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Thermally treated fungal manganese oxides for bisphenol A degradation using sulfate radicals

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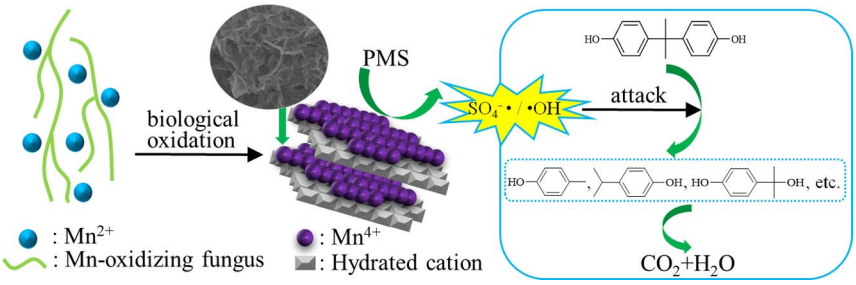
GRAPHICAL ABSTRACT



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ABSTRACT

Fungal manganese oxide (FMO) is ubiquitous in the environment and believed to be a promising material for catalysis due to its biological reactive mineral phases. In this study, layered FMO was produced by Mn (II)-oxidizing fungus. After thermal treatment at various temperatures, FMO was converted to manganese oxides (MnO_2, Mn_2O_3 and Mn_3O_4) with different morphologies. The as-obtained manganese oxides were adopted as catalysts for peroxymonosulfate (PMS) activation to produce sulfate radicals, which were highly efficient for bisphenol A (BPA) degradation. Catalytic evaluation showed that FMO calcined at 400 °C performed well for BPA degradation. Complete BPA degradation and 97% TOC removal in solution could be achieved in 30 min. Effects of catalyst dosage, PMS loading, solution pH and reaction temperature on BPA degradation efficiencies were also investigated. In recycle study, remarkable decrease of BPA degradation efficiency was found, which was ascribed to the coverage of catalytic reaction intermediates on the surface of catalyst. Fortunately, heat treatment could recover the catalyst with a complete BPA degradation efficiency over regenerated catalyst. Furthermore, a BPA degradation pathway was proposed based on intermediates identification by GC-MS and LC-MS.

1. Introduction

Biogenic Manganese oxide (BMO) is one of the most reactive mineral phase in aquatic and terrestrial environments, which plays an important role in the biogeochemical cycle of many elements [1,2]. Oxidative precipitation, where Mn (II) is oxidized by direct or indirect biological process to insoluble Mn (III, IV) oxides, is believed to deposit by microorganisms in natural conditions [3,4]. To date, it has been proved that many bacteria (*Pseudomonas putida* [5], *Leptothrix discophora* [6]) and funguses (*Basidiomycete* [7], *Ascomycete* [8]) can oxidize Mn (II) and produce BMO. Compared to Mn (II)-oxidizing bacteria, funguses have attracted much more attention not only for their high Mn (II) tolerance but also for species richness in the environment [9]. The widespread Mn (II)-oxidizing funguses contribute to the production of

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fungal manganese oxide (FMO). These Mn oxides are poorly crystalline minerals such as birnessite, vernadite (layer structure), and todorokite (tunnel structure) [8]. Due to their promising adsorption abilities and oxidative capacities for heavy metal ions and organic pollutants, particular attention has been paid to the application of FMO for heavy metals cycling and environmental remediation [10–12]. With unusual and well defined morphology, the favorable catalytic properties of biogenic manganese oxides are ascribed to the nature of its random layer stacking MnO_6 with poor crystallinity and the presence of an average valence state of $Mn^{3.3+}$ (containing a mixture of Mn^{2+}, Mn^{3+} and Mn^{4+}) [13,14]. High specific surface area is also reportedly important with a preponderance of edge sites being accessible for reaction [15]. With no need for excessive energy, microorganisms produce biogenic manganese oxides under environmentally benign conditions (at neutral pH, ordinary temperature and pressure), which is expected to be eco- and cost-effective system for functional material synthesis [16].

Bisphenol A (2, 2-bis (4-hydroxyphenyl) propane, BPA) is widely used in industry as a starting material to manufacture polycarbonate plastics, which is also known as a typical environmental pollutant of endocrine disruptors [17–19]. Currently, enormous quantities of BPA are easily discharged into aquatic environment from a vast array of plastics. Due to the strong durability and constant accumulation of BPA, serious ecology deterioration may be caused [20,21]. Conventional strategies for BPA removal from water include microbiological biodegradation [22], adsorption [23], ozonation catalytic degradation [24], photocatalytic degradation [25,26], etc. However, the disadvantages of time-consuming or relatively low efficiency limit the application of above-mentioned approaches. Recently, sulfate radical ($SO_4^{-\cdot}$) with high redox potential about 2.5–3.1 V has demonstrated great ability in oxidizing organic compounds [27–30]. Due to the efficient and quick degradation of organic pollutants into simple compounds, the application of sulfate radical-based advanced oxidation process is suggested to be a promising technology for BPA removal.

Commonly, production of sulfate radical can be achieved from activation of peroxymonosulfate (PMS) by energy system (thermal, photochemical or sonochemical), homogeneous catalyst system with transition metal ions (Co^{2+}, Mn^{4+}, Fe^{2+}, etc.) as well as heterogeneous catalyst system [31–33]. Previous studies have shown that Co^{2+}/PMS and Co_3O_4/PMS systems show outstanding performances for the degradation of organic pollutants [34–36]. However, toxic Co^{2+} dissolves in water may cause secondary contamination. Thus, manganese oxides are considered as alternative choices for PMS activation owing to their abundance in nature, low toxicity as well as stability in acidic solution. Through synthesizing various manganese oxides as heterogeneous catalysts for sulfate radical activation, Wang's research group suggested that the crystal structure, morphology and oxidation states all affected the catalytic activity of manganese oxides [37–39]. However, compared with chemically synthesized manganese oxides, less is known about biogenic manganese oxides in the activation of PMS for organic pollutants degradation. It is interesting and of great importance to investigate the characteristics of BMO for catalytic application.

In this study, a Mn (II)-tolerance fungus was cultured to remove Mn (II) ions in water and precipitated biological manganese oxide simultaneously. The obtained FMO and thermally treated FMO were applied for PMS activation to degrade a typical organic pollutant of BPA. In addition, several key parameters such as catalyst dosage, PMS loading, the initial pH of BPA solution and reaction temperature were investigated. Furthermore, recycle study was studied using optimized catalyst and reaction conditions. The possible BPA degradation pathway was also proposed based on GC–MS and LC–MS analysis.

2. Materials and methods

2.1. Culture and identification of microorganism

All reagents used in this work were of analytical grade and purchased from Chengdu Kelong reagent Co., China. *Pleosporales* sp. *Y-5* was isolated from the rotted wood in grove, Sichuan Province, P.R. China. Cultures were incubated in a medium containing 0.25 g L^{-1} sodium acetate, 0.15 g L^{-1} yeast extract, 15 g L^{-1} agar, 1 mL L^{-1} trace element stock (10 mg L^{-1} $CuSO_4{\cdot}5H_2O$, 13 mg L^{-1} $Na_2MoO_4{\cdot}2H_2O$, 44 mg L^{-1} $ZnSO_4{\cdot}7H_2O$, 20 mg L^{-1} $CoCl_2{\cdot}6H_2O$) supplemented with 100 mg L^{-1} Mn^{2+} (added as $MnSO_4{\cdot}H_2O$), pH 7.0, temperature 30 °C.

Genomic DNA was extracted from the mycelia using a rapid fungus genomic DNA isolation kit (Sangon Biotech, Shanghai, China). The Internal Transcribed Spacer (ITS) DNA was amplified by polymerase chain reaction (PCR) using primers ITS1 and ITS4 (ABI Veriti PCR System 9902, Applied Biosystems, China), according to the protocol of Sasaki et al. [40]. The PCR product was purified and subjected to a two-way sequencing by Sangon Biotech Company.

2.2. Preparation of FMO

Pleosporales sp. *Y-5* was incubated into 100 mL liquid medium containing 100 mg L^{-1} Mn^{2+} on an orbital shaker for 4 days (180 rpm, 30 °C). After that, FMO was collected by filtration, repeatedly washed with distilled water and lyophilized using a FD-1A-50 freeze-drier (Boyikang, Beijing, China) for 48 h.

Previous studies showed that oxidation state transition of manganese oxide could be achieved by calcination of MnO_2 at different temperature [37,41]. To obtain manganese oxides with different oxidation states, FMO was calcined at 400, 500 and 1000 °C for 4 h, respectively, in a muffle furnace in static air. The obtained samples were denoted as FMO-400, FMO-500 and FMO-1000, respectively.

2.3. Material characterization

Thermogravimetric analysis (TGA) was conducted with a Mettler-Toledo TGA/DSC1 machine in an air atmosphere (40 mL min^{-1}) with a ramping rate of 10 °C min^{-1} from room temperature to 1000 °C. The morphologies of FMOs were observed by using scanning electron microscopy (SEM, Zeiss Merlin Compact-61-78) and transmission electron microscopy (TEM, FEI Tecnai F30). Powder X-ray diffraction (XRD) patterns of FMOs were obtained on an X-ray diffractometer (Rigaku D/max-TTR III) with Cu Kα radiation (λ = 1.54056 Å) at 40 kV, current 40 mA. N_2 adsorption-desorption was measured at −196 °C using a Micromeritics ASAP 2020 instrument. The specific surface area was calculated by Brunauer-Emmett-Teller (BET) method. X-ray photoelectron spectroscopy (XPS) analysis was carried out on Thermo Scientific ESCALAB 250Xi (Al-Kα X-ray source). Fourier transform infrared (FT-IR) spectra was recorded on IR Prestige 21 (Shimadzu). The zeta potential was measured by Zetasizer Nano-ZEN3690 (Malvern).

2.4. Catalytic performance evaluation

BPA was chosen as a model organic pollutant to evaluate the catalytic efficiency of FMOs. In a typical run, 20 mg of catalyst was dispersed in 100 mL aqueous solution containing 50 mg L^{-1} BPA by constant stirring at 350 rpm. Prior to addition of 0.2 g L^{-1} of PMS ($KHSO_5{\cdot}0.5KHSO_4{\cdot}0.5K_2SO_4$, Huaxia Reagent Company, China) (purity ≥ 98%), the suspension was stirred for 1 h to reach adsorption equilibrium. To avoid the influence of indoor light, the beaker was covered with aluminum foil. At certain reaction time intervals, 0.75 mL of aqueous sample was withdrawn and mixed with 0.75 mL of methanol (MeOH) immediately to quench the reaction. After filtration with 0.2 μm syringe filter to remove solid catalyst, the remaining BPA concentration was quantified by Alltech HPLC system equipped with a UV

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