Emerging Nanotechnologies and Water
--- An Overview of Opportunities and Issues ---

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NanoSafe, Inc. (Supported by US Army Research Office
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Presentation Overview

- What is nanotechnology?
- What are nanomaterials? (*Where/how are they used?*)
- How does nanotechnology benefit the environment?
- What risks does nanotechnology bring?
- What are the critical regulatory (and risk) issues?
- Where is nanotechnology headed?
- What is a nano-enabled product?

*How might nanotechnology impact natural waters, and waste water processing?*
Nanotechnology Federal Spending

$1.4 billion in 2016

$24 billion since the inception of the NNI in 2001

"...the ability to understand and control matter at the nanoscale will lead to new innovations that will improve our quality of life and benefit society."

Application & Implications

http://www.nano.gov/about-nni/what/funding

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Nanotechnology: What is it?

US National Nanotechnology Initiative definition:

Nanotechnology: the understanding and control of matter at dimensions between approximately 1 and 100 nanometers, where unique phenomena enable novel applications.

(Encompassing nanoscale science, engineering, and technology, nanotechnology involves imaging, measuring, modeling, and manipulating matter at this length scale.)

Nanomaterial: a material having at least one dimension between 1 and 100 nanometres.
Mean diameter = 200+ nm

However:

- New EPA reporting rule addresses key definition issues
European Commission Definition (2011 draft):

Nanomaterial means a natural, incidental or manufactured material containing particles, in an unbound state or as an aggregate or as an agglomerate and where, for 50% or more of the particles in the number size distribution, one or more external dimensions is in the size range 1 nm–100 nm.,

specific cases... where warranted... number size distribution threshold of 50% may be replaced... between 1 and 50%;

and/or has internal or surface structures in one or more dimensions in the size range 1 nm–100 nm;

and/or has a specific surface area by volume greater than 60 m²/cm³, excluding materials consisting of particles with a size lower than 1 nm.
A criticism of with number size definitions...

- 10 nm diameter
- 0.005% mass
- 99+% mass
- 10 cm diameter

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How Small are Nanomaterials?
Nanotechnology examples: Gold Nanocages

Nano-scale Multi-Component Systems

*Electron Transport in Photocatalytic Titanium Dioxide*

Vaidyanathan Subramanian, Eduardo E. Wolf, and Prashant V. Kamat

*J. AM. CHEM. SOC.* 2004, 126, 4943-4950
Quantum effects: Quantum dots (CdSe)

(quantum confinement of excitons)

What is a quantum dot?

- Nanocrystals
- 2-10 nm diameter
- Semiconductors

TEM micrograph

http://33rdsquare.com

http://www.greenspine.ca/media/quantum_dots_c.jpg

2.0 nm  2.5 nm  3.0 nm  4.0 nm

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These are all nano-scale properties
Nanotechnology: Why is size important?

- 26.96 grams of coin silver
- Dia. = 40 mm
- Surface area = 27.70 cm²

- 26.96 grams of coin silver
- Dia. = 1 nm
- Surface area = $11.4 \times 10^7$ cm²

1-nm silver "nano" particles

4 ¼ sq inch

2.8 acres
Nanomaterials/Nanotechnology Benefits and Applications

**Fundamental issues/questions:**
- What/how are nanomaterials used?
- How much is used?
- What is the potential for release?
- If released, is there exposure?
- If there is exposure, are they toxic?

*New EPA reporting rule, effective May 12, 2017*
Major Materials

Nano-scale silver

Nano-scale titanium dioxide

Number of Products

- Silver: 383
- Carbon: 87
- Titanium: 179
- Silicon/Silica: 52
- Zinc: 36
- Gold: 19

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http://www.nanotechproject.org/cpi  - accessed Jan 26, 2017

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Consumer Products Inventory

An inventory of nanotechnology-based consumer products introduced on the market.

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Dissolution of nano-silver

- Nano-specific effects are debatable.
- But: Differentiating particle from ion effects is experimentally challenging.

Mass or Volume Used?

- Potential for high-volume use
- Little potential for release
Mass or Volume Used?

Titanium dioxide

Used in:
- foods
- paints and other coatings
- sunscreens
- cosmetics
- glass
- energy conversion
- photocatalytic agents

- projected annual global production ~ 260k metric tons (2015)
- varying proportions of production are micrometer-scale

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Photocatalytic Nano-Scale TiO$_2$

Destructive redox reactions with organic materials

Oxidation of NO$_x$ & SO$_x$

Band gap = 3.2 eV

Solar or artificial radiation

$\lambda \leq 388$ nm

Solar Spectrum

doping, modifications

NANOSAFE INC.
Photo-catalytic Nano-Scale Titanium Dioxide in Cement

LSU Basketball Practice Facility

Air France Headquarters

Dives in Misericordia Church

University at Albany School of Business

I-35 Gateway

Test Roadways
Photocatalytic Nano-Scale TiO₂

ROS are also very toxic to aquatic organisms

band gap = 3.2 eV

Solar Spectrum

λ ≤ 388 nm

doping, modifications
Photocatalytic TiO2 and Toxicity

Significant increase in toxicity under solar radiation

**Daphnia magna 48h LC50**

- 4 hr simulated solar radiation
  - LC50 = 29.8 µg/L
- Lab lighting
  - LC50 = >~300 mg/L (?)

**48-h Ceriodaphnia bioassay**

- LC50 0.35
- LC50 = 25.4

**Japanese medaka 96h LC50**

- 4 hr simulated solar radiation
  - LC50 = 2.42 mg/L
- Lab lighting
  - LC50 = 292 mg/L

**Hyalella azteca bioassay**

- LC50 29.9
- LC50 = 631

These examples illustrate:

- Particulate nanomaterials may dissolve, releasing toxic free constituents
  - nano-silver, CD/Se Q-dots

- Nanomaterials can have nano-specific toxic potential
  - photocatalytic TiO$_2$ (*based on intended function*)

- Release of nanomaterials (or dissolved constituents) will vary with application & volume/mass used
  - silver-impregnated fabrics vs. Q-Dot TV screens

Regulation of nanomaterials requires determination of volume or mass used, release potential, fate of released substances, & toxicity
Federal Regulations and Agencies

FFDCA - FDA

FIFRA - EPA

TSCA - EPA

OSHA - OSHA

CAA & CWA - EPA

FFDCA - FDA
(Cosmetics and dietary supplements only)

CPSA - CPSC

RCRA - EPA

Pre-Market

Use

End-of-Life


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<table>
<thead>
<tr>
<th>REGULATION</th>
<th>APPLICABILITY</th>
</tr>
</thead>
<tbody>
<tr>
<td>Toxic Substances Control Act</td>
<td>Use of &quot;new&quot; chemical substances must be reported</td>
</tr>
<tr>
<td>TSCA EPA</td>
<td>Use of &quot;existing&quot; substances does not require reporting</td>
</tr>
<tr>
<td></td>
<td>** A &quot;new&quot; substance is one that is not listed in the TSCA inventory</td>
</tr>
<tr>
<td>Federal Insecticide, Fungicide, and Rodenticide Act</td>
<td>Regulation is triggered by <strong>claims</strong> of pesticidal properties</td>
</tr>
<tr>
<td>FIFRA EPA</td>
<td></td>
</tr>
<tr>
<td></td>
<td><em>Inherent pesticidal activity does not trigger regulation. Claim such as: &quot;Kills germs on contact&quot;, or &quot;Kills ants dead!&quot; are pesticidal claims.</em></td>
</tr>
</tbody>
</table>

**New EPA reporting rule, effective May 12, 2017**
Nanomaterials: The Regulatory Challenges

- What is "nano"?

- When should “nano-ness” be regulated? E.g.;
  - is silver in 10 nm particles “just” silver?
  - are carbon nanotubes more than just carbon?
  - are fullerenes identical to carbon black?

- Product application is uncertain
  - opportunity for proactivity, but; what should be tested??

- Rate of invention is extremely high
  - new substances, forms and structures, combinations nearly unlimited variation

- Nanomaterials are a new class of substances
  - particulate, fibrous, particle and fiber matrices
  - NOT (typically) soluble (or transiently particulate)
All Regulation is Based on Risk Assessment

\[
\text{RISK} = \text{EXPOSURE} \times \text{TOXICITY}
\]

\[
\text{TOXICITY} = \text{EXPOSURE} \times \text{TIME}
\]

EXPOSURE
multiple concentrations

TIME = 48h, 96h, 7d, 10d, ...
(acute, chronic, life-cycle,...)

Standardized Regulatory Testing
(U.S.EPA, OECD, EU, ASTM, etc.)
## USEPA Standard Test Guidelines

<table>
<thead>
<tr>
<th>Code</th>
<th>Test Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>850.1710</td>
<td>Oyster BCF</td>
</tr>
<tr>
<td>850.1730</td>
<td>Fish BCF</td>
</tr>
<tr>
<td>850.1735</td>
<td>Whole sediment acute toxicity invertebrates, freshwater</td>
</tr>
<tr>
<td>850.1740</td>
<td>Whole sediment acute toxicity invertebrates, marine</td>
</tr>
<tr>
<td>850.1790</td>
<td>Chironomid sediment toxicity test</td>
</tr>
<tr>
<td>850.1800</td>
<td>Tadpole/sediment subchronic toxicity test</td>
</tr>
<tr>
<td>850.1850</td>
<td>Aquatic food chain transfer</td>
</tr>
<tr>
<td>850.1900</td>
<td>Generic freshwater microcosm test, laboratory</td>
</tr>
<tr>
<td>850.1925</td>
<td>Site-specific aquatic microcosm test, laboratory</td>
</tr>
<tr>
<td>850.1950</td>
<td>Field testing for aquatic organisms</td>
</tr>
</tbody>
</table>

**Group B—Terrestrial Wildlife Test Guidelines.**

<table>
<thead>
<tr>
<th>Code</th>
<th>Test Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>850.2100</td>
<td>Avian acute oral toxicity test</td>
</tr>
<tr>
<td>850.2200</td>
<td>Avian dietary toxicity test</td>
</tr>
<tr>
<td>850.2300</td>
<td>Avian reproduction test</td>
</tr>
<tr>
<td>850.2400</td>
<td>Wild mammal acute toxicity</td>
</tr>
<tr>
<td>850.2450</td>
<td>Terrestrial (soil-core) microcosm test</td>
</tr>
<tr>
<td>850.2500</td>
<td>Field testing for terrestrial wildlife</td>
</tr>
</tbody>
</table>

**Group C—Beneficial Insects and Invertebrates Test Guidelines.**

<table>
<thead>
<tr>
<th>Code</th>
<th>Test Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>850.3020</td>
<td>Honey bee acute contact toxicity</td>
</tr>
<tr>
<td>850.3030</td>
<td>Honey bee toxicity of residues on foliage</td>
</tr>
<tr>
<td>850.3040</td>
<td>Field testing for pollinators</td>
</tr>
</tbody>
</table>

**Group D—Nontarget Plants Test Guidelines.**

<table>
<thead>
<tr>
<th>Code</th>
<th>Test Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>850.4000</td>
<td>Background—Nontarget plant testing</td>
</tr>
<tr>
<td>850.4025</td>
<td>Target area phytotoxicity</td>
</tr>
<tr>
<td>850.4100</td>
<td>Terrestrial plant toxicity, Tier I (seedling emergence)</td>
</tr>
<tr>
<td>850.4150</td>
<td>Terrestrial plant toxicity, Tier I (vegetative vigor)</td>
</tr>
<tr>
<td>850.4200</td>
<td>Seed germination/root elongation toxicity test</td>
</tr>
<tr>
<td>850.4225</td>
<td>Seedling emergence, Tier II</td>
</tr>
<tr>
<td>850.4230</td>
<td>Early seedling growth toxicity test</td>
</tr>
</tbody>
</table>

**Group E—Toxicity to Microorganisms Test Guidelines.**

<table>
<thead>
<tr>
<th>Code</th>
<th>Test Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>850.5100</td>
<td>Soil microbial community toxicity test</td>
</tr>
<tr>
<td>850.5400</td>
<td>Algal toxicity, Tiers I and II</td>
</tr>
</tbody>
</table>

**Group F—Chemical-Specific Test Guidelines.**

<table>
<thead>
<tr>
<th>Code</th>
<th>Test Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>850.6200</td>
<td>Earthworm subchronic toxicity test</td>
</tr>
<tr>
<td>850.6800</td>
<td>Modified activated sludge, respiration inhibition test for sparingly soluble chemicals</td>
</tr>
</tbody>
</table>

**Group G—Field Test Data Reporting Guidelines.**

<table>
<thead>
<tr>
<th>Code</th>
<th>Test Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>850.7100</td>
<td>Data reporting for environmental chemistry methods</td>
</tr>
</tbody>
</table>
Some Key Requirements for a "Valid" Test

- Fully "dissolved" substance
- Absence of colloids/particles
- Consistent levels of exposure
- Measured, confirmed, documented levels of exposure
- Un-altered substance
  - No variation in physical/chemical form or structure
- Known or measured physical/chemical properties
- Toxicity documented using concentration-response metrics
  - EC50, LC50, LOEC, etc.
TiO₂ Settling rate  
(100 mg*L⁻¹)

Degussa/Evonic p25 TiO₂, ~21 nm dry, 80% anatase, 19% rutile  
- fully characterized by NIEHS/NTP  
- widely used worldwide in exploratory fate/effects studies
Variation in Exposure During Assays

Varying mass concentration

Varying primary particle size

Formation of particles

"20% Rule"

Variation in media concentration of 20% or greater suggests the need for testing modifications (more later)

Effect of rate of addition of dilution water

"Effect of ionic strength"

Diameter (nm, DLS)

Time (h)

0 5 10 15 20 25

1 2 3


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Review of OECD/OPPTS-Harmonized and OPPTS Ecotoxicity Test Guidelines for Their Applicability to Manufactured Nanomaterials

by

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Janeek J. Scott-Fordsmand, National Environmental Research Institute, Denmark
Kath Stewart, AstraZeneca UK Limited, UK
Screening Criteria & Release Potential (Tiers 1 and 2)

Abraded Concrete:

- **TiO2** - primarily matrix-bound
Fate / Persistence/Hazard) (Tiers 2, 3, 4)

Settling Behavior of TX-Active Cement Acid-Treated Fractions

- **Small fraction**
- **Large fraction**

Settling time (min) vs. absorbance
Environmental Risk Posed by Nanotechnologies?

- Are they toxic?
  - Silver, copper, zinc, cadmium (other metals)
    - little or unconfirmed nano-specific toxicity
    - impact is dependant on solubilization of toxic ionic species
  - Titanium dioxide, *zinc oxide* |
    - Can be highly toxic depending on the presence of UV radiation
      * Titanium dioxide will not dissolve, zinc oxide dissolves and is toxic as ionic zinc
  - Are they released in wastewater stream or natural waters?
    - Silver from treated fabrics
    - Titanium from foods, cosmetics, personal care products (toothpaste)
    - These will be retained in WWTP solids - potential impacts to land applications

The ability to detect and quantify nanomaterials in complex media is extremely limited
FIGURE 5. SEM analysis of (A) nanoscale TiO2, (B) microscale TiO2, (C) an aggregate of primary TiO2 material, (D) mineral-containing Ti in a biosolid sample, (E) TiO2 in toothpaste as a representative consumer product, and (F) nanoscale TiO2 in WWTP tertiary effluent. EDX insets were provided for some SEM images, but all solids shown were confirmed to contain Ti and O.


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# Nanotechnologies in Wastewater Treatment

<table>
<thead>
<tr>
<th>Nano-material</th>
<th>absorption</th>
<th>photocatalysis</th>
<th>non-photo catalysis</th>
<th>oxidation</th>
<th>filtration</th>
</tr>
</thead>
<tbody>
<tr>
<td>carbon nanotubes</td>
<td>titanium dioxide</td>
<td>iron 2+/3+</td>
<td>titanium dioxide</td>
<td>iron oxides</td>
<td>iron 2+/3+</td>
</tr>
<tr>
<td>fullerenes</td>
<td>iron oxides</td>
<td>zinc oxide</td>
<td>dioxide</td>
<td>iron oxides</td>
<td>iron 2+/3+</td>
</tr>
<tr>
<td>graphene/graphite</td>
<td>fullerenes</td>
<td></td>
<td></td>
<td></td>
<td>almost any</td>
</tr>
<tr>
<td>iron oxide</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>titanium dioxide</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>alumina</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>polymers (dendrimers)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Nano-function</td>
<td>high surface area</td>
<td>band-gap</td>
<td>semiconductor behavior</td>
<td>re-doxy potential</td>
<td>nano-porosity</td>
</tr>
<tr>
<td>functionalization</td>
<td></td>
<td>semiconductors</td>
<td></td>
<td></td>
<td>nano-fibrosity</td>
</tr>
<tr>
<td>nano-combinations</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

- Most application are in experimental/developmental stages
- Many application involve "nanoizing" existing technologies
- Nanomaterials are used in almost unlimited combination

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Broad Conclusions

- Nanomaterials are being used in all sectors of the economy
  -- and can be identified in products and waste streams
  -- direct impacts have not been documented

- Nanotechnology development will continue to accelerate

- The regulatory process is slowly adapting
  -- TSCA changes may enable closer scrutiny

Water and wastewater processes will both benefit and be at risk from emerging nanotechnologies.

Thanks for listening!
Adapting OECD Aquatic Toxicity Tests for Use with Manufactured Nanomaterials: Key Issues and Consensus Recommendations

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10Experimental Toxicology and Ecology, BASF SE, D-67056 Ludwigshafen, Germany
11Office of Pollution Prevention and Toxics, United States Environmental Protection Agency, Washington, D.C. 20460, United States

Supporting Information

ABSTRACT: The unique or enhanced properties of manufactured nanomaterials (NNs) suggest that their use in nanocomposite products will continue to increase. This will result in increased potential for human and environmental exposure to NNs during manufacturing, use, and disposal of nanocomposites. Scientifically based risk assessment for NNs necessitates the development of reproducible, standardized hazard testing methods such as those provided by the Organization for Economic Cooperation and Development (OECD). Currently, there is no comprehensive guidance on how best to address testing issues specific to NNs, particularly their behavior or ecological properties. This paper summarizes the findings from an expert workshop convened to develop a guidance document that addresses difficulties encountered when testing NNs using OECD aquatic and sediment test guidelines. Critical components were identified by workshop participants that require specific guidance for NN testing: preparation of suspensions, dose matrix, the importance and challenges associated with maintaining and monitoring exposure levels, and the need for reliable methods to quantify MNs in complex media. To facilitate a scientific advance in the consistency of nanotoxicology test results, we identify and discuss critical considerations where expert consensus recommendations were and were not achieved and provide specific research recommendations to resolve issues for which consensus was not reached. This program will enable the development of prescriptive testing guidance for NNs. Critically, we highlight the need to quantify and properly interpret and express exposure during the biosynthesis used to determine hazard classes.

INTRODUCTION

The rapid advancement and implementation of nanotechnology has inspired vigorous debate about the adequacy of current regulatory frameworks for ensuring the safe deployment of manufactured nanomaterials (NNs). The OECD Aquatic Toxicity Test Guidelines were the first peer-reviewed guidelines specifically designed and validated for testing of nanomaterials under aquatic exposure. However, several test parameters were found to be challenging to address in a standardized manner. The workshop was convened to develop a guidance document that addresses difficulties encountered when testing NNs using OECD aquatic and sediment test guidelines. Critical components were identified by workshop participants that require specific guidance for NN testing: preparation of suspensions, dose matrix, the importance and challenges associated with maintaining and monitoring exposure levels, and the need for reliable methods to quantify MNs in complex media. To facilitate a scientific advance in the consistency of nanotoxicology test results, we identify and discuss critical considerations where expert consensus recommendations were and were not achieved and provide specific research recommendations to resolve issues for which consensus was not reached. This program will enable the development of prescriptive testing guidance for NNs. Critically, we highlight the need to quantify and properly interpret and express exposure during the biosynthesis used to determine hazard classes.

TI02 type and form
Composite w/concrete
As-delivered?
During construction?
During Use?
End-of-life?
Form-specific
Fate processes
Transformation/modification
Toxicity / hazard
Are there novel considerations
specific to this nanotechnology?
e.g. release, fate, transport, MoA

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