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Report

Emerging Issues in Nanoparticle Aerosol Science and Technology

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Introduction

The field of aerosol science and technology covers the basic principles that underlie the formation, measurement and modeling of systems of small particles in gases. These systems play an important role in nature and industry. Many government agencies including EPA, NIOSH, DOE, NOAA and NIST have substantial aerosol research activities. The EPA is responsible for the establishment of ambient air quality standards for particulate matter, NIOSH is concerned with workplace exposure to particulate matter, the DOE has responsibility for nuclear reactor safety which includes emissions of radioactive particles in reactor accidents and NIST plays a role in providing primary calibration standards for particle size and chemical content. Aerosol technology plays an important role in inhalation therapy and in counter-terrorism, fields likely to be of interest to NIH including NIEHS. Industry uses aerosol processes for the manufacture of powdered materials of many different kinds including reinforcing fillers, pigments and catalysts and in the manufacture of optical fibers. The Particle Technology Forum sponsored by AIChE, includes industry participation and has an interest in aerosol technology. Finally, the American Association for Aerosol Research (AAAR), the principal professional society in the field, with a membership of about one thousand, sponsors regular research meetings in the field.

Aerosols of interest to government agencies and industry cover a wide range of particle sizes, shapes and chemical compositions. The Workshop focused on Nanoparticle Aerosol Science and Technology (NAST), a new subdiscipline in which a basic understanding of the relevant science and technology is only now emerging. Nanoparticle aerosols refer to particles smaller than 100 nanometers (0.1 μm) which may be present as individual particles or as aggregates. Nanoparticles may have unusual mechanical, optical, biochemical and catalytic properties that make them of special interest. Novel experimental and theoretical methods are under development and/or needed to characterize their formation and behavior.

Advances in NAST have applications in many fields that include (but are not limited to) (1) characterization and control of ultrafine aerosols emitted by air pollution sources, (2) industrial production of nanoparticle reinforcing fillers such as carbon black and fumed silica and catalysts such as titania, (3) start-up companies that manufacture specialty nanoparticle products by aerosol processes, (4) atmospheric dynamics of fractal-like nanoparticle aerosols (e.g. diesel emissions), (5) nanoparticle emissions from aerosol control technologies including filters and electrostatic precipitators, (6) nanoparticle formation in the upper atmosphere by entry of bodies

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from space and by emissions from solid fuel rockets, (7) control of workplace exposure to ultrafine aerosols, (8) manufacture of optical fibers, (9) manufacture of composites composed of blends of nanoparticles and molecular polymers (e.g. rubber), (10) fabrication of nanoparticle coatings, (11) on-line measurement of nanoparticle chemical composition and (12) contamination control in the microelectronics industry as line features shrink.

These applications are undergoing rapid changes. To help develop a coordinated effort, about 30 participants from university research groups, government agencies and industry with backgrounds in nanoparticle aerosols were brought together at the Workshop. The goals were (1) to review the current status of the field and identify research needs and (2) to set up a group that can serve as a prototype to promote research and development in NAST. The Workshop structure was as follows: as a scientific discipline, NAST depends on: (1) Nanoparticle aerosol characterization methods (2) Basic principles of nanoparticle aerosol formation and (3) Computational simulation of nanoparticle aerosol behavior. Two major fields of application of these methods and principles include: (4) Aerosol reaction engineering and (5) Atmospheric nanoparticles (ultrafine aerosol). Thus the Workshop was organized into five Panels that covered the basic areas and two major fields of application. The reports prepared by the Panels constitute the main body of the Workshop document. The Executive Summary that includes all of the recommendations by the individual Panels and brief statements on their applications follows. The Chair and Co-Chair wish to express their appreciation to Mr. Adamos Dalis (UCLA) and Prof. Rajdip Bandyopadhyaya (IIT Kanpur) for their editorial assistance.

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Experimental Methods and Instrumentation

Physical Characterization:

D.Y.H. Pui (Chair), R.C. Flagan, S.L. Kaufman, A.D. Maynard and J.F. de la Mora

Chemical Characterization:

S.V. Hering (Chair), J.L. Jimenez, K.A. Prather, A.S. Wexler and P.J. Ziemann

The role of nanoparticles, whether for improved materials, for semiconductor fabrication, for pharmaceuticals, for environmental assessment or for evaluation of global climatic, depends on their chemistry as well as their physical characteristics. The unique functionality of nanoparticle-based materials and devices depends directly on size- and structure-dependent properties, as do the environmental impacts and health consequences of airborne nanoparticles. Often chemistry is key to elucidating sources and formation mechanisms of nanoparticles, both in the atmosphere and in industrial applications.

Physical characterization of aerosol nanoparticles is critical to the advancement of the underlying science and development practical nanotechnologies. Nanoparticle size must be tightly controlled to take full advantage of quantum size effects in photonic applications, and agglomeration must be prevented. Agglomeration can only be prevented if the number concentrations are tightly controlled, which requires that the rate of new particle formation be quantitatively determined. Real-time measurements of particle size distributions and particle structure are, thus, enabling technologies for the advancement of nanotechnology. Key areas of research to improve our physical characterization capabilities include: (1) rapid aerosol nanoparticle measurements; (2) detection, characterization, and behavior in the low nanometer (<5 nm) size regime; (3) particle standards for size, concentration, morphology and structure; (4) charging behavior and technology throughout the ultrafine and nanoparticle size regimes; (5) distributed nanoparticle aerosol measurements; (6) integral parameter measurement; (7) off-line morphological, structural, and chemical characterization of nanoparticles; (8) on-line morphological and structural characterization of nanoparticles; (9) nanoparticle size distribution measurements with high detection efficiencies.

Ultrafine particles present an exceptional challenge for chemical analyses because their mass is small, of the order of attograms. Recent years have seen many advances in on-line analyses of the chemical constituents of ultrafine particles. Most prominent are the advances in particle mass spectrometry, with systems providing data on single particle composition, or size- and composition-resolved data for an ensemble of particles. While much has been accomplished, much is needed to improve our analytical capabilities for the ultrafine particle size fraction. Key areas of research include (1) unbiased sampling of ultrafine particles, both into particle beam systems and for sampling into a liquid stream for subsequent on-line analyses (2) identification and quantification of organic fraction at the compound or compound-class level; (3) better chemical characterization of light-absorbing particles; (4) means for quantification and calibration, especially for organic compounds; (5) morphological and surface chemistry determinations; (6) fast response systems; (7) systems that can operate under extreme conditions of temperature or pressure; and (8) better and more rapid on-line data reduction schemes. Importantly, the systems must be more accessible, and easier to use. They need to be made smaller in size, lower in weight and cost, especially for targeted needs.

Assessment of the role of nanoparticles in global climate, in health implications of aerosol nanoparticle exposure, whether due to ambient or occupational exposures, must await entirely new chemical and physical characterization technologies that enable distributed measurements. Full understanding of the sources of nanoparticle aerosols, whether in the environment, in industrial processes, in offices and homes is needed. This will require larger numbers of sensors that are simplified, robust and below the cost of the laboratory instruments in use today. Such instruments must not require an aerosol scientist to operate them, and should be reliable enough to provide the continuous data records.

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Fundamentals of Nanoparticle Aerosol Formation

S.K. Friedlander (Chair), M.R. Zachariah (Co-Chair), G.W. Mulholland, C.M. Sorensen, R.P. Turco

Particles form in gases by various mechanisms. Some of the major routes are considered in this section. Small solid particles often appear as aggregates which are also discussed.

Physics of Nucleation

Studies at the end of the nineteenth century by C.T.R. Wilson showed that at least three types of nuclei were activated when atmospheric air was saturated by adiabatic expansion: (i) existing aerosol particles at saturation ratios near unity, (ii) ionized gas molecules at higher values of the supersaturation and (iii) water molecules themselves at much higher supersaturations. Advances in theory and instrumentation may permit detailed characterization of atmospheric nuclei, not possible before. Applications are to reducing major uncertainties in the global radiation balance, especially the poorly understood 'indirect effects' produced by nuclei that modify the global cloud cover and the hydrologic cycle. Other important applications are to aerosols produced when hot vapors from pollution sources are injected into the atmosphere.

Research Recommendations

(1) Use of modern computational methods, including quantum theory, to calculate thermodynamic properties of atmospheric nanoclusters, charged and neutral.
(2) Effects of atmospheric nanoparticles on global climate through their role as fog and cloud condensation nuclei.
(3) Theoretical and experimental studies of the effects of turbulence on nucleation rates.

Chemical Nucleation: Soot Formation

There are many examples of chemically reacting gases that generate nanoparticles through complex chemical reaction pathways. Soot formation is the case that has been studied most because of its environmental and industrial importance. Controversy remains concerning the chemical steps that lead to particle formation, and the chemical nature and structure of the soot particles that are usually present as nanoparticle chain aggregates. Although experimental and conceptual difficulties abound, the payoff in fuel efficiency and environmental protection continue to stimulate research in this field.

Research Recommendations

(4) Well-characterized soot generators (reactors), the characterization of soot primary particles and their aggregates and proven chemical mechanisms for describing soot inception are needed.

Collision-Coalescence Formation Processes: Aggregate Dynamics

Over the last few decades, we have learned much about how small solid particles of inorganic oxides form, based on collision-coalescence concepts. This is a limiting case in which the chemical reactions that result in condensable molecules are very rapid compared with particle collision and coalescence; the equations of aerosol dynamics can be generalized to incorporate the rate of coalescence of particles in contact. Solid particles so formed then aggregate. Molecular dynamics (MD) show promise for calculating coalescence rates and characterizing the dynamic properties (e.g. elastic modulus and tensile strength) of aggregates. Measurements of *the dynamic behavior* (in addition to morphology) of chain aggregates should be possible with modern instrumentation. Applications are to (1) formation of primary solid particles and their aggregates in commercial synthesis of nanopowders and in emissions from industrial sources, e.g. coal fired power plants and incinerators, (2) nanocomposite materials such as rubber that incorporate aggregates and (3) dynamics of aggregates in flowing gases and at surfaces.

Research Recommendations

(5) Computational statistical mechanics (e.g. molecular dynamic and Monte Carlo techniques) calculations of (i) coalescence rates of nanoparticles in contact and (ii) individual nanoparticle thermodynamic properties (e.g. surface tension) and transport properties (e.g., solid state diffusion coefficients).
(6) Experimental studies of individual nanoparticle chain aggregates and their networks under stress, to measure their tensile strength and elastic modulus.
(7) MD studies of the effects of tension on nanoparticles chains to determine elastic modulus, tensile strength and locus of fracture. Compare calculations with data.

Aggregate Morphology

The ability to characterize aggregate morphology quantitatively, for example through the fractal dimension, has motivated research on aggregate structure and led to the discovery of 'superaggregate' structures with fractal dimensions that differ from the substructures. Also important is the measurement of the optical cross-section of soot agglomerates over a wide wavelength range for applications to radiant transport through smoke and postflame gases.

Research Recommendations

(8) Understand the growth kinetics and morphologies of superaggregate structures.
(9) Determine aggregate transport properties over the entire range of Knudsen numbers based on primary particle and aggregate sizes.
(10) Measure optical cross sections of soot agglomerates from the uv to at least 10 micrometers, for high temperature soot in the flame and under post-flame conditions as a function of agglomerate size and morphology.

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Computational Simulation of Aerosol Behavior

J.H. Seinfeld (Chair), S. Pandis(Co-Chair) and G. Ahmadi, D.R. Chen

Accurate and reliable models for simulating transport, deposition, coagulation, and dispersion of nanoparticles and their aggregates are needed for the development of design tools in a variety of technological areas. The applications of these computational tools include development of nanoparticle instrumentation (sampling, sensing, dilution, focusing, mixing, etc.), simulation of nanoparticle behavior in complex geometry passages (human respiratory track, aerosol transport/delivery systems, energy systems, etc.), and design of chemically reactive systems (combustors, aerosol reactors, etc.). Computational models for atmospheric nanoparticles are used for the quantification of the radiative effect of nanoparticles on the radiative balance of our planet (indirect effect of particles on climate) and for the design of abatement strategies for both nanoparticles and particle mass concentration.

Our panel identified the following research needs:

(1) Better understanding of the variation of hydrodynamic, surface, Brownian, electrical, and thermophoretic forces with nanoparticle size, shape and flow conditions
(2) Development of reliable aerosol dynamic models for simulating transport, deposition, coagulation of nanoparticles of different shapes including aggregates.
(3) Development of efficient numerical schemes for simulating nanoparticle transport and deposition capable of integration with CFD codes.
(4) Development of efficient schemes for the simulation of atmospheric nanoparticles together with the rest of the particulate matter (PM).
(5) Algorithm development of the simulation of atmospheric nanoparticles at different spatial scales (centimeters to thousands of kilometers).
(6) Evaluation of the atmospheric nanoparticle simulation tools against comprehensive ambient datasets.
(7) Development of computationally efficient schemes for the simulation of the compositional mixing state of atmospheric nanoparticles.

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Aerosol Reaction Engineering (ARE)

D.E. Rosner (Chair), T.T. Kodas (Co-Chair), P.D. Christofides, R.B. Diemer Jr., G. Fotou and S.E. Pratsinis

Many industrial applications of ‘tailored’ nanoparticles (including nanotubes) have been identified in recent years including their use in fabricating: selective catalysts or membranes, electronic/magnetic information storage or chemical sensor (detection) devices, optical films/fibers, additives for the control of rheological or thermophysical properties, energetic chemical propulsion fuel additives, etc. Remarkable applications are being rapidly discovered, and the increased availability of low-cost nanoparticles for exploratory R&D, made possible by the ARE-research recommended here, will accelerate these advances.

To capitalize on the unique properties of nanoparticles in applications, they will have to be synthesized in large quantities under carefully controlled conditions. For this purpose, ‘aerosol’ (gas-to-particle) chemical reactors will be attractive because of their ability to satisfy the demands of: high product purity, scale-up potential, improved control of phase identity/composition, and small environmental ‘footprint’. However, because of the stringent new demands associated with tailored nanoparticle synthesis, the science/technology base for designing/optimizing/controlling particle-producing reactors is presently inadequate. Developing this frontier area of chemical reaction engineering will pay dividends by facilitating the emergence of this new branch of ‘specialty chemicals’, and provide valuable guidance for the many start-up companies needed to supply the diverse and rapidly growing nanoparticle requirements of industry. Specific research recommendations are included for each of the following 16 ARE-areas:

(1) nanoparticle precursor selection, (2) ‘reduced’ kinetics of precursor evolution to embryonic particles, (3) models of ‘chemical’ nucleation rates, (4) nanoparticle growth by heterogeneous reactions, (5) modeling brownian coagulation rates, (6) improved rate-laws for nanoparticle restructuring, (7) modeling phase

change within nanoparticles, (8) improved prediction of nanoparticle transport properties, (9) development/implementation/testing of efficient EAD-simulation methods, (10) testing 'ARE' models against well-instrumented laboratory experiments, (11) model-based control of aerosol reactors, (12) model-based scale-up, (13) anticipating and controlling deposition, (14) health/safety precautions for handling nanoparticle products, (15) surface modification/coating opportunities, (16) property-resolved nanoparticle reactor-separators.

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Atmospheric Nanoparticles (Ultrafine Particles)

C. Sioutas (Chair), P.H. McMurry (Co-Chair),
P. Biswas, W.C. Hinds, and W.E. Wilson

Atmospheric ultrafine particles (UFP) – with diameters less than about 0.10 μm–are formed from gases by a variety of gas-to-particle conversion processes. There are at least three sources of gases, which may be converted to particles. UFP may be formed at high temperature sources and emitted directly to the atmosphere. Some processes may emit hot supersaturated vapors, which undergo nucleation and condensation while cooling to ambient temperatures. Chemical reactions in the atmosphere may lead to chemical species with very low saturation vapor pressures. These chemical species may form particles by a variety of nucleation processes. Although the UFP mass fraction is usually very small, this size range contains the highest number of ambient particles and an appreciable portion of total surface area. Because of their increased number and surface area, UFP are particularly important in atmospheric chemistry and environmental health. Toxicological and epidemiological evidence linking respiratory health effects and exposures to UFP has been increasing over the last several years. Recent epidemiological studies demonstrated a stronger association between health effects and UFP compared with accumulation mode or coarse particles. More recent toxicological studies indicate that ultrafine particles are most potent in inducing oxidative stress. UFP also localize in mitochondria where they induce major structural damage. Moreover, the concentrations of these particles in ambient air are spatially inhomogeneous and very dependent on the proximity to their sources. This inhomogeneity strongly affects population exposures, with a portion of the population (depending on activity pattern, profession, lifestyle, commuting time, etc.) being exposed acutely to very high UFP concentrations.

The science of atmospheric nanoparticle aerosols is relatively new, and despite recent progress much remains to be done. There is a fundamental lack of information on how nanoparticles form and grow to sizes that can serve as cloud condensation nuclei (CCN) and affect the earth's climate and albedo. There is also lack of knowledge on the fraction of these particles that are formed by direct emission, nucleation of hot vapor upon cooling, or atmospheric formation of gases that undergo nucleation. UFP physical, chemical and toxicological properties are poorly understood. Many of the measurements that are needed require instrumentation that currently does not exist. Little information is also available on the concentrations or physical/chemical properties of UFP in places where people live and work: in community air, in homes, in schools, in workplaces, or in vehicles. To determine UFP health effects, it is essential to develop and deploy technologies that can assess the nature and extent to which people are exposed to these particles. The complexity of the sources and nature of UFP suggest that much effort will be needed to either discover and/or refine our understanding of linkages between exposures and health effects. Studies focusing on health effects are also needed to identify mechanisms by which UFP induce cellular damage and generate oxidative stress, how they penetrate the cellular structure, and how the above effects depend on chemical composition. This information is essential in reducing emissions of UFP that are a risk to public health. Finally, there is a need to better detect UFP that pose a biological threat. Examples include viruses, nanometer sized fragments of biological particles, and condensed chemical agents on UFP surfaces.

In summary, our panel identified the following topics for research:

(1) photochemically-driven nucleation in the atmosphere, (2) nucleation and rapid growth that occurs as hot pollutant exhaust gases mix with cooler air in the ambient environment, (3) growth rates of freshly nucleated atmospheric ultrafine particles, (4) chemical and physical transformations of atmospheric ultrafine particles, (5) improvements in measurement technology for ultrafine particles, (6) atmospheric measurement needs for ultrafine particles, (7) measurement and characterization of ultrafine particle emissions from sources, (8) source apportionment of ultrafine particles, (9) population exposure assessment, (10) dosimetry, (11) health effects of UFP, (12) control technology, UFP and homeland security.

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