



Review

Analytical characterisation of nanoscale zero-valent iron: A methodological review



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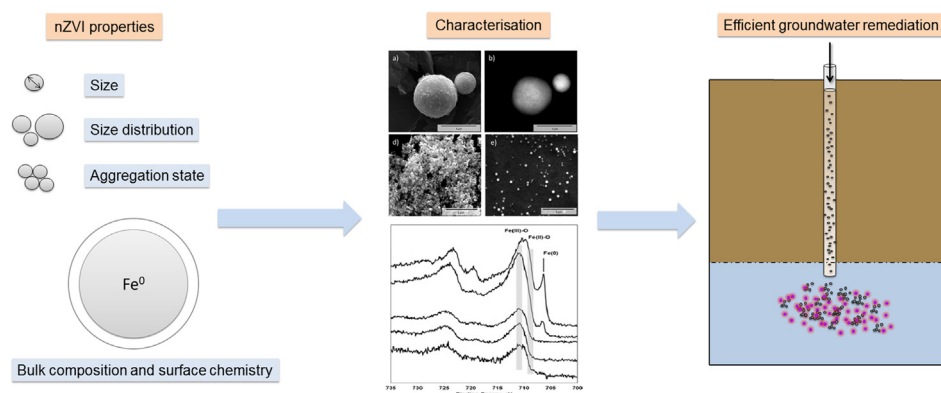
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HIGHLIGHTS

- The use of nanoscale zero-valent iron (nZVI) for environmental remediation is increasingly being investigated.
- The efficacy of nZVI materials is related to their size and surface properties.
- The characterisation of nZVI is extremely challenging due to their high reactivity.
- This review covers the fundamental principle and the type of information that various techniques can provide.
- The necessity of a multi-technique characterisation approach is discussed.

GRAPHICAL ABSTRACT



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ABSTRACT

Zero-valent iron nanoparticles (nZVI) have been widely tested as they are showing significant promise for environmental remediation. However, many recent studies have demonstrated that their mobility and reactivity in subsurface environments are significantly affected by their tendency to aggregate. Both the mobility and reactivity of nZVI mainly depends on properties such as particle size, surface chemistry and bulk composition. In order to ensure efficient remediation, it is crucial to accurately assess and understand the implications of these properties before deploying these materials into contaminated environments. Many analytical techniques are now available to determine these parameters and this paper provides a critical review of their usefulness and limitations for nZVI characterisation. These analytical techniques include microscopy and light scattering techniques for the determination of particle size, size distribution and aggregation state, and X-ray techniques for the characterisation of surface chemistry and bulk composition. Example characterisation data derived from commercial nZVI materials

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is used to further illustrate method strengths and limitations. Finally, some important challenges with respect to the characterisation of nZVI in groundwater samples are discussed.

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1. Introduction

In recent years, manufactured nanoparticles (MNPs) have attracted increasing interest for their potential applications in the treatment of contaminated soil and water. In comparison to traditional macro- and micro-scale materials, MNPs possess significantly higher surface-to-volume ratios, which frequently translate into unexpected surface effects and unique beneficial properties. In addition, by virtue of their small size, MNPs can potentially be used directly in the field for *in situ* treatment via injection at almost any location and depth in soil and groundwater systems [1]. Ideally, for this type of application, MNPs are expected to feature several key properties including: (i) high reactivity for the removal of targeted contaminants; (ii) high mobility in porous media (i.e. aquifers, soil); (iii) high reactive longevity after injection and (iv) low toxicity to the biota in the surrounding environment [1]. These properties are the main drivers when designing MNPs for the purpose of soil and groundwater remediation. However, it is quite clear that in practice

some of these properties are difficult if not impossible to achieve simultaneously. For instance, high reactivity usually translates to short longevity, and potentially greater ecotoxicological impact. In addition, these nanoparticles must also be produced and delivered at a cost that remains sufficiently low to compete with conventional technologies.

Due to their purportedly low cost, highly reactive surface sites and high *in-situ* reactivity, the most widely studied MNPs for environmental remediation are nanoscale zero-valent iron (nZVI) [2–4]. Although most of the lab-scale, pilot-scale and field demonstration studies targeted soil and groundwater remediation, nZVI has also been successfully applied for the stabilisation of biosolids [5], the removal of phosphorus [6,7], the decolouration of dyes [8,9], as a membrane anti-fouling agent [10] and for the treatment of nuclear waste, explosives and herbicides [11–13].

Many studies suggested a core–shell structure for nZVI [14,15] as illustrated in Fig. 1. The core consists primarily of zero-valent iron while the mixed valent oxide shell resulted from the oxidation of the core metallic iron [16]. nZVI materials have been shown to exhibit high reactivity in remediating aquifers contaminated by non-aqueous phase liquids (NAPL) and many other hazardous compounds [3,17,18].

Effectively delivering the nZVI to the contaminant source zone is essential for the success of *in situ* nZVI remediation. However, there are currently a number of issues limiting the potential effectiveness of nZVI for this application. In fact, many laboratory and pilot-scale field studies have demonstrated that the mobility and reactivity of nZVI are substantially limited in natural porous systems such as soils and also groundwater aquifers [19–22]. The limited mobility and reactivity can be explained by three basic mechanisms [23–25]:

- Due to their magnetic properties [26], nZVI particles have a natural tendency to aggregate which generates significant losses in reactivity and decreases environmental mobility [27];
- As nZVI are prone to surface oxidation, the formation of corrosion product precipitates (including $\text{Fe}(\text{OH})_2$, $\text{Fe}(\text{OH})_3$, Fe_3O_4 ,

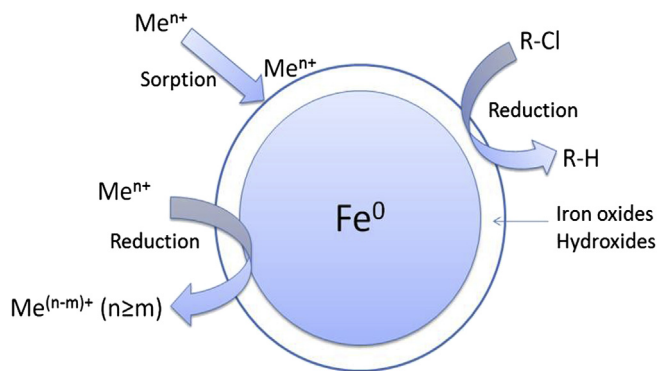


Fig. 1. The core–shell model of zero-valent iron nanoparticles (adapted from Ref. [16]). The core is made of metallic iron while the shell consists mostly of iron oxides and hydroxides. Thus ZVI nanoparticles exhibit characteristics of both iron oxides (e.g., as a sorbent) and metallic iron (e.g., as a reductant).

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