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Effect of surfactant SDS on the morphology and photocatalytic performance of Zn$_2$GeO$_4$ nanorods

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Abstract

The continuous discharge of organic dye effluents from textile industries causes severe global water pollution. A sustainable and effective route needs to be developed for the treatment of textile effluent in order to enable environmental protection and water recycling. In particular, the nanomaterials-based photocatalytic degradation of organic compounds is a promising approach to minimize water pollution. Herein, a facile hydrothermal method was reported for the preparation of Zn$_2$GeO$_4$ nanorods (NDs) with unique size, shape, and surface chemistry using sodium lauryl sulfate (SDS) as the surfactant. The crystal structure, size, and shape of the synthesized NDs were characterized by X-ray diffraction pattern (XRD) and field-emission scanning electron microscopy (FESEM). Initially, the XRD pattern revealed that SDS plays a crucial role in the formation of highly pure Zn$_2$GeO$_4$ NDs with rhombohedral crystalline nature. It was clearly noticed that increasing SDS concentration results in the formation of Zn$_2$GeO$_4$ NDs with decreased size ranges (100 nm). Conversely, the size of Zn$_2$GeO$_4$ NDs increased at higher SDS concentrations. The photocatalytic activity of Zn$_2$GeO$_4$ NDs was evaluated by the degradation of methyl orange (MO) in aqueous solution. Under light irradiation, the Zn$_2$GeO$_4$ NDs prepared by using different concentrations of SDS exhibited varied photocatalytic performance. Among the tested samples, Zn$_2$GeO$_4$ NDs prepared with 0.1 g of SDS showed the best photocatalytic activity with a MO decomposition rate of 94.6% within 60 min. This study suggests that SDS can be used to modulate the morphology and photocatalytic performance of Zn$_2$GeO$_4$ NDs, and the resultant Zn$_2$GeO$_4$ NDs can serve as a photocatalyst in wastewater treatment.

1. Introduction

In recent years, continuous expansion of water pollution demands alternative semiconductor-based photocatalysts to eliminate the organic pollutants in wastewater, especially for the degradation of organic dyes [1–4]. Most of the contemporary research work on photocatalysis has been focused on the dioxide- and disulfide-based nanocatalysts, including SiO$_2$ nanowires, NDs and nanotubes [5–7], TiO$_2$ nanoparticles and nanotubes [8–12], MnO$_2$-based nanostructures [13], MoS$_2$ nanotubes and nanopetals [14, 15], WS$_2$ nanosheets and NDs [16], SnS$_2$ nanosheet arrays [17], and so on. As compared with dioxides and disulfides, zinc germanate (Zn$_2$GeO$_4$) is received great attention because of its higher bandgap width (4.68 eV) and unique physicochemical properties [18, 19]. The Zn$_2$GeO$_4$-based nanomaterials hold the ability to generate maximum electrons upon light irradiation, which ultimately results in the increased reduction and oxidation of organic pollutants [20]. Furthermore, the Zn$_2$GeO$_4$-based nanomaterials are less prone to the photo corrosion effect than sulfide catalysts [21]. The unique structural properties of Zn$_2$GeO$_4$ helps them to be easily doped with different active ingredients [22, 23]. These advantages make the Zn$_2$GeO$_4$-based nanomaterials as a promising semiconductor-based photocatalyst [24]. Owing to their stupendous thermal and chemical stability, the Zn$_2$GeO$_4$-based nanomaterials have been used for bright white-bluish luminescence and deep UV detection [25].
At present, much effort has been devoted to fabricating the Zn$_2$GeO$_4$ nanowires, nanotubes, and NDs with diverse physicochemical properties and improved photodegradation performance [13–15]. Conventional methodologies, such as chemical vapor transport [26], catalyst-free evaporation deposition [27], hydrothermal/solvothermal approach [28, 29], hydrolysis of zinc and germanium double alkoxides [30], and solid-state synthesis [31], are available for the preparation of Zn$_2$GeO$_4$. Among these methods, the hydrothermal approach has been validated to be a low cost and simple process, and widely used in the fabrication of well-defined micro/nanostructures [32, 33].

The surfactants are always used in nanomaterial preparation not only to minimize the surface tension of liquids, it also plays a vital role in controlling the structural and functional properties of nanomaterials [34, 35]. The surface absorption behavior of surfactants on different crystal planes of nucleating centers directs the shape and size of nanomaterials [36, 37]. A cationic surfactant, cetyltrimethylammonium bromide (CTAB) has been used for the preparation of Zn$_2$GeO$_4$ [38, 39]. However, it was noticed that the addition of CTAB does not influence the growth and morphology of Zn$_2$GeO$_4$ [39]. The anionic sodium lauryl sulfate (SDS) possesses high water-oil balance value, as well as a good emulsifying and dispersing effect in the aqueous medium. To the best of our knowledge, SDS has not been utilized as a surfactant in the preparation of Zn$_2$GeO$_4$. Hence, in the present investigation, SDS was used as the surfactant to prepare Zn$_2$GeO$_4$ NDs through a facile one-step hydrothermal method. The effect of SDS contents on Zn$_2$GeO$_4$ NDs shape and size were studied through exclusive instrumentation. Further, the photocatalytic ability of Zn$_2$GeO$_4$ NDs on the degradation of methyl orange (MO) was evaluated towards wastewater treatment applications. The illustration of research strategy is shown in Scheme 1.

### 2. Experimental section

#### 2.1. Materials

Zinc acetate dihydrate (Zn(OOCCCH$_3$)$_2$·2H$_2$O, 99.0%), germanium oxide (GeO$_2$, 99.99%), SDS (C$_{12}$H$_{25}$O$_4$NaS, 99.0%), MO (ACS reagent, dye content 85%, C$_{14}$H$_{14}$N$_4$NaO$_3$S) were purchased from Shanghai Aladdin Biochemical Technology Co., Ltd.

#### 2.2. Preparation of Zn$_2$GeO$_4$ NDs

For the preparation of Zn$_2$GeO$_4$ NDs, Zn(OOCCCH$_3$)$_2$·2H$_2$O (4.53 g, 20.64 mmol), GeO$_2$ (1.08 g, 10.32 mmol), and different concentration of SDS (0, 0.05, 0.10, 0.20, 0.40, 0.70, and 1.00 g, referred as A0, B0.05, C0.10, D0.20, E0.40, F0.70, and G1.00, respectively) were mixed with 50 ml deionized water. Further, the mixture was stirred using a hot plate at room temperature for 1 h. Then, the reaction mixture pH was adjusted to 8–9 by dropwise addition of NaOH (1M) and stirred continuously for another 1 h. Subsequently, 50 ml of completely blended reaction mixture solution was transferred to a Teflon lined stainless steel autoclave and kept in a furnace at 180 °C for 12 h. After cooling down to room temperature, the final Zn$_2$GeO$_4$ NDs samples were collected using centrifugation and washed with deionized water followed by anhydrous ethanol for several times. Finally, the
samples were dried under reduced pressure at 60 °C for 12 h to obtain the white Zn2GeO4 powders for further studies.

2.3. Characterization
The prepared Zn2GeO4 NDs were characterized initially by powder x-ray diffraction pattern (XRD) and field-emission scanning electron microscopy (FESEM) analyses. The XRD patterns were recorded on XRD-7000 (Shimadzu, Japan) equipped with a graphite monochromatized Cu-Kα radiation (λ = 1.5406 Å) at an accelerating voltage of 40 kV and a current of 30 mA. The FESEM micrographs were obtained with JSM-7800F FESEM (10 kV, JEOL, Japan).

2.4. Photocatalytic activity of Zn2GeO4 NDs
The photocatalytic properties of Zn2GeO4 NDs were assessed by performing the MO degradation assay. Briefly, 20 mg of as prepared Zn2GeO4 NDs samples were separately added to 100 ml of the MO aqueous solution (15 mg l−1). The suspension was magnetically stirred in the dark condition for 30 min to establish the adsorption/desorption equilibrium. Then, the degradation of MO was carried out under the irradiation of a 500 W UV lamp. After irradiation, 4 ml of sample was collected from the suspension every 15 min. The samples were centrifuged at 10,000 rpm for 3 min to remove the photocatalyst. Finally, the resulting clear aliquot was analyzed in a UV-visible absorption spectrophotometer (U-3310) to record the absorbance changes of MO. The degradation rate of MO was calculated based on the maximum absorption intensity ratios of predetermined and initial time.

3. 3. Results and discussion

3.1. Characterization of the Zn2GeO4 NDs
Figure 1 displays the XRD patterns of as-prepared Zn2GeO4 NDs. The strong diffraction planes were noticed at (111), (300), (220), (410), (223), (600), (520), (333), (603), (523), (710), (606), (630), (713), (633), (416) clearly show that the prepared seven Zn2GeO4 NDs samples are in rhombohedral phase (JCPDS85-0454). The XRD patterns also indicate that the Zn2GeO4 NDs have high purity and crystallinity. Similar XRD patterns have been reported earlier, where the Zn2GeO4 ND synthesized via microwave-assisted solution-phase method was in a pure rhombohedral phase crystalline nature [40]. The XRD peaks of Zn2GeO4 NDs prepared with 0.1 g of SDS (C0.10) are broader than other samples. The crystalline sizes (D) of Zn2GeO4 samples are calculated using Scherrer’s equation (1):

\[ D = \frac{K \lambda}{B \cos \theta} \]  

(1)

Where D is the radius (size) of grain, K is the Scherrer constant, λ is the diffraction wavelength, B is the half-height width of the diffraction peak, θ is the Bragg diffraction angle. The crystalline size of resulting A0, B0.05,
C0.10, D0.20, E0.40, F0.70, and G1.00 samples were 124, 117, 71, 155, 172, 197, and 322 nm, respectively. Our results distinctly imply that the crystalline size of C0.10 was the smallest among all other Zn2GeO4 samples. Furthermore, figures 2(A)–(G) displays the FESEM images of all Zn2GeO4 samples. It was noticed that the C0.10 sample is the smallest with good integrity and length of about 100 nm. The A0, B0.05, D0.20, E0.40, and F0.70 samples were similar in size, with a length of about 240 nm and a diameter of around 130 nm. The size of G1.00 sample was high with poor integrity. The results obtained from FESEM micrographs were inconsistent with the crystalline sizes calculated by the Scherrer’s equation. The XRD and FESEM analyses specify that the particle size and integrity of Zn2GeO4 NDs were largely dependent on the concentration of SDS. The size of Zn2GeO4 NDs decreases first with increasing SDS concentration, and increases in excessive SDS concentration. Upon addition of 0.1 g SDS, the FESEM micrograph displayed the formation of smaller Zn2GeO4 NDs with corresponding large surface area.

Figure 2. FESEM images of the A0, B0.05, C0.10, D0.20, E0.40, F0.70, and G1.00 samples.
3.2. Effect of SDS contents on the morphologies of Zn$_2$GeO$_4$ NDs

In the absence of SDS surfactant, GeO$_2$ dissolves in an alkali solution to form a germanic acid anion. The germanic acid anions react with zinc ions to form Zn$_2$GeO$_4$ cores, followed by crystal precipitation into Zn$_2$GeO$_4$ NDs. At lower concentration, SDS exhibits as a monomer, attracts zinc ions and participates in the process for the formation of Zn$_2$GeO$_4$ cores (equations (2)-(4)), and the schematic growth mechanism of Zn$_2$GeO$_4$ NDs can be illustrated in figure 3. The presence of SDS on the outer layers of Zn$_2$GeO$_4$ cores and NDs could inhibit the lateral growth of NDs. Upon increasing the SDS concentration, it forms micelles in aqueous media. The critical micelle concentration (CMC) of SDS in pure water was found to be 2.34 g l$^{-1}$ [41]. In the presence of salts, the surface tension of SDS solution was reduced, and its CMC value becomes lower [42]. The SDS amount used for the preparation of C0.10 and D0.20 was close to its CMC concentration. Thus, for the A0, B0.05, and C0.10 samples, the higher SDS content in the reaction system could inhibit the growth of Zn$_2$GeO$_4$ NDs more significantly and results in the formation of NDs with a smaller size range. For the D0.20, E0.50, F0.70, and G1.00 samples, the increased SDS contents could form rod-, spherical-, or lamellar-like micelles in the reaction mixtures. The self-assembled SDS micelles show weak effect on the crystal growth of Zn$_2$GeO$_4$ cores.

$$2xZn^{2+} + ySDS = Zn_{2x}(SDS)_y^{2x-y}$$  \[ (2) \]

$$GeO_2 + 4OH^- = GeO_4^{2-} + 2H_2O$$  \[ (3) \]

$$Zn_{2x}(SDS)_y^{2x-y} + xGeO_4^{2-} = xZn_2GeO_4 + ySDS$$  \[ (4) \]

3.3. Photocatalytic performance of Zn$_2$GeO$_4$ NDs

The C0.10 sample was chosen for the preliminary evaluation of photocatalytic performance. Figure 4 shows the UV-visible absorption spectra of MO treated with Zn$_2$GeO$_4$ NDs and exposed to UV irradiation at different time intervals. The absorption spectrum of MO shows a peak at about 465 nm. After UV irradiation, the absorption intensity decreases sharply with the increasing irradiation time. The absorption peak of MO almost disappears.

![Figure 3. The structural formula (A) and spherical micellar model (B) of SDS.](image1)

![Figure 4. The time-dependent UV-visible absorption spectra of MO after UV irradiation and removal of the Zn$_2$GeO$_4$ NDs by centrifugation.](image2)
after irradiation for 60 min, which depicts the effective degradation of MO. A possible mechanism of the photocatalytic degradation of MO by Zn$_2$GeO$_4$ NDs is proposed. Under UV irradiation, the Zn$_2$GeO$_4$ NDs are excited to generate the photogenerated electrons ($e^{-}$) and holes ($h^{+}$). The surface-adsorbed O$_2$ or H$_2$O accepts $h^{+}$, forming reactive OH radical. The OH radical decomposes MO [43].

The photocatalytic ability of Zn$_2$GeO$_4$ NDs was then measured by calculating the photocatalytic degradation rate of MO using the following equation (5):

$$\omega_t = \frac{C_0 - C_t}{C_0} \times 100\%$$

Where $C_0$ is the initial concentration of MO, $C_t$ is the concentration of MO at time $t$, and $\omega_t$ is the photocatalytic degradation rate at time $t$. Figure 5 shows the degradation rate of MO photocatalyzed by A0, B0.05, C0.10, D0.20, E0.40, F0.70, and G1.00 samples. The C0.10 sample with the smaller size range exhibits rapid photocatalytic degradation rate against MO, while the A0 and G1.00 with large particle sizes show much slower photocatalytic rates. For the C0.10 sample, its photocatalytic degradation efficiency towards MO is 62.4% at 30 min and 94.6% at 60 min under UV irradiation. The photocatalytic degradation efficiency of C0.10 sample towards MO is higher than that of ZnO nanoneedle- and CaWO$_4$ nanoparticles-based photocatalysts [44, 45].

Langmuir Hinshelwood (L-H) model [46] was further used to study the kinetics of photocatalytic degradation of MO. The photocatalytic degradation rate can be expressed as equation (6):

$$R = -\frac{dC_t}{dt} = h_r \theta = h_r \frac{H C_t}{1 + H C_t}$$

Where $R$ is the reaction rate, $\theta$ is the surface coverage rate, $h_r$ is the reaction rate constant, $H$ is the adsorption coefficient of MO, and $C_t$ is the concentration of MO at time $t$. When $C_t$ is small, the $H C_t$ product is negligible and hence the integration of equation (6) is:

$$\ln \frac{C_t}{C_0} = -K t + B$$

Equation (7) represents that the photocatalytic degradation of MO by Zn$_2$GeO$_4$ NDs follows the first-order kinetic model. Therefore, the first-order reaction kinetic model was used to fit the photocatalytic degradation process of MO. The fitting curves were shown in figure 6.

Figure 7 shows the degradation rate constant of MO photocatalyzed by Zn$_2$GeO$_4$ NDs. For the A0, B0.05, C0.10, D0.20, E0.40, F0.70, and G1.00 samples, the respective MO degradation rate constants are calculated to be 0.40, 2.17, 2.84, 1.12, 0.74, 0.84, and 0.48 h$^{-1}$. The C0.10 sample exhibits the best photocatalytic degradation activity among these samples, and its reaction constant is about 7 times higher than that of Zn$_2$GeO$_4$ NDs prepared in the absence of SDS. The improved photocatalytic performance may be associated with the small particle size and the corresponding large specific surface area of the C0.10 sample, which provides more reactive sites for MO degradation.
4. Conclusion

In summary, the Zn$_2$GeO$_4$ NDs were successfully prepared by a one-step hydrothermal method using SDS as the surfactant. The influence of SDS concentration on morphological features of Zn$_2$GeO$_4$ NDs were clearly evidenced through XRD and FESEM analyses. Based on these results, the optimal SDS concentration was fixed as 0.1 g, which generates uniformly distributed Zn$_2$GeO$_4$ NDs with rhombohedral crystalline nature. Furthermore, the prepared Zn$_2$GeO$_4$ NDs were used for the effective degradation of MO under UV irradiation. Among the tested samples, the Zn$_2$GeO$_4$ NDs prepared with 0.1 g of SDS showed best photocatalytic decomposition of MO in aqueous medium. Thus, the overall results provides a straightforward hydrothermal method for the efficient preparation of Zn$_2$GeO$_4$ NDs towards promising applications in the textile industry effluent treatment. The main drawback of the Zn$_2$GeO$_4$ NDs-based photocatalyst is its difficulty in recycling. In the future, the Zn$_2$GeO$_4$ nanostructures will be fabricated on the magnetic particles to magnetically recycle the photocatalyst.

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Conflict of interest

On behalf of all authors, the corresponding author states that there is no conflict of interest.

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