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Perspectives of soil nanoremediation: the case of nano zerovalent iron and metal(loid) contaminants

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The use of nano zero-valent iron in environmental remediation has gained much attention in the last two decades. While these engineered nanoparticles have been successfully used in groundwater remediation, their potential in soil remediation remains limited, mainly due to the associated higher costs and comparable efficiency when compared to conventional iron-based materials, e.g., iron grit. Additionally, there is still a limited number of studies describing their efficiency under field conditions, possible toxicological issues, including toxicity to humans, LCA of the technology, and its economic viability in general. This perspective article aims to describe the mechanisms behind this technology and critically review the potentials and drawbacks of nZVI use in the remediation of soil contaminated with metals and metalloids as its potential for its widespread use in soil remediation is probably still limited.

Soil remediation using Fe-based materials

The contamination of soil with metal(loid)s is a global problem worldwide. Due to their non-degradability and persistence in the environment, the remediation of soils is thus limited to their extraction or stabilization. Soil washing/flushing techniques remove the contaminants from soils, but these approaches can be disruptive and create risks of subsequent groundwater contamination¹. Phytoextraction, i.e., the use of plants for extracting the contaminants from soils, is due to its low efficiency, most probably a dead-end^{2,3}, but the use of plants to stabilize contaminants in soils, i.e., phytostabilization, especially in combination with other stabilizing amendments is still a promising alternative⁴ (Fig. 1).

Stabilization of metal(loid)s in soils, including As, Cd, Cr, Cu, Ni, Pb, Zn, etc., has been the focus of many studies. It has gained attention among academia and industry as a potential alternative to excavation and land-filling, with results showing substantial improvements in soil properties and reduced contaminant mobility, bioavailability, and toxicity. This method, in general, represents a realistic and viable alternative for soil remediation since contaminant-immobilizing amendments decrease contaminant mobility, bioavailability, and bioaccessibility through various processes, i.e., adsorption, surface precipitation, structural incorporation, and ion exchange⁵. Besides other amendments, e.g., compost, biochar, clay minerals, lime, etc., Fe-based materials have been successfully applied for stabilizing metal(loid)s in soils for some time now, but comprehensive field studies are still scarce, despite being crucial for evaluating their efficiency and cost in real-world

conditions⁶, with some exceptions, e.g., the Difpolmine project in France, possibly the largest project in Europe using iron grit for As stabilization. Increasing the sorption capacity of soils can be achieved either directly by the addition of various Fe oxides or indirectly by their precursors, i.e., zero-valent Fe (ZVI) in the form of iron grit, iron sulfates, or iron mud, etc.^{5,7}.

For over 20 years now, zero-valent Fe nanoparticles (nZVI), i.e., particles with one dimension under 100 nm, have been successfully used due to their unique properties for the remediation of groundwater contaminated mainly by halogenated organic compounds, selected metals, nitrates, arsenates, oil, etc.^{8–10}. They represent engineered analogs of naturally formed nanoparticles found in the environment characterized by their high reactivity^{11,12}. Their potential use for the remediation of soils contaminated with metal(loid)s was highlighted over a decade ago⁸, and numerous studies have been performed since then, especially at the laboratory scale. On the other hand, the number of tests performed on soils in the field under natural conditions showing the efficiency and associated costs of the technology is still limited. This perspective article summarizes relevant results from studies involving nZVI and metal(loid)-contaminated soils and identifies possible gaps and perspectives. This work deals only with soils contaminated with metals and metalloids, so other soil contaminants will not be discussed here.

The behavior of nano zero-valent iron in soils

The nZVI particles are, like other nanoparticles, usually smaller than 100 nm in diameter. They are highly reactive in oxic and aqueous

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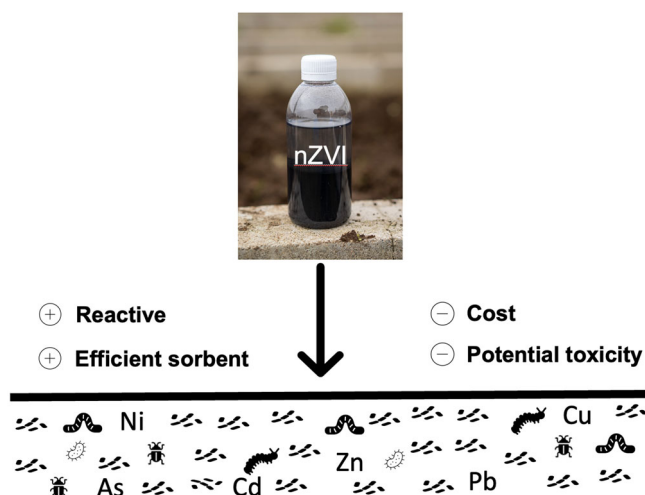


Fig. 1 | Potentials and drawbacks of nZVI use in soil remediation.

environments, such as soils, forming an outer shell formed by Fe oxyhydroxides around the Fe(0) core. In order to produce nZVI material that would be easily manageable and applicable, nZVI producers provide forms stabilized by various coatings, e.g., carboxymethyl cellulose, polyacrylic acid, guar gum, etc., or Fe oxyhydroxides in suspension^{13–15}. Fast nZVI oxidation can result in reduced reactivity, and surface passivation is thus essential for a successful application of nZVI in certain conditions¹⁶. This can be achieved by synthesizing various composites, e.g., with biochar¹⁷, zeolites¹⁸, etc.

Additionally, the interactions of nZVI with soil organic matter significantly influence its behavior and efficiency, as the evolution of the iron oxyhydroxide shell can be altered by dissolved organic matter in soils¹⁹. The actual composition of the Fe oxyhydroxides in the nZVI shell after it is equilibrated with the soil is mainly dependent on (i) the original nZVI coating; (ii) soil physico-chemical parameters, e.g., Eh, pH; (iii) composition of soil microorganisms communities, (iv) nZVI aging in soil; and include mainly the formation of ferrihydrite, magnetite, goethite and possibly lepidocrocite^{13,14,20}, which can have different geochemical and adsorption properties. It is not possible to generalize which Fe oxyhydroxides will be formed in soils after nZVI application, and this highlights the need for further long-term studies involving soils with contrasting properties and microorganism communities.

The physical-chemical properties of engineered nanoparticles are one of the most critical factors controlling their environmental behavior. In this context, the processes at the soil-root interface play a vital role in the behavior of engineered nanoparticles and contaminants^{21,22}. Different parameters such as soil type, organic matter content, pH, Eh, ionic strength, and aqueous chemistry can change aggregation kinetics, transformation, and subsequent behavior of engineered nanoparticles and their efficiency²³. Natural organic matter alters its stability through electrostatic and steric interactions. The transformation process of engineered nanoparticles is controlled by a combination of factors, depending on the particles' characteristics and the environmental receptors^{10,24,25}. Additionally, the changes in the hydraulic conditions in soils after nZVI application need to be considered²⁶. However, studies describing the mobility of engineered nanoparticles in general, including nZVI, in the soil environment are often contradictory, showing that this area is still open to discussion.

Metal(loid) stabilization in soils treated with nZVI

The processes responsible for immobilizing metal(loid)s on the nZVI surface include mainly adsorption and, to some extent, reduction. The nZVI particles can remove various metal(loid)s simultaneously; however, the type of adsorption reaction involved in the process depends on the redox potential of metal species. While metals with a standard redox potential more negative than Fe(0) are rather adsorbed on the Fe oxyhydroxides

formed in the nZVI shell, e.g., Cd, Zn, other metal(loid)s with a standard redox potential much more positive than nZVI can be reduced and precipitated when in contact with the zero-valent core, e.g., Cr, As, U, etc., or the combination of the two mechanisms, e.g., Pb, Ni¹⁰. The specific retention mechanisms, including the forms of the Fe oxyhydroxides in the shell and metal(loid) speciation after interactions with nZVI can be identified by various solid state analyses, i.e., X-ray photoelectron spectroscopy (XPS), X-ray absorption spectroscopy (XAS) techniques, such as X-ray absorption near edge structure spectroscopy (XANES) and extended X-ray absorption spectroscopy (EXAFS), X-ray diffraction (XRD), scanning electron microscopy (SEM) coupled with energy dispersive X-ray spectroscopy (EDS), and transmission electron microscopy (e.g., HR-TEM)^{10,14,20}. However, recovering and separating the nZVI particles from the soil after application for further investigations is problematic, and specialized separation techniques, e.g., magnetic separation, must be used²⁷. Sulfidation of nZVI particles can lead to promising results as it can sequester metals as sulfides, which could be resistant to subsequent reoxidation^{28,29}.

Unfortunately, most studies on metal(loid) stabilization in soils using nZVI are only laboratory-based. However, the first obtained results were promising for subsequent field applications at a larger scale³⁰. Arsenic is well known for its high affinity to Fe oxyhydroxides, and nZVI could thus present an attractive and efficient amendment. In one of the first studies, Gil-Díaz et al. evaluated As stabilization in a soil treated with nZVI, and the application significantly decreased As availability determined by various extraction procedures; however, the studied soil was spiked with As, which does not represent natural conditions³¹, which is a common issue in several similar laboratory investigations. The dominant mechanism seemed to be, in this case, As adsorption followed by possible reduction and diffusion through the thin oxide nZVI layer, resulting in an intermetallic phase with the Fe(0) core after breaking of As–O bonds at the particle surface³². Chromium(VI) is another good target metal for remediation by nZVI. Its reducing properties result in the transformation of Cr(VI) into Cr(III), which lowers its mobility and availability in the environment, which has also been shown in contaminated soils^{33–35}. The efficiency of the stabilization process depends on the (i) pH of the treated soil. As expected, anionic metal(loid)s (e.g., As, Cr) are preferentially retained in acidic conditions, cationic metals (e.g., Cd, Pb, and Zn) are better immobilized in near-neutral-alkaline soils; and (ii) the presence of several metal(loid)s and their competition for nZVI-sorption sites³⁶. However, when the stabilization efficiency of nZVI is compared with common iron-based materials (micro- or macro-sized)⁷, it is not possible to justify the costs associated with nZVI when used extensively on large, contaminated sites.

Toxicity and interactions with plants and microorganisms

It has been suggested that the generation of Fe²⁺ and reactive oxygen species by nZVI results in cell membrane disruption, and together with oxidative stress, these are the main mechanisms contributing to nZVI cytotoxicity and altering the taxonomic and functional composition of indigenous microbial communities^{14,37,15}. These unwanted effects can result in severe toxicity for soil biota, especially in soils with low organic matter contents³⁸. These drawbacks can be alleviated by the co-addition of inorganic and especially organic materials, e.g., compost, biosolids, bentonite, etc., which are beneficial for microbial communities and earthworms in nZVI-treated soils^{39–43}.

In general, iron-based nanomaterials have not shown significant toxicity toward bacteria and plants at concentrations lower than 50 mg/L and at 5 mg/L for earthworms, except for ball-milled nZVI⁴⁴. Higher nZVI concentrations (500 and 1000 mg/kg) inhibited growth and respiration and increased avoidance of earthworms and oxidative stress in *E. fetida* as a result of nZVI application⁴⁵. It is evident that the potential nZVI toxicity depends on environmental conditions, physico-chemical characteristics of the soil, concentration, aggregation, and reactivity of nZVI, and contamination types and levels^{37,46}. Generally, it is possible to assume that plants can take up Fe from nZVI into their aboveground parts^{13,47}. While Wojcieszek et al. (2023) indicate that the Fe in the aerial parts is mainly in the form of particles and not originating from dissolved species, this question

remains open, and the exact mechanisms need to be clarified using advanced techniques, e.g., synchrotron Mössbauer and isotope techniques, which is currently under investigations. Counteractive effects of arbuscular mycorrhiza and nZVI on plant physiology and metal(loid) uptake in the arbuscular mycorrhiza fungal–root–nZVI system need to be taken into account, and its presence is usually beneficial for alleviating the potential stress caused by nZVI to plants¹³.

The nZVI dose, as for any other material, is one of the crucial factors influencing its potential toxicity. For example, nZVI additions higher than 1–2% (w/w), which have been commonly studied under laboratory conditions (e.g., refs. 20, 31, 36, 38, 43, 45, 48), would not be economical and could potentially aggravate the associated toxicological risks. As for other engineered nanoparticles, another important point that needs to be taken into account is the potential toxicity and health-related risks of nZVI to humans, e.g., nZVI has the potential to induce cardiovascular disease through oxidative and inflammatory mediators produced from the damaged lung epithelium in chronic lung diseases⁴⁹ or directly damage DNA⁵⁰. Nevertheless, risks associated with ingesting the nanoparticles from the soil seem to be minimal.

nZVI composites

Few recent studies suggest that nZVI composites with biochar can be beneficial for selective sorption as well as for the stability of the amendments in soils^{17,51,52}. Biochar-supported nZVI improved the stability, mobility, and stabilization efficiency of nZVI in a Cr(VI)-contaminated soil⁵³. Using nZVI-biochar composites also helps limit the nanoparticles' aggregation in the soil and thus possibly increases the efficiency and reduces nZVI leaching through the soil profile. Additionally, using such composites could improve the selectivity of the amendments and possibly reduce application costs as more nZVI surfaces will be available for sorption due to limited aggregation and the influence of the biochar as another sorbent. The properties and preparation conditions of the biochar, e.g., pore structure, functional groups, feedstock composition, and pyrogenic temperature, are crucial for the resulting properties of the composite⁵³. Other possible materials improving the efficiency through limiting nZVI particle aggregation include e.g., zeolites¹⁸, bentonite⁴², vermiculite⁵⁴, etc.

The materials used for synthesizing the composites should be chosen in accordance with the target contaminants and specific soil conditions. When biochar is used, biowaste materials used as feedstock should be cost-efficient and improve the LCA of the final product. For example, the use of pyrolyzed biosolids, e.g., sewage sludge, could be promising as it would somehow reduce the footprint of this waste^{43,55}; however, the presence of various contaminants in the sludge must be carefully considered.

Concluding remarks

Since knowledge about engineered nanoparticles and nZVI regarding their interaction with biota and toxicity is still limited, their full-scale application and use for remediation remains problematic as there are still several open questions concerning phytotoxicity, ecotoxicity, and bioaccessibility of engineered nanoparticles in general that need to be evaluated in more detail. Additionally, recent studies suggest that the efficiency of nZVI for stabilizing metal(loid)s does not justify the costs associated with their use in practice at a large scale, for instance, when compared to larger-sized ZVI (iron grit), which is generally a waste product^{43,56}. Additional studies are still needed, especially from field applications, evaluating LCA of the technology, and its economic viability in general. Knowledge about the potential toxicity and risks associated with human exposure remains still limited. Also, the development of different composite materials with nZVI, e.g., with biochar from biosolids, could provide additional benefits and increase the cost efficiency of the technology. This does not mean that nZVI cannot be practically used in other applications, i.e., groundwater remediation, preparation of functional advanced materials and composites for particular use cases, etc. Still, its potential for widespread use in soil remediation is, in my opinion, limited.

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Author contributions

M.K. wrote the main manuscript and is the only author.

Competing interests

The author declares no competing interests.

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