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Combined Effect of Sonication and Electron Beam Irradiation on the Photocatalytic Organic Dye Decomposition Efficiency of Graphitic Carbon Nitride

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Abstract

The photocatalytic efficiency of graphitic carbon nitride (g-C₃N₄) for aqueous rhodamine B (RhB) decomposition was investigated. To examine the combined effects of sonication and electron beam (EB) irradiation on the photocatalytic efficiency, g-C₃N₄ was sonicated in 1,3-butanediol and subsequently irradiated with EB. The photocatalytic efficiency was improved by low-dose EB irradiation owing to the generation of structural defects that acted as active reaction sites. Sonication before EB irradiation induced mild exfoliation and further improved the photocatalytic efficiency. Prolonged sonication enlarged the improvement, primarily by increasing the specific surface area of g-C₃N₄. The positive effect of sonication was remarkable for g-C₃N₄ irradiated with low-dose EB compared to that for g-C₃N₄ irradiated with higher-dose EB. The difference between the sonication effects could be ascribed to electrostatic interactions and the resultant agglomeration among g-C₃N₄ particles after EB irradiation. High-dose EB irradiation caused electrification followed by coarsening of the particles, whereas low-dose EB irradiation did not produce these results and led to positive effects owing to EB-induced g-C₃N₄ structural alteration.

Keywords: g-C₃N₄; sonication; electron beam; rhodamine B; photocatalytic decomposition

1. Introduction

Graphitic carbon nitride (g-C₃N₄, Figure 1) is a carbon nitride polymorph with a graphite-like layered crystal structure that consists of heptazine ring (C₆H₇) units linked with N atoms. g-C₃N₄ behaves as a photocatalyst under visible light illumination. Its band gap of approximately 2.7 eV corresponds to visible light wavelengths, which allows g-C₃N₄ to utilize solar light efficiently. g-C₃N₄ can catalyze various photoinduced reactions, including hydrogen formation from water, pollutant degradation, and conversion of organic molecules [1–18]. Additionally, g-C₃N₄ is a metal-free photocatalyst comprising ubiquitous elements (C and N). It can be prepared from various commercially available nitrogen-containing organic materials, including melamine, urea, cyanamide, and dicyandiamide, through thermal condensation in air. Hence, the use of the g-C₃N₄ photocatalyst is potentially beneficial to various industrial and environmental processes.

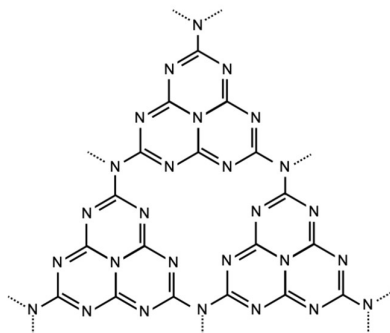


Figure 1. Typical structure of g-C₃N₄.

A well-known shortcoming of g-C₃N₄ photocatalysts is their low photocatalytic reaction efficiency owing to their high electron-hole recombination rates and small specific surface areas. g-C₃N₄ photocatalytic reactions are induced by visible light illumination, generating a charge-separation state via electronic excitation. The excited electrons and holes trigger reduction and oxidation, respectively. However, if electrons and holes recombine rapidly through radiative and non-radiative relaxation, the reaction efficiency is reduced. In addition, the specific surface area of g-C₃N₄ is generally small. Therefore, the space on the g-C₃N₄ surface that contributes to the photocatalytic reaction is limited. Consequently, improving the photocatalytic reaction efficiency is important for expanding the range of g-C₃N₄.

Electron beam (EB) irradiation is a potential method for improving the reaction efficiency of g-C₃N₄. However, studies of the effects of EB irradiation on g-C₃N₄ are limited. Zhang et al. studied the effect of EB irradiation on the photocatalytic efficiency of g-C₃N₄ [19]. They found that EB irradiation improved the photocatalytic efficiency of rhodamine B degradation in aqueous solutions. The loss of pyridinic N and increased tertiary N content were also observed, suggesting that the structural alteration of the tri-s-triazine structure of g-C₃N₄ through EB irradiation resulted in altered photocatalytic efficiency. Picho-Chillán et al. studied the photocatalytic degradation of Direct Blue 1 using g-C₃N₄ irradiated with a low-dose EB [20], finding that EB irradiation increased the adsorption capacity of the dye. Moreover, the photocatalytic dye decomposition efficiency of the EB-irradiated g-C₃N₄ in the presence of H₂O₂ was higher than that of intact g-C₃N₄. Mendes et al. investigated the structural changes in g-C₃N₄ during EB exposure using transmission electron microscopy [21] and found that N species were removed by the EB. Mohapatra et al. studied the effect of EB on the photocatalytic performance of g-C₃N₄ for H₂ generation and Cr⁶⁺ reduction [22]. They reported that N vacancies were generated through EB irradiation, which led to an improvement in photocatalytic efficiency. We also studied the effect of EB irradiation on the structure and reactivity of g-C₃N₄. We found that relatively low-dose EB irradiation improved the photocatalytic efficiency, whereas the efficiency degraded when a higher dose of EB was used [23]. These results indicate that EB-induced formation of defects through N elimination improves g-C₃N₄ photocatalytic efficiency. The enhancement of photocatalytic performance through N defect formation has also been reported using methods other than EB irradiation [5–7,17,18,24–26].

Another strategy for improving photocatalytic efficiency involves exfoliation to create C₃N₄ nanosheets. This exfoliation technique has been applied in numerous studies of g-C₃N₄ photocatalysts. For example, nanosheet fabrication was carried out in some of the above-mentioned studies [6,9,10,15,18] using various methods, such as thermal treatment in air. Liu et al. developed a novel exfoliation method that employed N₂-assisted treatment [27] and cogrinding with sugar [28,29]. As a simpler method, sonication of g-C₃N₄ in an organic liquid can be applied to achieve exfoliation. For example, Yang et al. examined the sonication of g-C₃N₄ in various solvents and found that isopropanol and *N*-methyl-pyrrolidone were promising solvents for preparing stable g-C₃N₄ nanosheet dispersions [30]. She et al. reported the preparation of a stable g-C₃N₄ nanosheet dispersion by sonication using 1,3-butanediol as the solvent [31]. These exfoliation treatments significantly

increased the specific surface area of g-C₃N₄, thereby enhancing its photocatalytic performance. Consequently, a synergistic improvement in the photocatalytic performance is expected from the combination of sonication and EB irradiation. However, studies of these combined effects are scarce.

Herein, the combined treatment of sonication and EB irradiation was examined for the first time to develop a novel technique for improving the efficiency of a photocatalytic reaction (decomposition of aqueous rhodamine B). In this study, g-C₃N₄ prepared from melamine was sonicated for various durations and irradiated with low- or high-dose EB. The textural and chemical characteristics of the samples were investigated to elucidate the effects of the combined irradiation/sonication.

2. Materials and Methods

2.1. Preparation of g-C₃N₄

The g-C₃N₄ was prepared using melamine (FUJIFILM Wako Chemicals, Japan). Melamine was placed in an alumina crucible and covered with an alumina lid. Subsequently, the crucible was heated in air in a muffle furnace at a heating rate of 5 °C/min and maintained at 550 °C for 2 h. A pale yellow product was obtained. The product was ball-milled for 24 h and sifted through stainless-steel sieves. g-C₃N₄ powder with a particle diameter between 1 mm and 180 µm was collected.

2.2. Sonication and Electron Beam Irradiation

The prepared g-C₃N₄ (1.0 g) was added to 25 mL of 1,3-butanediol (Tokyo Chemical Industry, Japan) and sonicated for 60–480 min using an ultrasonic homogenizer (LUH150, Yamato Scientific, Japan) [31]. The ultrasonic frequency was set at 20 kHz. The solvent was then vaporized at 140 °C using a thermostatic drying oven. This procedure was repeated until a sufficient amount of g-C₃N₄ was collected for EB irradiation.

For the EB irradiation, each sample (approximately 5 g) was placed in a polyamide-polyethylene bag (10 × 15 cm) before or after sonication and sealed with a household vacuum sealer. The sample was packed to form a sufficiently thin (1–2 mm thick) particle layer for EB transmission. EB irradiation was conducted at the NHV Corporation Co., Ltd., Kyoto, Japan. The EB dose used for irradiation was 200 or 600 kGy. Irradiation was conducted at ambient temperature. Hereafter, the samples are named based on the sonication time (“S”) and EB dose (“E”). For example, the sample sonicated for 60 min and irradiated with 200 kGy EB is denoted as “S60E200.”

2.3. Characterization

The crystal structures of the samples were elucidated using X-ray diffractometry (XRD, D8 Discover, Bruker, USA) with Cu K_α X-ray. The elemental analysis and classification of chemical bonds were performed by X-ray photoelectron spectroscopy (XPS) (JPS-9200, JEOL, Japan) with Mg K_α X-ray. Indium foil was used to mount the samples for XPS experiments. Peak deconvolution of the high-resolution C 1s and N 1s was performed using CasaXPS software [32]. The specific surface areas of the samples were determined using N₂ adsorption/desorption measurements (Autosorb, Quantachrome Instruments, USA). The Brunauer–Emmett–Teller (BET) adsorption isotherm was used to calculate the specific surface areas [33].

2.4. Photocatalytic Decomposition of Organic Dye

The photocatalytic degradation of RhB was monitored by colorimetric RhB concentration measurements. Portions of a 10 mg/L aqueous RhB solution (50 mL) were taken, and 0.1 g g-C₃N₄ was added to each solution. The mixture was then illuminated with visible light from an Xe light source (MAX-303, Asahi Spectra, Japan). Visible light illumination was performed using a quartz rod immersed in the sample mixture. The wavelength of the light was 400–600 nm. Air bubbling was continued throughout the photocatalytic reaction. The g-C₃N₄-containing solution was sampled every 10 min; g-C₃N₄ was removed from the sample by centrifugation, and the supernatant RhB concentration was determined by colorimetric quantitative analysis (V-630, JASCO, Japan).

3. Results and Discussion

3.1. Characterization of g-C₃N₄

Figure 2 shows the powder XRD patterns of intact and sonicated samples before EB irradiation (S0E0–S480E0). The XRD patterns of S480E200 and S480E600 are also shown. Two major peaks assigned to the (100) and (002) reflections, corresponding to the in-plane packing and interlayer stacking, respectively, were observed at approximately 13° and 27°. Comparison of these diffraction patterns shows that sonication and subsequent EB irradiation slightly affected the entire g-C₃N₄ crystal structure. Although a slight shift in the (002) peak was observed, the main features of the diffraction pattern did not change after sonication and EB irradiation. These results also indicate that sonication under the present treatment conditions did not cause intensive interlayer expansion to create nanosheets with a few layers and probably leads to mild exfoliation. If exfoliation proceeded intensively through sonication, the (002) peak should have shifted significantly as the interlayer distance increased.

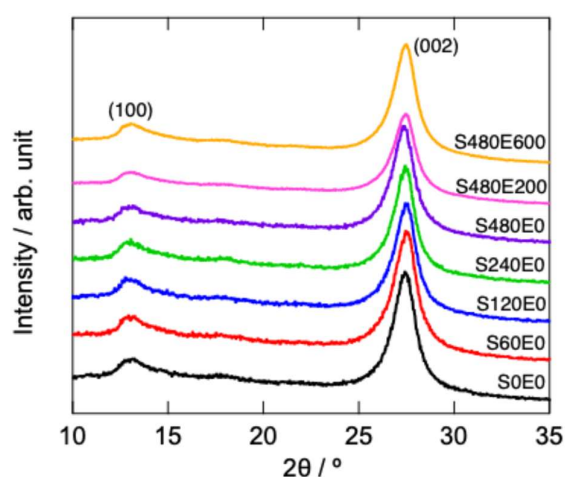


Figure 2. Powder XRD patterns of intact and sonicated g-C₃N₄ samples. Diffraction patterns of S480E200 and S480E600 are also shown.

The specific surface areas of the samples as functions of sonication time are shown in Figure 3. The specific surface areas tended to be small, even after sonication (approximately 10–20 m² g^{−1}). The specific surface areas of the EB-irradiated g-C₃N₄ were larger than those of the unirradiated g-C₃N₄ in all cases. In addition, the EB dose affected the specific surface area. Prolonged sonication increased the specific surface areas of the EB(200 kGy)-irradiated samples (S0E200–S480E200). In contrast, the specific surface areas of the EB(600 kGy)-irradiated samples (S0600–S480E600) exhibited marked fluctuations depending on the sonication time. A similar tendency was observed in our previous work [23]; the moderate structural change that occurred through low-dose EB irradiation contributed to an increase in the surface area, whereas excess EB irradiation led to more destructive structural changes and a resultant decrease in the specific surface area.

The fluctuation in the specific surface area in the case of EB irradiation at 600 kGy can be understood based on the coarsening of the relatively small particles caused by EB-induced electrification. Small g-C₃N₄ fragments formed through prolonged sonication aggregated significantly by EB-induced electrification compared to moderately sonicated g-C₃N₄. The EB-induced electrification of materials has been previously reported [34,35]. Oxidation by EB causing electron detachment from metal cations has also been reported for Co₃O₄ (the transformation of Co²⁺ to Co³⁺) [36]. Ionization is a typical initial process that occurs via radiation; thus, electron detachment from g-C₃N₄ is possible through EB irradiation. If g-C₃N₄ is electrified by EB-induced charging, the g-C₃N₄ particles could aggregate via electrostatic interactions, causing coarsening. However, the

understanding of the impact of ionizing radiation on the physicochemical properties of g-C₃N₄ remains insufficient, and further investigation of the irradiation effect is necessary.

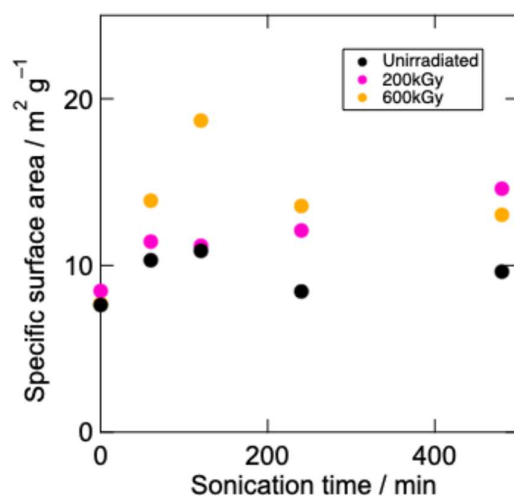


Figure 3. Specific surface area of unirradiated and EB-irradiated g-C₃N₄ as a function of sonication time.

Figure 4 shows typical C 1s and N 1s high-resolution XPS spectra of the EB-irradiated samples after sonication for 60 min. The C 1s spectra reveal that four types of C exist in the samples [23,37]: unexpectedly formed C–C + C=C; C–N possessing hydrogen-containing amino groups; (N)₂–C=N contained in triazine rings; and other species, including oxidized C (the XPS survey also detected a small amount of O). Although the peaks corresponding to defect moieties generated through EB-induced N elimination are not specifically identified, the species exhibiting C–NH/C–NH₂ peaks (denoted as “C–NH/C–NH₂ + defect C” in Figure 5(A, C)) may include also defect carbons. The N 1s spectra also exhibited features similar to those of intact and EB-irradiated g-C₃N₄ without sonication. They consist of three types of N atoms: pyridinic C–N=C, N–(C)₃ connecting three carbon atoms, and hydrogen-containing amino groups. Although charge-up-induced peak shifts were observed, the typical features of the C 1s and N 1s spectra were consistent with previously reported results.

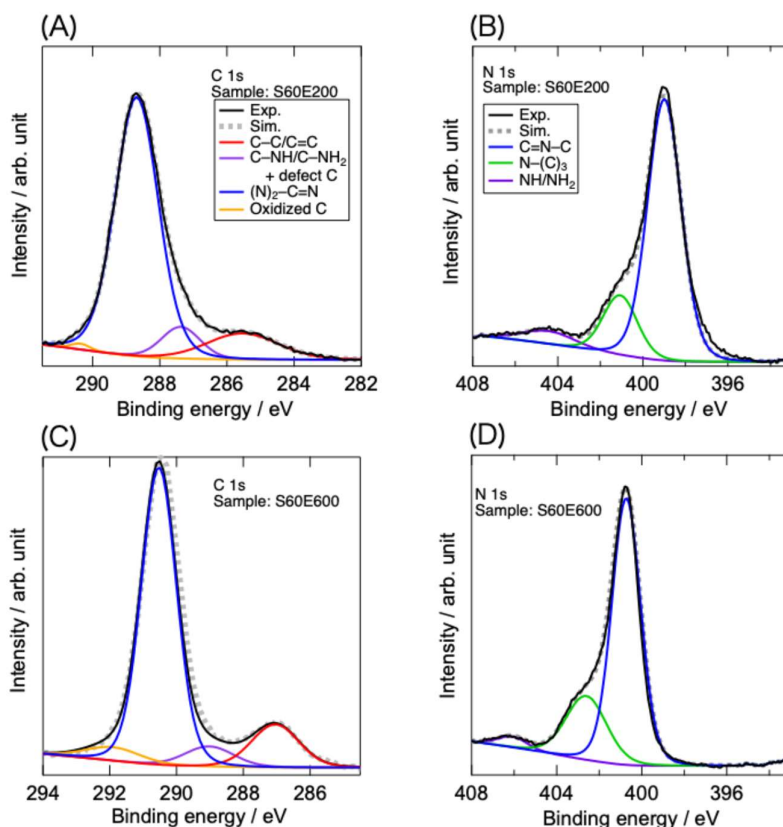


Figure 4. Typical high-resolution XPS of g-C₃N₄ after sonication for 60 min and subsequent EB irradiation: C 1s spectra (A, C) and N 1s spectra (B, D).

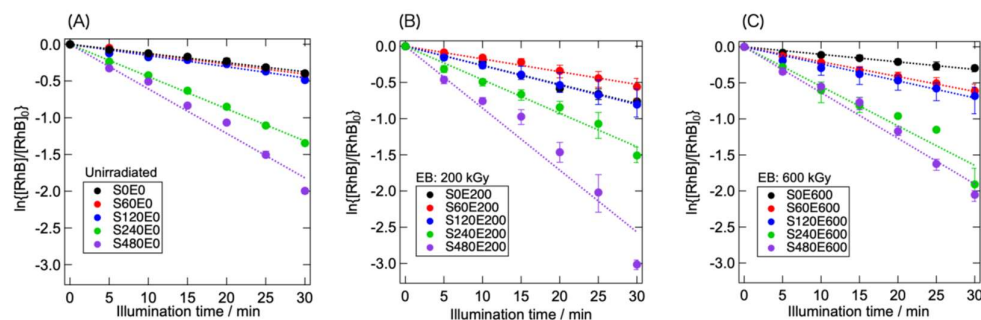


Figure 5. Variation of RhB concentration measured using unirradiated g-C₃N₄ (A) and EB-irradiated g-C₃N₄ (B, C) as a function of illumination time.

EB irradiation altered the composition of C and N in the samples, as previously reported [23], whereas sonication did not cause obvious changes. Similarly, the abundance ratios of C and N with different chemical bonding states were altered by EB irradiation but were not significantly affected by sonication. Previous results have revealed that pyridinic N is the predominant species eliminated upon EB irradiation, and that N vacancies and C defects form through N elimination [19–23]. This structural alteration could be related to the improvement in the photocatalytic efficiency. In the present study, the change in the chemical structure of g-C₃N₄ was mainly caused by EB irradiation, and the impact of sonication on the chemical structure was minor.

3.2. Photocatalytic RhB Decomposition Using Treated g-C₃N₄

Figure 5 shows the RhB concentration as a function of the illumination time under visible light. The effects of sonication and EB irradiation on the rate of photocatalytic RhB decomposition can be observed. A comparison of the RhB decomposition rates of S0E0, S0E200, and S0E600 revealed that S0E200 exhibited the highest reaction rate. This difference in the reaction rate has already been reported in an earlier study [23], where moderate structural modification with low-dose EB irradiation improved the photocatalytic reaction efficiency. The preceding sonication altered the reaction rate depending on sonication time. Sonication for up to 120 min had a relatively minor effect on the photocatalytic efficiency, but prolonged sonication markedly accelerated the photocatalytic RhB decomposition.

Comparing S480E0, S480E200, and S480E600, the influence of EB irradiation on the reaction efficiency was obvious. S480E200 exhibited the highest reaction efficiency, indicating that low-dose EB irradiation effectively improves the photocatalytic reaction efficiency of sonicated g-C₃N₄. In contrast, the reaction efficiency of S480E600 was similar to that of S480E0 despite its larger specific surface area (Figure 3). Notably, although S120E600 had the highest specific surface area among the samples examined, it exhibited a relatively low reaction efficiency. This suggests that the combined effect of EB-induced structural changes and surface area alteration through sonication dominated the reaction efficiency. The structural and textural changes induced by low-dose EB irradiation had a positive effect. However, the positive effect of EB irradiation at a higher dose (600 kGy) was limited; under these conditions, electrostatic particle coarsening and EB-induced destructive structural alteration influenced the reaction efficiency as much as did the change in textural properties (increase in the specific surface area).

4. Conclusions

In this study, we investigated the combined effects of EB irradiation and sonication on the photocatalytic efficiency of g-C₃N₄. Prolonged sonication before EB irradiation improved the g-C₃N₄ reaction efficiency. The effect of the subsequent EB irradiation on the photocatalytic efficiency depended on the EB dose and is summarized as follows:

- Low-dose EB irradiation further improved the reaction efficiency of sonicated g-C₃N₄, as it does that of g-C₃N₄ that is not sonicated.
- Higher-dose EB irradiation did not markedly increase the reaction efficiency compared to that of unirradiated g-C₃N₄ despite its larger specific surface area.

Therefore, the photocatalytic efficiency can be determined based on the complex counterbalance between the EB- and ultrasonic-induced textural and structural alterations. Sonication and high-dose EB irradiation likely caused opposite changes in the textural features, that is, moderate particle refining and electrostatic coarsening, and led to variations in the specific surface area. Additionally, as previously reported, high-dose EB irradiation results in destructive structural alterations through N elimination, decreasing photocatalytic efficiency.

The aforementioned chemical radiation process and its effects on g-C₃N₄ have not been thoroughly elucidated. The primary reaction induced by ionizing radiation is electron detachment to form positively charged species, and EB-induced attractive interparticle interaction (electrification followed by particle coarsening) explains the change in the specific surface area. However, the related processes that take place in g-C₃N₄ remain unclear. To clarify the effects of irradiation on the textural features of g-C₃N₄, further study is needed on this radiation-chemical process.

Author Contributions: Conceptualization, T.T.; methodology, A.H. and T.T.; validation, A.H. and T.T.; investigation, A.H., S.S., K.S., and T.T.; resources, A.F. and T.T.; data curation, A.H. and T.T.; writing—original draft preparation, T.T.; writing—review and editing, A.H. and A.F.; visualization, A.H. and T.T.; supervision, A.F. and T.T.; project administration, T.T.; funding acquisition, A.F. and T.T. All authors have read and approved the published version of the manuscript.

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Conflicts of Interest: The authors declare no conflicts of interest.

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