

A High-Performance Electropositive Filter

Fred Tepper and Leonid Kaledin

Reverse osmosis (RO) membranes have become the industry standard for purification of any feed water source. They are used extensively for producing high-purity, low-conductivity process water. They are also used for producing ultrapure water from fresh water because RO is presumed to be the ultimate purifier.

Because RO filters are readily fouled by colloidal particles and biofilms, some method of prefiltration is used to protect them. Backwashable ultraporous membranes (UP) are used as prefilters in medium- and large-volume systems, and disposable nonwoven depth filters are used for smaller RO systems. Ion-exchange resin and granular carbon beds are also used upstream of RO, but such prefilters are themselves often fouled by particulate contaminants such as humic and fulvic acids.

A multibarrier RO system has been developed by Kinetico–Pall for point-of-

use (POU) drinking water applications (1). That device passed the National Sanitary Foundation's (NSF) certification to the Environmental Protection Agency's (EPA) standards (2). A recent article showing data on POU systems suggests that RO is unreliable for removing bacteria to the EPA standard (3). The authors believe that leakage is due to minor flaws in the membrane. For POU applications, they propose using a small "high-flow" hollow-fiber membrane postfilter.

SEPARATION BY ELECTROADHESION

We developed a new type of filter (NanoCeram brand), that filters particles by electroadhesion. An electropositive nanoalumina fibrille, only 2 nm in diameter, is the active component. It is end-bonded to a microglass fiber (Photo 1), and the composite is wet formed into a nonwoven filter. The filter's average pore size is between 2 and 3 μm . Cilia-like nanoalumina fibrilles are exposed to fluid passing through the media, providing a highly active surface that adsorbs electronegative particles even at high flow rates (~1 cm/sec). Most particles are electronegative and become more so at submicron size (particularly nanosize).

Microglass is electronegative in a neutral solution. Table 1 shows how the zeta potential becomes electropositive for increasing ratios of nanoalumina. The zeta potential reaches +32 mV when nanoalumina reaches 25 weight percent. Beyond 40% nanoalumina, there isn't enough space on the microglass; any excess fills the pores of the media,



NanoCeram brand cartridges
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increasing the pressure drop. We have standardized on 35% nanoalumina when using 0.6- μm microglass fibers. Cellulose and polyester fibers are added to provide sufficient flexibility to allow the media to be pleated.

Calculations show that electropositive forces on the surface of nanoalumina can modify the path of a small particle from as far away as 1 μm , attracting it until it attaches to the nanoalumina. The result is a shrinking of about 50% of the aperture in the 2–3- μm pore, through which the particle must traverse with no effect on its flow path. Because the particle has to traverse hundreds of pores before exiting a single layer of media, it has a very high probability of being adsorbed. The filter can therefore retain colloidal particles, small bacteria, and viruses at flow rates far beyond that of UP membranes. Particles larger than 3 μm are retained mostly on the filter's surface.

NanoCeram filters come in two different design formats: a pleated

PRODUCT FOCUS: ALL BIOLOGICALS

PROCESS FOCUS: FEED WATER PURIFICATION IN MANUFACTURING

WHO SHOULD READ: PROCESS DEVELOPMENT, MANUFACTURING, QA/QC

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LEVEL: INTERMEDIATE



Photo 1: Nano alumina fibrilles on a 0.6 µm-diameter microglass fiber

single-layer cartridge about 0.8 mm thick and a multilayer design in which the same thickness of media is wrapped around a perforated core. The pleated version is commercialized. Wrapped multilayer configurations are easily fabricated and offer higher retention of virus-size particles, but at the cost of higher pressure drop, so they are best suited in final polishing.

A single-layer filter has a very high dirt-holding capacity (DHC). We have shown (4) that the media's DHC is 20 or more times greater than data reported for 1-µm pore size microglass, melt-blown, and membrane media (5).

Comparisons with Electropositive

Media: A single layer of NanoCeram will filter >6 LRV (log retention value) of *Escherichia coli* or *Klebsiella terrigena* (~0.5 µm). Table 2 compares retention of *Brevundimonas diminuta* (0.3 µm) by one layer of NanoCeram compared with a competitor's electropositive medium. That medium comprises two copleated layers about 0.4 mm thick. Filter discs 25 mm in diameter were cut from its cartridge and used as a pair. The filters were challenged with a concentrated solution of *B. diminuta* at a flow rate of 10 mL/cm²/min. Samples of effluent were taken initially and then at 60 and 130 mL. Note that at pH 7.2, retention of the NanoCeram was still above 99.9% in the 130–140-mL fraction, whereas the other medium slipped below 98%. When the two media were tested at pH 9.2 or in the presence of salt water, the differences in retention were even more striking. At pH 9.2, NanoCeram's retention for the sample taken at 60 mL was 99.9% compared with 73% for the other media. In sea water, the two were 99.7% and 72%, respectively.

Table 3 shows a similar set of data, but this time using MS2 virus (25 nm), a bacteriophage often used as a virus simulant in filter testing. Neither filter can remove viruses to potable water standards. Nevertheless, NanoCeram

Table 1: Zeta potential and specific surface conductance of NanoCeram filters

NanoCeram Loading, wt%	Zeta Potential, mV	Surface Conductance, nS	MS Removal (%)
0	-35	0.92	8
5	-12	0.06	29
10	7	0.10	94
15	23	0.55	>99.9999
25	32	0.67	>99.9999
40	29	0.42	>99.9999
50	23	0.3	>99.9999

Table 2: Filtration of *B. diminuta* bacteria by electropositive media

Media	Thickness mm	Basic Weight g/m ²	Challenge Water			<i>B. diminuta</i> Removal, %		
			pH	TDS ^a g/L	BD ^b CFU ^c /mL	0–10 mL	60–70 mL	130–140 mL
NanoCeram	0.8	200	7.2	0	7 × 10 ⁵	99.997	99.97	99.93
			9.2	0	1.3 × 10 ⁶	99.99	99.9	
			7.2	30	1.2 × 10 ⁶	99.9	99.7	
			9.2	30	5.1 × 10 ⁵	99	98.5	
Other Media, Two Layers	0.8	210 ^d	7.2	0	7 × 10 ⁵	98.6	97.7	97.7
			9.2	0	1.3 × 10 ⁶	93.8	73	
			7.2	30	1.2 × 10 ⁶	92	72	
			9.2	30	5.1 × 10 ⁵	92	84	

^a total dissolved solids (TDS); ^b *Brevundimonas diminuta* (BD); ^c CFU = coliform forming unit; ^d two layers

Table 3: Filtration of MS2 phage by electropositive media

Media	Thickness mm	Basic Weight g/m ²	Challenge Water			MS2 Removal, %		
			pH	TDS ^a g/L	MS2 PFU ^b /mL	10 mL	60 mL	130–140 mL
NanoCeram	0.8	200	7.2	0	3 × 10 ⁵	99.0	98.0	94.4
			9.2	0	6 × 10 ⁵	90	90	
			7.2	30	5 × 10 ⁵	97	97	
			9.2	30	4 × 10 ⁵	96	88	
Other Media, Two Layers	0.8	210 ^c	7.2	0	6 × 10 ⁵	99.3	92	62
			9.2	0	3 × 10 ⁵	60	13	
			7.2	30	5 × 10 ⁵	4	6	
			9.2	30	4 × 10 ⁵	0	0	

^a total dissolved solids (TDS); ^b plaque forming unit (PFU); ^c two layers

removes more than 90%, whereas the other media type is virtually transparent to them when stressed at higher pH or with a high salt content.

COMPARISON WITH DEPTH FILTERS

To protect RO membranes from fouling, manufacturers suggest that a challenge stream be less than 1 NTU (nephelometric turbidity units) and have a silt density index (SDI) lower than 3. Pleated cartridges were challenged with

A2 test dust, which has an average particle size of about 5 µm. This test was accelerated by challenging the filters with a very high level of dust (250 NTU). Table 4 compares dust retention with that of filters from two other manufacturers. The NanoCeram filtered the dust to less than the detectable limit of our turbidometer (0.01 NTU). Measured SDIs were 0.5 and 0.2, both substantially below the recommended 3.0 level for SDI. The effluent from the 1 µm absolute filters of the two other

Table 4: Filtration efficiency of pleated cartridges

Manufacturer	Type	Flow Rate, gpm	Type of Water	Turbidity, NTU		SDI ₃₀ ^a
				In	Out	
Argonide	P2.5 × 10	4	A2 dust ^b in RO water	252	<0.01	0.2 ± 0.3 ^c
			Municipal water	0.87	<0.01	0.5 ± 0.1 ^d
A	1 µm absolute, 2.5-in × 10-in	4	A2 dust ^b in RO water	239	60	ND ^e
			Municipal water	0.54	0.10	4.4 ± 0.2 ^f
	0.35 µm absolute, 2.5-in × 10-in	4	A2 dust ^b in RO water	239	55	ND ^e
			Municipal water	0.57	0.14	4.6 ± 0.2 ^f
B	1 µm standard, 2.5-in × 10-in	4	Municipal water	1.3 ± 0.1 ^g	0.4 ± 0.1 ^g	Not tested
			A2 dust ^b in RO water	243	23	ND ^e
			Municipal water	1.63 ± 0.2 ^g	<0.01 ^h	5.5 ± 0.2 ^f
	5 µm standard, 2.5-in × 20-in	4	Municipal water	1.5 ± 0.7 ^g	1.1 ± 0.4 ^g	ND ^e

^a Silt density index (SDI₃₀); ^b ISO 121030-1, A2 fine test dust from PTI Inc.; ^c average of six; ^d average of four ^e Not done because turbidity of filtered water was too high — should be <1 NTU; ^f average of three measurements; ^g average over three hours test; ^h after 5 minutes of continuous flow.

Table 5: Bacteria removal by silver-treated pleated NanoCeram 2.5 × 5-in cartridges at 2 gpm

Day	Filtered Tap Water, Gallons	Bacteria	Bacteria Concentration, CFU/mL ^a	Treated LRV ^b	Untreated LRV
1	60	BD ^c	5.4 × 10 ⁴	5.4	2.8
2	360	BD	6 × 10 ⁵	6.7	2.6
3	720	BD	7 × 10 ⁵	6.7	4.7
4	1080	BG ^d	2.1 × 10 ⁶	7.8	4.6
5–6	Stagnation				
7	1380	BG	5 × 10 ⁵	6.6	4.6
8	Stagnation				
9	1740	BG	1.7 × 10 ⁵	>5.0	3.0
10	1860	BG	3 × 10 ⁵	>5.1	4.7
11	2160	EC ^e	7 × 10 ⁵	>5.7	4.6
12–13	Stagnation				
14	2400	EC	2.1 × 10 ⁶	>6.3	4.3
15	2880	EC	5 × 10 ⁶	>6.4	4.7

^a Coliform forming unit (CFU); ^b Log retention value (LRV); ^c *Brevundimonas diminuta* (BD); ^d *Bacillus globigii* (BG), variant Niger; ^e *Escherichia coli* (EC)

manufacturers could not be measured for SDI because the turbidity level was too high. When we tested the latter two cartridges with our municipal water, which tends to average only about 1 NTU, the SDI was still excessive.

A 2.5 × 10-in. NanoCeram cartridge was tested by a customer for carbon dust loading. This test involved recirculating a mixture of 5 g carbon dust (3–5 µm) through the filter at a flow of 18 Lpm (liters per minute) while noting any leakage of carbon into the effluent. The customer's metric was that an acceptable filter had to retain all 5 g, with no detection of carbon in the effluent. The NanoCeram cartridge

passed that standard. An additional 50 g of carbon dust was added to the tank, and the resulting mixture was passed through the same cartridge, again with no evidence of carbon leakage. Then an additional 100 g of carbon was added. The mixture in the tank was almost clear when the test was terminated because the pump overheated. The filter had retained nearly all 155 grams of carbon, with none detected in the effluent. By test's end, the flowrate had declined to about 10 Lpm.

Once we produced our own filters, we started to use them as replacement for the 2.5–10-in. sediment filter (a meltblown) in our own "Aqua FX" RO system. So far, in

Table 6: MS2 retention projected for a cylindrical filter 2.5-in diameter, 10-in high

No. of Layers	Thickness, mm	MS2, LRV ^a	Initial Pressure Drop ^b , psi (bar)
1	0.8	>2.5	3 (0.2)
2	1.6	>5.0	6 (0.4)
3	2.4	>7.0	9 (0.6)

^a Log removal value (LRV); ^b flowrate is 1 gpm

about six months of service (~2000 gallons of water), we have seen no changes in conductivity of the product water. Before its use, it was necessary to replace the sediment filter about once a month because of excessive increases in conductivity. Ion-exchange and carbon filters also required frequent changeout because of sediment that bypassed the original filter, causing discoloration and apparent contamination.

In another test, a 2.5–5-in. cartridge was challenged by our municipal water. At a flow rate of 1.5 gpm (gallons per minute), the filter removed colloidal matter from 6800 gallons to less than 0.01 NTU. At that point, there was leakage of turbidity, reaching about 0.2 NTU until the test was terminated at about 11,000 gallons as a result of excessive pressure drop.

Silver-Treated NanoCeram: A

proprietary silver solution was added to a 2.5–5-in. cartridge, and the filter was then challenged with various bacteria during an ongoing test series — now up to 2880 gallons (Table 5). Its retention of different bacteria was compared with that of an unimpregnated cartridge. Each filter was challenged by untreated municipal water containing residual chlorine. In the first three days, both filters were challenged with *B. diminuta*. Several days later *Bacillus globigii* was used, and on day 11, we changed to *E. coli*. Note that in every case, the treated filter exceeded the untreated one by a considerable margin, and in some cases the improvement was up to 4 LRV.

MULTILAYER FILTERS

Multilayer filters provide a deep bed capable of high virus retention and also providing sufficient sites for interfering particles such as humic acid. Figure 1 shows retention of MS2 as a function of filter thickness and for two different flow rates. The results show some experimental

data points compared with those projected from a model we developed using 30-nm latex spheres. The model is very useful in projecting filter retention and filter life (breakthrough curves) as a function of particle concentration and flow rate. The effect of pH has been found to be minimal between the values of 5 and 9. Data show that virus can be retained to 6 LRV using a filter 2.7 mm thick (four layers) at 40 mL/cm²/min flow. At 10 mL/cm²/min, only three layers would suffice, provided that the virus concentration is less than about 10⁴ PFU/mL (plaque-forming units/mL).

Table 6 shows the MS2 retention as a function of layer thickness projected for a tubular filter 2.5-in diameter × 10-in. high. The calculations presume a nominal flowrate of 1 gpm. The pressure drop for each variation is also shown. A three-layer filter would have a very high initial retention, and at a ΔP lower than 1 bar. But because a very small virus will slowly migrate through the medium, more layers would be needed to achieve the same high levels of retention. Efforts are currently under way to develop a cartridge that will provide six months' worth of drinking water and meet EPA guidelines (2).

AIR FILTRATION

The high DHC and high efficiency we see in water is also seen when NanoCeram is used as an air filter. A highly porous NanoCeram is efficient for filtering liquid aerosols and dry particles (NaCl aerosol) from an air stream. These lower-pressure-drop formulations have pore sizes ranging from 30 to 40 μm. Yet they can retain 99.995% of 0.3 μm particles at a pressure drop equivalent to HEPA. If a thin layer of such media is used as a prefilter for HEPA, it will reduce the rise in ΔP that ordinarily occurs when HEPA is loaded. The resulting filter life is then about five to 10 times greater than that of bare HEPA.

We also tested such filters by challenging them with aerosolized bacteria. In comparative testing of filters with aerosolized *E. coli*, we achieved >99.9998% retention compared with 99.992% for a HEPA media.

HEALTH AND SAFETY CONSIDERATIONS

Potential hazards of nanoscale materials in general and fibrous ones in particular have caused concern in the industry. Our only filter component that has yet to be used in potable water filters is the nanoalumina. We measured 50 μg/L in the effluent, which is below EPA's 200 μg/L level and below that of the aluminum content found in many sources of water.

Boehmite, the same mineral form as nanoalumina, has been used for decades as an orally ingested analgesic that can be purchased over the counter. Our form has a higher surface area and dissolves more rapidly. We measured the dissolution rate of the nanoalumina in simulated stomach acids (pH = 3.5 with pepsin) and found that it dissolves in about 16 hours at room temperature and probably much more rapidly at body temperature. The oral ingestion of nanoalumina from our filters would therefore not appear to be a health or safety concern.

RELIABLE OPTIONS

NanoCeram is an electropositive filter media with a high capacity for particles even in the presence of salt and alkaline. It filters bacteria, protozoa, viruses, endotoxins, DNA/RNA, latex spheres, submicron metals, activated carbon dust, natural organic matter, and fine test dust (mostly silica). It has a high dirt-holding capacity, far exceeding that of microglass, meltblown, and membranes, providing users with longer intervals between filter change-out. A pleated single-layer cartridge has a flow rate comparable with that of a conventional depth filter of about 2-μm pore size. However, it is comparable in bacteria retention to a 0.2-μm absolute membrane. The media's bacteria retention can be increased further by adding a silver compound.

Nonwoven fibrous (depth) filters are inherently more reliable than membranes. They can be pictured as a series stack of filters, providing redundancy in depth. A defect at one level is compensated by layers beneath, in contrast with membranes, for which a point defect can result in substantial increases in leakage. Another major advantage is their higher flowrates. Their principal deficiency is a relatively lower filtration efficiency than that of membranes. Electropositive media can bridge the gap, providing high retention at moderate to high flow rates as well as a high DHC. Such media are particularly beneficial for filtering submicron particles.

One of the most important applications for NanoCeram is to protect RO membranes from being fouled by particles. Such fouling is predominantly caused by ultrafine particles, including bacteria that can coat the surface of a membrane. Other types of filters, including activated carbon (GAC) and ion-exchange (IX) beds, can be similarly fouled by ultrafines. GAC is susceptible to fouling by humic acid, and IX beds are often fouled by colloidal iron oxides. NanoCeram media filter both types of particles.

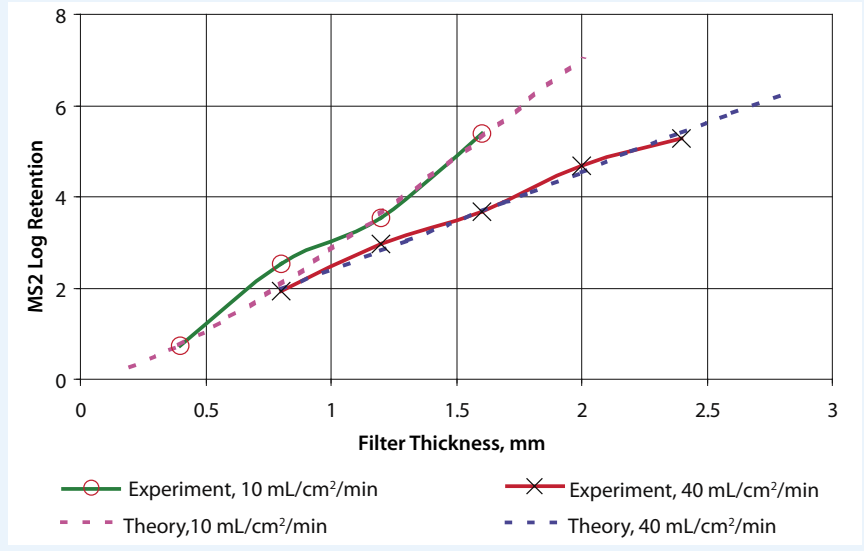
A multilayer version of the filter is a candidate for polishing water downstream of an RO membrane. It is easy to fabricate and would cost less than hollow-fiber cartridges.

Electropositive media also can benefit ultraviolet and ozone treatment systems by filtering organic particulates upstream of those devices, improving their efficiency. Another important application is the polishing of hazardous particles from waste streams.

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Figure 1: Comparison of MS2 retention with model



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Corresponding author **Fred Tepper** is
president and **Leonid Kaledin** is a senior
scientist at Argonide Corporation, 291 Power
Court, Sanford, FL 32771; 1-407-322-2500, fax
1-407-322-1144, fred@argonide.com;
www.argonide.com.

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