ULTRA HIGH EFFICIENCY PLEATED DEPTH FILTER

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Overview & Summary

- A dual pleated layer depth filter cartridge with electropositive properties (NanoCeram[®] or "NC") is capable of greater than 6 LRV of virus and 7.5 LRV of E coli retention, at high flowrate and with high dirt holding capacity.
- An alternate cartridge includes powdered activated carbon, (NanoCeram-PAC or "NC-PAC") is capable of retaining both small particles and soluble contaminants with high efficiency.
- Both have wide application for purifying drinking water and in industrial, food and drink and pharmaceutical manufacture.
- Data are presented comparing the performance of these filters as compared to a number of different depth filter media's as well as membranes.
- Dual layer NC or NC-PAC is capable of better virus retention than most of the depth media as well as a charged 0.1 micron membrane at flowrates that are ~ 25 times higher than the tightest of the depth media or the membrane
- The retention of small particles or virus can deteriorate substantially when in the presence of salt or at high alkalinity

About Argonide

- Founded in 1994 to develop nano technology products;
- Invented the NanoCeram[®] filter media ("NC") in 2000, based on a nano alumina fiber
- Invented a combined chemical and biological filter NanoCeram[®]-PAC ("NC-PAC")
- Invented an air filter media capable of retaining high levels of virus

Electron Microscopic Image NanoCeram® Fibers

The active ingredient of the filter media is a nano alumina (AlOOH) fiber, only 2 nanometers in diameter.



The nano fibers are dispersed and adhere to glass fibers and appear as a fuzz on the microglass fiber.

The composite fiber is formed into a nonwoven media by wet processing. The water filter's pore size is ~ 2 microns.

Since the nano alumina fibers are dispersed and fixed in place, particles have easy access to their charged surface.

Zeta Potential & Virus Removal

NanoCeram [®] Wt % on Glass	Zeta potential, mV	MS2 Retention (%)
0	-35	8
5	-10	29
10	7	94
15	12	>99.9999
32	50	>99.9999

Zeta potential becomes positive with increasing nano alumina and this causes a marked increase in retention of small particles such as virus (MS2).

T4 Phage crowded onto NC fiber composite



Courtesy E. Helmke, Alfred Wegener Institute, Germany

Sample prepared by immersion of media into concentrated T4 solution. Three phages are encircled as examples, where the icosahedral head and tail are seen. The high density of electropositive charges results in attraction and adhesion of colloidal particles such as virus.

PAC Filter

- Powdered activated carbon (PAC) or any nanoparticle can be retained in the NanoCeram structure
- The ~ 8 µ PAC particles are held in place by electroadhesive forces due to nano alumina fibers
- No adhesives are used
- There is minimal shedding of particles
- The result is a carbon filter media (NC-PAC) with very high dynamic sorption capacity for sub-micron particles as well as physiosorbable contaminants

Chlorine Removal Comparison of PAC



Chlorine attacks most membranes. NC-PAC is several hundred times more efficient for retaining chlorine than other commercially available carbon media.

Filter Characteristics and Virus Retention of NC and NC-PAC

Media	Thick,	Basis weight q/m^2	Challen	ge water	MS2 removal ^{b,c} ,	
			рН	TDS ^a g/L		
NC-PAC	0.8	220	7	0	<mark>3.5</mark>	
One layer			9.5	0	<mark>2.3</mark>	
			7	30	<mark>2.2</mark>	
NC-PAC	0.8+0.8	220+220	7	0	<mark>>6.5</mark>	
Two layers	=1.6	=440	8.5	0	<mark>>6.4</mark>	
			7	30	<mark>>6.6</mark>	
			8.5	30	<mark>>6.4</mark>	
NC	0.8	220	7	0	<mark>3.0</mark>	
One layer			9.5	0	<mark>2.3</mark>	
			7	30	<u>2.2</u>	
NC	0.8 + 0.8	220+220	7.0	0	<mark>6.0</mark>	
Two layers	=1.6	=440	8.5	0	<mark>5.5</mark>	
			7	30	<mark>4.8</mark>	
			8.5	30	<mark>4.1</mark>	

Notes: a) Total Dissolve Solids (TDS) – sea salts; b) 5 ml aliquots were passed through 25 mm disc at rate of 10 ml/min and collected into sterile vial; c) green color stands for high LRV (>5), yellow color stands for moderate LRV from 2 to 5.

Filter Characteristics and Virus Retention Competitive Depth Media

Media	Pore size,	Thick,	Basis $u_{\rm arg}^2$	Cha	llenge water	MS2
	μm	IIIII	weight, g/m	рН	TDS ^a g/L	LRV
Manufacturer A	1.1	5.0	1140	7	0	<mark>>6.5</mark>
				8.5	0	1.2
				7	30	0.2
Manufacturer A	5.0	5.0	1140	7	0	<mark>0.7</mark>
				8.5	0	0.1
				7	30	0.1
Manufacturer B	0.6	2.6	1430	7	0	<mark>>6.6</mark>
				9.5	0	<mark>5.2</mark>
				7	30	0.3
Manufacturer C	0.8	4.1	1571	7	0	<mark>6.8</mark>
				9.5	0	<mark>4.8</mark>
				7	30	<mark>2.3</mark>
Manufacturer C	1.3	4.1	1480	7	0	<mark>5.1</mark>
				9.5	0	0.1
				7	30	0.7
Manufacturer C	1.4	4.1	1470	7	0	1.0
				9.5	0	0.2
				7	30	1.2
Manufacturer D	0.1	0.15	60	7	0	0.3
charged memorane						

Notes: a) Total Dissolve Solids (TDS) – sea salts; b) 5 ml aliquots were passed through 25 mm discs at rate of 10 ml/min and collected into sterile vials; c) green color stands for high LRV (>5), yellow color stands for moderate LRV from 2 to 5 and red color stands for very low LRV (<2).

Virus Retention of NC, NC-PAC and Electropositively Charged Membrane



Filtered volume through 25 mm discs, mL

Filtration of Sub-Micron Organic Particles



Membranes and NC media are challenged with humic acid, a particle smaller than a virus. Note that both NC and NC-PAC had far better retention than even a 0.025μ membrane (which clogged almost immediately).

Virus retention by mixed cellulose ester membrane filters

Mean pore size, mm	Flow velocity ^a , cm/min	MS2 removal, %
0.025	2 ^b	<mark>73± 3</mark> °
0.1	10	<mark>1 ± 11</mark>
0.22	10	<mark>0 ± 6</mark>
0.45	10	<mark>4 ± 6</mark>
1.2	10	<mark>1 ± 7</mark>

Notes: a) Fifteen mL aliquotes of MS2 solution at input concentration of $3.5*10^{6}$ PFU/mL were passed through 25 mm diameter membrane at flow rate 40 mL/min through ~4 cm² surface area (corresponds to 10 mL/min) of 1.2 mm, 0.45 mm, 0.22 mm and 0.10 mm membranes; b) Pressure drop for the 0.025 µm membrane was higher than 40 psi at the above flow rate therefore we were using ~5 times lower flow rate through the 0.025 mm membrane, i.e., 8 mL/min (or flow velocity ~2 cm/min); c) red color stands for very low MS2 retention (<1 LRV or <90%).

Is there a flaw in Absolute membrane ratings?

- The previous slide suggests that there is major slippage of biological particles through membranes that have smaller pores than the size of the particles
- This was observed by Ch. Wick and P. E. McCubbin¹. MS2 has a reported MW of 2 million Daltons. Yet they found that MS2 virus passed through filters rated at 300 K Daltons
- The phenomena of "grow-through" of bacteria is known to occur through media rated smaller than their nominal size
- We suggest that biological particles are comprised of viscous fluid encapsulated by membranes, so that they are not rigid particles but are sufficiently plastic to penetrate
- Adsorption of such particles by electroadhesion rather than by mechanical filtration, could circumvent this flaw

1. Charles Wick and P. E. McCubbin, U S Soldier and Biological Command, Filtration Characteristics of MS2 Using Various Molecular Weight Filters – 1999- ECBC Rept TR-054

Virus Retention Conclusions

- NC-PAC in two layers has equivalent or better virus retention than other depth filters including the smallest pore size ones
- NC has lesser virus retention than NC-PAC, but it is still much greater than other depth media with the exception of the finest grade
- Both NC and NC-PAC have better retention than the 0.1 µ pore size charged membrane, even at neutral pH.
- The virus retention of the competitive depth filters falls substantially or is nil when in the presence of highly alkaline or conductive (salty) matrixes, while NC and NC-PAC are minimally affected by salt or alkalinity.

Bacteria Retention by NC and NC-PAC Media

Media	Thick,	Basis	Challe	nge water	E. coli	B. diminuta	
	mm	g/m^2	pН	TDS ^a g/L	LRV	LRV	
NC-PAC	0.8	220	7	0	>6.0	<mark>>8.7</mark>	
One layer			9.5	0	<mark>≥6.3</mark>	<mark>>6.2</mark>	
			7	30	>6.2	<mark>>5.5</mark>	
NC-PAC	0.8+0.8	220+220	7	0	>7.5	<mark>8.0</mark>	
Two layers	=1.6	=440	9.5	0	<mark>>6.5</mark>	<mark>>6.2</mark>	
			7	30	<mark>>6.5</mark>	<mark>>5.9</mark>	
			8.5	30	<mark>>6.5</mark>		
NC	0.8	220	7	0	<mark>>6.0</mark>	<mark>4.5</mark>	
One layer			9.5	0	<mark>>4.5</mark>	4.7	
			7	30	<u>4.2</u>	<u>5.2</u>	
NC	0.8+0.8	220+220	7.0	0	>7.5	<mark>>7.6</mark>	
Two layers	=1.6	=440	9.5	0	<u>6.3</u>	<mark>>6.2</mark>	
			7	30	>7.0	<mark>>5.9</mark>	
			8.5	30	<mark>>7.0</mark>		

Notes: a) Total Dissolve Solids (TDS) – sea salts; b) 5 ml aliquots were passed through 25 mm discs at rate of 10 ml/min and collected into sterile vials; c) green color stands for high LRV (>5), yellow color stands for moderate LRV from 2 to 5.

Bacteria Retention by Competitive Depth Media

Media	Pore	Thick,	Basis	Cha	llenge	E. coli	B.
	size,	mm	weight,	W	ater	removal ^{b,c}	diminuta
	μm		g/m^2	pН	TDS ^a	LRV	removal ^{b,c}
					g/L		LRV
Manufacturer A	5.0	5.0	1140	7	0	1.1	
				8.5	0	<mark>1.2</mark>	
				7	30	1.5	
				8.5	30	<mark>1.8</mark>	
Manufacturer B	0.6	2.6	1430	7	0		<mark>8.0</mark>
				9.5	0		<mark>>6.5</mark>
				7	30		<mark>>5.5</mark>
Manufacturer C	0.8	4.1	1770	7	0		<mark>>7.6</mark>
				9.5	0		<mark>>6.5</mark>
				7	30		<mark>>5.</mark> 5

Notes: a) Total Dissolve Solids (TDS) – sea salts; b) 5 ml aliquots were passed through 25 mm discs at rate of 10 ml/min and collected into sterile vials; c) green color stands for high LRV (>5), red color stands for very low LRV 2.

Bacteria Retention of Depth Media Conclusions

- Better than 6 LRV of E coli and >5.9 LRV of B diminuta were retained by two layers of NC and NC-PAC whether in alkaline or in the presence of salt
- E coli retention by Manufacturer A's 5 µm media was poor
- The loss of retention at high pH or with salt was not significant in the case of the competitive depth media with particles much larger than a virus. The presumption is that these larger particles are retained primarily by mechanical filtration, rather than being dependent on electroadhesion.

Flowrate of Various Depth Media



Retention and Dirt Holding Capacity of Test Dust

Media	Pore	Number	Thick,	Total	Flow	Dirt	β ratio
	size,	of layers	mm	Weight	velocity (in	holding	-
	μm			σ/m^2	cm/s) at	capacity ^{a,b}	
				5/111	$\Delta P=3 psi$	mg/cm ²	
NC	2.0	1	0.8	220	0.226 ^c	58	>25,000
					0.111 ^d		
NC-PAC	2.0	1	0.8	220	0.286 ^c	57	>25,000
					0.114 ^d		
Manuf. A media	1.1	1	3.8	1280	0.014	49	>25,000
Manuf. A media	5.0	1	5.0	1140	0.230	119	<mark>360</mark>
Manuf. B media	0.6	1	2.6	1430	<mark>0.006</mark>	22	>25,000
Manuf. C media	0.8	1	4.1	1770	0.007	13	1000
Manuf. C media	1.4	1	4.1	1470	0.015	27	<mark>270</mark>
Manuf. D	0.1	1	0.14	60	0.010	34	>25,000
Charged membrane							

Notes: a) A2 fine dust produced by PTI, Inc.; b) to terminal pressure of 30 psi at input concentration of 250 NTU (0.4 g/L); c) single layer; d) double layer; e) red color stands for low flow velocity and β value

Flowrate Remarks

- With the exception of Manufacturer A's coarsest media (~5 μm), both NanoCeram and NC-PAC (both 2 μm pore size) in two layers have a higher flowrate than any of the other media that were tested.
- A single layer of NC or PAC has a higher flowrate than any of the depth media but one (Manuf. A- 5 µm).
- At DP=3 psi, a single layer of NC or NC-PAC has a flux approximately 130 LPM/m^{2.} This is 25 times as great as the 0.1 µm membrane as well as the smallest pore size media tested, and yet both NC's have much greater virus clearance

Turbidity Retention of Media's



Dirt Holding Capacity Conclusions

- With the exception of Manufacturer A's 5 micron depth filter, NC and NC-PAC retain more test dust than any of the other media's tested.
- Of all of the depth media, only NC or NC-PAC is thin enough to be pleated – Both NC and NC-PAC, when in a pleated cartridge, would have higher dirt holding capacity than any of the competitive planar media tested including Manufacturer A's 5 micron media
- Several grades of depth media show early leakage of test dust therefore substantially reducing their β value as compared to NC and NC-PAC

Bio testing of NC and NC-PAC cartridges

Cartridge	Cartridge	Cartridge	Number of	Flowrate,	MS2,	Bacteria	removal	Cyst
	Height,	Diameter,	layers	GPM	removal,	Bacteria	LRV	removal,
	cm	cm			LRV ^{a,e}	Ductoriu		LRV ^{a,r}
PAC2.5x5	12	7	1	2	<mark>3.4</mark>	КТ ^ь	<mark>7.1</mark>	>3.7
P2.5x10	24.5	7	1	4	<mark>3.1</mark>	КТ ^ь	>7.1	<mark>>3.7</mark>
DP2.5x10 ^c	24.5	7	2	4	<mark>>6.0</mark>	E. coli	<mark>7.4</mark>	ND ^d
PAC2.5x20	50	7	1	8	<mark>>4.5</mark>	E. coli	7.3	>3.7
P4.5x10	24.5	11	1	8	<mark>>4.5</mark>	E. coli	7.5	>3.7
P4.5x20	50	11	1	22	<mark>>4.3</mark>	KT ^b	>5.0	>3.7

Notes: a) Logarithm retention value; b) Raoutella terrigena (deposited as Klebsiella terrigena); c) double pleated; d) ND – not done; e) green color stands for high LRV (>5), yellow color stands for moderate LRV from 2 to 5 and red color stands for very low LRV (<2); f) WQA tested single layer NC-PAC for Cryptosporidium (cysts) using latex sphere surrogates. The cartridges exceeded the drinking water requirements of >99.95% (>3.3 LRV) retention according to NSF/ANSI 53 International Standard.

Third party testing of cartridges

Initial Retention Efficiency per ASTM F795-88 using Black Iron Oxide Contaminant^a at High Alkalinity (pH 9.5) and Elevated Temperature (65°C)

Cartridge ^b	Port		Particle size range ^d , microns						
		0.1-0.25	0.25-0.5	0.5-0.6	0.6-0.8	0.8-1.0	>1		
Argonide P2.5x10 ^c Cartridge	Upstream particle count	283849	168736	46095	19008	8710	13959		
	Downstream particle count	67	21	15	<1	<1	<1		
	Net Efficiency, %	<mark>99.98</mark>	<mark>99.99</mark>	<mark>99.97</mark>	<mark>>99.995</mark>	<mark>>99.99</mark>	<mark>>99.993</mark>		
Manuf. E P2.5x10 ^c Cartridge	Upstream particle count	349554	282356	85009	38698	25328	27382		
	Downstream particle count	123199	24671	3484	2479	896	114		
	Net Efficiency, %	<mark>64.76</mark>	<mark>91.26</mark>	<mark>95.90</mark>	<mark>93.59</mark>	<mark>96.46</mark>	<mark>99.58</mark>		

Data Courtesy of DCS Control

Notes: a) Test was done by IBR Laboratories, Grass Lake, MI; b) Flow Rate – 4 USGPM; c) c/w double 222 o-rings & alignment fins; d) Upstream/Downstream numbers appearing in the Table, are the number of test particles for a size range counted by the laser device MC100S s/n M1029.

Conclusions about Cartridge Testing

- With NC and NC-PAC, the results obtained to date show excellent scalability from discs to cartridges
- Bacteria retention was higher than 5 LRV with a single pleated layer and higher than 7.4 LRV with double layer (NC)
- WQA tested single layer NC-PAC pleated cartridges for Cryptosporidium (cysts) using latex sphere surrogates. They exceeded the drinking water requirements of >99.95% retention for cysts
- Testing by IBR for ultrafine particles shows very high retentivity by NC over the whole particle size range, achieving 99.98% retention of particles between 0.1 and 0.25 microns, *at pH 9.5 and 65° C!*

Comparable Endotoxin Removal

Endotoxin removal by NanoCeram filters via the LAL method

Challenge	Filter	Filtration	Filtered	Influent	Effluent	Retention
waters	thickness, mm	rate,	volume per	Endotoxin	Endotoxin	factor ^b ,
		ml/cm ² /min	sq. cm,	concentration	concentration	%
			ml/cm ²	EU/ml	EU/ml	
Water +	1.5 = 2 layers	10	0.8	10	0.12	<mark>98.8</mark>
endotoxin	1.5 = 2 layers	10	0.8	235	< 0.12	>99.95
	1.5 = 2 layers	2	1.4	$4.8 \cdot 10^5$	$9.7 \cdot 10^3$	<mark>98.0</mark>
5% Dextrose in	1.5 = 2 layers	2	1.4	$4.2 \cdot 10^5$	<0.6	>99.9999
Endotoxin free						
water						
1% Liposyn ^a II	1.5 = 2 layers	2	1.4	$4.2 \cdot 10^5$	$9.7 \cdot 10^3$	<mark>97.7</mark>
in Endotoxin						
free water						

Notes: a) Liposyn II is an intravenous fat emulsion. A 10% composition contains: 5-wt% safflower, 5-wt% soybean oil, up to 1.4–wt% egg phosphatides (as emulsifier) and 2.5-wt% glycerin b) green color stands for high endotoxin retention (>99%), yellow color stands for moderate retention from 90% to 99%.

Endotoxins, which are a serious particulate contaminant in pharmaceuticals, can be as small as a virus and are difficult to filter. NC has a high endotoxin removal efficiency

Safety & Environmental

- There are significant concerns about the impact of nanomaterials on health, safety and on the environment.
- Nanoalumina fibers also known as boehmite (AIOOH) fibrilles, have been used in antacids such as Maalox for many years before being replaced by Al(OH)_{3.} The latter is lower in cost and dissolves more rapidly in stomach acids.
- Boehmite nanofibers have also been used extensively in vaccines. In that application they are called adjuvants. They have been FDA approved for 63 years as a primary component of baby shots and flu vaccines. More than 2 billion doses have been used in humans and countless more in veterinary vaccines.
- Numerous studies of occupational exposures of all aluminas, including boehmite, used in industry are essentially inert in the lungs*
- Aluminum in all forms, shed or dissolved from NC filters has been shown to be <20 ppb (via ICP/MS).

(*Dinman, Graduate School of Public Health, Univ Pittsburgh, 1990, in Alumina Chemicals Science and Technology Handbook, Amer Ceramic Soc)

Emulsified Oil Removal by NC

•Low levels of oil or oil emulsions are often an undesirable contaminant

- •Current technology is not effective for polishing low levels of emulsified oil after oil skimming and coalescing systems.
- •NanoCeram or NanoCeram-PAC can retain emulsified oil



Dual Layer NC and PAC Cartridges



Dual pleated layer 2.5 and 5" diameter cartridges fit in standard housings

Lenticular Filter Replacement



The dual pleated layer NC (or NC-PAC) cartridge on the left is a drop in replacement for the lenticular filter (right)

NC and NC-PAC as Alternates to Membranes

- There is a large and growing market for fine particle depth filters as well as for MF/UF membranes
- Approximately 20% of all membranes sold are for filtering particulate
- MF/UF membranes are used to produce pure and ultrapure water in a number of industries:
 - > Microelectronics
 - Food & Drink
 - Pharmaceutical manufacture
 - Automotive manufacture
 - Protecting RO membranes
- NanoCeram filters have substantial advantages over charged depth filters as well as MF/UF membranes
 - Less expensive than membrane cartridges or charged membrane
 - Flux tens to hundreds of times greater than membranes
 - Higher filtration efficiency
 - Greater dirt holding capacity then most depth media and all membranes tested
 - No water waste as compared to membranes that might be used in tangential flow

Removing Colloidal Iron and Copper Two Case Studies

- Colloidal iron can pass through a 25 nm pore size membrane and would not be intercepted by any of the competitive depth media
- Toyota has a 5 million gallon chilled water system in their Kentucky plant, with over 20 Miles of 4" pipe and larger + seasonal flow and dead legs
- Significant corrosion was observed in the piping system, causing grave concern and cost in the event of replacement
- The corrosion is caused by iron bacteria that use colloidal iron as a food source
- NC removed all measurable colloidal iron, manganese and iron bacteria in Toyota's pilot test
- Another user found that NC double pleated cartridges, used in a chiller, retained both iron and copper (probably as oxides or hydroxides), while Manufacturer A's lenticular was transparent to both these metals.

Features and Benefits of NanoCeram

- NanoCeram[®] are cost effective filters for removal of sub-micron particles from water
- NC-PAC has somewhat better particulate retention than NC and at the same time has a high dynamic efficiency for retaining chlorine
- NC and NC-PAC's performance exceeds depth filters and MF/UF membranes in virtually all respects
- A single layer NC or NC-PAC filter covers the gamut of particle size ranges from micron to a few nanometers and does so at moderate to high flowrate and dirt holding capacity.
- And it can be used as an prefilter to protect RO in fresh water and seawater

We create and make great things

THANK YOU

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