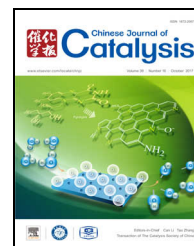


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## Article

# Synthesis of Ag/AgCl/Fe-S plasmonic catalyst for bisphenol A degradation in heterogeneous photo-Fenton system under visible light irradiation



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## ABSTRACT

A novel plasmonic photo-Fenton catalyst of Ag/AgCl/Fe-S was synthesized by ion exchange and photoreduction methods. The obtained catalyst was characterized by X-ray diffraction, X-ray photoelectron spectroscopy, scanning electron microscope imaging, and Brunauer-Emmett-Teller measurements. Moreover, the photocatalytic activity of Ag/AgCl/Fe-S was investigated for its degradation activity towards bisphenol A (BPA) as target pollutant under visible light irradiation. The effects of  $\text{H}_2\text{O}_2$  concentration, pH value, illumination intensity, and catalyst dosage on BPA degradation were examined. Our results indicated that the Ag/AgCl material was successfully loaded onto Fe-sepiolite and showed a high photocatalytic activity under illumination by visible light. Furthermore, active species capture experiments were performed to explore the photocatalytic mechanism of the Ag/AgCl/Fe-S in this heterogeneous photo-Fenton process, where the major active species included hydroxyl radicals ( $\cdot\text{OH}$ ) and holes ( $\text{h}^+$ ).

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## 1. Introduction

Safety concerns in relation to bisphenol A (BPA) have drawn attention owing to its widespread use in producing epoxy resins and polycarbonate plastics [1]. As a representative endocrine disruptor, BPA can damage the fecundity of animals and humans [2] by disrupting endocrine effects in the reproductive systems and cause the death of some types of cells [3–5]. Thus, a rapid and efficient method of removing BPA is urgently required.

Among various technologies for removing BPA, the heterogeneous photo-Fenton process is considered to be an effective and affordable method, owing to its generation of highly active radicals and the easy separation of the catalysts from treated

wastewater [6,7]. In the past few decades, much attention has been paid to investigating heterogeneous photo-Fenton processes for organic pollutant degradation under UV irradiation. However, UV light-based heterogeneous photo-Fenton processes are limited in their practical applications because UV light accounts for only 3%–5% of solar light energy [8]. Thus, the development of heterogeneous photo-Fenton catalysts that operated based on visible light is of great importance for practical applications.

The combination of photo-Fenton catalysts and plasmonic materials might overcome this problem. Plasmonic materials based on silver/silver halide (Ag/AgX, X = Cl, Br, I) composites can strongly absorb visible light because of their surface plasmon resonance [9–11] and are extensively used as visible-light

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photocatalytic materials [12–15]. However, Ag/AgX composites suffer from high charge carrier recombination rates, causing losses of photocatalytic efficiency [16]. Additionally, recent evidence has suggested that iron oxides, blended with semiconductor composites, can act a selective acceptor and inhibit the recombination of electron/hole pairs [17–19]. For example, Yang et al. [20] synthesized a  $\text{Fe}_3\text{O}_4/\text{rGO}/\text{TiO}_2$  visible light catalyst, in which the photo-induced electrons from  $\text{TiO}_2$  could rapidly transfer to  $\text{Fe}^{3+}$ , accelerating the redox transformation between  $\text{Fe(III)}$  and  $\text{Fe(II)}$ . On the basis of the above reports, we attempted to add Ag/AgCl composites into a heterogeneous photo-Fenton system. We expected that the Ag/AgCl might promote the charge transfer between  $\text{Fe(III)}/\text{Fe(II)}$  by promoting photogenerated electrons and enhancing the photo-Fenton catalytic activity under visible light irradiation. The choice of catalyst support is also important for preparing efficient heterogeneous photo-Fenton catalysts. Sepiolite is a zeolite-like clay mineral that has a high specific surface area and good chemical stability [21,22]. Thus, in this work, sepiolite was selected as the support material to enhance the synergistic effects between Ag/AgCl and hydroxy-iron, and fabricated a quaternary-composite photo-Fenton catalyst. We investigated the photocatalytic activity and stability of our synthesized Ag/AgCl/Fe-Sepiolite (Ag/AgCl/Fe-S) photocatalyst using BPA as a target contaminant. In addition, the possible photocatalytic mechanism involved in the photo-Fenton system was discussed. This work provides new insights into the preparation of visible-light responsive photo-Fenton catalysts.

## 2. Experimental

### 2.1. Materials

The sepiolite sample used in this study was obtained from Hunan Province, China, and the clay particle size was approximately 100-mesh. All chemical reagents were of analytical grade and used without further purification. Deionized water was used throughout the experiments.

### 2.2. Catalyst preparation

Acidified sepiolite was prepared by pouring 50 g of raw sepiolite powders into 1 L of  $\text{HNO}_3$  solution (2 mol/L) under continuous stirring at 40 °C for 2 h. This mixture was then filtered and washed with deionized water repeatedly until the supernatant pH value was approximately 7, and the resulting solid was vacuum dried at 100 °C overnight.

The Ag/AgCl/Fe-S catalyst was prepared by ion exchange and photoreduction methods. First, Fe-sepiolite was fabricated as follows.  $\text{Na}_2\text{CO}_3$  (0.2 mol/L) was added dropwise into 0.2 mol/L  $\text{Fe}(\text{NO}_3)_3$  solution under stirring at 25 °C until the molar ratio of Na/Fe was 1:1. This solution was aged for 24 h at 25 °C and then added into an aqueous suspension containing 2 wt% of the above-treated sepiolite under stirring at 60 °C until the final Fe/clay ratio was equal to 5 mmol/g of sepiolite. After aging for 12 h at 60 °C, the mixture was centrifuged and washed by deionized water. The resulting precipitates were

dried in air overnight at 70 °C to obtain Fe-sepiolite. Subsequently, the Fe-sepiolite was used to fabricate the AgCl/Fe-S. A 1-g portion of Fe-sepiolite was dispersed in 100 mL of deionized water and a 20-mL  $\text{AgNO}_3$  solution (0.15 g of  $\text{AgNO}_3$  dissolved in 20 mL of water) was added to the mixture with vigorous magnetic stirring for 12 h at 25 °C. Then, a 20-mL KCl solution (0.15 g of KCl dissolved in 20 mL of water) was added into the mixture, which was stirred for a further 30 min. The resulting product (AgCl/Fe-S) was filtered, washed, and dried at 70 °C. Finally, Ag/AgCl/Fe-S was prepared via a photoreduction method. A 1-g portion of the AgCl/Fe-S was dispersed in 100 mL of deionized water. An  $\text{AgNO}_3$  solution (30 mg of  $\text{AgNO}_3$  in 5 mL of water) was then added to the mixture, and the reaction stirred in the dark for 30 min. This mixture was irradiated with visible light ( $\lambda > 400$  nm) for 30 min to partially reduce the  $\text{Ag}^+$  ions to  $\text{Ag}^0$  species. The final product was gathered by centrifugation and dried at 70 °C. For comparison, pure Ag/AgCl was also prepared by the same method without the addition of Fe-sepiolite. All of the prepared samples were crushed and screened through a 200-mesh sieve.

### 2.3. Characterization

The XRD patterns of the prepared materials were acquired with a diffractometer (Rigaku D/max-2550 VK/PC) equipped with  $\text{Cu K}\alpha$  radiation at 40 kV and 50 mA. XPS measurements were performed on a K-alpha X-ray photoelectron spectrometer (PHI Quantera II, UIVAC) using a monochromatic Al  $\text{K}\alpha$  X-ray radiation source at 1486.71 eV. The morphologies of the products were observed with a scanning electron microscope (SEM, JSM-6360LV, JEOL). The nitrogen adsorption-desorption isotherms were determined with a NOVA 2200e instrument. Prior to the adsorption tests, the samples were outgassed for 12 h at 150 °C. The Brunauer-Emmett-Teller (BET) method was used to calculate the specific surface areas of the samples. The light absorption properties were measured with a UV-vis diffuse reflectance spectrophotometer (Shimadzu, UV-2550) with a wavelength range of 200–800 nm.

### 2.4. Photocatalytic reaction and analytical methods

All the experiments were conducted in a photoreaction apparatus (BL-GHX-V, Shanghai Depai Biotech. Co. Ltd., China), in which a 500 W xenon lamp equipped with a 420-nm cutoff filter was applied as the visible light source. The light source was positioned inside a cylindrical Pyrex vessel surrounded by a jacket with circulating water. The photocatalytic activities of the studied catalysts were evaluated from their ability to degrade BPA under various conditions. An appropriate amount of the catalyst was added into 100 mL of BPA solution (10 mg/L), and the initial pH of solution was adjusted by addition of NaOH or  $\text{HNO}_3$  solutions. Prior to irradiation, the solution was magnetically stirred in the dark for 30 min to establish an adsorption-desorption equilibrium. The reaction was started when  $\text{H}_2\text{O}_2$  was added to the solution and the light source was turned on. During the photocatalytic process, samples were taken from the reaction mixture at fixed intervals, and filtered immediately

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