Abstract. Research about the fate and behavior of engineered nanoparticles in the environment is despite its wide applications still in the early stages. The fast-growing area of nanoparticle research and the high level of uncertainty create a big challenge for describing clearly the recent state of the current scientific knowledge. Therefore, in this study the certain knowledge, the known uncertainties and the identified knowledge gaps concerning mobility of engineered metal oxide nanoparticles in porous media are analyzed. The mobility of nanoparticles is mainly investigated in model laboratory studies under well-defined conditions, which are often not realistic for natural systems. In these model systems, nanoparticles often retain in the pore system due to aggregation and sedimentation. However, under environmental conditions, the presence of natural organic matter may cause stabilization or disaggregation of nanoparticles and favors therefore higher mobility of nanoparticles. Additionally, potential higher mobility of particles using preferential flow paths is not considered. Knowledge of the long-term behavior of nanoparticles concerning disaggregation, dissolution or remobilization in soils under environmental conditions is scarce. Scientific uncertainty itself is rarely mentioned in the research papers. Seldom known methodically uncertainties in nanoparticle characterization are referred to. The uncertainty about the transferability of the results to environmental conditions is discussed more often. Due to the sparse studies concerning natural material or natural pore systems, certain conclusions concerning the mobility of nanoparticles in the soil environment are not possible to drawn.

Keywords: nanoparticles, metal oxide, porous media, soil, environmental research, scientific uncertainty

1. Introduction

“There is a high level of uncertainty in the research concerning the fate and behavior of nanoparticles in the environment” is often stated. Research about the fate and behavior of engineered nanoparticles in the environment is despite its wide applications still in the early stages. As far as is known, no engineered nanoparticle has been directly detected in the environment [1]. Up to now, any risk assessment failure because of the limited knowledge and the huge uncertainties about the expected environmental concentration of engineered nanoparticles in the environment [2]. Scientific uncertainties can be considered from different perspectives. One possibility is to distinguish between (a) the uncertainties related to the basic research like the measuring uncertainty, not yet validated, contradictory, inconsistent, or not reproduce able data and (b) the uncertainties related to applied research questions as e.g. in the risk assessment like transferability of model data or data from model systems to environmental systems, the uncertainties in scaling issues, e.g. spatial heterogeneity or lack of reliable data. Of course, there are several more definitions and perspectives of uncertainties, e.g. in psychology, ethics and social sciences. For this meta-analysis, these two perspectives of scientific uncertainty will be considered.
The fast-growing area of nanoparticle research and the high level of uncertainty create a big challenge for describing clearly the recent state of the current scientific knowledge [3]. One way to analyze the knowledge of a specific topic is to distinguish between certain and uncertain knowledge and knowledge gaps. First descriptions of the uncertainty of the behavior and effects of nanoparticles in the environment includes investigations of the measurement uncertainties in characterizing nanoparticles with different methods [4, 5] and scientific research on environmental, health, and safety risks [6]. Uncertainties have primarily been observed in the areas of test conditions, effect estimations, characterization, and exposure of nanoparticles [6]. However, this list remains descriptive and does not allow for any conclusions about the kind, sources or consequences of these uncertainties.

Metal oxide nanoparticles are because of their expected toxicity and high production volumes one of the nanoparticle classes, from which the main risk for the environment is expected [2]. One way nanoparticles enter the environment is via the industrial and house-hold wastewater to the effluents of sewage plants and through sewage sludge [7-10]. The nanoparticles can then reach aquatic and terrestrial environments [8]. Soils may play a key role for the fate and behavior of nanoparticles, because soils can act as nanoparticle-sink as well as nanoparticle-source, e.g. for groundwater, organisms, and plants [3]. Because one key parameters of the fate of nanoparticles in the environment is their mobility, the meta-analysis was carried out in order to characterize the certain knowledge, the known uncertainties and the knowledge gaps in the field of metal oxide nanoparticle transport in porous media.

2. Material and Methods

To characterize the knowledge about metal oxide nanoparticle transport in porous media, the literature was screened in March 2012 for peer-reviewed research articles dealing with experiments of the topic “transport of metal oxide nanoparticles in porous media”. Searches were carried out in Web of Science with search terms like nano*, transport, deposition, porous media, soil. Searching for the terms nano*, (transport or mobility or retention) in the article topic leads together with the term porous media to over 600 results, together with the term soil to around 400 results. Scanning these articles, research papers dealing with metal oxide nanoparticle transport in water saturated porous media like glass beads, sand or soil material and only one paper per author were selected. After this specification, 13 papers were left for the meta analysis, which are shown in Table 1 (next page).

The meta-analysis was carried out concerning the following topics: nanoparticle characteristics (type, coatings, size), porous medium (material, grain size, porosity), experimental conditions (ionic strength, pH, particle concentration, presence of organic substances, Darcy velocity, application time), measurement methods of particle size and effluent concentration, considered models, presented results and drawn conclusions, mentioned or discussed uncertainty related to transport of nanoparticles in the environment.

3. Results and discussion

3.1. Nanoparticle characteristics

The nanoparticle characteristics used in the selected studies are summarized in Table 2. Most studies used titanium dioxide or zinc oxide as studied nanoparticles. In three studies, more than one nanoparticle type was investigated. Titanium dioxide and zinc oxide nanoparticles were chosen because they are the most widely used metal oxide nanoparticles, which are present in a variety of applications and consumer products (e.g. sunscreens and paints) with high production volumes. This is about 3000 respectively 550 t/year TiO₂ and 550 respectively 55 t/year ZnO worldwide and in Europe [11]. It is expected that the ongoing use of both nanoparticles lead to relevant inputs of 1,28 µg TiO₂/kg·year and 0,09 µg ZnO/kg·year (mode values) and a concentration of 0,4 – 4,8 µg TiO₂/kg in soils in Europe [9, 10]. Other metal oxide
nanoparticles as aluminum oxide or iron oxide nanoparticles are produced in lower amounts, both about 55 t/year worldwide [11]. According to Som and colleagues, TiO₂ and ZnO are belonging to the most environmental relevant nanoparticles [12].

Table 1: Selected peer-reviewed research articles in alphabetic order dealing with the topic of transport of metal oxide nanoparticles in porous media

| first author | year | article title | journal |
|--------------|------|---------------|---------|
| Ben-Moshe [13] | 2010 | Transport of metal oxide nanoparticles in saturated porous media | Chemosphere |
| Chen [14] | 2011 | Transport and Retention of TiO₂ Rutile Nanoparticles in Saturated Porous Media under Low-Ionic-Strength Conditions: Measurements and Mechanisms | Langmuir |
| Chowdhury [15] | 2011 | Mechanisms of TiO₂ nanoparticle transport in porous media: Role of solution chemistry, nanoparticle concentration, and flow rate | Journal of Colloid and Interface Science |
| Fang [16] | 2009 | Stability of titania nanoparticles in soil suspensions and transport in saturated homogeneous soil columns | Environmental Pollution |
| Godinez [17] | 2011 | Aggregation and transport of nano-TiO₂ in saturated porous media: Effects of pH, surfactants and flow velocity | Water Research |
| Jiang [18] | 2012 | Transport and deposition of ZnO nanoparticles in saturated porous media | Colloids and Surfaces A |
| Joo [19] | 2009 | Influence of Carboxymethyl Cellulose for the Transport of Titanium Dioxide Nanoparticles in Clean Silica and Mineral-Coated Sands | Environmental Science & Technology |
| Kanel [20] | 2011 | Influence of pH on the transport of nanoscale zinc oxide in saturated porous media | Journal of Nanoparticle Research |
| Lecoanet [21] | 2004 | Laboratory assessment of the mobility of nanomaterials in porous media | Environmental Science & Technology |
| Li [22] | 2011 | Transport and deposition of CeO₂ nanoparticles in water-saturated porous media | Water Research |
| Petosa [23] | 2012 | Transport of two metal oxide nanoparticles in saturated granular porous media: Role of water chemistry and particle coating | Water Research |
| Solovitch [24] | 2010 | Concurrent Aggregation and Deposition of TiO₂ Nanoparticles in a Sandy Porous Media | Environmental Science & Technology |
| Zhao [25] | 2012 | Transport and Retention Behavior of ZnO Nanoparticles in two Natural Soils: Effect of Surface Coating and Soil Composition | Journal of Nano Research |

About half of the studied nanoparticles were uncoated, bare particles when introduced in the porous system. The other half was coated with various organic polymers, organic acids or surfactants. In technical applications, most nanoparticles are functionalized [26]. However, the characteristics of released nanoparticles are unclear [1]. Released nanoparticles may be embedded into or sorbed to a matrix [26]. They may have their engineered coating and aged coating [27], no coating or will become a substitute coating of e.g. natural substances after released in the environment. Summarized, the surface properties of engineered nanoparticles released and transformed or aged in the environment are still unclear [26, 28, 29]. Coatings may have a tremendous effect via charge or steric stabilization on the stability and therefore on the aggregation behavior and the mobility of nanoparticles [1, 24, 30-33]. Therefore, one important factor of the mobility of nanoparticles, the exact surface properties under environmental conditions, is not known [29].
Table 2: Selected nanoparticle characteristics used in the selected research studies

| character     | nanoparticle specification (quantity)                                                                 |
|--------------|-------------------------------------------------------------------------------------------------------|
| type         | TiO₂ (9), ZnO (5), Fe₂O₃ (1), CuO (1), CeO₂ (1)                                                     |
| coating      | without coating (6), CMC (2), SDBS (1), Triton X-100 (1), acetic acid (1), dispersant and polishing additives (1), polymer (1), citrate (1) |
| primary size | < 10 nm (3); 10-49 nm (11); 50-99 nm (3); > 99 nm (3)                                               |
| size in suspension | < 100 nm (9); ~100-500 nm (14); ~500-1000 nm (3); >1000 nm (2)                                    |

Nanoparticles are commonly defined as particles with at least two or three dimensions between 1 and 100 nm (e.g. [1, 34]). The primary sizes of the nanoparticles used in these studies are according to the manufactures or determined by microscopic methods mainly in this range. However, the nanoparticle sizes in the experimental suspensions with ionic strengths in the environmental range were about one order of magnitude bigger. One reason could be the size measurement with DLS, where the hydrodynamic radius is determined. But mainly the bigger size is due to aggregation. These studies are determining therefore transport of aggregated nanoparticles.

3.2. Porous media

Selected characteristics of the porous media are summarized in Table 3. The porous media used are mainly well-defined systems as glass beads and clean industrial sand. Only in two studies, real soil material was used. By using defined mineral surfaces (SiO₂), uniform and defined surfaces are constructed. In environmental systems, a big variety of different mineral surfaces concerning charge and surface functional groups contribute unspecific and specific sorption sites, which affect the retention of nanoparticles in soils. By using clean silica sand or glass beads, these factors are neglected. Additionally, by excluding any natural organic matter from such model systems, an important stabilizer for particles (natural colloids as well as engineered nanoparticles) is not present in these systems, which is always present in natural systems and may increase the mobility of nanoparticles in soils [31, 32, 35-37].

The grain sizes in the studies with glass beads and sand are ranging from fine sand to coarse sand with a respective porosity of the packed material. In such model systems, well-defined pore systems can be established, which allows generally a high reproducibility of the experimental data. By modeling these data, specific transport mechanism could be analyzed. However, in natural system, a great variety of particle sizes and shapes are present. Additionally only sparse data for smaller fractions like silt and clay or mixtures are available; and no data exist with aggregated material, secondary pore systems, or biological aggregates. Cornelis and colleagues assumed a colloid-mediated nanoparticles transport in soils [38], so smaller grain sizes in transport experiments should be taken into account.

Table 3: Selected characteristics of the porous media used in the selected research studies

| character     | porous media specification (quantity)                                                                 |
|--------------|-------------------------------------------------------------------------------------------------------|
| material     | glass beads (2); clean silica sand (9); soil material (2)                                            |
| average grain size | < 250 μm (2); 250-500 μm (6); 500-750 μm (5); > 750 μm (2)                                        |
| porosity     | < 0.30 (3); 0.30-0.39 (6); 0.40-0.49 (5); > 0.49 (2)                                               |

It is uncertain, if results of such model systems can be transferred to natural systems, when such important factors like different mineral compositions, a broad range of grain size and porosity, presence of natural organic matter, colloid-facilitated transport and preferential flow paths are not considered.
3.3. Experimental conditions
A summary of the experimental conditions of the selected studies is shown in Table 4. The ionic strengths used in the experiments are in the board range of the relevant aquatic and terrestrial systems. In some studies also the effects changes in the ionic strength are studied. This is also very probably for natural systems, e.g. in cases of heavy rainfall or flooding events. All studies conducted their experiments in a neutral pH range from 6-8, which is also most common for aquatic and terrestrial systems. Higher and lower pH values are also considered in some studies, which are relevant especially for soils, where pH values can be significant higher or lower. Darcy velocities ranging from below 0,01 up to over 1,00 cm/min, which is in the range which could be expected in natural soil systems.

Table 4: Selected experimental conditions used in the selected research studies

| character               | experimental set-up specification (quantity)                                                                                                                                 |
|-------------------------|---------------------------------------------------------------------------------------------------------------------|
| ionic strength          | most 0,1 - 100 mM NaCl (some studies used KCl, NaBr, NaNO₃), and / or 0,01 - 10 mM CaCl₂                              |
| pH                      | < 6 (5); 6-8 (13); > 8 (4)                                                                                           |
| particle concentration  | 0-10 (5); 11-50 (8); 51-100 (3); >100 (1)                                                                           |
| organic substance       | without (10), with soil organic substance (2), Aldrich HA (1)                                                        |
| Darcy velocity [cm/min] | Up to 0,01 (3); 0,11-0,50 (5); 0,51-1,00 (4); > 1,00 (6)                                                            |
| time of NP application  | 1-5 (5); 6-10 (5); > 10 (3)                                                                                          |

In most studies, the particle concentrations ranges between 5 and 50 mg/l, however, concentrations up to 800 mg/l were also used. The environmentally relevant concentrations for surface water for different nanoparticles are expected in the pg/l to low µg/l range [9, 10, 39]. Considering sewage sludge as a source for nanoparticles in soils, one has to anticipate with a load of mg/kg per year [9]. At high particle concentration, aggregation is favored and a lower mobility would be determined compared with conditions with lower particle concentration. One hint for the aggregation in the used nanoparticle suspension is the considerable higher particle size (in most cases one order of magnitude) in suspension compared to the primary size. Using particle concentrations in the range for the aquatic pathway of nanoparticles to soils is challenging, because the detection methods for most methods are not sufficiently low [9, 10, 28, 39]. However, it is questionable is the results are transferable to natural conditions with several orders of magnitude lower particle concentration.

Organic acid was added to the system only in one study. As discussed in the section 3.2, excluding natural organic matter, an important stabilizer for nanoparticles is omitted. It is assumed that organic molecules (short acids or fulvic and humic acids) will play an important role in stabilizing nanoparticles in environmental systems [31, 32, 35-37].

The period of time of the nanoparticle applications ranged between 1 and mostly 10 pore volumes. This seems an appropriate time for model studies. Only a few studies considered subsequent remobilization steps, which could be relevant for environmental systems, e.g. in case of heavy rainfall or flooding events. No long time behavior is considered.

3.4. Measurement methods
Selected measurement methods used in the studies are presented in Table 5. The effluent concentration was mostly measures by UV-vis or ICP-MS, -AES, or –OES. Measuring with ICP-MS, -AES, or –OES, the total concentration is given as result. No information is available about the distribution between particular
and dissolved form. It had been shown that from UV-vis spectra, nanoparticle size and concentration can be determined [40]. However, most studies indicate only the particle concentration in the effluent. No further information of a possible change in particle size or the dissolution of nanoparticles, or a possible aging of nanoparticle coating was given. Additional experiments determining these parameters in a corresponding suspension and in the effluent could strengthen such data and their interpretation, but this is only partly done.

Table 5: Selected measurement methods used in the selected research studies

| character                        | methods (quantity)                                                                 |
|---------------------------------|------------------------------------------------------------------------------------|
| effluent NP concentration       | UV-vis (7); ICP-AES, ICP-OES and ICP-MS for Zn and Ti (5), Fluorescence Spectroscopy (1) |
| particle size in suspension      | DLS (13), NTA (1), laser diffraction (1)                                           |

The particle size in suspension is measured by DLS. With DLS, particle sizes from ~ 3 to 1000 nm in mg/l concentration range can be measured. Though particle size is calculated by calculating their movement from the measurement of the scattered light, many assumptions and simplifications have to be made, coming to a routine measurement result (detailed description see e.g. [39]). One of the assumptions is, that the measured particles are spherical, which should be proofed before the measurement. However, DLS was used for size determination also for non-spherical particles (e.g. [14]). Because DLS provide intensity based results, the bigger particles are overemphasized [34]. This results in difficulties in the interpretation of polydisperse suspension. Therefore giving the polydispersity index together with the particle size would be a help to interpret the results. With NTA, the particles size from 30 up to 1500 nm at lower concentrations (µg/l concentration range and lower) can be determined. However, if the particle concentration in the sample is too high, it have to be diluted, which alters its diffusion behavior and aggregation state [39]. Cross validation and additional specifications like size distribution or polydispersity index could strengthen the data, until better methods are available [34, 41].

3.5. Discussed theories and models

From the selected papers, in 11 studies transport theories and models are discussed and some specifications were calculated, as summarized in Table 6. Even if it is stated that the theories are not able to predict the transport behavior of nanoparticles [18], some specifications mainly from the classical colloid filtration theory were calculated and compared for different experimental conditions or for different nanoparticles. The interpretation of such results is difficult, because the models were not able to completely describe the observed nanoparticle transport in the model systems. The transfer to environmental systems is questionable because of the unconsidered factors as discussed above.

Table 6: Theories and models discussed and calculated specifications in the selected research studies

| theories and models                           | specification (quantity)                               |
|----------------------------------------------|-------------------------------------------------------|
| classical colloid filtration theory (Yao et al 1971, a.m.) | attachment efficiency (6), deposition rate coefficient (5), \( L_{\text{max}} \) (4), collector efficiency (3) |
| DLVO-theory                                   | interaction energy (4)                                 |

3.6. Main findings and conclusions

The main findings are summarized in Table 7. If investigated, a dependence of ionic strength, ion valence, pH value, presence of humic acid, surface coatings or surfactants were determined, which could be
expected from classical colloid theories like the colloid filtration theory [42, 43] and the DLVO theory. In
the conclusions, as transport determining mechanisms are mainly mentioned aggregation followed by
DLVO interactions, straining of particles, blocking effects and disaggregation. Further it is concluded that a
potential risk for groundwater exist, as well as that bare nanoparticles exhibited a very limited mobility.

Table 7: Main findings and conclusions of selected studies

| main results / conclusions | specification (quantity) |
|---------------------------|-------------------------|
| mobility/deposition/retention of nanoparticles in porous media depends on | ionic strength (10), ion valence (4), pH (8), presence of humic acid, surface coatings or surfactants (5), nanoparticles characteristics (2) |
| transport determining mechanisms | aggregation (4), DLVO interactions (3), straining (2), blocking (2), disaggregation (2), deposition (1), ripening (1), pore plugging (1) |
| potential risk for | groundwater (3) |
| limited mobility | for bare nanoparticles (3) |

3.7. Mentioned uncertainties

The mentioned uncertainties in the selected research papers are summarized in Table 8. Uncertainty itself is
rarely mentioned. More often the sources (e.g. lack of analytical methods) or consequences (e.g.
questionable transferability) of uncertainties are considered. Only in two studies, uncertainties related to
basic research are discussed. These are measuring uncertainties related to the size determination by DLS
and NTA and the discussion of reasons of contradicting results of studies with different nanoparticles. The
uncertainties related to applied research are mostly discussed in the conclusions or the sections dealing with
environmental consequences. Mainly the transferability of the results to environmental conditions is
discussed with different foci.

Table 8: Mentioned uncertainties in selected studies

| uncertainties | specification (quantity) |
|---------------|-------------------------|
| measurement uncertainties | size (1) |
| contradicting results | different characteristics of nanoparticles (1) |
| transferability to environmental conditions | without specification (4), due to presence of organic molecules (3), preferential pathways (2), high variability (1) |
| transferability to other nanoparticles | type (2) |

3.8. Certain and Uncertain Knowledge and Knowledge Gaps

Certain knowledge about the mobility of metal oxide nanoparticle in porous media is restricted to model
systems with a limited number of studies with a limited number of nanoparticles. Generally, nanoparticle
concentrations are used, which are much higher than the expected environmental concentrations [9, 10, 33].
The water saturated porous media consist mainly of clean sand without any natural organic matter or other
minerals and in absence of smaller grain sizes or any secondary pores. The identified factors controlling
the mobility are ionic strength, ion valence, pH, presence of humic acid, surface coatings or surfactants,
which could be expected from classical colloid theories. It is concluded, that various mechanisms as e.g.
aggregation and disaggregation, pore plugging and deposition are affecting the dynamic nanoparticle
transport, indicating that classical filtration theory often fail to describe completely the transport behavior
[14, 15, 17, 18].
The uncertainties in the fields of basic research could be found in the small amount of papers dealing with our topic, in the measurement uncertainty, the sparse characterization of the systems and the fragile validation of the results. Cross validation of particle size determination to validate the data is rarely done. By measuring the effluent concentration only, any possible changes in nanoparticle characteristics as dissolution, aggregation or transformation or aging of coatings are not considered. Because the mobility of different nanoparticles with different sizes and coatings differ significantly [13, 23, 25], comparability from one nanoparticle type to the other type or size or coating is questionable. Therefore, a broad validation of single findings is difficult to date. Defining reference materials and procedures would be helpful in order to compare results.

The uncertainties in the fields of applied research are mostly the transferability of the results to environmental conditions. Because most studies were conducted in the absence of natural organic matter, other minerals than silicium dioxide, smaller grain sizes or secondary pore systems which may affect the retention in soils and sediments, the transferability of these data from model systems to environmental systems is questionable. A good half of the authors mentioned or discussed this uncertainty in their papers. Major knowledge gaps are the unknown form and the unknown amount of engineered nanoparticles at their release in the environment and their transformation and aging under environmental conditions [1, 26, 33, 44]. The fact, that the surface characteristics, which greatly affect the nanoparticle mobility, are not known for environmental conditions, is discussed very rarely in the selected studies.

3.9. Sources and Consequences of Uncertainties and Knowledge Gaps

The main source of these uncertainties and knowledge gaps is caused by the lack of available methods to separate, characterize and detect engineered nanoparticles in environmental media at environmental concentrations [1, 26, 33, 45]. One of the biggest challenges is to separate and characterize the small amount of engineered nanoparticles in environmental matrices, which contain high amounts of highly heterogenic natural nanoparticles [34].

Because of the lack of analytical methods, the form of nanoparticles at release and the surface properties transformed and aged in the environment are not known [1, 26, 33, 44]. Therefore, one important factor of the mobility of nanoparticles in the environment, the exact surface properties, is still unclear [29]. The characteristics of the chosen bare or coated nanoparticles may be not relevant under environmental conditions.

As consequences of these uncertainties knowledge gaps, any risk assessment, regulation or management concerning engineered nanoparticles based yet on modeling data or studies in model systems, which transferability to environmental conditions is questionable as discussed above. Studies under more natural conditions are challenging to date.

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