
NANOTRANSPORT-Project



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NANOTRANSPORT

The Behaviour of Aerosols Released to Ambient Air from Nanoparticle Manufacturing
- A Pre-normative Study

Publishable final activity report

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<p>Title:</p> <p>Publishable final activity report</p> <p>- the NANOTRANSPORT project</p>					
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<p>Summary:</p> <p>The objective of the NANOTRANSPORT project was to investigate physical changes which NP aerosols undergo after release into the workplace environment under realistic scenarios. This information is essential to understand the characteristics of NPs when they reach a human receptor after transport over a distance from a NP source. Knowledge obtained from the study was used to develop recommendations to EC regarding:</p> <ul style="list-style-type: none"> - Test aerosols for nanotoxicology studies; - Testing of filters and protective equipment in the workplace; - Research priorities. <p>The final activity report provides brief information on major achievements and key project actions carried out during the whole project period from September 2006 to April 2008.</p>					
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Project summary

Project objectives

The NANOTRANSPORT project addresses the behaviour of aerosols released to ambient air from nanoparticle (NP) manufacturing. We intend to elucidate and document the need for standardised test aerosols adapted to the scope of nanotoxicology and occupational health studies.

The objective of NANOTRANSPORT was to investigate physical changes which NP aerosols undergo after release into the workplace environment under realistic scenarios. This information is essential to understand the characteristics of NPs when they reach a human receptor after transport over a distance from a NP source. Knowledge obtained from the study was used to develop recommendations to EC regarding:

- Test aerosols for nanotoxicology studies;
- Testing of filters and protective equipment in the workplace;
- Research priorities.

Project participants

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Work performed

During the whole project period from September 2006 to April 2008, we completed all the tasks proposed, including:

- ü a state-of-the-art study to identify critical parameters governing the behaviour of NP aerosols (responsible contractor: DNV);
- ü a scenario development workshop gathering leading experts to develop realistic exposure scenarios at NP manufacturing sites (responsible contractor: DNV/UniK);

- ü experimental work to generate test aerosols and investigate their behaviour released in an exposure chamber under different conditions defined in the exposure scenarios (responsible contractor: UniK/Grimm);
- ü recommendations to EC based on experimental results and conclusions (responsible contractor: UniK);
- ü Dissemination of project findings through presentations at national and international conferences, publications at scientific journals and the project website (responsible contractor: all partners).

Project website

<http://research.dnv.com/nanotransport/>

Key conclusions of the project

- | There is considerable evolution of nanoaerosols over time: their average size increases, while their concentration decreases;
- | Natural background aerosols are scavengers for NPs;
- | The time scale for size evolution depends on concentration and primary size of the NPs and that of the background aerosol - it may range from a matter of a few minutes up to half an hour;
- | Nanoparticles will be physically/chemically present in size classes other than those in which they were originally emitted;
- | Filtration efficiency for primary NPs < 80 nm is usually sufficiently high, but their agglomerates may be in the Most Penetrating Particle Size (MPPS) range of between 80-200 nm.

Impacts of the project results

- | The agglomeration dynamic of primary Pt nanoparticles released in a simulated workplace environment was thoroughly characterised in this study, using both experimental data and aerosol dynamic modelling. The results are of importance for defining standard test conditions for inhalation toxicological studies.
- | We observed that agglomerates formed under different scenarios of NP release (e.g. source concentration of NP, ratio of NP and background particles, continuous or discontinuous release, particle size) are different in terms of particle size distribution and attachment to ambient particles. Consequently, the test condition for inhalation toxicological study should be adapted to the specific release mechanisms in workplaces.
- | Due to fast agglomeration of NPs and attachment to the background aerosol, workers in manufacturing places are most likely exposed to agglomerates rather than primary nanoparticles. Information on toxicity of agglomerates and stability of agglomerates

are needed, in addition to the toxicity of NP in primary form.

- | As to monitoring of NP in workplace air, NP may not be detected in the size range where they were originally emitted; the size range of agglomerate should also be considered. Moreover, measurement equipments able to identify particle chemical composition need to be applied in order to detect NPs attached to background aerosols.
- | For testing of filter efficiency, not only the size range of primary NPs need to be tested, but also the size range of agglomerates, as our results show that agglomerates may be presented in size range of the most penetrating particle size (MPPS) of filters.

1 Project activities and main achievements

1.1 Development of model exposure scenarios

Exposure scenarios were proposed on the basis of the literature study, visits to relevant companies, experiences of the project partners and discussions with invited experts at the scenarios development workshop. By compiling all the information the project partners defined model exposure scenarios, and developed the experimental program accordingly.

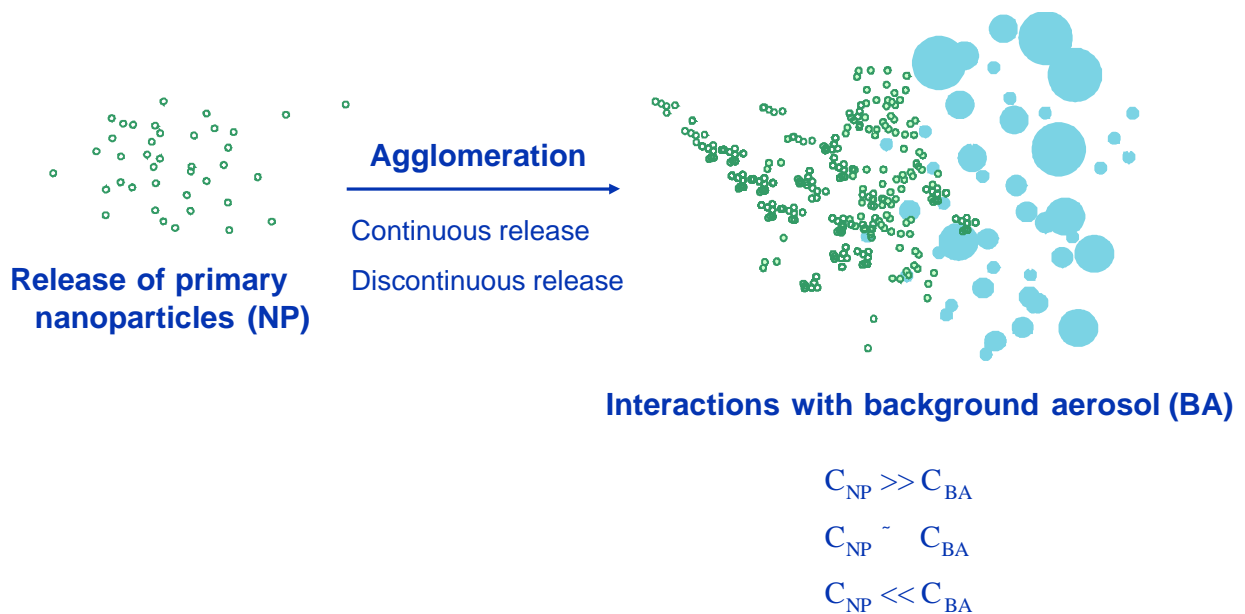


Fig. 1: Model exposure scenarios used for the study (C_{NP} : nanoparticle concentration, C_{BA} : background concentration)

The NANOTRANSPORT project focused on exposure scenarios related to release of primary nanoparticles (NP) from manufacturing. Model exposure scenarios described in Fig. 1 were used. Redispersión of powder formed NPs was not the scope of this study. When primary NPs

are released in ambient air, agglomeration may occur among NPs and also between NPs and background aerosols. Both a continuous release by leakage and a pulse-wise discontinuous release, e.g. in cleaning operations or in the case of an accident, were considered relevant. Background aerosols in workplace air are often presented in a concentration range which is similar or even higher than the engineered NPs detected. The agglomeration between background aerosols and engineered NPs at different concentration ratios were also included in the exposure scenarios. According to reports from literature the realistic concentration range of background aerosols is between 10^3 to 10^5 particles/ cm^3 .

Our experimental results of NP release in simulated workplace environment show that the proposed scenarios are highly relevant in occupational setting. Particle size distribution of released primary NPs evolves considerably. The time scale of size evolution is mainly dependent on the concentration of NP and background aerosols, in the order of few minutes to about an hour. At a lower concentration of $< 10^4$ particles/ cm^3 the agglomeration process of Pt ($d=10$ nm) is very slow, in the order of an hour, and therefore, workers may be exposed to primary NPs. When NP concentration is higher than 10^7 particles/ cm^3 , agglomeration occurs within seconds; even workers standing close to the source are very likely exposed to agglomerates rather than primary NPs. This conclusion is of importance for defining test aerosols for nanotoxicological studies.

The experiments also confirm that homogeneous agglomeration among NPs and heterogeneous agglomeration between NPs and background particles are the two main mechanisms affecting the particle size and number concentration. Which of the two mechanisms is prevailing in a given environment is dependent on the concentration ratio between NPs and background particles. When background concentration is very low, e.g. in clean rooms, homogeneous agglomeration is predominant. When the background concentration is higher than NP concentration, the normally larger background particles act as scavenger to NPs.

1.2 Selection of measurement metrics and modifiers

Literature study on critical parameters governing the behaviour of nanoaerosols was performed in order to select metrics and modifiers for the experimental programme. Identified parameters important for toxicological properties and aerosol dynamic behaviour are summarized in table 1.

Table 1: Selected metrics and modifiers important for nanotoxicology study and aerosol dynamics

Metrics

- Chemical composition
- Particle size distribution
- Agglomerate size and stability
- Morphology
 - Fractal dimension and aspect ratio
- Surface area
- Charge
- Surface properties
 - Reactivity, hydrophobicity, functional groups,
- Crystal structure
- Biopersistence
- Solubility
- Conductivity

Modifiers of metrics

- Residence time
- Concentration
 - mass, number, surface area
- Convective flows
- Humidity
- Temperature
- Existing background aerosols
- Sorption of organic molecules

As the NANOTRANSPORT project focused on the physical changes of NPs and on the agglomeration dynamic, spherical Pt NPs and background particles are selected as model test particles. Chemical changes of test NPs after release were not considered for this study. For on-line monitoring, particle size number distribution is regarded as the most important metric relevant for NP toxicology and aerosol dynamics. Particle surface area concentration, another important metric for toxicity, can be estimated from particle size number distribution. The use of spherical particles simplified the calculations. Off-line techniques were used for a thorough physical chemical characterisation of test aerosols generated at the source and after release in the exposure chamber. The selected modifiers in table 1 were controlled or varied in experiment to simulate workplace conditions.

Results of experimental work show that the key parameters controlling the evolution of NP size and number concentration with time are:

- the concentrations of NP release,
- the ventilation rate, the concentration and
- size of background particles.

Coagulations among NPs and between NP and background aerosols were identified as the two most important mechanisms changing particle size and particle number concentration. The attachment of NP to background aerosol can in addition induce a change in the chemical composition, which becomes different from the original NP at the source. Agglomeration stability turned out to be another important factor that may affect particle toxicity, as the agglomerates formed through Brownian collision are held together by weak van de Waal's forces and deagglomeration may occur in biological tissue.

1.3 Experimental results - Aerosol dynamics of nanoparticle in workplace exposure

An experimental program was designed to investigate aerosol dynamic behaviour of freshly generated test nanoparticles when released in an exposure chamber under different simulated workplace environment defined in model exposure scenarios.

Experimental set-up

Experimental set-up

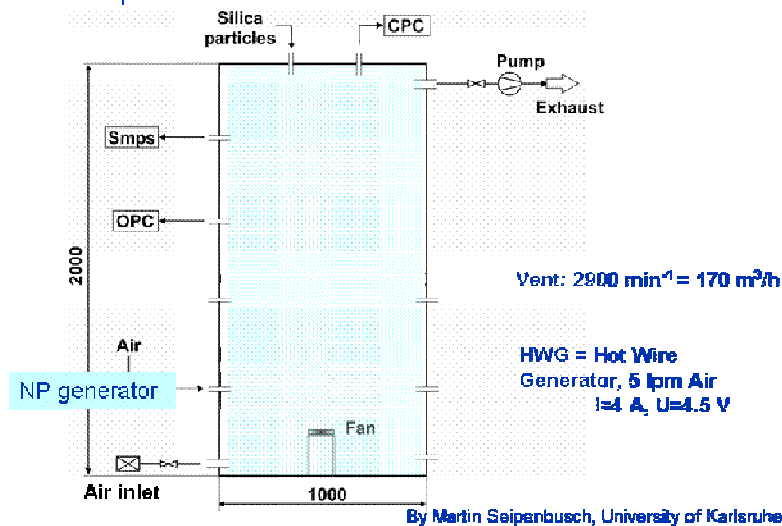


Fig. 2: Experimental set-up used for study the behaviour of nanoparticles after release in simulated workplace environments

The experimental set-up in Fig. 2 was built at UniK with technical and instrumental support from Grimm Aerosol Technik GmbH. Physicochemical properties of Pt nanoaerosol were characterized both at the source (Fig. 3) and after release in the exposure chamber. Particle-size distribution of NP aerosol is monitored on-line at different places in the chamber. One example is given in Fig. 4 on time evolution of particle size distribution after discontinues release in a clean exposure chamber. Parameters, such as, convective flow rate, concentrations of NPs and background aerosols were varied to simulate different exposure scenarios. Using the designed experimental setup and monitoring equipment, the agglomeration procedure of freshly generated NP was quantitatively documented in number size distribution.

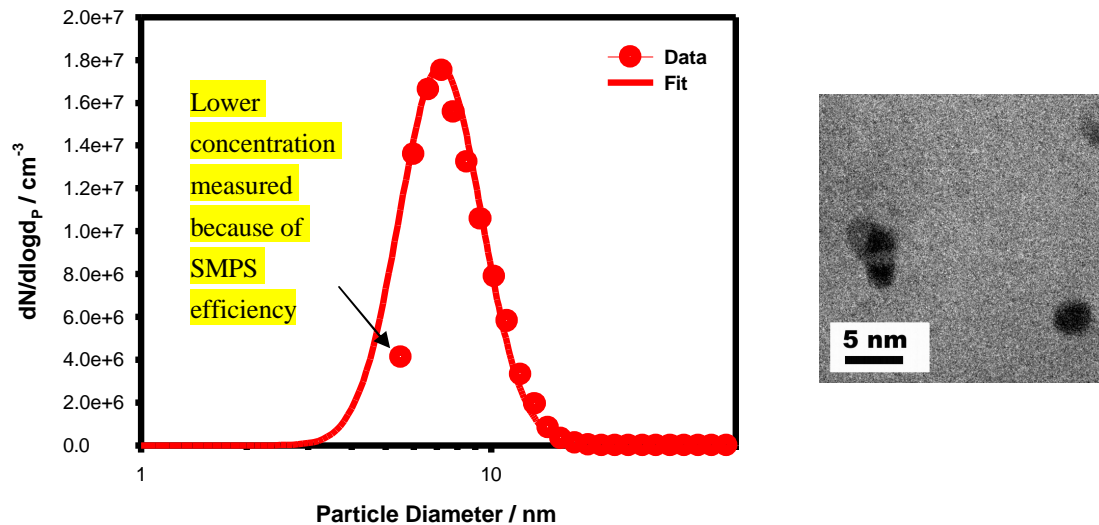


Figure 3: Particle size distribution (left) and TEM image (right) of the generated Pt particles.

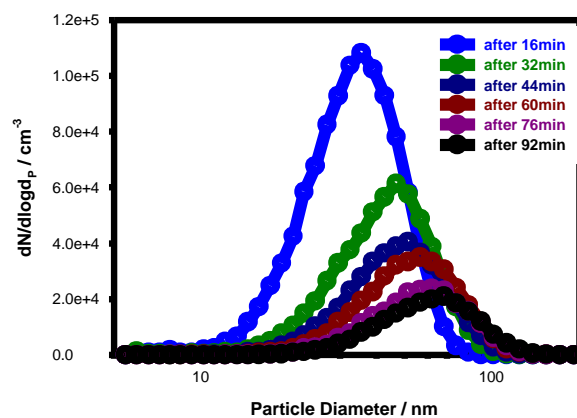


Fig. 4: Size distributions of the Pt particles after release in a clean exposure chamber, measured with scanning mobility particle sizer (SMPS).

Major experimental results

The experimental results, together with our general knowledge background of aerosol science, lead to a number of key conclusions regarding the evolution on nano-aerosols with time. Nano-aerosols emitted from an NP source evolve considerably with time, both due to auto-agglomeration and/or due to attachment to pre-existing background aerosol particles. Which one of these two mechanisms will prevail depends on the given workplace scenario (i.e. the strength of a primary NP source relative to background concentration, and the elapsed time during transport from source to receptor). For simplicity and without limiting the conclusions, we disregard the attachment of NP to macroscopic surfaces such as “walls” or workbenches. The influence of NP deposition is under 14% as shown by our experimental results.

Aerosol dynamic processes are driven largely by concentration, with size (or size differences) being a relatively minor factor. If the NP concentration is high compared to the typical background concentration, then auto-agglomeration will initially dominate the evolution of

size and concentration, forming a significant new population of agglomerates NP. If source and background are comparable in concentration, then both mechanisms are likely to run concurrently, producing both agglomerates of NP and mixed particles; if the source is relatively weak, the scavenging will prevail.

Both mechanisms cause the average particle size to increase significantly on a time scale ranging from minutes to about an hour, while the number concentration decreases, first rapidly and with decreasing concentration more slowly. The auto-agglomeration process stabilizes typically in the 100-200 nm size range, where collision processes slow down. The attachment of small NP to relative larger background aerosol particles leaves the size of the latter practically unchanged. The scavenged NPs therefore merge into the ambient aerosol between about 0.1 and 1 μ m. The attachment of NP to pre-existing background aerosols as well as auto-agglomeration thus lead to the physical/chemical presence of NPs in size classes well above the one in which they were originally produced and emitted.

The time scale for size evolution depends on concentration and primary size of the NPs and that of the background aerosol - it may range from a matter of a few minutes up to an hour. An aerosol dynamic model was derived based on coagulations through Brownian collisions among NPs, as well as between NP and background aerosols, described using Smoluchowski equation. The model was applied to fit the experimental data. The good agreement between experimental data and model calculation demonstrates the possibility to predict the concentration of NP in a given workplace environment.

The aerosol agglomeration and/or attachment process is driven predominantly by thermal ("Brownian") collisions. Hence the adhesion forces within the agglomerates will be mostly of the van der Waals type and thus fairly weak. Adhesion within an agglomerate may be enhanced by adsorption of water vapor; however this should not affect its behavior once it becomes immersed into cellular fluid.

Filtration of nanometer sized aerosol particles can be done with high efficiency using state of the art filter media. The effectiveness of filters for NP increases continually down to sizes on the order of 1-2 nm. However, the shift of nanoparticles to larger size classes by the two above-mentioned mechanisms may transfer them into the size range where filters are least efficient, the most penetrating particle size (MPPS), typically in the range of about 80-200 nm.

1.4 Recommendations

Recommendations are developed based on experimental results of the study, taken into account the accepted practices in production and use of NPs and relevant research needs described in literature.

Suitable test aerosols for nano-toxicology studies

Based on the results of the study, two scenarios must be considered in emulating exposure to NP in the workplace. In one case, the receptor is located immediately at the source of a NP aerosol (the nose near the "leak", so to speak); in the other, the receptor is located further away (e.g. a person working elsewhere in the lab). Scenario 1 requires testing with primary, un-agglomerated NP, while Scenario 2 requires aged aerosols, either auto-agglomerated or

adhering to neutral particles, or both. Scenario 2 may well be the more prevalent case for many exposure situations. Studying the deposition and effects of “aged” nano-aerosols is thus at least as important as those of un-agglomerated particles from a primary NP source.

A point of clarification with regard to the above mentioned scenarios: the range within which a receptor will likely be exposed to predominantly un-agglomerated NP (the “near-field exposure” radius) can be expressed in terms of the average distance traveled by the aerosol during the time required for the primary aerosol to become significantly agglomerated. The “near-field” exposure radius is thus a function of the primary emitted concentration (hence the agglomeration rate) as well as air velocity. Analogous considerations apply to the far-field exposure scenario.

For either scenario it becomes critical to control the state of agglomeration of the particles at the receptor: either as little agglomeration as possible, or a significant but defined state of agglomeration with corresponding, well defined structural characteristics of the aged particles.

The state of agglomeration matters of course for exposure assessment studies, because deposition is governed by aerosol particle size. The agglomeration stability and sizes are also important for toxicological studies. Currently our knowledge about the toxicological effects of the NP agglomeration is rather limited. Further research need to be done to investigate the potential for deagglomeration in cellular environments, or how the structure of the airborne agglomerate carries over into the liquid state.

Liquid NP suspensions or pre-prepared powders are generally difficult to aerosolize with controlled particle sizes. Particle structures and agglomerate strengths obtained via redispersion are generally not representative of those formed in the aerosol state, except in cases where NP redispersion is the release mechanism to be investigated.

For exposure and toxicology studies it is therefore recommended to produce and age NP directly via on-line aerosol processes, including aging the presence of a relevant background aerosol. Aerosol processes best ensure NP structures and size distributions representative of workplace releases into the air. Only after the effects of agglomerate structure in cellular tissue environments, the transport processes of agglomerates in such environments, possible deagglomeration mechanisms etc. have been understood may it become possible to relax these requirements.

Testing of filters & protective equipment

The mechanisms of particle filtration in air are well understood. In particular we know that the efficiency of filters increases steadily with decreasing particle size in the submicron range. Recent studies confirm this down to sizes of about 1 or 2 nm. We can thus conclude that a filter deemed sufficient for larger particles will be even more effective in capturing “true” NP (<10 nm).

On the other hand, the growth of aged NP by auto-agglomeration or their attachment to ambient background particles can shift their size into the range of the Most Penetrating Particle Size (MPPS) of typical filter media, which is typically in the range of 80 - 200 nm. If adequate protection against nano-aerosols is required, it becomes critical to assure adequate filter efficiencies also in the MPPS region.

The methods for filter testing, and in particular for testing at the MPPS, are well developed and have been incorporated into various standards; aerosol generation and testing equipment for the submicron size range such as mobility spectrometers and CPCs are also available on the market. Mobility spectrometers classify irregularly shaped particles by their mobility size, which is also the relevant parameter for diffusion deposition in filters.

The chemical or surface composition of the particles is of no importance for the deposition efficiency in filters and other protective equipment and therefore, does not have to be considered in the choice of test aerosols.

A notable exception to this statement are nano-fibers and carbon nano-tubes (CNTs), which by virtue of their highly non-spherical shape do not behave in the same way as isometric particles during deposition in a filter. Filtration behavior of fibrous particles merits a more detailed investigation. The agglomeration state of CNTs is again critical for such tests.

Metrology of nano-aerosols

The fact that aging nano-aerosols can move up the size scale rapidly by various agglomeration processes has important implications in choosing suitable metrological strategies for purposes of workplace assessments.

Aged NP aerosols are not recognizable by a specific particle size range, such as " <10 nm". Moreover, it is not sufficient to look for NP in the "nano size range" where they may have originally been generated/emitted, unless one measures near a primary source. Agglomerated or scavenged NP will populate a size range where they become nearly indistinguishable from ubiquitous background aerosol by straightforward size distribution measurements. It is well known that additional ENP concentrations are often marginal, unless there are gross emission sources.

One alternative are highly selective real-time (!) analytical techniques with some kind of species sensitivity. This would allow detecting the presence of a NP even when it is attached to other particles. Today such methods are not broadly available and those which exist, such as single particle mass spectrometry or aerosol catalysis, are untested in the context of workplace assessments. A significant effort is necessary in this critical area, akin to the development of real-time detection for bio-aerosols, which seemed nearly impossible a decade ago but has made great strides since then for well known reasons.

An alternative strategy to hazard assessment of NP emissions is based on the use of aerosol dynamic models to predict relevant information for specific workplace scenarios such as concentrations and size range of the NP aerosol at the receptor. Such models have been developed in past years to predict the evolution of an aerosol during catastrophic releases of radioactive material. Although not yet validated for such new applications, aerosol dynamic codes require as input

- the background aerosol size distribution,
- the characteristics of an emission source (strength, primary size range), hence the identification of release mechanisms for each specific NP hazard;
- and assumptions about an age distribution and possible dilution of the aerosol during transport to the receptor. This can be done via model scenarios.

Finally, the prevalence of aged and therefore agglomerated NP brings up the question of their ability to fragment, which presumably has a strong influence on biological effects. Thus there is a need for effective techniques to quantify agglomerate strength in aerosol particles.

Open questions & priorities for future research

As a result of the preceding analyses, a number of important open questions can be formulated.

The *release mechanisms and sources of NP* into ambient workplace air need to be characterized and typified regarding source strength, size range and agglomerate structure. This is relevant for the development of realistic aerosol generators, realistic doses, as well as for the effective use of predictive aerosol dynamic models.

The development of *model aerosol sources* for exposure and toxicological studies needs more focus and effort. Simultaneously, a systematic effort should be undertaken to use aerosol sources for such studies, rather than powders or liquid suspensions and to advance the appropriate techniques for their application. Model aerosol sources are required for relevant classes of species and release mechanisms, including aging and scavenging methods where appropriate.

Existing *predictive dynamic models* for nano-aerosol evolution between source and receptor need to be adapted, validated and further developed as needed. While the basic mechanisms are known, the specific input parameters for the workplace environment are not.

Real-time methods for highly selective, species-specific NP detection in the aerosol state are not broadly available and require a major effort, including the sustained funding of basic research to develop and validate new concepts. This includes methods capable of detecting NP attached to background particles as well as new methods to characterize the surface chemical state of airborne particles.

The fate of *structure and strength of nano-agglomerates* during their transition from the aerosol to the cellular liquid phase, and the influence of these structural parameters on their toxicological effects needs a concerted investigation. Techniques for quantifying the strength of homogeneous and heterogeneous agglomerates, especially on-line methods would be very useful.

2 Dissemination and use

Oral presentations at national or international conferences

1. Wu Q., Seipenbusch M., Kasper G., Spielvögel J., Grimm H., Weitzenböck J. (2008): Presentation on NANOTRANSPORT project. Workshop on Research Projects on the Safety of Nanomaterials, 17-18 April 2008, Brussels, Belgian.
2. Seipenbusch M., A. Binder, und G. Kasper (2008): Veränderung der abscheidungsrelevanten Parameter von Nanopartikeln zwischen Freisetzung und Rezeption am Arbeitsplatz. Jahrestreffen des ProcessNet (Dechema) Fachausschusses Gasreinigung, 19 February 2008, Germany.

3. Wu Q., Seipenbusch M., Kasper G., Grimm H., Weitzenböck J. (2007). Selection of metrics relevant for inhalation health risk study of nanoparticle aerosols. Euro NanOSH Conference 2007-Nanotechnologies: A Critical Area in Occupational Safety and Health. 3-5 December 2007, Helsinki Finland.
4. Seipenbusch, M., Binder, A., Kasper, G., Grimm, H.J. and Wu Q. (2007). Aerosol Dynamics of NP in Workplace Exposure. Euro NanOSH Conference 2007-Nanotechnologies: A Critical Area in Occupational Safety and Health. 3-5 December 2007, Helsinki Finland.
5. Wu, Q. and Marion S. (2007). Managing risks of nanomaterials- the example of nanoparticle exposure at workplace. Teknologirådets åpne høring om nanomaterialer, risiko og regulering. Oslo, 22. September 2007.
6. Wu, Q. and Weitzenböck, J.R. (2007). A new approach to assessing the risk of using nanomaterials in industry. NANOMAT Conference, Bergen, Norway, 5-7 June 2007.
7. Kasper, G. (2007). EU-Projekt Nanotransport: Szenarien zu Transportvorgängen und Veränderungen nanopartikulärer Aerosole zwischen Emissionsquelle und Rezeptor. BASF-Symposium Expositionsmessung luftgetragener Nanopartikel am Arbeitsplatz, 17.04.2007, Ludwigshafen.

Poster presentation at conferences:

1. A. Binder, M. Seipenbusch, G. Kasper, J. Spielvogel, H.J. Grimm and Q. Wu (2007): Aerosol Dynamics of Nanoparticles in Workplace Exposure. European Aerosol Conference 2007 in September 2007 in Salzburg

Papers submitted to peer reviewed journals

Seipenbusch et al. 2008. Aerosol dynamics of nanoparticle in workplace exposure.

Workshops organized by the project consortium

1. Workshop on development of nanoparticle exposure scenarios on 22 February 2007 in Karlsruhe, Germany
2. Final NANOTRANSPORT project workshop on 16 April 2008 in Brussels, Belgian

Project website:

Project information and downloadable documents will be available on the website of the project <http://research.dnv.com/nanotransport/>.

Future plans for using and disseminating

The project partners are going to participate three more conferences this year to present the project results:

1. 11th InterNational Inhalation Symposium: Benefits and Risks of Inhaled Engineered Nanoparticles, June 11 - 14, 2008, Hannover, German;
2. NanoImpactNet WP1 Workshop on nanorisks: I: Minimal analytical characterization of NP needed for hazard assessment of NP in biological matrices; II: Standardization of materials

and protocols, June 19-20, 2008, Dublin Ireland.

2. International Conference on Safe production and use of nanomaterials, Nanosafe 08, 3-7. November 2008 in France.

All the three project partners are active participants in the new EU project NANODEVICE under FP7. The knowledge and experiences gained within the NANOTRANSPORT project will be transferred to the NANODEVICE project.

We will also use the new established NanoImpactNet (the European Network on the Health and Environmental Impact of Nanomaterials) to disseminate the findings of the NANOTRANSPORT project.