



## Original Research Paper

## Enhanced reactivity of nanoscale zero-valent iron prepared by a rotating packed bed with blade packings



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## ABSTRACT

Nanoscale zero-valent iron (nZVI) was prepared by continuously pumping aqueous  $\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$  (0.1 mol/L) and  $\text{NaBH}_4$  (0.2 mol/L) into a rotating packed bed (RPB) with blade packings. A rotational speed of 1400 rpm, liquid flow rates of 0.6 L/min, and a temperature of 21 °C were used. nZVI that was prepared in this way had a mean grain size of 11.5 nm and a BET surface area of 17.1  $\text{m}^2/\text{g}$ . The reactivity of the thus prepared nZVI was measured using the degradation of Reactive Red 2 (RR2) in the presence of dissolved oxygen. At pH 4 and 30 °C with an nZVI dosage of 0.30 g/L, the prepared nZVI degraded approximately 92% of the RR2 in 5 min; this efficiency of degradation greatly exceeded that obtained using nZVI that was purchased from Centron Biochemistry Technology (CBT).

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## 1. Introduction

In 1965, Vivian et al. [1] studied for the first time the effect of centrifugal acceleration on the stripping of carbon dioxide from water using a packed column that was attached to a centrifuge. This packed column, which had a height of 0.305 m and a diameter of 0.152 m, was packed with Raschig rings with a nominal size of 0.02 m. The centrifugal acceleration was varied from 10 to 63  $\text{m}/\text{s}^2$  by varying the rotational speed of the centrifuge. Their results indicated that the liquid-side volumetric mass transfer coefficient depended on the centrifugal acceleration to the power of 0.41–0.48, suggesting that mass transfer could be improved by increasing the centrifugal acceleration. Additionally, consistent with the penetration theory, the liquid-side mass transfer coefficient varied with the centrifugal acceleration to the power of 1/6 [1]. The effective interfacial area may increase with the centrifugal acceleration [1]. Therefore, the liquid-side volumetric mass transfer coefficient could be significantly improved by increasing the centrifugal acceleration.

Based on the above discussion, a promising alternative means of intensifying mass transfer is to bring liquid into contact with gas under a very high centrifugal acceleration by rotating the doughnut-shaped packings. In 1981, Ramshaw and Mallinson [2] exploited the centrifugal acceleration to enhance the efficiency of gas–liquid separation. Accordingly, they invented the rotating packed bed (RPB) for distillation and absorption. This novel tech-

nology is referred to as “Higee” (an acronym for high gravity). When the liquid flows through the rotor, it is subjected to a high centrifugal acceleration of at least 300  $\text{m}/\text{s}^2$ , determined by the rotational speed, reducing the tendency to flood below that in a conventional packed bed [3]. Accordingly, the RPB can be operated at a higher gas or liquid flow rate [3]. Also, packings with a larger specific area (2000–5000  $\text{m}^2/\text{m}^3$ ) and a higher voidage (90–95%) can be used [3]. Since the centrifugal acceleration is higher (2000–10,000  $\text{m}/\text{s}^2$ ), a thinner film and smaller droplets can be formed [4]. The mass transfer would be enhanced by a factor of 10–100, so the required equipment is physically smaller than that required for the conventional packed bed, and the capital and operating costs are consequently lower [3]. The RPB has been widely used for distillation [4], VOCs absorption [5–15],  $\text{CO}_2$  absorption [16–24],  $\text{O}_3$  absorption [25–27], ozonation [28], reactive precipitation [29–32], and stripping [33].

Over the last few years, we successfully used the RPB with blade packings to absorb VOCs, as presented in Fig. 1 [8]. According to the results, the RPB with blade packings exhibited superior operating characteristics, including a low pressure drop and a high mass transfer efficiency [8]. We have presented more results concerning the RPB with blade packings for removing a single VOC by absorption [8–10,12,15]. In the relevant studies, the VOCs were methanol, ethanol, isopropyl alcohol, 1-butanol, acetone, methyl ethyl ketone, ethyl acetate, and methyl acetate. The RPB with blade packings could effectively process a gas stream that contained a single VOC, with a high removal efficiency. Recently, we used the RPB with blade packings to remove VOCs from binary mixtures by absorption [13–14].

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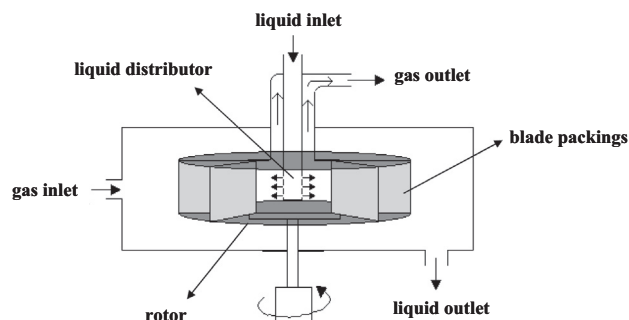


Fig. 1. RPB with blade packings.

In 2004, Chen et al. [34] investigated micromixing in the RPB with structured packings. They proposed that the mass transfer would be improved as the micromixing efficiency was increased. According to our previous results [8], mass transfer of the RPB with blade packings was comparable to that of the RPB with structured packings, implying that the micromixing efficiency of the RPB with blade packings would be comparable to that of the RPB with structured packings. Additionally, precipitation process has been found to be significantly affected by micromixing, because the reaction time of precipitation is on the same order of magnitude as the characteristic time for micromixing [35]. Accordingly, the RPB with blade packings would be expected to be able to prepare nanoparticles. However, preparing nanoscale zero-valent iron (nZVI) in the RPB with blade packings has rarely been discussed. In this study, nZVI were prepared in an RPB with blade packings, in which the reductive precipitation of iron with borohydride occurred. This study elucidates the characteristics and reactivity of nZVI that was prepared by reductive precipitation in an RPB with blade packings.

## 2. Experimental

### 2.1. Chemicals

Ferrous chloride ( $\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$ , 98%) and sodium borohydride ( $\text{NaBH}_4$ , 98%) were purchased from Alfa-Aesar. Reactive Red 2

( $\text{C}_{19}\text{H}_{10}\text{Cl}_2\text{N}_6\text{Na}_2\text{O}_7\text{S}_2$ , 40%) was obtained from Sigma-Aldrich. Hydrochloric acid (HCl, 37%) was supplied by Scharlau. All stock and working solutions were prepared in deionized water with a resistivity of 18.2  $\text{M}\Omega \text{ cm}$ , which was obtained using a Direct-Q 3 UV-R Tap to Pure & Ultrapure water purification system (Merck Millipore).

### 2.2. Preparation of nZVI

The RPB with blade packings had an inner radius of 1.95 cm, an outer radius of 6.25 cm, and an axial height of 2.95 cm. The packings comprised 12 blades within the RPB, separated by angles of  $30^\circ$ . Each blade had a depth of 4.30 cm and a height of 2.95 cm. Each blade was covered with a stainless steel wire mesh that was formed from interconnected filaments with a mean diameter of 0.22 mm and a mean mesh diameter of 3 mm. The packed blades had a specific surface area of  $93 \text{ m}^2/\text{m}^3$  and a voidage of 0.99. The rotational speed of the RPB with blade packings was varied from 600 to 1800 rpm, providing 17–149 times the acceleration due to gravity at the arithmetic mean radius. nZVI was prepared by reductive precipitation in the RPB with blade packings, according to the following chemical reaction [36].

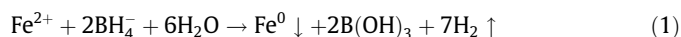


Fig. 2 schematically depicts the experimental setup for preparing nZVI in the RPB with blade packings by the above chemical reaction. The initial reactant was  $\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$  and the reductant was  $\text{NaBH}_4$ .  $\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$  (40 g) was dissolved in 2 L of deionized water that did not contain dissolved oxygen. Tank A contained this as-obtained solution with a pH of 3.4 and an  $\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$  concentration of 0.1 mol/L.  $\text{NaBH}_4$  (15 g) was dissolved in 2 L of deionized water that did not contain dissolved oxygen. This solution with a pH of 10.6 and an  $\text{NaBH}_4$  concentration of 0.2 mol/L was then placed in tank B. To increase the production rate of nZVI, solutions from both tanks A and B at  $21^\circ\text{C}$  were introduced into the packed bed through the liquid distributors at an identical flow rate of 0.6 L/min, which was maintained using flowmeters. Accordingly, the molar ratio of  $\text{NaBH}_4$  to  $\text{FeCl}_2$  was 2:1, consistent with stoichiometric ratio in Eq. (1). According to previous investigation [29],

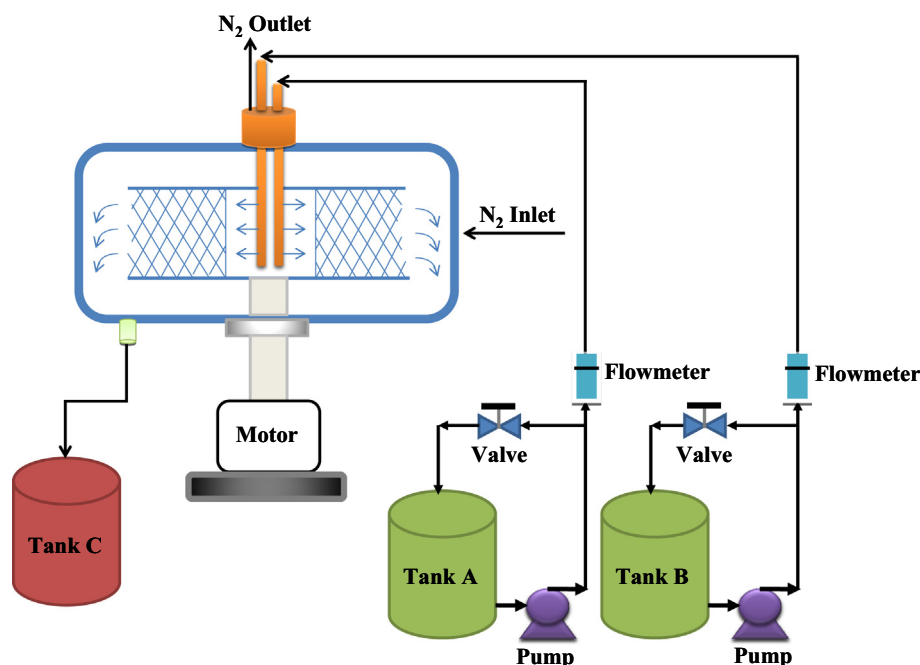


Fig. 2. Experimental setup for preparation of nZVI.

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