



Synthesis and characterization of AgCl@graphitic carbon nitride hybrid materials for the photocatalytic degradation of atrazine

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Abstract

Mesoporous graphitic carbon nitride (pg-C₃N₄) was prepared via pyrolysis of dicyandiamide and urea in an air atmosphere. AgCl@pg-C₃N₄ nanocomposites were synthesized using a modified precipitation technique. pg-C₃N₄ and AgCl@pg-C₃N₄ nanocomposites were characterized using XRD, BET, UV–Vis spectroscopy, PL, TEM, and XPS techniques. The results reveal that AgCl was well dispersed on the surface of the pg-C₃N₄. The photocatalytic degradation of atrazine under visible light irradiation was used to measure the photocatalytic performance of the pg-C₃N₄ and AgCl@pg-C₃N₄ nanocomposites. The photocatalytic activity of the AgCl@pg-C₃N₄ nanocomposite has higher photocatalytic activity than the pg-C₃N₄ nanoparticles. The reason for the high photocatalytic activity of the AgCl@pg-C₃N₄ nanocomposite may be due to the synergic effect between AgCl and pg-C₃N₄. The photocatalytic degradation of atrazine follows a first order reaction. © 2014 Elsevier Ltd and Techna Group S.r.l. All rights reserved.

Keywords: Mesoporous graphitic carbon nitride; AgCl; Visible photocatalyst; Degradation of atrazine

1. Introduction

The widespread use of pesticides has caused many problems. For example, atrazine and amitrole have been indicated as thyroid gland disruptors, endocrine disruptors, and possible carcinogens [1–4]. Atrazine and amitrole have been detected in groundwater and drinking water supplies [5–7]. Removal of these two pesticides must be performed using nonconventional methods due to their high solubilities in water [1,6,8]. Electro-Fenton and activated carbon methods have been used to remove amitrole [9,10]. A nanofiltration method was used for the removal of atrazine [11]. From an economic point of view, this approach is not a commercially viable method. However, a photocatalytic method is a suitable method for the removal of pesticides from an economic and environmental point of view.

TiO₂, WO₃, and ZnO have wide band gaps, so they can be used for environmental protection and clean energy under UV irradiation [12–14]. A variety of visible-light-driven photocatalysts have been developed to improve their absorption from the UV to the visible regions [15]. Two methods have been applied to improve the photocatalytic activity from the UV to the visible region. The first method is doping cations or anions [16], controlling the shape or structure of the photocatalyst [17], designing semiconductor composites, and noble metal loading [18–21]. The other method is developing novel, sunlight-based photocatalysts, such as BiVO₄ [22] and BiOX (X=Cl, Br, I) [23–25]. These developed, novel, sunlight-based photocatalysts are still composed of metal-based inorganic solids, and they must have sufficient photocatalytic stability and suitable band edge positions. Recently, a polymer-like semiconductor material (g-C₃N₄) has been used as an efficient photocatalyst for hydrogen production and environment purification [26–30]. This polymer-like semiconductor material (g-C₃N₄) is a cheap, stable material that is abundant and has a controllable surface, so it can be treated as a new material for environmental applications and solar

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energy. However, there are many drawbacks for the use of $g\text{-C}_3\text{N}_4$ in the photocatalysis field, such as a low surface area, high $e\text{-}h$ recombination rate, and lack of absorption above 460 nm. Therefore, the photocatalytic activity of $g\text{-C}_3\text{N}_4$ must be improved. Researchers found that three strategies seem to be very efficient. The first one is the enhancement of photocatalytic activity and adsorption ability by controlling the textural structure of $g\text{-C}_3\text{N}_4$ [31–38]. The second one is the chemical doping of anions or cations, which changes the electronic structure of $pg\text{-C}_3\text{N}_4$ and extends the absorption range [39–44]. The third one is a coupling of $pg\text{-C}_3\text{N}_4$ with another photocatalyst, such as TiO_2 [45], TaON [46], Bi_2WO_6 [47], etc. To the best of our knowledge, there have been no reports on the photocatalytic degradation of atrazine by $\text{AgCl}@pg\text{-C}_3\text{N}_4$ under visible light. In this work, a novel $\text{AgCl}@pg\text{-C}_3\text{N}_4$ photocatalyst was prepared and characterized. Visible photocatalytic degradation of atrazine using the $\text{AgCl}@pg\text{-C}_3\text{N}_4$ photocatalyst was studied.

2. Experimental

2.1. Preparation of mesoporous graphite-like C_3N_4 photocatalysts

All reagents that were used in this research were pure and used without any further purification. Urea and dicyandiamide were purchased from Sigma-Aldrich. The preparation method depended on the pyrolysis of dicyandiamide and urea in an atmosphere of air. The details of the preparation method were as follows: adding 5 g of urea to 3 g of dicyandiamide, mixing them carefully, and heating the mixture using a Muffle Furnace at 530 °C for 4 h. The resulting yellow, solid materials were washed by HNO_3 (0.1 mol) and deionized water to remove any residue adsorbed on the surface of the mesoporous graphite-like C_3N_4 photocatalyst. Finally, the resulting pure materials were dried for 12 h at 100 °C.

2.2. Synthesis of $\text{AgCl}@pg\text{-C}_3\text{N}_4$ nanocomposites

$\text{AgCl}@pg\text{-C}_3\text{N}_4$ nanocomposites were synthesized using a modified precipitation technique, which involved the dispersion of the as-synthesized $pg\text{-C}_3\text{N}_4$ in 30 ml of distilled water using sonication for 30 min, adding 0.6 g of silver nitrate to an aqueous solution of $pg\text{-C}_3\text{N}_4$ dropwise, and stirring the resulting mixture for 3 h at room temperature. Finally, an aqueous solution of KCl was added to the above mixture to maintain a molar ratio of 1 to 1 for Ag^+ to Cl^- . The resulting mixture was stirred for 24 h at room temperature. The mixture was filtrated and washed many times with distilled water and ethanol, then dried under vacuum for 12 h at 80 °C. A series of $\text{AgCl}@pg\text{-C}_3\text{N}_4$ nanocomposites with different weight percentages of AgCl were prepared and labeled as $\text{AgX}@pg\text{-C}_3\text{N}_4\text{-wt}\%$, where wt% is the weight percentage of AgCl.

2.3. Photocatalyst characterization

A Bruker Axis D8 using Cu $K\alpha$ radiation ($\lambda = 1.540 \text{ \AA}$) was used to determine the X-ray diffraction patterns of the $pg\text{-C}_3\text{N}_4$ and $\text{AgCl}/pg\text{-C}_3\text{N}_4$ nanocomposites. A Nova 2000 series Chromatech apparatus was used to measure the BET surface area of the $pg\text{-C}_3\text{N}_4$ and $\text{AgCl}/pg\text{-C}_3\text{N}_4$ nanocomposites before the samples were heated under vacuum for 3 h at 200 °C. A UV/Vis/NIR spectrophotometer (V-570, JASCO, Japan) was used to measure the band gap energy of the $pg\text{-C}_3\text{N}_4$ and $\text{AgCl}/pg\text{-C}_3\text{N}_4$ nanocomposites. A JEOL-JEM-1230 microscope was used to determine the shape and particle size of the $pg\text{-C}_3\text{N}_4$ and $\text{AgCl}/pg\text{-C}_3\text{N}_4$ nanocomposites. Before measurement, the samples were dispersed in ethanol for 30 min. Then, a small portion of the samples were placed on a carbon-coated copper grid. A Thermo Scientific K-ALPHA, X-ray photoelectron spectroscopy (XPS), England, was used to determine the elemental nature of the sample. The photoluminescence emission spectra of the $pg\text{-C}_3\text{N}_4$ and $\text{AgCl}/pg\text{-C}_3\text{N}_4$ nanocomposites were measured using a Shimadzu RF-5301 fluorescence spectrophotometer.

2.4. Photocatalytic activity

The photocatalytic performance was measured under visible light irradiation via the photooxidation of atrazine. A 300-W Xenon lamp was used as the irradiation source, and it was completely covered with an optical cut-off filter to remove any light that has a wavelength below 420 nm. An aqueous solution of 100 ppm atrazine was kept in the dark for 30 min prior to the illumination step to ensure complete adsorption–desorption equilibrium was reached. The change in the concentration of atrazine was measured using high-pressure liquid chromatography (Shimadzu LC 20 A) with a C18 column UV detector. To confirm the complete oxidation of atrazine into carbon dioxide, chloride, and nitrate ions, the concentrations of chloride and nitrate ions were measured using ion chromatography (DX-300) with a CDM-II conductivity detector and an AS4A-SC column. To confirm the presence of carbon dioxide gas as one of the final products from the photocatalytic oxidation of atrazine, the gases that were obtained from the photocatalytic reaction were passed over a 0.2 M NaOH solution. Then, a barium nitrate solution was added to them, and the white precipitate produced was analyzed using XRD.

3. Results and discussion

3.1. Characterization of $pg\text{-C}_3\text{N}_4$ and $\text{AgCl}/pg\text{-C}_3\text{N}_4$ nanocomposites

Fig. 1 shows the XRD patterns of the $pg\text{-C}_3\text{N}_4$ and $\text{AgCl}/pg\text{-C}_3\text{N}_4$ nanocomposites with different AgCl weight percentages. The results reveal that the characteristic peak for $pg\text{-C}_3\text{N}_4$ appeared in all samples. There is no peak for AgCl due to the low AgCl content or good dispersion of AgCl on the surface of $g\text{-C}_3\text{N}_4$. Additionally, increasing the weight percentage

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