



Dark ambient degradation of Bisphenol A and Acid Orange 8 as organic pollutants by perovskite SrFeO_{3-δ} metal oxide

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H I G H L I G H T S

- Perovskite SFO prepared by high temperature and high-energy ball milling process.
- SFO metal oxide shows good efficiency in degrading and mineralizing BPA.
- Rapid decoloration of AO8 was achieved in the presence of SFO metal oxide.
- O₂^{•-} is the predominant ROS for dark oxidative degradation of BPA and AO8.

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Current advanced oxidation processes (AOPs) are chemically and energetically intensive processes, which are undesirable for cost-effective and large-scale system water treatment and wastewater recycling. This study explored the Strontium Ferrite (SFO) metal oxide on the degradation of highly concentrated organic pollutants under dark ambient condition without any external stimulants. The SFO particles with single perovskite structure were successfully synthesized with a combined high temperature and high-energy ball milling process. An endocrine disruptor, Bisphenol A (BPA) and an azo dye, Acid Orange 8 (AO8) were used as probe organic pollutants. BPA was completely degraded with 83% of mineralization in 24 h while rapid decoloration of AO8 was achieved in 60 min and complete breakdown into primary intermediates and aliphatic acids occurred in 24 h under the treatment of dispersed SFO metal oxide in water. Such efficient degradation could be attributed to the enhanced adsorption of these anionic pollutants on positively charged ball-milled SFO metal oxide surface, resulted in higher degradation activity. Preliminary degradation mechanisms of BPA and AO8 under the action of SFO metal oxide were proposed. These results showed that the SFO metal oxide could be an efficient alternative material as novel advanced oxidation technology for low cost water treatment.

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1. Introduction

Advanced oxidation processes (AOPs) have received great attention for water treatment in recent years due to the high efficiency in generating reactive oxidative species (ROS) such as hydroxyl radical, superoxide anion radical and singlet oxygen, which can rapidly oxidize and remove a wide range of organic pollutants from water

[1,2]. AOPs can be categorized into two main groups, namely photo and dark oxidation. Photocatalysis is widely known as an efficient method to destroy organic pollutants under UV or sunlight irradiation [1,3]. However, the use of photocatalyst is greatly inhibited during night time or in the absence of light illumination. Therefore, dark oxidation methods such as ozonation, ultrasonication and Fenton process were studied by other researchers as means to avoid the need of light illumination [2,4–6]. However, those dark processes are currently less effective in removing organic pollutants due to low total organic carbon (TOC) removal [4]. Furthermore, they require additional chemicals, high electrical power and/or pH adjustment for efficient operation. Thus, it remains crucial to explore on other material alternative which can initiate robust

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oxidative process without the aids of external stimulants while achieving a high mineralization capability.

Perovskite mixed metal oxides had been demonstrated to oxidize and remove effectively several organic compounds [7,8]. Strontium Ferrite ($\text{SrFeO}_{3-\delta}$, SFO) metal oxide with perovskite structure was found to possess redox behavior [9] and catalytic activity for the oxidation of organic compounds such as toluene and methane [10,11]. This study thereby aimed to explore the capability of SFO to degrade water pollutants under dark ambient condition for the first time. The submicron size perovskite SFO metal oxide particles were synthesized from a combination of conventional high temperature solid state reaction and high-energy ball milling process and the physicochemical properties were characterized systematically using XRD, SEM, BET and zeta potential analyses. Two organic compounds were chosen as probe organic pollutants, namely Bisphenol A (BPA) and Acid Orange 8 (AO8), respectively. BPA is an endocrine disruptor and is regarded as an emerging carcinogenic environmental pollutant that could not be removed easily by the conventional water treatment process [12,13] while AO8 is a highly soluble azo dye pollutant found in textile wastewater and has high resistance to photo oxidation [14]. Despite both BPA and AO8 being challengingly difficult to be removed by conventional AOPs, the experimental results in this study demonstrated that the milled SFO metal oxide can efficiently degrade both pollutants in dark, hence demonstrating the use of SFO as a potential candidate of AOP for water treatment.

2. Materials and methods

2.1. Reagents

Starting materials for the synthesis of SFO metal oxide included the strontium carbonate (SrCO_3 , 99.9+ %) purchased from Sigma-Aldrich and iron (III) oxide ($\alpha\text{-Fe}_2\text{O}_3$, hematite, 99.945%) from Alfa Aesar. The targeted organic pollutants including Bisphenol A (BPA, $\geq 99\%$) and Acid Orange 8 (AO8, 65%) were purchased from Aldrich. All reagents were used as received without further purification. All the solutions were prepared using Milli-Q deionized water ($18.2 \text{ M}\Omega \text{ cm}^{-1}$).

2.2. Synthesis of SFO metal oxide

SFO metal oxide was synthesized through a combined high temperature solid state reaction and high-energy ball milling process. The starting materials (SrCO_3 and $\alpha\text{-Fe}_2\text{O}_3$) were first homogenized by wet milling in ethanol at 150 rpm using agate milling media. The weight ratio of ethanol-to-starting materials was set as 2:1. The wet milled mixture was then dried at 100°C before subjected to two cycles of solid state annealing at 1200°C for 24 h with oxygen ambient in a Carbolite bottom loading furnace. After each annealing cycle, the powder was grounded in an agate mortar for re-mixing purpose. In order to reduce the particle size, the SFO powder after solid state reaction was further ball-milled at 200 rpm for 3 h in a Retsch Planetary Ball Mill PM400 system. Tungsten carbide bowl and balls with a diameter of 20 mm were selected as the milling media. The weight ratio of ball-to-SFO powder was set as 20:1.

2.3. Characterization of SFO metal oxide

The morphology of SFO metal oxide was observed through a LEO 1550 Gemini scanning electron microscope (SEM) at an accelerating voltage of 5 kV. X-ray diffraction (XRD) pattern was recorded with a Shimadzu XRD-6000 diffractometer using monochromated $\text{Cu K}\alpha$ irradiation ($\lambda = 1.5418 \text{ \AA}$), operating at 40 kV and 40 mA. The N_2 adsorption/desorption isotherm of the SFO metal oxide at liquid nitrogen temperature (77 K) was obtained on a Quantachrome

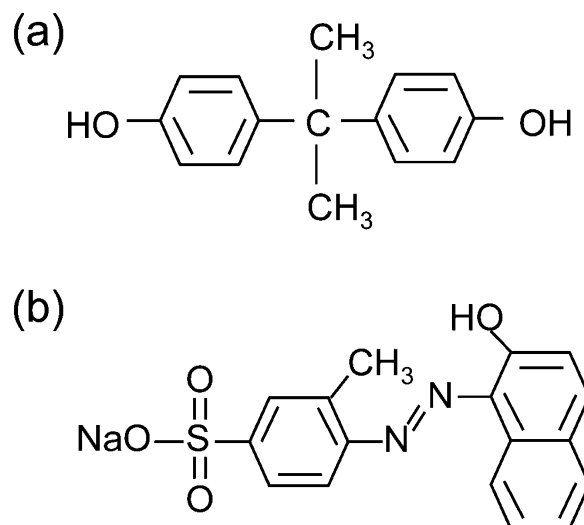


Fig. 1. Chemical structure of (a) BPA and (b) AO8.

Autosorb-1 analyzer after the samples were degassed at 200°C for 21 h. BET surface area was calculated from the adsorption isotherm. Surface charge of the SFO metal oxide in water was determined from zeta potential measurement using a Malvern ZetaSizer Nano ZEN3600 fitted with a 633 nm red laser.

2.4. Degradation experiment

Fig. 1 shows the chemical structure of BPA and AO8. The initial concentrations of BPA and AO8 were $440 \mu\text{M}$ and $150 \mu\text{M}$, respectively. The degradation of targeted pollutants was carried out in a simple dark batch reactor at room temperature. The dispersions of milled SFO metal oxide were magnetically stirred during the degradation process. At designated intervals, 4.5 mL of liquid samples were withdrawn, centrifuged and filtered to remove the SFO particles. The concentration of BPA and AO8 was analyzed by measuring the absorbance at 276 nm and 487 nm respectively using a Shimadzu UV2400 UV-vis spectrophotometer. HPLC analyses were performed using an Inertsil ODS-3 column and a series 200 UV/vis detector at 225 nm to identify possible intermediate byproducts formed during the degradation of BPA. The mobile phase of acetonitrile/water (80/20, v/v) was maintained at a flow rate of 1 mL/min. Assignment of the HPLC peaks of the samples was done by matching their retention time with that of the standard chemical solutions containing 2-phenyl-2-propanol, *p*-isopropylphenol, acetophenone, *p*-hydroxyacetophenone, *p*-hydroxybenzaldehyde, *p*-hydroxybenzoic acid and hydroquinone because these chemical compounds had been reported as main intermediate byproducts in the degradation of BPA by other advanced oxidation processes [15–17]. These standard chemical solutions were analyzed via HPLC under similar analytical conditions. The remaining TOC in the sample solutions was monitored using a Shimadzu ASI-V TOC analyzer. The adsorbed BPA and intermediate byproducts on the surface of milled SFO metal oxide were investigated through X-ray photoelectron microscopy (XPS) surface analysis. The XPS analysis was conducted in ultra-high vacuum (1×10^{-8} mbar) in a Kratos AXIS spectrometer using monochromatic $\text{Al K}\alpha$ (1486.71 eV) as X-ray radiation source, with a pass energy of 40 eV. Curve fitting was performed via the Lorentzian–Gaussian method (LG (30)) with Shirley background subtraction. Ion chromatography (IC) analysis was done with a Dionex ICS-1000 ion chromatography system (equipped with a 4 mm \times 250 mm IonPac AS15 anion-exchange column and a conductivity detector) to identify the lowest-molecular-weight carboxylic acids such as acetic, formic

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