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Activated electric arc furnace slag as an efficient and reusable heterogeneous Fenton-like catalyst for the degradation of Reactive Black 5

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ABSTRACT

This study aims to explore the feasibility of using iron slag derived from an electric arc furnace (EAFS) as a catalyst to degrade Reactive Black 5 (RB5). Raw EAFS (R-EAFS) subjected to 0.1 M NaOH solution treatment and thermal treatment was denoted as activated EAFS (A-EAFS). A-EAFS was characterized and examined for its capability to degrade RB5 in a dark-batch reaction under different catalyst dosages (0.01–0.06 g), H₂O₂ dosages (2–12 mM), initial pH values (2–7), initial concentrations (25–150 ppm), and reaction temperatures (303–323 K). The optimal conditions (94% within 60 min) for RB5 degradation were 0.2 g L⁻¹, 8 mM H₂O₂, and pH 3. The degradation rate of A-EAFS for RB5 exceeded 94%, which was higher than that of R-EAFS. Recycling of A-EAFS after 10 reuses suggested that this stability is ascribed to the regeneration of the major constituent's ferrous ion magnetite and maghemite to decompose H₂O₂ into hydroxyl radical catalytically. Therefore, A-EAFS is an alternative heterogeneous catalyst in the abatement of organic dyes from polluted water through a heterogeneous Fenton-like reaction.

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1. Introduction

The presence of chemical compounds (e.g., dyes) in wastewater has become a global concern because of its serious threat to public health and hydrosphere. An estimated 1 ton of textile products consumes approximately 21–377 m³ of water, and the World Bank indicated that textile dyeing and treatment processes account for 17–20% of industrial water pollution [1,2]. Given their resistance to detergents, dyes become converted into complex organic molecules that are chemically stable, nonbiodegradable, carcinogenic, mutagenic, and toxic even at low concentrations [3]. Thus, these contaminants must be detoxified prior to their release into the environment.

In abiding with the less than 0.5 ppm discharge limit set by the United States Environmental Protection Agency law [4], advanced oxidation processes (AOPs) have been considered as a green and efficient choice for depurating contaminated wastewater. Among all the intensively studied AOPs, Fenton or Fenton-like reactions produce hydroxyl radical (HO•) activated by ferrous ion (Fe²⁺) with a redox potential of 2.80 eV at room temperature and pressure. This radical can decompose persistent organic dyes. However, the economic expediency of homogeneous Fenton processes is

limited by their post-treatment requirement prior to iron sludge formation, sensitivity to a narrow operational pH range of 2.5–3.5, and difficult regeneration of lost iron catalyst.

To solve these drawbacks, many studies have developed efficient and economical heterogeneous Fenton-like catalysts that incorporate metal oxides inside several catalyst support materials [5–7]. Despite their potential, heterogeneous Fenton processes lack recyclability of iron oxide. Consequently, the addition of a fresh catalyst is needed to sustain an excellent catalytic activity. Therefore, the use of an iron-rich source material that easily recovers is ideal to maintain an effective heterogeneous Fenton-like catalytic activity.

Byproducts of steel industries are known iron-rich sources. Electric arc furnace (EAF) plays an important role in making modern steel and dominates the overall steel production in Malaysia. Direct smelting of iron-containing materials, such as ferrous scrap (steelworks cut-offs from vehicles, capital, and post-consumer scrap), is a major feedstock for the EAF [8]. With respect to end-products, slag is formed from lime to collect undesirable components in the EAF slag (EAFS), which is classified as steel waste. Approximately 20–40 kg of EAFS is generated per ton of steel produced.

EAFS has been used as a raw material for road construction [9], cement production [10], and ceramic tile making [11]. EAFS typically contains more than 25% iron oxide, such as magnetite

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(Fe₃O₄) or other complex oxides (SiO₂, Al₂O₃, and CaO) [6]. Thus, EAFS can be easily recovered through magnetic separation [12] and serve as a promising candidate heterogeneous catalyst in environmental applications [13]. Iron phase with respect to wustite (FeO), magnetite, and maghemite (γ -Fe₂O₃) exhibits unique properties, including electrical, magnetic, catalytic, and low toxicity. In particular, magnetite works efficiently as a heterogeneous catalyst in Fenton-like reactions because of its high Fenton reactivity. Magnetite uniquely contains both Fe³⁺ and Fe²⁺ in the spinel structure. Meanwhile, maghemite has a similar structure to magnetite [13]. According to the classical Haber–Weiss mechanism, the presence of Fe²⁺ occupied in the spinel structure initiates the decomposition of H₂O₂ into HO• radicals [14]. The accommodation of both Fe²⁺ and Fe³⁺ allows for reversible oxidization and reduction (redox) without structural change. In addition, magnetite can be easily separated from the reaction medium by using an external magnetic field because of its inherent magnetic properties [13].

Inspired by environmentally benign concepts, this study investigated the utilization of the byproduct EAFS to degrade Reactive Black 5 (RB5). Widely used in textile production, RB5 was chosen as a model pollutant of reactive dyes. R-EAFS, A-EAFS, and UA-EAFS were explored by characterizing their respective cycles of reaction, and their effects on various factors were also discussed. The stability study on A-EAFS would provide not only interesting candidates but also insights into the recyclability and durability of this steel waste for heterogeneous Fenton-like reaction applications.

2. Materials and methods

2.1. Materials

High-quality RB5 (98% Merck) and H₂O₂ (30%, Merck) were prepared and used without purification. EAFS was collected from a metal steel factory in Penang, Malaysia. pH controllers with alkaline (0.1 M of NaOH) and acid (0.1 M HCl) solutions were prepared. The alkaline solution was also used for washing purposes. Double distilled water was used throughout the preparation of the dye solution.

2.2. Preparation of material

2.2.1. Preparation of activated EAFS

Raw EAFS (R-EAFS) was ground and sieved to obtain a particle size of 40–50 μ m. Subsequently, R-EAFS particles were soaked in 0.1 M NaOH for 4 h and washed several times with distilled water to remove adhering dust and fine materials. The supernatant was decanted by centrifuging at 500 rpm for 10 min. Finally, the samples were dried at 100 °C for 24 h and then calcined at 400 °C for 2 h prior to use as activated EAFS (A-EAFS).

2.2.2. Catalytic activity procedure

RB5 was degraded through a heterogeneous Fenton-like reaction in a 250 mL conical flask with 200 mL of different RB5 concentrations (25–150 ppm) at different temperatures (30–50 °C) and pH (2–7). Prior to batch runs, reactions were initiated by adding certain amounts of H₂O₂ (2–12 mM) into the aqueous solution and shaken at 120 rpm in an isothermal water bath shaker in the dark. At regular intervals, withdrawn samples were filtered through 0.2 μ m Millipore syringe filters to remove suspended particles and then analyzed using a UV–vis spectrophotometer (UV-1700 Pharmaspec Shimadzu). The concentration of RB5 was determined by measuring the absorbance of the solution at λ_{max} 598 nm. The spent A-EAFS was washed five times with distilled water and then dried at 60 °C in the oven for the next cycles. The same optimal condition catalytic reaction was repeated for 10 cycles, and the sample was indicated as used activated EAFS (UA-EAFS).

Table 1
XRF analysis chemical composition for R-EAFS and A-EAFS.

| Component | Chemical composition, wt.% | |
|--------------------------------|----------------------------|--------|
| | R-EAFS | A-EAFS |
| Fe ₂ O ₃ | 32.65 | 33.45 |
| CaO | 28.25 | 29.80 |
| SiO ₂ | 22.06 | 22.09 |
| Al ₂ O ₃ | 6.13 | 5.74 |
| MnO | 4.09 | 4.21 |
| MgO | 2.95 | 2.90 |
| Na ₂ O | 0.03 | - |
| P ₂ O ₅ | 0.89 | 0.40 |
| SO ₃ | 0.22 | 0.20 |
| Cl | 0.01 | 0.01 |
| K ₂ O | 0.03 | 0.02 |
| TiO ₂ | 0.79 | 0.79 |
| V ₂ O ₅ | 0.56 | 0.57 |
| Cr ₂ O ₃ | 0.84 | 0.81 |
| NiO | 0.01 | 0.01 |
| CuO | 0.02 | 0.02 |
| ZnO | 0.25 | 0.23 |
| SrO | 0.06 | 0.05 |
| ZrO | 0.01 | 0.02 |
| Nb ₂ O ₅ | 0.02 | 0.02 |
| BaO | 0.12 | 0.13 |
| PbO | 0.02 | - |
| LOI | 0.01 | 0.008 |

The homogeneous A-EAFS was tested on filtrate solution. A known amount of RB5 stock solution was added into the filtrate solution to keep its initial concentration constant prior to re-initiation of reaction by H₂O₂. Sampling of RB5 concentration was carried out periodically and the concentration of RB5 was determined as previous procedure.

2.3. Characterization of catalysts

The chemical contents of R-EAFS and A-EAFS were analyzed with an X-ray fluorescence spectrometer (XRF) using a Rigaku RIX 3000 X-ray Fluorescence (XRF). Mineral phases were determined through X-ray diffraction (XRD) of the powders with a Philips PW 1710 in the 2 θ range of 10°–90°.

The morphologies of the sample surface were imaged and observed via scanning electron microscopy (SEM, Philips XL30S). Meanwhile, the percentage elemental composition of the sample was easily determined via SEM combined with energy dispersive X-ray (EDX) spectroscopy.

The surface area and pore size distributions of the catalyst were determined using the Micromeritics ASAP 2020 model in accordance with the Brunauer–Emmett–Teller (BET) method. Total pore volume was determined on the basis of nitrogen adsorption/desorption at a relative pressure of 0.99 by using the Barrett–Joyner–Halenda (BJH) model.

3. Results and discussion

3.1. Characterization of R-EAFS, A-EAFS, and UA-EAFS

3.1.1. XRF

XRF measurements were performed to investigate the loss of ignition (LOI) as well as chemical content in R-EAFS and A-EAFS. LOI analysis revealed that both samples had low moisture content and impurities (approximately 0.01 wt.%). Most moisture and impurities were vaporized at a high temperature during the formation of slag from molten steel [15]. Table 1 shows the chemical compositions of R-EAFS and A-EAFS. Fe₂O₃, CaO, and SiO₂ were the major components of R-EAFS and A-EAFS. However, the treatment

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