Research and Demonstration of Electrospun Nanofiber Filters: Multifunctional, Chemically Active Filtration Technologies for Small-Scale Water Treatment Systems

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Our motivation: Water quality in Iowa small drinking water systems

**Arsenic**
(8% samples > MCL)

Red: ≥ 0.01 mg/L (MCL)
Blue: 0.001-0.009 mg/L

**Atrazine**
(8% of samples)

*Iowa Statewide Rural Well Water Survey Phase 2 (SWRL2): Results and Analysis. UI Center for Health Effects of Environmental Contaminants (2009)*
There is a niche for nanotechnology in small systems

**A Water Treatment Engineers Introduction to Reactive Nanomaterials**

<table>
<thead>
<tr>
<th>Must harness</th>
<th>How?</th>
<th>Water quality benefit</th>
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</thead>
<tbody>
<tr>
<td>High reactivity</td>
<td>Specific surface area &amp; size effects</td>
<td>Efficiency toward historically recalcitrant pollutants</td>
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<tr>
<td>Scale</td>
<td>Size- &amp; morphology-directed synthesis</td>
<td>“Small” promotes point-of-use &amp; decentralized technologies</td>
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<td>Tunable properties</td>
<td>Surface &amp; bulk chemical modification</td>
<td>Tailored treatment to source water quality</td>
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<tr>
<td>“Bottom up” processing</td>
<td>Design &amp; fabrication of hetero-nanostructures</td>
<td>Multi-functionality shrinks the process train</td>
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</tbody>
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**Caveats:**
1. Nanomaterials will not fulfill promise in treatment applications as suspensions
2. The most reactive nanomaterial may not be optimal for application
EPA STAR Small Systems Project: Chemically reactive filtration technologies

**Overarching objectives:**

1) **Fabricate** multi-component nanofiber mats via electrospinning,

2) **Optimize** performance as chemically reactive filtration materials

3) **Demonstrate** application in POU and POE systems
Electrospinning of chemically active (inorganic) nanofibers for treatment

Polymer based sol-gel solution (TiO$_2$)
Titanium tetraisopropoxide (TTIP) & poly(vinyl pyrrolidone) (PVP)

Flow rate (~0.1 mL/h)
Applied Voltage (~10 kV)

Lab scale
Annealing into Oxide

High Voltage Power Supply

http://nano.mtu.edu/electrospining_start.html
Electrospinning yields mats ideal as reactive coatings

Scalable

TiO$_2$ 2.0 kV x700 SE(U)

120 μm

0.2 μm

100 nm

22 h

TiO$_2$

6
Nanofiber size can be tailored during synthesis

Light source:
- Suntest Solar Simulator
- 1000W Xe (λ>305 nm)

Solution:
- pH 7
- 0.1 g/L TiO₂

- [Phenol]/[Phenol]₀ vs. Time (min)
- k<sub>obs</sub>(30) = 0.0793 min⁻¹ (d = 30 nm)
- k<sub>obs</sub>(60) = 0.0541 min⁻¹ (d = 60 nm)
- k<sub>obs</sub>(150) = 0.0352 min⁻¹ (d = 150 nm)
- k<sub>obs</sub>(210) = 0.0230 min⁻¹ (d = 210 nm)

Decreasing diameter

Voltage: 12 kV
- PVP: 12%
- Diameter: 30 10 nm

Voltage: 10 kV
- PVP: 12%
- Diameter: 60 10 nm

Voltage: 10 kV
- PVP: 15%
- Diameter: 150 25 nm

Voltage: 8 kV
- PVP: 15%
- Diameter: 300 30 nm
Nanofiber crystallinity and grain size tunable during annealing

Optimal ratio: 26:74 (0.5)% rutile:anatase
Surface-doped Au/TiO$_2$ is highly photoactive nanofiber catalyst for organic oxidation

Annealed at 650 °C

Aerioxide P25

$k_{\text{obs}}$ for phenol decay (min$^{-1}$)

0.5

0.4

0.3

0.2

0.1

0.0

Au loading (% w/w)

0

2

4

6

8

10

12

$t_{1/2} \sim 2$ min

$t_{1/2}(\text{phenol})$

$\lambda_{\text{cut-on}}$ (nm)

1.5

5

30

1200

pH 7; Suntest 0.1 g/L TiO$_2$

$t_{1/2}(\text{P25}): 10$ min

$t_{1/2}(1.5\% \text{Au/TiO}_2): 15$ min

Atrazine
Ongoing work: Iron oxide and carbon sorbent nanofibers

\[ \alpha-\text{Fe}_2\text{O}_3 \]

Hematite Nanotubes

- Adsorbed chromate (mg/g)
  - pH 6
  - 0.1 g/L

- Aqueous chromate (mg/L)
  - NF
  - NP
Ongoing: performance testing in flow through systems

Filtration housings

Water Matrix

Reject (cross flow)

Permeate/ Filtrate (dead end & cross flow)

PT = Pressure transducer
FM = Flow meter
Take home points on progress to date

• Electrospinning is highly versatile and scalable route to reactive nanofiber mats

• Nanofiber routes may be a responsible route to environmental applications of nanotechnology

• Challenges remain in optimizing reactivity (e.g., surface area) and robustness

• Future work will explore long term performance testing, reactive longevity and regeneration
Acknowledgements

Cwiertny Group members
Dr. Danmeng Shuai: Electrospinning
Rebekah L. Oulton: CNT/O_3
Edgard M. Verdugo: CNT DBP
Katie Greenstein: Electrospinning
Jason Haase: CNT microfiltration
Kathy Peter: Electrospinning
Caylyn Lanzl: Nano iron oxide reactivity
Dr. Shen Qu: Cattle growth promoters
Undergrads: Connor Redmond, Matt Tarnoff, Sara Kaalberg, Meaghan O’Connor

Collaborators
Dr. Howard Fairbrother: Johns Hopkins
Dr. Nosang Myung: U.C. Riverside
Dr. Yong-Ho Choa: Hanyang University (Korea)
Dr. Rich Valentine: Iowa
Dr. Gene Parkin: Iowa
Dr. Tim Mattes: Iowa
Dr. Vicki Grassian: Iowa
Jon Durst: Iowa
Michael J. Nalbandian: U.C. Riverside
Kevin Wepasnick: Johns Hopkins
Dr. Jonas Baltrusaitis: Iowa CMRF
Dr. Gayan Rubasinghe: Iowa

Funding