

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Introduction

The rapid progress of industrialization and global economic development has significantly improved living standards but has also led to the uncontrolled release of organic contaminants into the environment [1]. Among these pollutants, synthetic dyes like methylene blue (MB), which are extensively used in industries such as textiles, printing, paper, and cosmetics, represent an environmental risk [2] the effectiveness of ZnO-Mn-Ce/biochar as catalyst and activated by H2O2 for the removal of Methylene Blue (MB. MB is a stable aromatic compound that is highly toxic, potentially carcinogenic, and resistant to natural biodegradation [3] pore size, and oxygen defects. Its presence in water bodies blocks sunlight penetration, which harms aquatic ecosystems by impeding photosynthesis and ultimately leading to oxygen depletion [4].

While conventional wastewater treatment technologies, including membrane filtration, chemical reduction, and adsorption, have been successfully employed, they often suffer from limitations such as high energy consumption, high operational costs, and the generation of secondary sludge that requires further disposal [3] pore size, and oxygen defects. In this context, photocatalysis has emerged as a promising, green, and sustainable alternative for the complete mineralization of organic pollutants into harmless substances such as carbon dioxide and water under mild reaction conditions, using solar or ultraviolet energy [2] the effectiveness of ZnO-Mn-Ce/biochar as catalyst and activated by H2O2 for the removal of Methylene Blue (MB.

ZnO is widely recognized as a highly efficient n-type semiconductor photocatalyst due to its wide direct bandgap of approximately 3.37 eV, high exciton binding energy of 60 meV, high electron mobility, and excellent chemical stability [5, 6]. Compared to other metal oxides like TiO_2, ZnO is often more cost-effective and exhibits better light absorption across a broader range of the solar spectrum, making it a viable alternative for degrading persistent organic compounds [3, 6] pore size, and oxygen defects. However, the practical application of ZnO is frequently hindered by two major challenges: the fast recombination rate of photogenerated electron-hole pairs and a wide bandgap that limits its efficiency under visible light irradiation [4, 7].

To address these limitations, recent research has shifted toward the development of innovative nanostructures and hierarchical architectures [6]. Controlling the morphology of ZnO at the nanoscale is essential because the photocatalytic process is a surface-mediated reaction consisting of the adsorption of reactants, reactive radical generation, and the subsequent oxidation of pollutants [3] pore size, and oxygen defects. In particular, the synthesis of porous ZnO nanostructures has attracted significant attention [7]. Porous materials provide a significantly larger specific surface area compared to bulk ZnO, creating a high density of active sites for the generation of reactive oxygen species (ROS), such as hydroxyl radicals (•OH) and superoxide radical anions ($\bullet O^{2-}$) [8, 9].

Studies have shown that the pore size and distribution are critical determinants of photocatalytic performance [3] pore size, and oxygen defects. Mesoporous ZnO (pore sizes between 2 and 50 nm) is particularly effective as it facilitates the rapid mass transport and diffusion of reactant molecules and dissolved oxygen into the internal active sites of the catalyst [3, 7] pore size, and oxygen defects. Furthermore, 3D hierarchical architectures assembled from porous nanosheets can enhance light harvesting through repeated internal scattering and reflection within the cavities, while simultaneously preventing the aggregation of particles during the reaction [8, 10].

Various synthetic strategies have been established to produce these nanostructures, including hydrothermal, sol-gel, and precipitation methods [6, 11] as a widely used material in optics, electronics, and medicine, requires a complete overview of different conditions for facile and easily reproducible syntheses. Two types of optimization of ZnO hydrothermal preparation from zinc acetate and sodium hydroxide solution are presented, which allowed for obtaining miscellaneous morphologies of materials. The first was a temperature-controlled synthesis from 100 to 200 °C, using citric acid as a capping agent. The formation of hexagonal rods at the lowest temperature was evidenced, which agglomerated to flower-like structures at 110 and 120 °C. It was followed

by transformation to flake-like roses at 160 °C, up to disordered structures composed of nanosized plates (>180 °C. Among these, hydrothermal synthesis has gained popularity as a fast, simple, and cost-effective approach that provides homogeneous volumetric heating, significantly accelerating the reaction rates compared to conventional heating [4, 7]. This study explores the synthesis of porous ZnO particles designed specifically for the efficient photocatalytic degradation of MB. By optimizing the surface properties and porous structure of the catalyst, this research aims to provide new technical and theoretical insights into the design of advanced materials for dye wastewater remediation.

Materials and methods

For the synthesis, 400 mg of zinc nitrate hexahydrate ($Zn(NO_3)_2 \cdot 6H_2O$), 100 mg of urea, and 100 mg of hexadecyltrimethylammonium bromide (CTAB) were dissolved in 20 mL of deionized water under continuous stirring. The resulting homogeneous solution was transferred into a 40 mL Teflon-lined stainless-steel autoclave and subjected to hydrothermal treatment at 200 °C for 6 h in a convection oven. After naturally cooling to room temperature, the obtained precipitate was separated by centrifugation, thoroughly washed several times with deionized water and ethanol to remove residual organic species, and dried overnight at 60 °C. The dried material was subsequently annealed in air at 300 °C for 1 h with a heating rate of 5 °C min^{-1} to obtain the final ZnO product.

The crystalline structure and phase composition of the synthesized ZnO-400 were examined using a SmartLab X-ray diffractometer (Rigaku Corp., Japan). The morphology and hierarchical architecture of the samples were investigated by scanning electron microscopy (SEM, Crossbeam 540, Carl Zeiss, Germany).

The photocatalytic activity of ZnO-400 was assessed using MB as a model organic dye. For each experiment, 2 mg of the catalyst was dispersed in 10 mL of MB solution (3×10^{-5} M) and stirred in the dark for 60 min to establish adsorption-desorption equilibrium. After the adsorption step, the suspension was exposed to UV light irradiation at 365 nm using an 18 W lamp, and aliquots were collected at predetermined intervals of 10, 20, and 40 min. The degradation efficiency was determined by measuring the change in the maximum absorption peak of MB at 664 nm using a UV-Vis spectrophotometer (Thermo Fisher Scientific Inc., USA).

Results and discussion

Before detailed characterization, preliminary screening experiments were carried out to select the optimal precursor mass and to evaluate the necessity of post-synthesis annealing. Using ($Zn(NO_3)_2 \cdot 6H_2O$) precursor masses of 100, 200, and 400 mg, MB degradation after 20 min of UV irradiation reached 48.9%, 27.8%, and 58.8%, respectively, identifying 400 mg as the most effective precursor mass (denoted ZnO-400). In addition, the non-annealed sample achieved only 5.1% degradation after 20 min, compared to 58.8% for the annealed ZnO-400 sample, confirming that post-synthesis annealing at 300 °C is essential for achieving high photocatalytic activity, likely due to the removal of residual organic species (urea, CTAB) that would otherwise block active sites on the catalyst surface. Based on these results, ZnO-400 was selected for detailed structural characterization and photocatalytic evaluation. The surface morphology and hierarchical structure were observed using SEM at various magnifications (Figure 1).

The SEM images of the ZnO particles reveal a distinctive hierarchical flower-like architecture composed of interconnected thin nanosheets. At lower magnifications, these structures appear as complex three-dimensional assemblies resembling flowers, where numerous “petals” radiate from a central core to form a textured framework [12]. Upon closer inspection at higher magnification, these petals are identified as ultrathin nanosheets or plates with highly irregular edges, contributing to a significantly complex and potentially porous surface morphology. High-resolution imaging specifically quantifies the thickness of these individual building blocks, with measurements showing

values of approximately 29.11 nm and 39.51 nm, confirming their nanostructural nature. This unique morphology suggests that the hydrothermal conditions, combined with the presence of urea and the surfactant CTAB as shape-directing agents, successfully facilitated the growth of a high-surface-area framework assembled from these nanoscale sheets [13]. Then, the crystalline structure and phase orientation of the synthesized ZnO-400 particles were characterized by XRD (Figure 2).

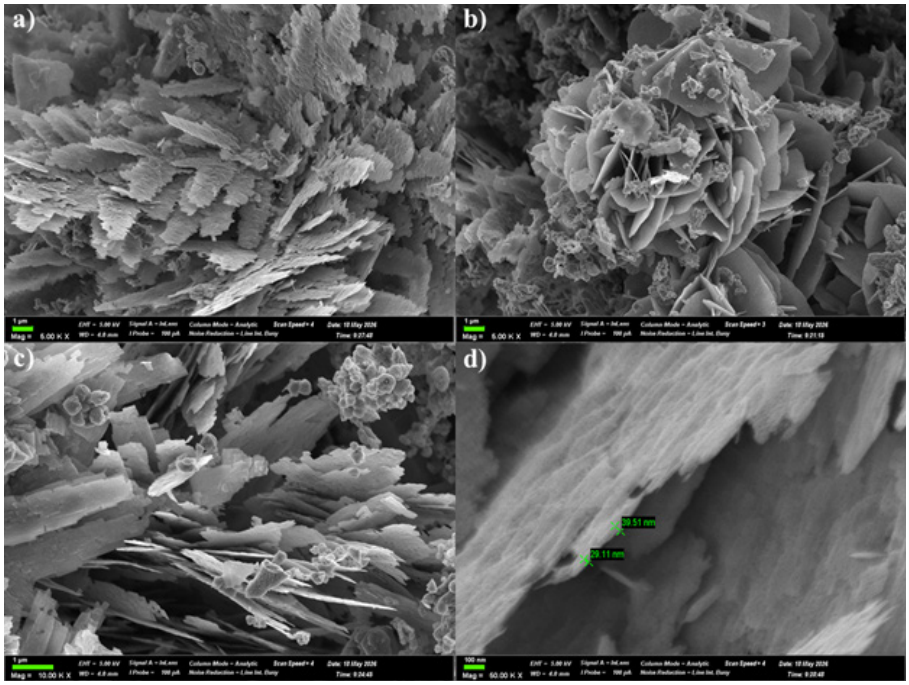

Figure 1 – SEM images of 400a-ZnO structures at different magnifications:
(a) and (b) 5,000; (c) 10,000; and (d) 50,000

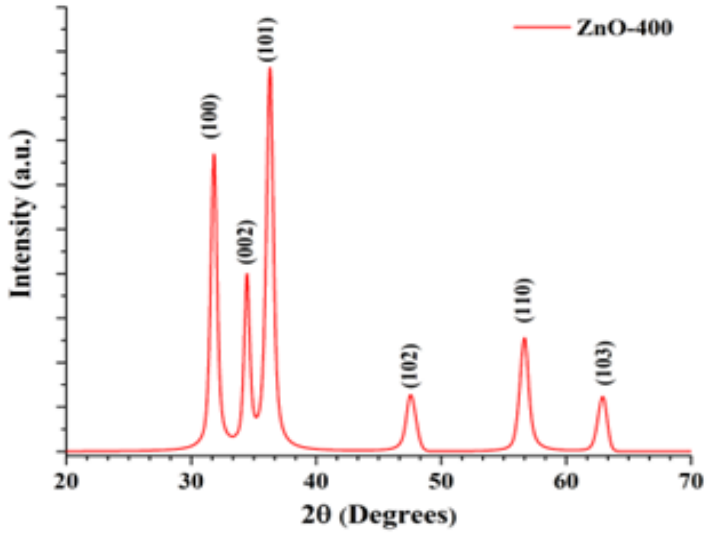

Figure 2 – XRD patterns of ZnO-400 particles

The XRD pattern for the ZnO-400 sample provides a detailed insight into the structural properties and phase purity of the synthesized material. The XRD pattern displays distinct diffraction peaks at 2θ values of approximately 31.8°, 34.4°, 36.3°, 47.6°, 56.6°, and 62.9°, corresponding to the (100), (002), (101), (102), (110), and (103) crystallographic planes, respectively. These reflections confirm the formation of the hexagonal wurtzite phase of ZnO and agree well with the ICSD reference card No. 01-076-8930 [14]. Notably, the (101) peak displays the highest relative intensity, a characteristic typical of the standard polycrystalline ZnO pattern with random grain orientation [4]. The average crystallite size of the synthesized particles was calculated using the Scherrer equation. It was found to be 12.51 ± 2.94 nm, confirming that the annealing at 300 °C was sufficient to achieve a well-ordered lattice. Furthermore, the absence of any extraneous peaks and the very low background noise in the pattern signify high phase purity, indicating that the washing steps and subsequent thermal treatment effectively removed all precursors, such as zinc nitrate, and organic additives like urea and CTAB.

The photocatalytic performance of ZnO-400 was assessed by monitoring the time-dependent degradation of MB under UV irradiation. The results were compared with the self-degradation control performed under UV light, as shown in Figure 3. The experimental procedure included a 60-minute dark adsorption phase (60 min) to establish an adsorption-desorption equilibrium.

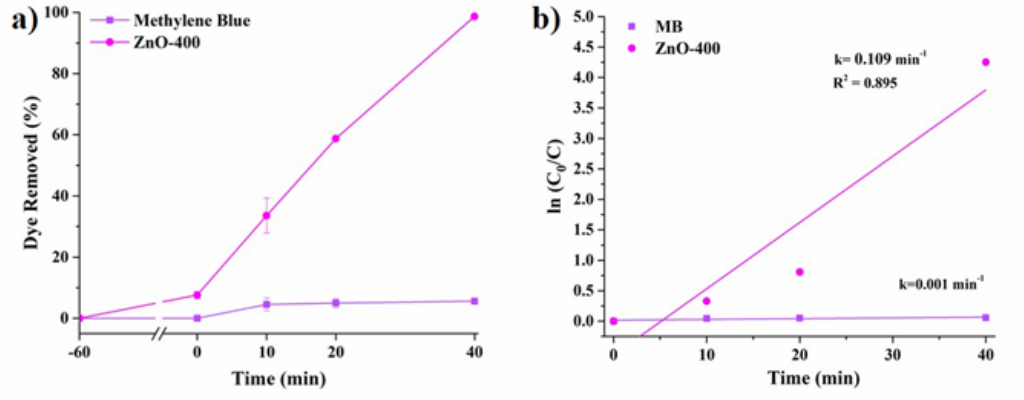

Figure 3 – Photodegradation of MB as a function
of irradiation time using ZnO-400 (0.2 g/L) (a) and kinetics (b)

Once light irradiation commenced, the ZnO-400 catalyst demonstrated a rapid and significant increase in photocatalytic activity, reaching (33.6 ± 5.8)% degradation within just 10 minutes and jumping to (58.8 ± 0.1)% by 20 minutes. By the conclusion of the 40-minute test, the ZnO-400 sample achieved a (98.7 ± 0.5)% degradation efficiency. In contrast, the control showed negligible self-degradation, reaching only (5.6 ± 0.3)% degradation after 40 minutes. This difference confirms that the dye is highly stable under light exposure and that the observed degradation is almost entirely facilitated by the ZnO catalyst. The excellent photocatalytic performance of ZnO-400 can be attributed to its hierarchical flower-like architecture composed of ultrathin nanosheets, which provides a large accessible surface area for dye adsorption and abundant active sites for the generation of reactive oxygen species, thereby promoting the efficient mineralization of the organic pollutants.

The photocatalytic degradation kinetics of MB were analyzed using the Langmuir–Hinshelwood model. The linear relationship between $\ln(C_0/C)$ and irradiation time (Figure 3b) indicates that the degradation process follows pseudo-first-order kinetics [15]. The reaction rate constant (k) for the ZnO-400 sample was determined to be 0.109 min^{-1}, which was 109 times higher than the rate constant

of the self-degradation of MB (k = 0.001 min^{-1}). This significant enhancement in the kinetic rate is directly related to the hierarchical flower-like morphology and the assembly of thin nanosheets identified in the SEM analysis, which provide a high specific surface area and abundant active sites for the generation of reactive oxygen species. By the 40-minute mark, the $ln(C_0/C)$ value for ZnO-400 reached approximately 2.3, indicating a rapid and efficient degradation process compared to the negligible changes observed in the catalyst-free control. The high linearity of the ZnO-400 plot suggests a stable and consistent catalytic process throughout the duration of the experiment.

The photocatalytic performance of ZnO-400 compares favorably with recently reported ZnO-based systems. Khao et al. achieved 99% MB degradation using hydrothermally synthesized ZnO nanoparticles after 75 minutes of UV irradiation at 254 nm with a catalyst loading of 0.375 g/L [16]. Saadi et al. reported a degradation efficiency of 72.3% for methylene blue using sol-gel-derived ZnO nanoparticles under UV irradiation, which is lower than the 90.8% achieved by ZnO-400 in only 40 min. [17]. Krobthong et al. compared ZnO nanoparticles prepared by precipitation and combustion, obtaining apparent rate constants lower than the rate constant of 0.109 min^{-1} determined for ZnO-400 [18]. Atta et al. demonstrated 75% MB removal in 3 hours using ZnO thin films under UV irradiation [19]. The strong performance of ZnO-400 is attributed to the hierarchical flower-like architecture assembled from ultrathin nanosheets, which provides a higher active surface area and more efficient light harvesting compared to the conventional nanoparticle or thin-film morphologies used in these studies.

Next, the effects of catalyst dosage and the presence of various anions on MB photodegradation were studied (Figure 4).

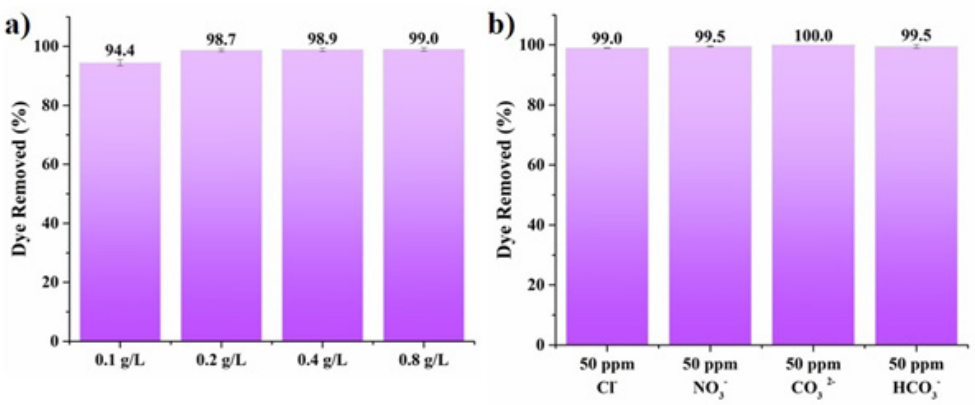

Figure 4 – MB photodegradation at different ZnO-400 catalyst dosages (a) and in the presence of various anions (ZnO 0.2 g/L) (b)

The effect of catalyst dosage on photocatalytic degradation was evaluated by varying the ZnO concentration from 0.1 to 0.8 g/L (Figure 4a). MB degradation increased from (94.4 ± 1.1)% to (99.0 ± 0.6)%, indicating that higher catalyst loadings provide more active sites and enhance reactive oxygen species generation. The most notable improvement occurred between 0.1 and 0.2 g/L, where degradation reached (98.7 ± 0.5)%. Beyond 0.2 g/L, only marginal gains were observed, suggesting that the system approached saturation and that factors such as light shielding and increased turbidity may limit further improvement [4, 14]. Therefore, 0.2 g/L can be considered a near-optimal catalyst dosage, providing high degradation efficiency while maintaining efficient light utilization and cost-effectiveness. The analysis of the interfering ions (Figure 4b) shows that MB degradation remained high in the presence of common anions (50 ppm), with degradation efficiencies of (99.0 ± 0.1)% for

Cl^-, (99.5 ± 0.2)% for NO_3^-, (99.5 ± 0.6)% for HCO_3^-, and 100% for CO_3^{2-}. The minimal variation among different ions indicates that coexisting anions have little influence on the photocatalytic process. This high tolerance to interfering species highlights the potential of the ZnO-400 catalyst for practical wastewater treatment applications.

The analysis of the scavenger radicals identifies the primary reactive species driving the dye degradation process by observing the reduction in degradation efficiency upon the addition of specific quenching agents (Figure 5) [1, 16].

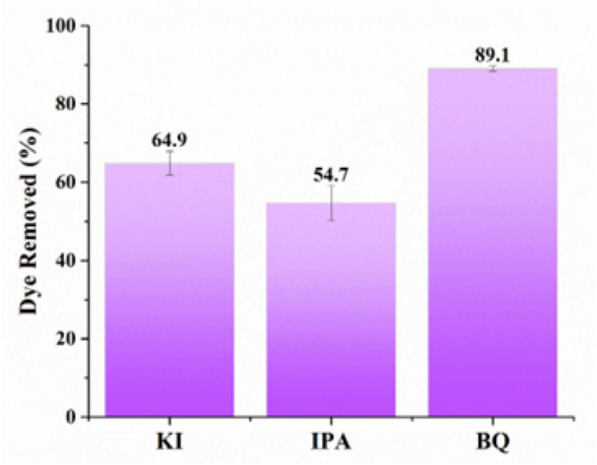

Figure 5 – Effects of radical scavengers (10 mM each)
on MB photodegradation by ZnO-400 (0.2 g/L).

The most significant suppression occurs with the addition of isopropanol (IPA), which drops the degradation efficiency to (54.7 ± 4.4)%, indicating that hydroxyl radicals (•OH) are the dominant oxidative species in the system. Potassium iodide (KI), used to scavenge holes (h^+) and surface-bound hydroxyl radicals, results in an MB degradation of (64.9 ± 3.1)%, suggesting that photogenerated holes also play a substantial secondary role in the reaction mechanism. Conversely, the addition of benzoquinone (BQ) has the least inhibitory effect, with the system still achieving (89.1 ± 0.6)% dye degradation, which reveals that superoxide radicals ($•O^{2-}$) are the least significant contributors to the overall degradation process. This hierarchy of inhibition (IPA > KI > BQ) clearly establishes a mechanism primarily governed by hydroxyl radical-mediated oxidation, supplemented by direct hole oxidation.

Conclusions

Porous flower-like ZnO structures were successfully synthesized via the hydrothermal method and exhibited excellent photocatalytic activity toward MB degradation. Structural characterization confirmed the formation of highly crystalline hexagonal wurtzite ZnO assembled from ultrathin nanosheets, providing a large active surface area for photocatalytic reactions. Under UV irradiation, the catalyst achieved (98.7 ± 0.5)% MB degradation within 40 min and followed pseudo-first-order kinetics with a rate constant of 0.109 min^{-1}. The optimal catalyst dosage was 0.2 g/L, and the photocatalytic performance was largely unaffected by the presence of common inorganic anions. Scavenger experiments revealed that hydroxyl radicals were the primary reactive species, with photogenerated holes also contributing to degradation. These findings demonstrate that the synthesized ZnO structures are promising photocatalysts for the efficient treatment of dye-contaminated wastewater.

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МЕТИЛЕН КӨГІН УК-СӘУЛЕ АСТЫНДА ЫДЫРАТУҒА АРНАЛҒАН ИЕРАРХИЯЛЫҚ ГҮЛ ТӘРІЗДІ ZNO СИНТЕЗІ ЖӘНЕ ОНЫҢ ФОТОКАТАЛИТИКАЛЫҚ БЕЛСЕНДІЛІГІ

Аңдатпа

Зерттеуде қоршаған ортаны тазартудағы фотокаталитикалық тиімділікті арттыру мақсатында несепнәр мен CTAB-ты пішін түзуші агенттер ретінде пайдалана отырып, гидротермиялық әдіспен кеуекті, иерархиялық гүл тәрізді мырыш оксиді (ZnO) құрылымы сәтті синтезделді. Сканерлеуші электрондық микроскопия (SEM) және рентгендік дифракция (XRD) нәтижелері аса жұқа нанопарақшалардан құралған жоғары кристалдық таза гексагональды вюрцит фазасының түзілгенін растады. Синтезделген ZnO-дың фотокаталитикалық қасиеттері 365 нм УК-сәуле әсерінде метилен көгін (MB) ыдырату арқылы зерттелді. Катализатор 40 минут ішінде бояғыштың (98.7 ± 0.5)%-ын ыдыратып, реакция жылдамдығының тұрақтысы 0.109 мин$^{-1}$ болатын жасанды бірінші ретті кинетикалық модельге сәйкес жоғары белсенділік көрсетті. Оңтайландыру зерттеулері катализатордың 0.2 г/л мөлшері тиімді екенін анықтады. Сонымен бірге, қатар жүретін иондардың әсерін зерттеу нәтижелері жүйенің әртүрлі қоршаған орта аниондарының (Cl^-, NO_3^-, HCO_3^-, CO_3^{2-}) қатысуында да жоғары тұрақтылық пен тиімділікті сақтайтынын көрсетті. Белсенді бөлшектерді тұзақтау тәжірибелері деградация механизмі негізінен гидроксил радикалдарының (•OH) тотығу әрекетімен жүзеге асатынын, ал фотогенерацияланған кемтіктердің (h^+) қосымша үлес қосатынын анықтады. Алынған

нәтижелер синтезделген кеуекті ZnO құрылымының коммуналдық ағынды сулардағы тұрақты органикалық ластағыштарды ыдыратуға арналған жоғары тиімді, тұрақты және экологиялық қауіпсіз фотокатализатор екенін көрсетеді.

Түйін сөздер: мырыш оксиді, гидротермиялық синтез, метилен көгі, фотокаталитикалық ыдырау, иерархиялық наноқұрылымдар.

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СИНТЕЗ И ФОТОКАТАЛИТИЧЕСКАЯ АКТИВНОСТЬ ИЕРАРХИЧЕСКОГО ЦВЕТКОПОДОБНОГО ZNO ДЛЯ ДЕГРАДАЦИИ МЕТИЛЕНОВОГО СИНЕГО ПОД ДЕЙСТВИЕМ УФ-ИЗЛУЧЕНИЯ

Аннотация

В данной работе методом гидротермального синтеза с использованием мочевины и цетилтриметиламмоний бромида (CTAB) в качестве структурообразующих агентов была успешно получена пористая иерархическая цветкоподобная структура оксида цинка (ZnO), предназначенная для повышения фотокаталитической эффективности в процессах очистки окружающей среды. Структурная характеристика с использованием сканирующей электронной микроскопии (SEM) и рентгеновской дифракции (XRD) подтвердила формирование высококристаллической чистой гексагональной фазы вюрцита, состоящей из ультратонких нанолистов. Фотокаталитическая активность синтезированного ZnO была исследована на примере деградации красителя метиленового синего (MB) под воздействием ультрафиолетового излучения (365 нм). Катализатор обеспечил высокую степень разложения красителя – (98.7 ± 0.5)% за 40 минут, при этом процесс подчинялся кинетической модели псевдопервого порядка с константой скорости реакции 0.109 мин$^{-1}$. Исследования по оптимизации показали, что дозировка катализатора 0.2 г/л является близкой к оптимальной. Тесты на совместное присутствие ионов продемонстрировали высокую стабильность системы и эффективность удаления загрязнителя в присутствии различных конкурирующих анионов окружающей среды (Cl^-, NO_3^-, HCO_3^-, CO_3^{2-}). Исследования с использованием ловушек активных частиц выявили выраженную иерархию ингибирования, показав, что механизм деградации преимущественно определяется окислением с участием гидроксильных радикалов (•OH), при дополнительном вкладе фотогенерированных дырок (h^+). Полученные результаты свидетельствуют о том, что синтезированная пористая архитектура ZnO является высокоэффективным, стабильным и экологически устойчивым материалом для удаления стойких органических загрязнителей из коммунальных сточных вод.

Ключевые слова: оксид цинка, гидротермальный синтез, метиленовый синий, фотокаталитическая деградация, иерархические наноструктуры.