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Ag_3PO_4/AgBr/Ag-HKUST-1-MOF composites as novel blue LED light active photocatalyst for enhanced degradation of ternary mixture of dyes in a rotating packed bed reactor

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ABSTRACT

This study focused on a continuous flow photocatalytic rotating packed bed for simultaneous photodegradation of methylene blue (MB), auramine-O (AO) and erythrosine (ER). Blue light emitting diode strips were used as light sources in this reactor for photocatalyst activation. Ag_3PO_4/AgBr/Ag decorated HKUST-1 MOF composite (HKUST-1-Ag_3PO_4/AgBr/Ag) as novel visible light photocatalyst was synthesized and characterized by XRD, FE-SEM, EDS and DRS analyses. Eight effective parameters include initial concentrations of MB, AO and ER, rotational speed, solution flow rate, aeration flow rate, HKUST-1-Ag_3PO_4/AgBr/Ag mass and time of illumination were optimized using a Taguchi design L18 ($2^1 \times 3^7$). The pH was optimized by one at a time method and fixed at 5.0. The maximum photocatalytic degradation efficacies of 92.01%, 89.57% and 89.96% for MB, ER and AO, respectively, were found at the optimum conditions: 75 min of illumination time, 15 mg/L of each dye, 0.4 g/L of HKUST-1-Ag_3PO_4/AgBr/Ag, 1000 rpm of rotational speed, 0.3 L/min of solution flow rate and 25 L/min of aeration rate. Kinetic study indicated that the Langmuir-Hinshelwood (L-H) model was well fitted to the experimental data of ternary photocatalytic degradation.

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1. Introduction

The different dye pollutants in the environment are creating a worldwide concern [1–5]. Synthetic dyes with complex molecular structures are difficult to be degraded with conventional methods like biological processes [2,6–8]. Among the various treatment methods, photocatalytic degradation is an efficient procedure for dyeing wastewater treatment because of its lower cost, operable under standard atmospheric condition, high efficiency, easy handling and no generation of harmful secondary by-products [9–13]. Photocatalytic degradation process by generation of highly reactive and nonselective hydroxyl radicals ($^{\bullet}OH$) is susceptible for dyes [14]. Therefore, finding new photocatalyst materials in order to improve degradation efficiency has always been the case. In the past decades, a large number of semiconductors, such as $TiO_2$and ZnO due to their high band gap, require the ultraviolet radiation which limits their application in terms of economics and environmental friendliness [15,16]. Therefore, visible light-active semiconductors and environmentally friendly photocatalysts such as $BiVO_4$, Bi_2S_3, Ag_3VO_4 and $Ag_2Mo_2O_7$ were developed [17–19]. Ag-based materials have emerged due to their unique crystal structures, and fascinating photocatalytic properties [20,21]. While the drawbacks, such as the serious deactivation, low efficiency, high electron-hole recombination rate and short stability, have hindered their practical application in photocatalysis [21,22]. Thus, composite and hybrid materials with others such as MOFs and Br^-, Cl^- and $PO_4{}^{3-}$ anions are needed to make an efficient, visible light active and stable photocatalyst [22,23]. In this case, Br^- and $PO_4{}^{3-}$ anions with cling to Ag can exhibit high stability and efficiency of Ag nanoparticles under successive blue LED illumination [24,25]. Metal–organic frameworks (MOFs) known as organic–inorganic hybrid materials with a three dimensional porous network and crystalline structures based on an organic linker and metal clusters have reasonable band gap, high stability, recoverable and structural control [26–29]. MOFs based photocatalyst favored photo activity due to large specific surface area, high pore volume

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and structural adaptivity [30,31]. In the present work, a new hybrid with HKUST-1 MOF and Ag_3PO_4/AgBr/Ag was synthesized as an efficient and stable material in visible light driven photodegradation of dyes in a rotating reactor for simultaneous photocatalytic degradation of ternary mixture of dyes under blue LED irradiation [32,33].

The stability, high photocatalytic activity, long-term durability and regeneration ability of the photocatalysts are important issues for practical application [34,35]. The instability upon photo-illumination is a major limitation of photocatalysts [36,37]. Generally, the photochemical stability of MOFs as photocatalysts under extended irradiation for long periods in not sufficient. Some of the MOFs can be instable in certain solvents and their structure decomposes when contact with some reagents [38]. The structure of MOFs has important role in stability during the photocatalytic degradation. Thus, modification of MOFs structure is a key factor to improve their stability as well as durability. Ag-based compounds such as Ag_3PO_4 have been shown high photocatalytic activity in photodegradation processes, while stability of their structure is still a challenge [37,39,40]. Some hybrid composite photocatalysts based on Ag_3PO_4, such as Ag_3PO_4/TiO_2, $Ag_3\ PO_4/Fe_3O_4$, Ag_3PO_4/SnO_2 and graphene oxide/Ag_3PO_4 have been synthesized to improve the performance and stability which indicated enhanced photocatalytic activity and stability compared to the pure Ag_3PO_4 catalyst [41,42]. The Ag_3PO_4 photocatalyst suffers from poor photo-stability in practical degradation process due it readily decomposes photo-chemically [34]. Thus, the development of composite photocatalysts with sufficient charge separation capability and high photocatalytic stability is necessary. In present study, the synthesis of HKUST-1-Ag_3PO_4/AgBr/Ag as stable and efficient photocatalyst was carried out by ultrasound-assisted solvothermal method. The existence of Cu in the HKUST-1 and Ag in Ag_3PO_4/AgBr/Ag structure have an important effect in enhancing the photocatalytic efficiency as well as the stability of photocatalyst through the enhanced carrier transfer and separation [43]. The composite of HKUST-1 and Ag_3PO_4/AgBr/Ag can be an ideal strategy to construct a stable and efficient complex photocatalytic system with extremely high photocatalytic performance and photo-stability rather than HKUST-1 and Ag_3PO_4/AgBr/Ag toward degradation of ternary mixture of dyes under visible-light illumination.

In photocatalytic degradation reactions, mass transfer rate and light distribution in a reactor play important roles in pollutant degradation. Mass transfer rate can be extensively intensified by a rotating photocatalytic reactor in a continuous flow mode by applying a centrifugal field for acceleration. The centrifugal force created a great shear force that caused the turbulency and micro mixing in a porous bed. Liquid film is converted to tiny droplet which enhance mass transfer dramatically [44]. Due to higher interfacial surface area, rotating photocatalytic packed bed reactor has less physical size and volume than conventional photocatalytic reactors, leading to significant reduction in the capital and operating costs. A strip blue LED which inserted around the reactor is a good shape distribution of light to produce high photocatalyst activity to achieve a very cost-effective and environmental friendly cleaning procedure [45–47].

In the present study, a ternary mixture of dyes was degraded using photocatalytic process, whereas in our previous investigation, binary mixture of dyes has been studied [48,49]. The HKUST-1-Ag_3PO_4/AgBr/Ag was synthesized as an efficient photocatalyst with high stability and lower band gap (1.3 eV) rather than previous photocatalysts which was used in our previous work. Also, Taguchi method than to other optimization method not only permit in saving the time and cost, but also leads to a fully developed process and provides systematic, simple and efficient approach for the optimization of the optimum design parameters.

2. Experimental

2.1. Materials and apparatus

Chemical reagents including sodium hydroxide, hydrochloric acid, silver nitrate, di-sodium hydrogen phosphate, sodium bromide and copper (II) nitrate hemipentahydrate were purchased from Merck Company (Darmstadt, Germany). Benzene-1, 3, 5-tricarboxylicacid and polyvinylpyrrolidone (PVP) was purchase from Sigma-Aldrich (USA). The concentration of solution was analyzed by UV–vis spectrophotometry (model V-530, Jasco, Japan). The pH was measured using a pH/Redox/Temperature meter (AL20 pH, AQUALYTIC, Germany). X-ray diffraction (XRD, Philips PW 1800) was recorded using Cu kα radiation (40 kV and 40 mA). The morphology of samples was analyzed using field emission scanning electron microscopy (FE-SEM: Sigma, Zeiss). Diffuse reflectance spectra (DRS) were collected with an Avant's spectrophotometer (Avaspec-2048-TEC).

2.2. Synthesis of HKUST-1 MOF

HKUST-1 was prepared by ultrasound-assisted solvothermal method as follows: benzene-1, 3, 5-tricarboxylicacid (3.6 mmol) and copper (II) nitrate hemipentahydrate (10 mmol) were sonicated for 1.0 h in 100 mL of solvent consisting of 80 mL DMF and 20 mL ethanol in a glass jar. Subsequently, the mixture was transferred into a Teflon-lined stainless autoclave, sealed and heated at 130 °C for 24 h to yield octahedral crystals. Finally, the solvent was removed and heated in an oven at 100 °C for 12 h to yield a porous material.

2.3. Synthesis of HKUST-1-Ag_3PO_4/AgBr/Ag

The synthesis of HKUST-1-Ag_3PO_4/AgBr/Ag was carried out by ultrasound-assisted solvothermal method as follows: Initially, 1.0 g of HKUST-1 MOF was well dispersed in water. Subsequently, 0.15 g of silver nitrate was added and sonicated continuously for 30 min and then mixed thoroughly with 0.09 g of di-sodium hydrogen phosphate, 0.6 g NaBr and 0.5 g PVP. The resulting mixture were sealed in Teflon-lined autoclave for hydrothermal treatment at 160 °C for 12 h. Finally, the obtained product was collected by centrifugation, washed with distilled water several times, and dried in oven at 70 °C.

2.4. Photocatalytic reactor setup

The reactor structure (Fig. 1) consists of two sections: rotating packed-bed and lateral equipment including: pump, tank, control box, flow meter, motor, aeration pump, a stirrer and control valves. The main part of the system is the rotating packed bed including porous bed, solution distributor, housing case and stripe blue LED. The porous bed is a one-piece torus-shaped unit, made of stainless wire mesh, has the inner radius, outer radius, and axial height of 3, 6, and 4 cm, respectively and was mounted to a shaft riding that supported by two bearings and connected to a motor controlled by a speed regulator to adjust the rotational speed. The rotating porous bed is covered with the housing case made of quartz glass with an inside diameter of 10 cm and axial height of 8 cm, and equipped with blue stripe LED (light source). The solution distributor consisted of a pipe with 4 vertical set of holes having 16 holes of 0.1 cm diameter. Liquid with a relatively high velocity exited from the liquid distributor and, then, sprayed onto the inner edge of the packed bed. The velocity of the liquid running out from the liquid distributor has to be sufficiently high to pass the gap between the liquid distributor and the inner edge of the packing [50].

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