Photocatalysis for Water Purification

Subjects: Materials Science, Composites

Contributor: Hartini Ahmad Rafaie, Nurul Infaza Talalah Ramli, Zuraida Khusaimi, Mohd Arif Mohd Sarjidan, Paweena Dulyaseree, Zul Adlan Mohd Hir

Endocrine-disrupting chemicals (EDCs) in the aquatic environment have garnered a lot of attention during the past few years. Due to their toxic behavior, which interferes with endocrine functions in both humans and aquatic species, these types of compounds have been recognized as major polluting agents in wastewater effluents. Therefore, the development of efficient and sustainable removal methods for these emerging contaminants is essential. Photocatalytic removal of emerging contaminants using silver carbonate (Ag_2CO_3)-based photocatalyst is a promising process due to the unique characteristics of this catalyst, such as absorption of a larger fraction of the solar spectrum, wide band gap, non-toxicity, and low cost. The photocatalytic performance of Ag_2CO_3 has recently been improved through the doping of elements and optimization variation of operational parameters resulting in decreasing the rate of electron–hole pair recombination and an increase in the semiconductor's excitation state efficiency, which enables the degradation of contaminants under UV or visible light exposure. This entry summarized some of the relevant investigations related to Ag_2CO_3 -based photocatalytic materials in the water recovery procedure suggests that the creation of a cutting-edge protocol is essential for successfully eliminating EDCs from the ecosystem.

Keywords: Ag2CO3 ; degradation ; endocrine disrupting compounds ; photocatalysis

1. Endocrine-Disrupting Chemicals (EDCs) Classification

EDCs are a kind of environmental pollutant that relate to the mixture of chemical agents that interfere with the human body's endocrine system; for instance, metabolic, immune systems, hormones, etc. As the EDC disruptors come from a variety of sources, people and aquatic life are regularly exposed through the air, food, and water. Additionally, EDCs can also enter the human body through the skin. Based on the characteristic functional groups in the target EDC substrates and their application fields, EDCs can be classified into a few types: (1) pesticides and herbicides (aromatic rings and heterocyclic rings containing chlorine substituents) ^{[1][2]}, (2) hormones and steroids containing phenolic hydroxyl/carbonyl groups (i.e., estron, 17 β -estradiol, estriol, and 17 α -ethyl estradiol ^[3]), (3) pharmaceuticals and personal care product ^{[4][5]}, (4) plasticizers (bisphenols, polystyrene (PS) and phenol-formaldehyde resin (PF) ^{[G][7]}), and (5) other organic pollutants (**Figure 1**).



Figure 1. Classification of EDCs.

The existence of EDCs in aquatic systems such as drinking water, wastewater, and underground water is a pressing environmental concern. It is suspected that a range of natural chemicals and man-made substances may interfere with the endocrine systems of both humans and wildlife, with varying sources of exposure and chemical properties. The proliferation of synthetic EDCs in the environment has added to the already vast array of chemicals that have been discharged worldwide, resulting in a more complex situation ^[8]. These compounds, which can be either natural or synthetic, are discharged into aquatic environments by multiple sources, including industrial discharge or drain water, domestic sewage effluents, livestock waste, and landfill leachates ^[9]. While the active components of some pharmaceuticals, cosmetics, and pesticides are crucial to identify the chemical constituents present in the environment, particularly those that are highly water-soluble. Generally, wastewater treatment is the largest contributor to EDC contamination, particularly in the form of untreated and treated sewage effluents and direct discharge to rivers.

In recent times, an increasing number of studies have drawn attention to the diverse health and environmental implications associated with endocrine-disrupting compounds. These compounds are known to interfere with the endocrine systems of humans and animals and can impact hormone synthesis, release, transport, metabolism, and excretion within the body ^[10]. For instance, the extensive use of pharmaceuticals and personal care products (PPCPs) in daily life has led to the presence of these compounds in rivers, groundwater, and even drinking water ^[4]. The occurrence of such pollutants in water resources can also be attributed to agricultural practices, accidental spills, leaching from building materials, disposal of prescription drugs, and chemical runoff from wastewater treatment plants, among other factors, aside from natural events. Moreover, phenolic compounds can be detected in water bodies due to various sources such as agricultural and industrial activities, synthetic resin and plastic waste, food packaging, paper coating, surfactants, adhesives, flame retardants, and dyestuffs ^[11]. These compounds can undergo a gradual biochemical transformation through microbial processes in the water stream, potentially leading to the formation of additional toxic byproducts. **Table 1** presents a concise summary of the reported harmful effects of EDC contaminants from various sources.

Potential EDCs	Sources of EDCs	Harmful Effect	Ref.
Pharmaceutical and personal care products (PPCPs)		• Display the potential to cause serious damage to the aquatic environment; for instance, endocrine conduit disturbance, brain injury, and carcinogenic diseases	
	antibiotics, analgesics, beta-blockers, lipid regulators, anti-inflammatory drugs, X-ray contrast media, and estrogens	 brain injury, and carcinogenic diseases Reproductive systems problem: polycystic ovarian syndrome (PCOS), prostate cancer, infertility Reproductive disorders, obesity, and postnatal immune disorders 	[<u>12]</u> [<u>13]</u>
		Reproductive disorders, obesity, and postnatal immune disorders	
		Obstructing human and wildlife immune systems	
Hormones and steroids	estrogens, androgens, glucocorticoids, and mineralocorticoids	• Possess immunotoxin effects including immunosuppressive properties as well as the possibility to increase autoimmune reactions, Myelotoxicity, stimulation of the reticuloendothelial system, depression of cell- mediated immunity, and natural killer cell activity	[14]

Table 1. Sources and harmful effects of endocrin	ne disrupting chemicals (EDCs)
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Potential EDCs	Sources of EDCs	Harmful Effect	Ref.	
Plasticizers and additive		Reduce the fertilization rate		
	polychlorinated biphenyls (PCBs), Phthalates (DEP, DEHP, DBP), Bisphenol A, Polyfluoroalkyl compound (PFOS, PFOA)	Increase the risk of miscarriage		
		Obesity and insulin resistance		
		Diabetes and liver abnormalities	[<u>9]</u> [15]	
		Increased the level of cholesterol		
		 Negative health effects mostly on male species, including decrease in sperm counts, toxicity to testes, prostate, and seminal vesicle 		
Pesticides and herbicides		 Obstructing the natural hormones due to their aptitude in binding with estrogen or androgen receptors. 		
		 Affect spermatogenesis leading to poor sperm quality, hence decreased male fertility 		
	polychlorinated biphenyls (PCBs), Dichlorodiphenyltrichloroethane (DDE), Organochlorines (DDT)	Increase the risk of breast cancer	[<u>9]</u> [16]	
	• Infl inve ma • Me	Influence the reproductive function of		
		invertebrates, reptiles, fish, birds, and mammals		
		Metabolic syndrome and diabetes		

2. EDC Removal from Water by Semiconductor-Based Photocatalytic Materials

The performance and stability of photocatalytic materials based on semiconductor-based materials have emerged as a critical issue in oxidative environments, particularly when exposed to light energy (photons), as they are utilized for combating endocrine-disrupting chemicals (EDCs). Recently, researchers have been focusing on enhancing the performance of semiconductor-based photocatalysts in degrading endocrine-disrupting chemicals (EDCs) by designing hybrid or ternary structures or manipulating the surface compositions and structures of photocatalysts.

TiO₂ and ZnO are considered the primary contenders for an outstanding semiconductor photocatalyst that can effectively eliminate low concentrations of toxic organic substances. This is primarily due to its non-toxic nature, cost-effectiveness, high activity, photostability, and commercial viability. However, TiO₂ and ZnO require UV light to initiate the photocatalytic process due to its bandgap energy, which only allows it to utilize around 3-5% of the solar radiation that reaches the earth's surface [17][18]. To overcome this limitation, researchers have been focusing on modifying these photocatalysts to reduce their bandgap energy, which would allow them to harvest visible light. Consequently, there have been several studies investigating modified TiO₂ and ZnO materials for EDC removal in aqueous media. For instance, Hayati et al. prepared a ZnO@AC@Fe₃O₄ composite mediated with peroxymonosulfate (PMS) for boosting the degradation efficiency of BP-A under UVC irradiation. The photocatalytic activity of ZnO@AC@Fe₃O₄ mediated with PMS produced a higher degradation rate of 95.6% BPA, and about 58.9% of TOCs were eliminated within 60 min of the reaction. It was proposed that the synergistic interaction between PMS-based oxidation photocatalysis contributed toward BPA removal in the composite process ^[19]. The optimization of several morphological and structural properties for improved photocatalytic performance requires the surface modification of TiO₂ nanoparticles (NPs). Batista et al. investigated the degradation of acetaminophen (APP) in the aqueous medium using magnetic photocatalysts based on carbon xerogel/TiO₂ composites. This composition showed a maximum degradation efficiency of 99.2 of APP, respectively ^[20]. The ability of carbonaceous materials to act as photosensitizers, coupled with their large surface area and favorable mechanical and electronic properties, has piqued the interest of researchers, leading to a surge in their functionalization [20]. In another study,

Silvestri et al. synthesized a combination of TiO₂ and Polypyrrole (PPy) for the degradation of pesticide 4-CP and pharmaceutical compound DCF. The performance of combination photocatalysts showed that the PPy-TiO₂ was able to convert more than 90% of DCF and 40% of 4-CP in just 60 min compared to TiO₂ alone. They postulated that the introduction of polymers constituent to TiO₂ facilitates the transfer of charges and prevents electron-hole recombination, hence improving efficiency $\frac{21}{2}$. While Monfared et al. reported that the combination of nano TiO₂ and the ability of polyaniline to excite the electrons under visible light (Eg = 2.8 eV) caused the formation of a nanocomposite with significantly better photocatalytic behavior especially under visible light [22]. Anjum et al. also reported an enhancement of photocatalytic degradation when combining ZnO-ZnS with polyaniline (PANI) [23]. This combination of semiconductor materials and conducting polymer provides a synergistic behavior between the polymer and catalyst and reduces the bandgap energy in the range of visible light, which was also agreed and reported by Thakare et al. [24]. Meanwhile, Baishnisha et al. prepared graphitic carbon nitride (g-CN)/CuO nanocomposite for the photocatalytic degradation of phenol under visible light irradiation [25]. The combination of g-CN/CuO resulted in high degradation efficiency (87.8%) within 120 min. They reported that the formation of heterojunction by combining other semiconductor or carbon materials is a recognized technique to promote the separation of photogenerated electron-hole pairs which exhibit enhanced photocatalytic performance and a high efficiency of utilizing solar energy. This also has been supported by Kumar et al., combining ZnO/SnO₂ with reduced graphene oxide (RGO) toward the elimination of different organic pollutants viz. pbromophenol, bisphenol A, and ofloxacin from water [26]. The nanocomposite containing 5 wt% reduced graphene oxide exhibited a maximum photocatalytic efficiency of approximately 98.64% and 98.50% for removing p-bromophenol and bisphenol A, respectively, when exposed to UV light for 180 min. Additionally, this same level of efficiency was observed for eliminating ofloxacin after 120 min of UV light exposure.

2.1. Silver Carbonate (Ag₂CO₃) Photocatalyst

In recent times, the excellent photocatalytic performance of silver-based semiconductors has led to their increased research attention. Notably, Ag_2CO_3 has emerged as a highly efficient material for degrading EDCs in wastewater due to its exceptional photocatalytic capabilities. Moreover, as Ag-based compounds, they are well known for antibacterial activity. Ag_2CO_3 , as a highly active visible-light-driven photocatalyst, has been recognized as a good photosensitizer since its narrow band gap (~2.17 V) is advantageous for sunlight absorption ^[27].

Table 2 presents recent research on the application of composite catalysts, including ternary and guaternary composites, under visible/solar light irradiation for the degradation of various pollutants. The degradation efficiency is dependent on factors such as the type and concentration of the pollutant, the amount of catalyst used, and the light source employed. Notably, the use of Ag₂CO₃-based catalysts for the degradation of EDCs under light irradiation has been a recent area of focus in these studies. Mergenbayeva et al. prepared Ag₂CO₃ microparticle photocatalysts for 4-t-BP degradation under solar light irradiation $\frac{[28]}{2}$. After 60 min, the complete degradation of 4-t-BP (5 ppm) was accomplished using Ag₂CO₃ (200 mg/L). They postulated that increasing the amount of the catalyst from 100 mg/L to 300 mg/L resulted in an increase in the final degradation efficiency from 41.6% to 100%. Similarly, Petala et al. observed that increasing Ag₂CO₃ photocatalyst concentration from 250 mg/L to 1000 mg/L led to an increase in ethyl paraben (EP) removal, while increasing EP concentration from 0.25 mg/L to 1.00 mg/L slightly lowered the photodegradation rate constant, kapp from 0.115 min⁻¹ to 0.085 min⁻¹ [29]. Rosman et al. investigated Ag₂CO₃/Ag₂O heterostructure over a ZnO photocatalyst, and results exhibited a 99.3% photodegradation of ibuprofen (IBF) solution under visible-light irradiation [30]. Accordingly, the effective interfacial charge transfers observed in the ZnO-Ag₂CO₃-Ag₂O sample were facilitated by the compact interfacial heterojunction. This was attributed to the synergistic effect resulting from the Ag₂CO₃/Ag₂O heterostructure. Another study conducted by Rosman and her colleagues worked on Ag₂CO₃-based photocatalytic membrane combined with poly (vinylidene fluoride) (PVDF) acting as ultrafiltration for degradation similar to the source of EDC, which is ibuforen classified under pharmaceutically active compounds (PhACs) [31]. This hybrid photocatalytic membrane resulted in a removal of 35.27% IBF under visible light irradiation. The use of Ag₂CO₃ as a photocatalyst is not restricted to foreign materials; it also involves incorporating polymer materials into the design of the photocatalyst to enhance its photocatalytic performance toward EDC removal [32]. Pirzada et al. conducted a study where they synthesized a heterostructure of Ag₂CO₃ with a perovskite metal ferrite to improve the stability and photocatalytic efficiency through the strong surface plasmon resonance effect of Ag nanoparticles on the surface [33]. They successfully achieved a degradation efficiency of approximately 59% for p-chlorophenol using a novel LaFeO₃/Ag₂CO₃ heterostructure photocatalyst under natural sunlight within 45 min. Notably, the nanocomposite significantly improved the stability of Ag₂CO₃, and no catalyst decomposition was observed even after multiple photocatalytic cycles.

Table 2. Photocatalytic degradation of EDCs of over-selected semiconductor-based materials from aqueous solution.

Semiconductor-Based Photocatalyst	EDCs Target Compounds	Light Source/Experimental Condition	Photodegradation Efficiency (%)	Ref.
ZnO@AC@FeO	bisphenol A	UV lamp (UV–C irradiation, λ = 254 nm, OSRAM 8 W 60 min irradiation time	95.6%	[<u>19]</u>
Carbon xerogel/TiO ₂ composites	acetaminophen	125 W mercury lamp, with a power density of 80 w/cm ² to 300 w/cm ² and a wavelength in the range of 266–578 nm ³ h irradiation time	99.2%	[<u>20]</u>
Polypyrrole-TiO ₂ composite	pesticide 4-CP and diclofenac	Xenon lamp (600 Wm ⁻²) 60 min irradiation time	90% of DCF and 40% of 4-CP	[<u>21</u>]
Polyaniline/titanium dioxide nanocomposite (PAni/TiO ₂)	benzene	5 (UV-C) lamps (8 W) with wavelength of 265 nm and 11 visible light lamps (8 W) 70 min irradiation time	50% under UV light 23% under visible light	[22]
ZnO-ZnS@polyaniline nanohybrid	2-chlorophenol	cool white visible light lamps (Sylvania s068) of total 104 W 240 min irradiation time	88%	[23]
Graphene-TiO ₂ - polyaniline nanocomposite	4-nitrophenol	Visible light 5 min irradiation time	100%	[<u>24]</u>
ZnO/SnO ₂ Z-scheme heterojunctions	p-bromophenol and bisphenol A	UV light (30 W, 365 nm) 180 min irradiation time	98.64% (p- bromophenol) and 98.50% (bisphenol A)	[26]
Ag ₂ CO ₃ microparticles	4-tert-butylphenol	Simulated solar 60 min irradiation time	100%	[28]
Ag ₂ CO ₃	ethyl paraben (EP)	solar simulator (Oriel, model LCS-100, Newport, Irvine, CA, USA) equipped with a 100 W xenon ozone-free lamp) 30 min irradiation time	90%	[<u>29]</u>
ZnO/Ag ₂ CO ₃ /Ag ₂ O	ibuprofen	Visible (a white light-emitting diode (LED) spotlight (>420 nm, 100 W) 480 min irradiation time	99.3%	[<u>30]</u>
PVDF-ZnO/Ag ₂ CO ₃ /Ag ₂ O nanocomposite membrane	ibuprofen	Visible (Light-emitting diode lamp, k = 420 nm, 100 W)	35.27%	[<u>31]</u>
LaFeO ₃ /Ag ₂ CO ₃ nanocomposites	p-chlorophenol	Visible 450 W xenon lamp operated at 400 W with 395 nm filter) 45 min irradiation time	59%	[33]

2.2. Synthesis Techniques of Ag₂CO₃-Based Photocatalyst

Today, there are several types of reported Ag-based semiconductors that have been reported, drawing attention toward the elimination of EDCs in wastewater. Basically, all Ag-based semiconductors are responsive in the visible light irradiation due to their small band gap energy (<3.0 eV). Moreover, it has been found as a talented photocatalyst for the elimination of organic contamination due to nontoxicity and unique structural properties. More recently, Ag₂CO₃ has been considered one of the Ag-based photocatalysts with the distinct advantage of being driven by visible light absorption and relatively narrow band gap (~2.1–2.7 eV) ^{[27][34]}. Nevertheless, Ag₂CO₃ alone suffers from the problem of photocorrosion during the photocatalytic reaction ^{[35][36]}. Therefore, various strategies have been researched and implemented to improve its photocatalytic activity and stability. Ag₂CO₃ has been designed and constructed by a heterojunction photocatalyst via coupling with another semiconductor or carbon element to enhance the separation of photogenerated electron–hole pairs to prevent the occurrence of photochemical corrosion ^[37]. Moreover, it should be noted that the photocatalytic performance of semiconductors is affected not only by their band gap, but also by their physicochemical properties such as crystal structure, morphology, and crystallinity. Due to their strong visible light absorption, great efforts have been devoted to the design, preparation, and characterization of Ag-based photocatalysts.

There are various approaches that have been conducted in synthesizing and designing Ag_2CO_3 -based photocatalysts. This synthesis method can be categorized into solution-based and vapor-based approaches. Physicochemical properties such as crystal structure, size, and morphology of photocatalysts are significantly influenced by the preparation method,

and this also results in the changes of charge career separation and band gap energy of the material. This implies that the good control of preparation conditions determines the efficiency of photocatalysis. A solution-based approach is the simplest and least energy consuming. Therefore, an extensive approach has been developed by researchers; for instance, co-precipitation, sol–gel, and the solid-state reaction method enhance the physicochemical properties of the photocatalyst, hence improving the photocatalytic reaction of EDC removal in aqueous media.

The co-precipitation method is one of the significant and familiar solution-based approaches for developing Ag₂CO₃-based photocatalyst. Yu et al. developed a series of Ag₂S/Ag₂CO₃ composite photocatalysts at different compositions (Ag₂S, Ag₂CO₃, 4%Ag₂S-Ag₂CO₃, 8%Ag₂S-Ag₂CO₃, 16%Ag₂S-Ag₂CO₃, 32%Ag₂S-Ag₂CO₃ and 40%Ag₂S-Ag₂CO₃ using the coprecipitation method ^[38]. This approach includes three steps: (1) mixing of Na₂CO₃ and Na₂S·9H₂O in deionized water followed by adding AgNO₃ dropwise, (2) stirring for 1 h, and (3) the drying and calcination of the precipitation. Moreover, a successive precipitation route has been introduced to prepare the core-shell-like composite of Ag₂CO₃@Ag₂S and Ag₂S@Ag₂CO₃ composite photocatalysts with 32 wt% Ag₂S. They reported that the coupling of Ag₂S into Ag₂CO₃ could produce a well-contacted Ag₂S/Ag₂CO₃ interface as represented in TEM results, which largely enhanced the photocatalytic activity and stability in decomposing methyl orange and phenol.

Another study conducted by Rui Zhang and co-workers also involved the synthesizing of Ag₂CO₃-based semiconductor photocatalysts via the co-precipitation method ^[39]. They have designed a Z-scheme heterostructure of $ZnFe_2O_4/Ag_2CO_3$ with an introduction of polyaniline (PANI) as a surface stabilizer to enhance the photocatalytic ability of the composite photocatalyst. For the first step, the $ZnFe_2O_4$ was prepared by mixing the $Fe(NO_3)_3 \cdot 9H_2O$ and $Zn(NO_3)_2 \cdot 6H_2O$ as a starting material in deionized water followed by hydrothermal and, finally, the drying process. The second step of the preparation of $ZnFe_2O_4/PANI/Ag_2CO_3$ was performed by mixing the $ZnFe_2O_4$, PANI, and AgNO_3 and undergoing an ultrasonic treatment under dark conditions; finally, the product was dried. They observed that $ZnFe_2O_4/PANI/Ag_2CO_3$, Ag_2CO_3 presents a regular rod-like structure, indicating that the addition of $ZnFe_2O_4$ and PANI has a great influence on the crystal growth of Ag_2CO_3 . For photocatalytic activity, they found that the introduction of PANI and $ZnFe_2O_4$ improved the separation efficiency of photogenerated carriers of Ag_2CO_3 .

Jindou Hu and colleagues constructed the Ag_2O/Ag_2CO_3 heterojunction by adjusting the alkalinity of a room temperature solid-state chemical reaction system ^[39]. A simple room temperature solid-state (RTSS) chemical reaction through changing the molar ratios of the starting materials, which are $AgNO_3$ and Na_2CO_3 , was introduced. It is a simple process that starts with the grinding of raw material and ends with a drying process. They observed that the molar weight of Na_2CO_3 is important in the formation of Ag_2O/Ag_2CO_3 composites, and the respective contents of Ag_2O and Ag_2CO_3 in the composites can be adjusted by the addition of an amount of Na_2CO_3 resulting in the change of physicochemical and photocatalytic properties of the prepared samples. **Table 3** represents a summary of previous research on the photocatalytic degradation of EDCs by Ag_2CO_3 -based photocatalysts that were prepared using different approaches.

Photocatalyst	Synthesis Technique	EDC Target Compounds	Light Source	Photodegradation Performance	Ref.
Ag ₂ S/Ag ₂ CO ₃ composite	Coprecipitation method	Phenol and Bisphenol A	300 W tungsten halogen lamp (Visible)	The highest photocatalytic activity (reaching 54% phenol degradation in 150 min) by Ag ₂ S@Ag ₂ CO ₃ photocatalysts 32%Ag ₂ S@Ag ₂ CO ₃ displayed higher decomposition rate than Ag ₂ CO ₃ for degradation of bisphenol A	[38]
LaFeO ₃ /Ag ₂ CO ₃ hetero-structure	co-precipitation method	p- chlorophenol	natural sunlight	LaFeO ₃ /Ag ₂ CO ₃ exhibited the highest photocatalytic activity with percentage degradation of 59% for p-chlorophenol within 45 min under natural sunlight irradiation.	[33]
ZnO/Ag ₂ CO ₃	facile in situ precipitation– deposition	phenol	Visible lamp and UV lamp	1 and 2% ZnO/Ag ₂ CO ₃ shows highest efficiency degrading phonol under UV irradiation compared to ZnO and Ag ₂ CO ₃	<u>[37]</u>

 Table 3. Photocatalytic degradation of EDCs by Ag₂CO₃-based photocatalysts prepared by various experimental conditions.

Photocatalyst	Synthesis Technique	EDC Target Compounds	Light Source	Photodegradation Performance	Ref.
ZnFe₂O₄/PANI/Ag₂CO₃	Hydrothermal/Co- precipitation method	bisphenol A	incandescent lamp (Visible)	ZnFe ₂ O ₄ /PANI/Ag ₂ CO ₃ exhibited the best photocatalytic ability of bisphenol A with percentage degradation of 86.36% under 40 min irradiation	<u>[39]</u>
Ag ₂ O/Ag ₂ CO ₃	Simple room temperature solid- state (RTSS) chemical reaction	Phenol and Bisphenol A	350W Xe lamp (Visible)	AAC-6 with mass ratio of 36.2 and 63.8 for Ag ₂ CO ₃ and Ag ₂ O exhibited the highest photocatalytic degradation performance with percentage degradation of 80% for Bisphenol A and 70% for Phenol after 60 min	[40]
N-doped carbon quantum dots (CQDs)/Ag ₂ CO ₃	Precipitation method	phenol	350-W Xe lamp with 420 nm long-pass (Visible)	Highest photocatalytic performance in phenol degradation was obtained over 3N-CQDs/Ag ₂ CO ₃ in 150 min	[41]

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