Nanoparticles for Remediation: Solving Big Problems with Little Particles



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Remediation with engineered nanomaterials (ENMs) promises more effective and cheaper approaches than conventional methods due to the increased reactivity of nanoparticles and the possibility of in situ treatment. Three examples of the use of ENMs in soil remediation are nanoscale zero-valent iron for the degradation of halogenated organic compounds, nanoscale calcium peroxide for the destruction of organics (e.g. gasoline) and nanoscale metal oxides for the adsorption of metals. However, these methods are very new, and more research is needed on the mobility of ENMs in the soil and their impact on the environment.

KEYWORDS: engineered nanomaterials, nZVI, adsorption, reduction, in situ remediation

INTRODUCTION

Maintaining and restoring the quality of air, water and soil is one of the great challenges of our time. Most countries face serious environmental problems, such as the availability of drinking water, the treatment of waste and wastewater, air pollution and the contamination of soil and groundwater. In many cases, conventional remediation and treatment technologies have shown only limited effectiveness in reducing the levels of pollutants, especially in soil and water (Rickerby and Morrison 2007). Nanotechnology promises a potential revolution in approaches to remediation.

Nanotechnology is a broad and interdisciplinary field dealing with structures and particles at the nanoscale. Nanotechnology can be defined as "research and technology development at the atomic, molecular, or macro-molecular levels using a length scale of approximately one to one hundred nanometers in any dimension; the creation and use of structures, devices and systems that have novel properties and functions because of their small size; and the ability to control or manipulate matter on an atomic scale" (US EPA 2007, p 5).

The terms *nanoparticle* (NP) and *nanomaterial* (NM) are used in the scientific literature to describe these small particles, and some confusion exists with respect to their exact definition; different organizations have come up with different definitions (Som et al. 2010). The term *engineered nanoparticles* (ENPs) is most often used as a collective term for all discrete particles that have one or more external dimensions at the nanoscale, while the term *engineered nanomaterials* (ENMs) is a collective term for ENPs and nanostructured materials. Nanostructured materials may

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E-mail: nicole.mueller@empa.ch be composed of discrete functional materials with one or more external dimensions at the nanoscale (e.g. polymer composites that contain ENPs), or they may be free of ENPs (e.g. nanoporous polymers, nanostructured bulk materials and nanoscale coatings).

Nanoparticles are not only produced deliberately for specialized materials and processes, they can also form as accidental by-products of industrial processes. They also occur naturally and are found in air, water, soil and sedi-

ments. Natural NPs can have an atmospheric, geogenic or biogenic origin (Banfield and Zhang 2001).

Since the 1990s the use of ENMs in a variety of environmental applications, such as water purification, wastewater treatment, indoor and outdoor air cleaning, and soil and groundwater remediation, has been investigated (Mueller and Nowack 2009). Various applications have been successfully demonstrated at the laboratory scale, but most of them still require verification of their efficacy and safety in the field. It is thus not surprising that to date few nanotechnological applications for environmental use have been commercialized. The high costs involved in developing materials and running pilot and field trials have also delayed progress.

However, some environmental applications of nanomaterials have entered the market or are under close investigation. These include:

- Nanoscale TiO₂ for the photocatalytic degradation of contaminants in air (e.g. NO_x, VOCs) and water (e.g. microorganisms, organic materials)
- Nanofiltration for wastewater treatment and drinking water purification (e.g. removal of hardness and desalination)
- Nanoscale zero-valent iron for soil and groundwater remediation

There is considerable commercial potential in environmental nanotechnologies. Boehm (2006) projected that the world market for applications of environmental nanotechnologies would reach approximately \$6 billion by 2010. The market for soil and groundwater remediation is expected to grow to around \$23.6 billion worldwide; the UK and Japan are expanding near-term markets, and central and eastern Europe countries will be important mid-term markets (Rickerby and Morrison 2007).

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NANOPARTICLES: **NEW PROPERTIES DUE TO SMALL SIZE**

Nanoparticulate materials are new but not new. The chemical composition of nanomaterials may be the same as an equivalent material in bulk form (particle size in the micrometre to millimetre range), but nanoparticles can display totally new characteristics due to their high surfaceto-volume ratio and because, at their small size, quantum effects come into play (Hochella and Madden 2005). For example, TiO₂ has been used as a white pigment in highopacity paints for a long time. By contrast, nanoscale TiO₂ particles are transparent to visible light (wavelength 400-800 nm). Aluminium in cans is harmless, whereas nanoscale aluminium is highly explosive and can be used in rocket fuels. Hematite particles with a diameter of 7 nm adsorb Cu ions at lower pH values than particles with a diameter of 25-88 nm, illustrating one way in which surface reactivity of iron oxide particles varies with decreasing diameter (Madden et al. 2006).

Nanomaterials may be significantly more reactive than larger particles because of their much greater surface area per unit of mass (Rickerby and Morrison 2007), and this effect is made use of in environmental remediation. Zerovalent iron (ZVI) has been used in reactive barriers at numerous sites all over the world for the removal of organic and inorganic contaminants (Nowack 2008). ZVI effectively dechlorinates many halogenated hydrocarbon compounds (Gillham and O'Hannesin 1994). The degradation is based on redox reactions, in which iron donates electrons to the contaminants, reducing them to less toxic compounds. ZVI can also decrease dissolved concentrations of nitrate, perchlorate, selenate, arsenate, arsenite and chromate (Nowack 2008).

The reaction rates of nanoscale zero-valent iron (nZVI) are 25-30 times faster than the reaction rates of granular iron in the micrometre to millimetre range, and the sorption capacity is also much higher (Li et al. 2006). nZVI particles can have surface areas per unit of mass up to 30 times greater than larger-size powders of granular iron and can be 10 to 10,000 times more reactive (US Navy 2010). FIGURE 1 illustrates the very high reactivity of nZVI: when it comes into contact with air, it ignites spontaneously. In contrast, the reactivity of granular iron with oxygen is very slow, leading to slow oxidation (rusting) of the surface.

SOIL AND GROUNDWATER REMEDIATION WITH NANOPARTICLES

Environmental remediation methods can be classified as adsorptive or reactive and as in situ or ex situ (Tratnyek and Johnson 2006; Hodson 2010 this issue; TABLE 1). The use of nanomaterials in all these scenarios has been investigated. In soil and groundwater remediation, in situ applications seem to be most promising as they are in general less costly. For in situ treatment, it is necessary to create either an in situ reactive zone with relatively immobile nanoparticles or a reactive nanoparticle plume that migrates to contaminated zones. For applications in topsoil, nanoparticles can be worked into the surface of the contaminated soil using conventional agricultural practices. These different approaches are shown in Figure 2.

Soil and groundwater contamination are closely linked. Methods targeting soil contamination indirectly affect the quality of the groundwater, and vice versa. We therefore summarize the nanotechnologies used for soil and groundwater remediation. Little work is being carried out on topsoil remediation using nanoparticles.



CLASSIFICATION OF REMEDIATION METHODS INVOLVING NANOPARTICLES

| | In situ | Ex situ |
|------------|---|--|
| Adsorptive | In situ sequestration of contaminants by adding binding agents, e.g. iron oxides | Extraction of contaminated solution, which is then treated with adsorbents, as in nanofiltration |
| Reactive | In situ reaction of nanomaterial, e.g. nZVI, with target contaminant | Extraction of contaminated solution, which is then treated with reactants, as in TiO_2 photo-oxidation |

Contamination of soil and groundwater with carcinogenic organic substances and/or metals occurs all around the world. Groundwater sources are also frequently contaminated with pesticides or halogenated compounds. Landfill leakage, agriculture and chemical accidents are the main sources of these pollutants.

Conventional remediation technologies include ex situ soil washing and pump-and-treat operations, and in situ thermal treatment, chemical oxidation and use of reactive barriers with iron (Hodson 2010 this issue). Soil and groundwater remediation is generally very expensive, and conventional methods are not always successful or they take a long time for the remediation to become effective. Karn et al. (2009) state that pump-and-treat methods require 18 years of operation on average, compared to 1-2 years for a treatment with nZVI. New and more effective applications are therefore needed. TABLE 2 gives examples of the current use of nanoparticles in remediation. Photocatalysis could be used in a pump-and-treat operation to purify groundwater, but this article will focus on in situ soil remediation via adsorption and redox reactions.



Nanoscale zero-valent iron ignites spontaneously FIGURE 1 when it comes into contact with air. © AQUATEST, CZECH REPUBLIC

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Adsorption

TABLE 2

Iron oxides can strongly adsorb metals in soils. Adding nanoscale metal oxides to soils will thus immobilize soil metals (Schorr 2007). A mixture of iron and iron oxide has also been shown to be effective in phosphate removal – even more effective than higher-cost products such as activated alumina – while being active for even longer periods (Schorr 2007). Green rust – a very reactive iron oxide – can be used to reduce Cr(VI) to Cr(III), which is not soluble and much less toxic than the mutagenic Cr(VI) (Rickerby and Morrison 2007). Carbon-based nanomaterials, such as dendrimers and polymers, are also currently being explored for the removal of metals and organics from soils and groundwaters (Mueller and Nowack 2009).

Iron oxide minerals can be used to adsorb not only metals but also arsenic (Rickerby and Morrison 2007). Arsenic in groundwaters in the Bengal region of Southeast Asia and elsewhere constitutes a major hazard to the health of millions of people who use these waters for drinking, cooking and irrigation (Smedley and Kinniburgh 2002). Iron oxide NPs can bind arsenic 5–10 times more effectively than larger particles (Rickerby and Morrison 2007). In laboratory tests, more than 99% of the arsenic in water was bound by 12 nm diameter iron oxide nanoparticles.

EXAMPLES OF THE USE OF NANOPARTICLES IN REMEDIATION

| Process exploited | Nanomaterials used | Target compounds |
|----------------------|--|---|
| Photocatalysis | TiO ₂ | Organic pollutants |
| Adsorption | Iron oxides, dendrimers | Metals, organic compounds, arsenic |
| Redox reactions | Nanoscale zero-valent iron (nZVI), nanoscale calcium peroxide | Halogenated organic compounds, metals, nitrate, arsenate, oil |

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FIGURE 2 In situ technologies used to treat polluted ground-water and soils: (1) injection of nZVI to form a reactive barrier; (2) injection of mobile nZVI to form an nZVI plume; (3) incorporation of NP into topsoil to adsorb or degrade pollutants

This method is thus some 2500 to 25,000 times more efficient than current systems, at least at the laboratory scale.

A new study by Pan et al. (2010) found that nanoscale magnetite particles can be used successfully to immobilize phosphate in soil by adsorption. They compared the immobilization efficiency of micro- and nanoscale particles stabilized by a coating of carboxymethyl cellulose and found that only nanoscale particles were able to penetrate the soil column. Non-stabilized 'nanomagnetite' could not pass through the soil column under gravity because it quickly agglomerated into microparticles. The coated NPs were more transportable due to their small size and the increased negative charge associated with the carboxymethyl groups. Consequently, a large proportion (72%) of the coated NPs could pass through the column due to their charge repulsion with the negatively charged soil particles. Transport over a certain distance is needed to achieve a good distribution of the reactive particles in the soil matrix and therefore an even adsorption capacity for phosphate.

Much research has been conducted on nanosorbents, and although the results are promising, there are only a few commercial applications so far. The problems with using nanosorbents in soils are similar to those encountered with conventional adsorbents (O'Day and Vlassopoulos 2010 this issue).

Redox Reactions

Nanoscale Zero-Valent Iron (nZVI)

The use of nZVI in groundwater remediation is the most widely investigated environmental nanotechnological technique (Nowack 2008) and has considerable potential benefits (Tratnyek and Johnson 2006; Klimkova et al. 2008). Field-scale commercial applications of nZVI have already become common in the United States (Karn et al. 2009), but there have been only a few projects in Europe so far (Mueller and Nowack 2010). A web-based list of the sites where nZVI has been applied is available at www. nanotechproject.org/inventories/remediation_map/.

Competition among suppliers (Tratnyek and Johnson 2006) has led to a significant drop in the price of nZVI materials. nZVI can be produced by top-down (milling) or bottom-up (chemical synthesis) processes (Li et al. 2006). Due to its high reactivity (FIG. 1), nZVI must always be handled as a slurry (FIG. 3). This requires some additional infrastructure above ground because the suspension has to be remixed immediately before its injection (FIG. 4).

There are two ways to use nZVI in groundwater and soil remediation (Tratnyek and Johnson 2006; Nowack 2008), as shown in FIGURE 2.

- nZVI is injected to form a reactive barrier of iron particles.
- nZVI is injected in surface-modified form (e.g. coated with polyelectrolytes, surfactants or cellulose/polysaccharides) to establish a plume of reactive iron, which destroys any organic contaminants within the aqueous phase.

Several studies have shown that nZVI as a reactive barrier is very effective in the reductive degradation of halogenated solvents, such as chlorinated methanes, brominated methanes, trihalomethanes, chlorinated ethenes, chlorinated benzenes and other polychlorinated hydrocarbons, in groundwater (Zhang 2003; Schorr 2007). nZVI has also been shown to be effective against pesticides and dyes (Zhang 2003). Efficient removal by nZVI of polycyclic aromatic hydrocarbons (PAHs) adsorbed to soils has been reported at room temperature (Chang et al. 2005, 2007), while under the same conditions only 38% of the polychlorinated biphenyls (PCBs) were destroyed because of the very strong sorption of PCBs to the soil matrix (Varanasi et al. 2007).

According to Tratnyek and Johnson (2006), nZVI used in real-world groundwater remediation has a particle size larger than 100 nm and is thus strictly speaking outside the standard definition of NP size. These authors also state that the mobility of nZVI will be less than a few metres under almost all relevant conditions as nZVI tends to aggregate, producing clusters that may approach several micrometres in size and thus be easily removed from the porewater. Companies are therefore functionalizing nZVI particles to stop them from aggregating, for example, with



FIGURE 3 nZVI slurry being poured into a well. © AQUATEST, CZECH REPUBLIC



FIGURE 4 Mixing station where the iron particles in the slurry are resuspended before injection. © ALENCO GMBH, GERMANY

surfactants or polymers. Other approaches combine the nZVI with carbon platelets or embed the nZVI in oil droplets to facilitate particle delivery into the contaminated area (Mueller and Nowack 2010). In the United States, it is common to combine the nZVI with other metals, such as palladium, to increase the reactivity. In Europe such bimetallic particles are not used due to their possible toxicity and the limited additional benefit (Mueller and Nowack 2010).

Tratnyek and Johnson (2006) state that high reactivity tends to correlate with low selectivity. For this reason, remediation with nZVI may be inefficient because nZVI particles may react with non-target substances, including dissolved oxygen, sulphate, nitrate and water. This also implies that nZVI will have a limited lifetime in porous media and reinjections of nZVI may be necessary, which makes the treatment more costly. On the other hand, the short in situ lifetime of nZVI may be beneficial by preventing unwanted exposure of the environment and humans to nZVI.

A field test at a site in Germany contaminated by chlorinated hydrocarbons was carried out by the Federal Institute for Geosciences and Natural Resources (Houben et al. 2006). Injection of nZVI particles caused significant changes in redox chemistry, while other physico-chemical and hydrochemical parameters of the groundwater were not negatively affected. The limited penetration depth and lateral dispersion of the nZVI resulted in the clean-up of only a small part of the test site. Results from other pilot sites have shown that the oxidation–reduction potential in a soil decreases significantly following nZVI addition, while the pH increases slightly (US EPA 2005).

In the United States, many of the contaminated sites are on military bases. The US Navy and NASA have used nZVI in remediation for a number of years and have had positive experiences. Information on different case studies (supervised by government bodies) and background information on nZVI can be found in US EPA (2005) and Mach (2004). Examples of stages in the field application of nZVI are shown in Figures 4, 5 and 6.

The use of nZVI has been shown to be successful in the remediation of groundwater in porous soils. However, not much is known about the efficiency of the treatment in soils that are not saturated with water. The main challenge



FIGURE 5 Injection wells at a contaminated site in the Czech Republic. © AQUATEST, CZECH REPUBLIC

to date is the limited mobility of the particles. The actual efficiency of nZVI remediation depends on the geo- and hydrochemistry of the site.

Nanoscale Calcium Peroxide

Nanoscale calcium peroxide has recently been used for the clean-up of oil spills (Karn et al. 2009). Several projects have been conducted in New Jersey, USA. Two American companies are using nano-sized calcium peroxide as an oxidant in the remediation of soils containing various organic contaminants, such as gasoline, heating oil, methyl tertiary butyl ether (MTBE), ethylene glycol and solvents. Nanoscale calcium peroxide is claimed to be highly efficient in removing aromatics and is also used in enhanced bioremediation. The oxygen produced in the reaction of calcium peroxide with water leads to an aerobic environment that supports natural bioremediation by aerobic organisms present in the soil.

Risks Associated with the Use of Nanomaterials

Decreasing particle size and increasing reactivity are properties that may render a substance more toxic. Thus the very properties that make nanoparticles technologically interesting may place them in a novel category of potentially toxic substances. A number of studies exist concerning the toxicity of nanoparticles. Generally these studies suggest that smaller particles are more toxic than larger ones, although large differences among different particle types exist (Oberdörster et al. 2007). It is thus not possible to easily extrapolate toxicity data from bulk material to nanoparticles.

Nanoparticles may be ingested, inhaled or taken up through the skin. Several studies have shown that nanosized particles can be taken up by a wide variety of mammalian cell types (Oberdörster et al. 2007). However, much is still unknown about the toxicity of nanoparticles to humans and the environment. Factors such as composition, structure, molecular weight, the melting/boiling point, the octanol-water partition coefficient, water solubility, activity, the particle aggregation/disaggregation potential, and surface coatings and structure may be important variables influencing the toxicity of different particles and their behaviour in the environment (Nowack and Bucheli 2007). The issue of toxicity and ecotoxicity is especially important for all nano-remediation techniques because in most of them free NPs are directly added to soil or groundwater, and thus environmental exposure definitely occurs.

Only a few studies are available on the toxicity of nZVI. Li et al. (2009) found that nano-iron can induce oxidative damage in fish embryos and disturb the antioxidative balance in fish adults. The risk associated with nZVI depends not only on its toxicity but also on the extent of exposure. Tratnyek and Johnson (2006) have concluded that exposure to nZVI will be minimal due to rapid agglomeration of the particles, limited particle mobility and fast oxidation to iron oxide. Phenrat et al. (2009) investigated this nZVI "ageing" effect and found that 11-month-old nZVI was less toxic to rodent microglia (a type of cell in the brain and spinal cord) and neurons than fresh nZVI. Fresh nZVI induced mitochondrial swelling and apoptosis (controlled cell death) and reduced the levels of adenosine triphosphate. They further found that the redox activity of aged nZVI was minimal and that fresh nZVI tended to sediment and agglomerate faster than aged nZVI.



FICURE 6 An nZVI slurry is pumped into the wells at a contaminated site in the Czech Republic. © AQUATEST, CZECH REPUBLIC

CONCLUSIONS

Nanoparticles can potentially be used for the remediation of soil and groundwater. The small particles are highly reactive and have great sorption capacity. However, technical challenges, such as the delivery of the particles to the target area, have to be solved. There are also concerns regarding the release of large quantities of manufactured nanoparticles into the soil prior to extensive human and ecological toxicity testing.

So far nZVI is the only application of nanomaterials in soil and groundwater remediation that has been successfully commercialized – at least in the United States. Possibly in a few years, remediation with nanoscale calcium peroxide will also be common, whereas soil and groundwater remediation methods relying on the adsorption of contaminants to nanoparticles are still far from market.

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