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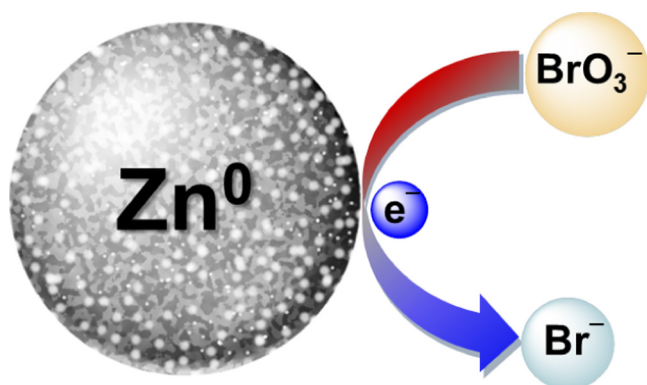
Efficient reductive elimination of bromate in water using zero-valent zinc prepared by acid-washing treatments



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GRAPHICAL ABSTRACT



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ABSTRACT

Although zero valent zinc (ZVZ) is a strong reductant, studies using ZVZ for bromate reduction are rare. In this study, ZVZ is prepared by acid-washing zinc powder with HCl and used to reduce bromate. The effect of acid-washing on the morphology of zinc powder is also examined. Zinc powder inefficiently reduces bromate, but ZVZ obtained by acid-washing zinc powder eliminates bromate and converts it to bromide. A higher dose of ZVZ enhances elimination efficiency perhaps because the formation of a passivation layer of zinc oxide could be scattered on the large surface of ZVZ. Elevated temperature also substantially improves both elimination efficiency and kinetics. The effect of pH is shown to have the most significant impact on the bromate elimination; elimination efficiency and kinetics are tremendously bolstered at pH = 3, whereas the elimination of bromate is completely suppressed under alkaline conditions. ZVZ can reduce bromate to bromide even in the presence of other anions and also be reused multiple times. Thus, ZVZ can be easily prepared and used to efficiently reduce bromate to bromide. The findings presented here are essential to the design and implementation of bromate elimination in water using zero-valent metals.

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1. Introduction

Chemical oxidation is extensively employed in water purification and wastewater treatment because such techniques (e.g., advanced oxidation processes (AOPs)) effectively and rapidly degrade organic pollutants. However, since water and wastewater

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are complex matrices and contain numerous compounds and ions, intensive chemical oxidation can lead to the formation of many by-products. Some of these by-products pose serious threats to human health and the environment. In particular, bromate (BrO_3^-) has received increasing attention because it is a 2B substance reported by the International Agency for Research on Cancer [1]. Conventionally, the formation of bromate is attributed to reactions of ozone with bromide [2]. However, recent studies have shown that bromate can be also generated from treatments of bromide-containing water by sulfate radical-based AOPs [3,4]. As AOPs are widely implemented in water treatment facilities [5], bromate is increasingly present in water supplies and should be eliminated to prevent its negative impacts.

Many methods have been reported for the elimination of bromate in water, including filtration [6], adsorption [7], and ion exchange [8]. However, these methods simply transfer bromate from one phase (*i.e.*, aqueous phase) to another (*i.e.*, solid phase) and its hazards persist. Thus, several studies have suggested that bromate can be eliminated by reducing it to bromide by hydrogenating bromate with H_2 and catalysts [9–12], or using zero-valent metals (ZVMs) [13–17]. Although hydrogenation of bromate by H_2 is possible, this approach typically demands continuous purging of H_2 , whose solubility in water is considerably low as well as the use of expensive borohydrides. Preparation of catalysts for hydrogenation is also complicated and involves with noble metals [18–22].

In contrast, elimination of bromate by ZVMs is relatively straightforward and easily implemented, and therefore many studies have successfully demonstrated bromate reduction by ZVMs [13–17]. As zero-valent iron (ZVI) is a versatile material and applied in various areas, most of these studies focused on development of ZVI for bromate reduction [15–17]. However, several ZVMs are also effective and more advantageous than ZVI for reductive reactions. In particular, zero-valent zinc (ZVZ) is a promising ZVM for bromate reduction because it is a more powerful reductant than ZVI [23]. In addition, only a single oxidation step can occur for ZVZ ($\text{Zn}^0 \rightarrow \text{Zn}^{\text{II}}$); therefore ZVZ is preferable for studying mechanisms of bromate reduction by ZVMs [23]. However, studies using ZVZ for reduction of bromate are very rare. Thus, we evaluate bromate reduction by ZVZ and also investigate various factors influencing reduction efficiency.

Because zinc is easily oxidized upon exposure to air, commercially available zinc powder requires acid-washing pre-treatments to remove the resulting zinc oxide layer to obtain ZVZ. The effect of acid-washing with different acid concentrations on bromate reduction is examined. Other effects on bromate reduction are investigated, including ZVZ dose, temperature, pH, and co-existing ions. Recyclability of ZVZ with and without acid-washing regeneration is also evaluated to determine whether ZVZ can be re-used.

2. Experimental

2.1. Materials

Chemicals used in this study were all commercially available and used without purification. Zinc powder, sodium bromate and sodium bromide were obtained from Sigma-Aldrich (USA). Hydrochloric acid (HCl) (37%) was purchased from Union Fine Chemicals, Taiwan. Sodium nitrate, sodium sulfate and sodium phosphate were received from Alfa Aesar (USA). Deionized (DI) water was prepared to less than $18 \text{ M}\Omega\text{-cm}$.

2.2. Reductive elimination of bromate in water using acid-washed ZVZ

The as-received zinc powder was acid-washed to obtain ZVZ by removing a passivation layer of zinc oxide on the surface of zinc powder as illustrated in Fig. 1(a). HCl was particularly selected as the most effective acid for the acid-washing treatment based on previous studies [13,14,24]. The acid-washing treatment started by adding zinc powder (*e.g.*, 1.2 g) to an HCl solution (10 mL) with a pre-set concentration (*i.e.*, 0.01–1 M). The mixture was immediately placed on an orbital shaker (300 rpm) and stirred for 2 min at ambient temperature. The acid-washed ZVZ was recovered by filtration and washed with oxygen-free DI water thoroughly. The effect of acid-washing on the morphology of zinc powder was revealed using scanning electronic microscopy (SEM) (JEOL JSM-6700, Japan).

Reductive elimination of bromate by acid-washed ZVZ was evaluated by batch-type experiments. Typically, acid-washed ZVZ (*e.g.*, 1 g (dry basis)) was added to a beaker containing 0.2 L of bromate solution with an initial concentration (C_0) of 10 mg L^{-1} (*i.e.*, $78.1 \mu\text{mol/g}$). The beaker was then placed on a stirring plate (300 rpm) and maintained at a desired temperature. After pre-set intervals, sample aliquots were taken from the beaker and filtered through disk membranes ($0.22 \mu\text{m}$). The reaction time for a batch experiment was set at 120 min. The filtrate was then injected into an ion chromatography system (Dionex ICS-1100, USA) to measure the remaining concentration of bromate (C_t) and the resulting concentration of bromide (C_t) at a given time t . The elimination efficiency (q_t) of ZVZ for reducing bromate and generating bromide was calculated via the following formula (Eq. (1)):

$$q_t = \frac{(C_0 - C_t) \times v}{W} \quad (1)$$

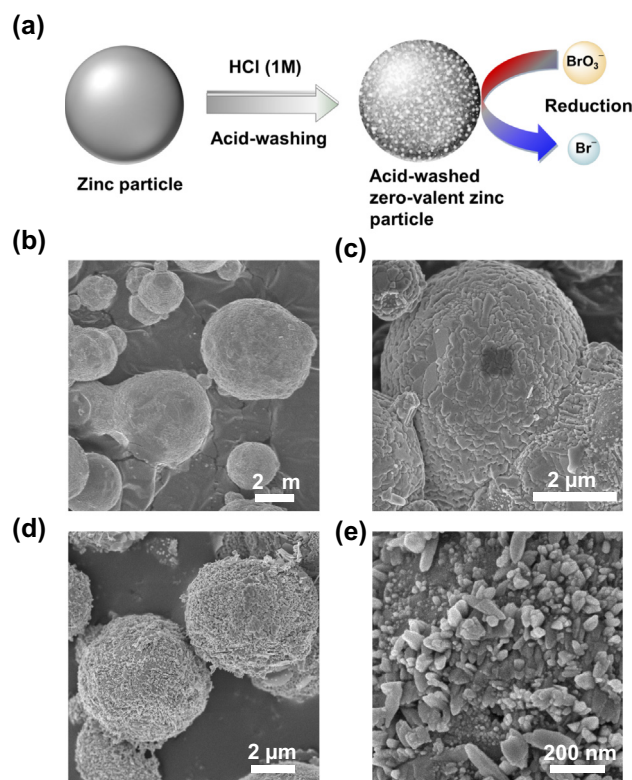


Fig. 1. (a) Illustration of preparation of ZVZ by acid-washing zinc particle and SEM images of (b), (c) pristine zinc particle and (d), (e) acid-washed ZVZ using 1 M HCl at different magnifications.

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