NANOCERAM® FILTERS

Winner of 2005 Space Foundation Hall of Fame Award

Argonide Corporation, Sanford, Florida
www.argonide.com

R&D AWARD WINNING MICROBIOLOGICAL FILTERS 2002

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

NASA

U.S. AIR FORCE CROSS INTO THE BLUE

DEPARTMENT OF ENERGY UNITED STATES OF AMERICA

DTRA

SUMMARY

- We invented a revolutionary microbial filter, capable of retaining sub-micron particles with high efficiency, yet it has a high flow rate
- The active ingredient in the filter is a nano alumina fiber that is highly electropositive
- The filter retains all types of particles, including silica, natural organic matter, metals, bacteria, DNA and virus.
- It has high capacity for micron size as well as nano size particles.
- Pleated filter cartridges are now available for sale.
- It has wide applications in chemical, microelectronic, and pharmaceutical manufacture, food and drink, cleaning coolants, prefilters for reverse osmosis and for cleansing drinking water.

About Argonide

- Founded in 1994 to develop nano technology products

- Initial products were nano metal and oxide powders developed by scientists in Siberia. R & D done by these scientists was cost shared by Argonide and the U S Department of Energy

- These scientists developed the nano alumina fiber and are currently investigating other nano fiber chemistries

- Fred Tepper, founder of Argonide, who had prior experience in respiratory filters, invented the NanoCeram® filter media based on the nano alumina fiber

- A U S patent has issued and others are in the pipeline

- The NanoCeram® technology received the 2005 Hall of Fame award from the Space Foundation

US GOVERNMENT SPONSORSHIP

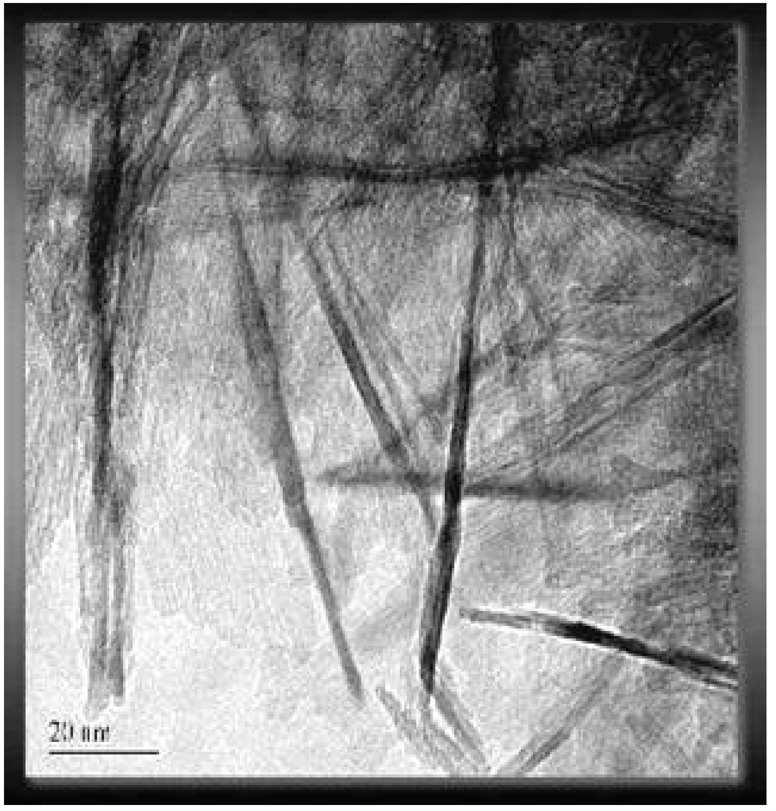
- **NASA (Phases I & II) – Filter for purifying recycled water for long duration space flight**
- **Department of Energy – Cooperative agreement to support Siberian scientists in nano technology**
- **Environmental Protection Administration – Development of point of use arsenic sorbent**
- **Tyndall Air Force Base - Portable water purifier**
- **Tyndall AFB – Air filter development**
- **Ciencia Corporation (prime contractor to DTRA) – Biological agent collection and concentration for Ciencia's detector**

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 highly electropositive.

Other components of pleated cartridges are cellulose, polyester and glass fibers plus an organic binder.

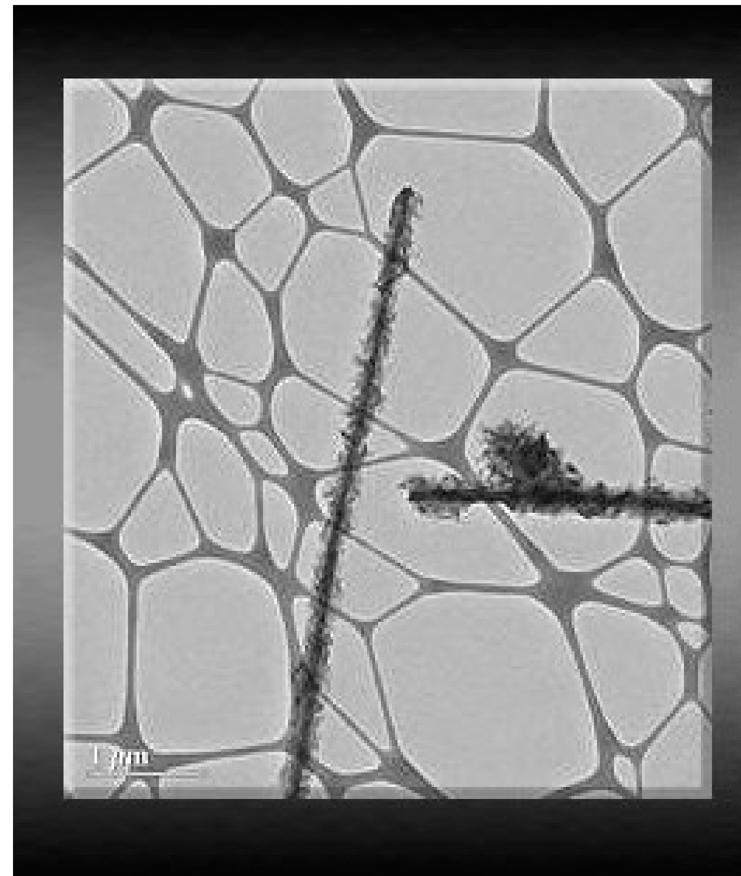


Nano Alumina On Microglass Fibers

The nano fibers are first dispersed and adhere to glass fibers. The nano alumina is seen as a fuzz on the two glass fibers.

Other fibers are added and the mixture is processed at a paper mill to produce a non-woven filter.

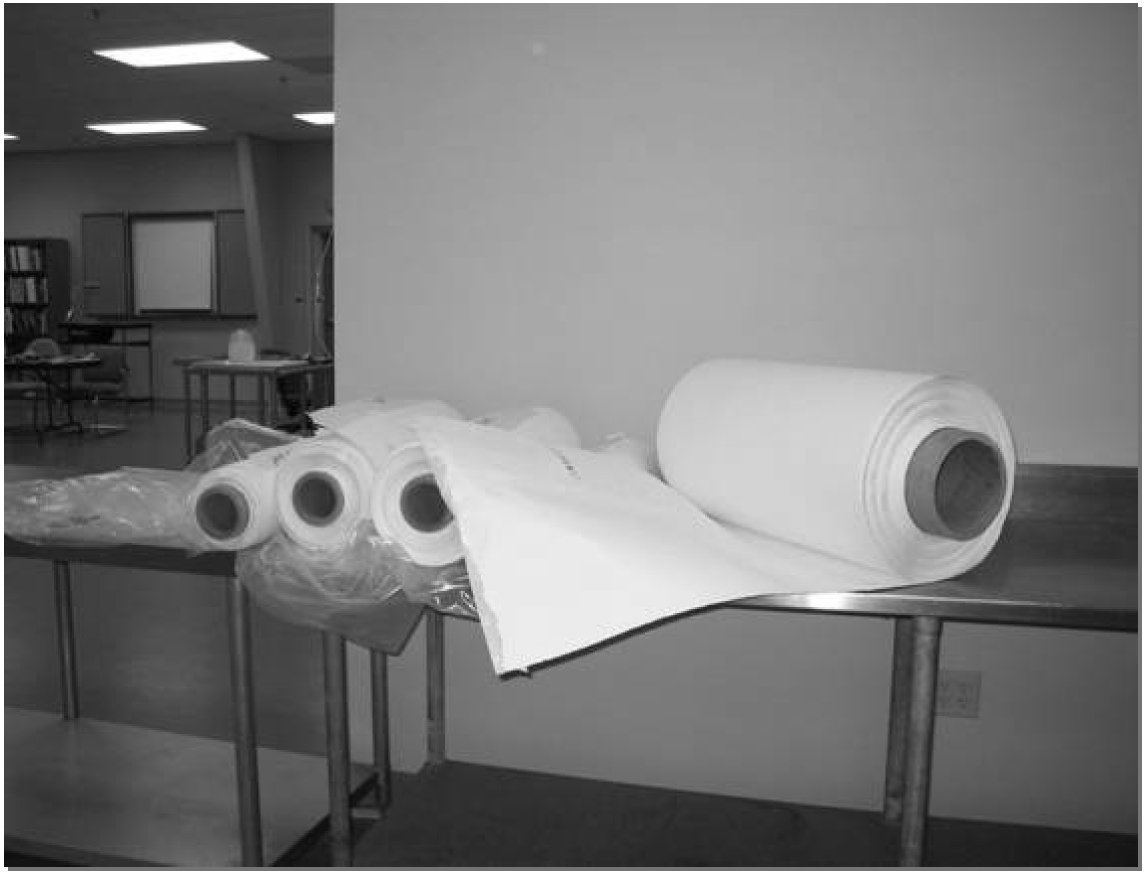
Because the nano alumina is dispersed, particles have easy access to the charged surface.



Features of NanoCeram® Filters

- Flow rates (flux) tens to hundreds of times greater than ultraporous membranes
- NanoCeram separates particles by charge rather than size
- Higher retentivity for virus than "Absolute" ultraporous membrane filters
- Endotoxin removal > 99.96%
- DNA removal > 99.5%
- Resistant to clogging by fine and ultra fine particles
- Pleated versions have 5-8 times higher dirt holding capacity than typical cartridges
- Filtration efficiency for micron size particles >99.995%

Pilot lot of NanoCeram media



A major cost barrier was overcome when continuous manufacture via paper-making technology was demonstrated

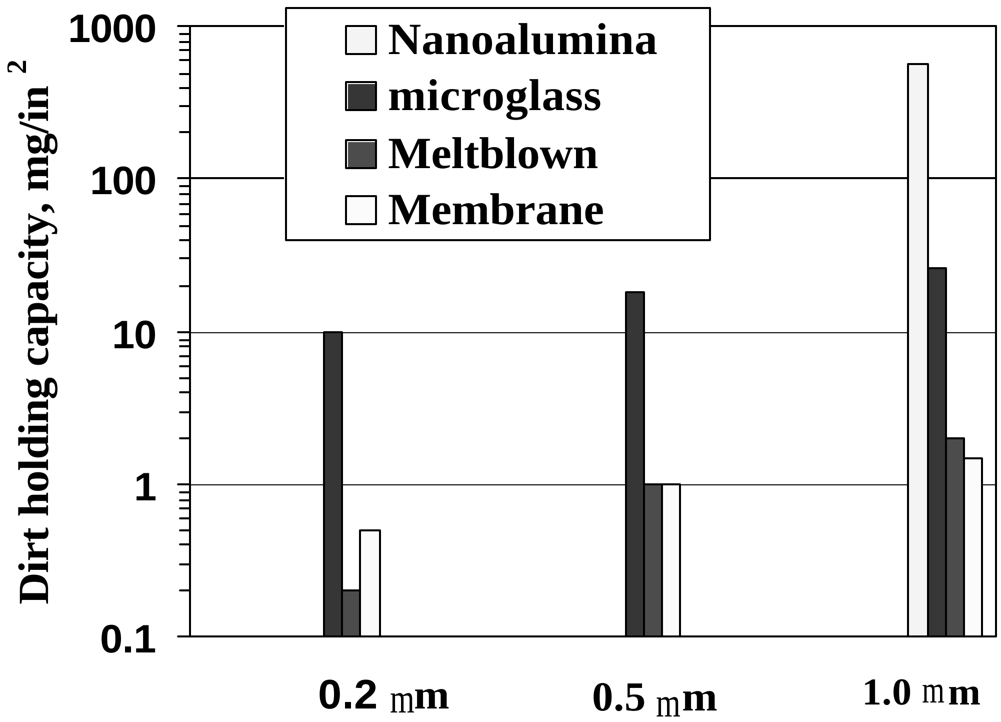
Zeta Potential & Virus Removal

NanoCeram® Wt % on Glass	Zeta potential, mV	MS2 Retention (%)
0	-35	8
5	-10	29
10	7	94
15	12	>99.9999
25	32	>99.9999
40	29	>99.9999
50	23	>99.9999

Zeta potential becomes positive with increasing nano alumina and this causes a marked increase in virus (MS2) retention

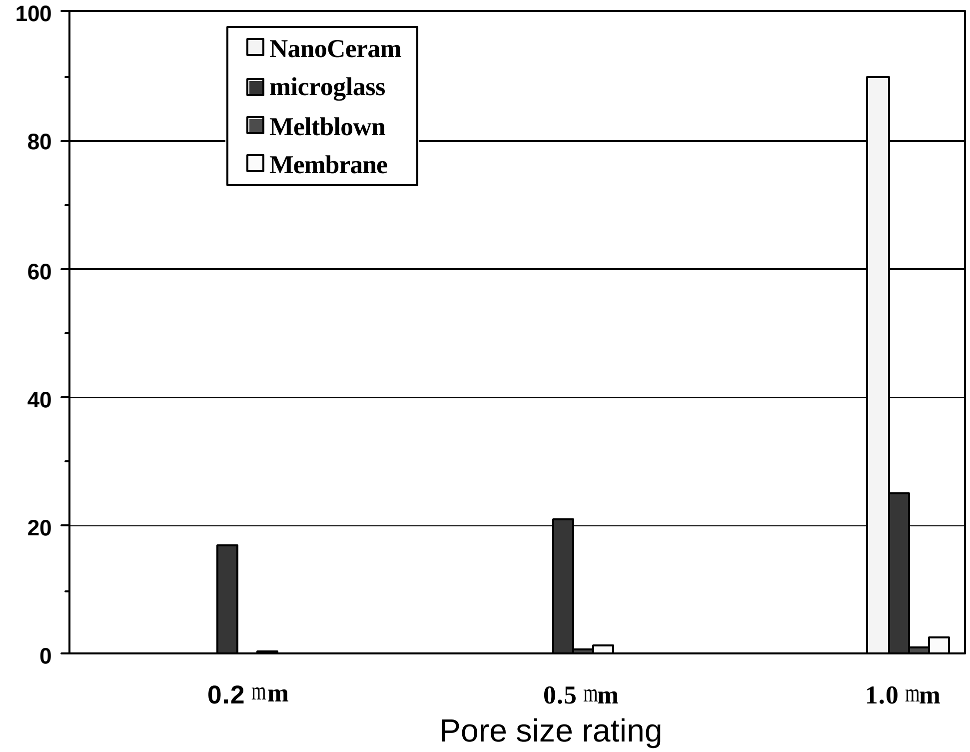
Filtering dirt particles

This compares the dirt holding capacity for A2 fine test dust (~1-4 µm) vs data presented by C. Shields for microglass, meltblown and membranes



It's dirt holding capacity of 574 mg/in^2 is almost twenty times greater than microglass filter media when compared at a pore size rating of 1 µm and far greater than that if compared at the smaller pore size ratings.

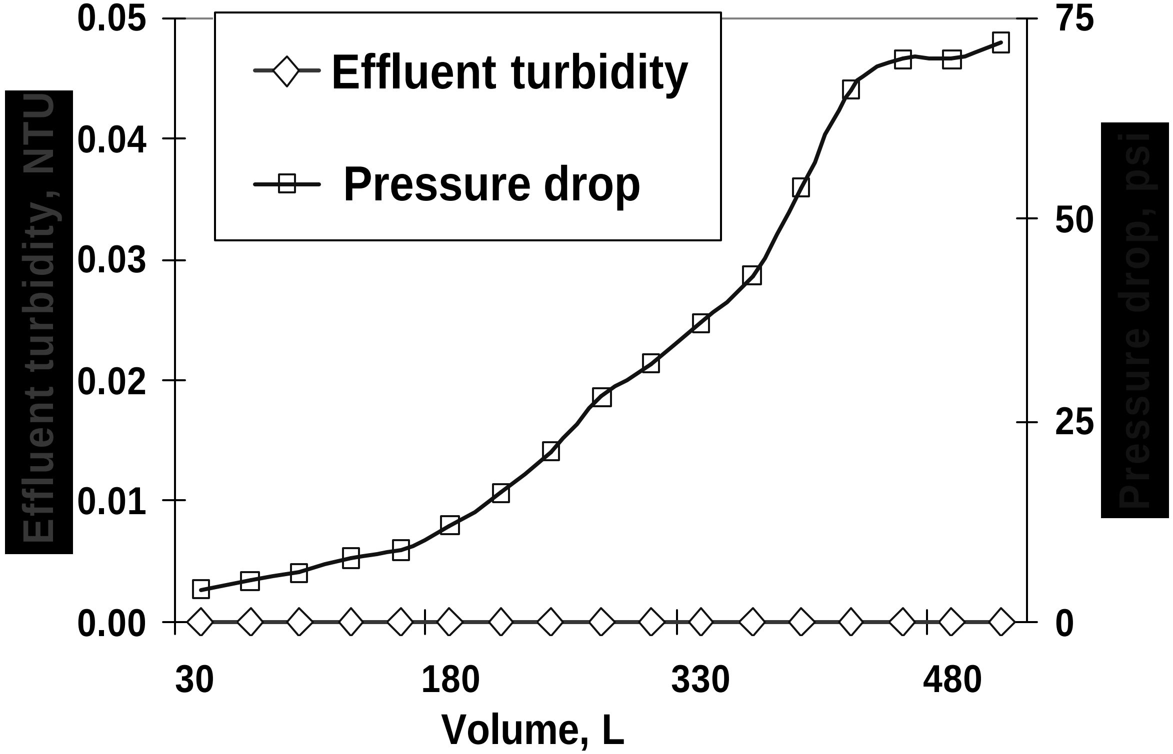
COMPARISON OF FLOW CAPACITY



This shows NanoCeram's flowrate, superimposed over Shields' data [1] for clean water flow of several types of filter media. The nominal pore size of NanoCeram is 2-3 µm average, providing a major benefit in flow vs. 1.0 µm pore size of all other medias. If compared at smaller pore sizes, the flow benefit of NanoCeram would be huge. Yet NanoCeram has higher filtration efficiency when compared to any of the others, irrespective of pore size rating.

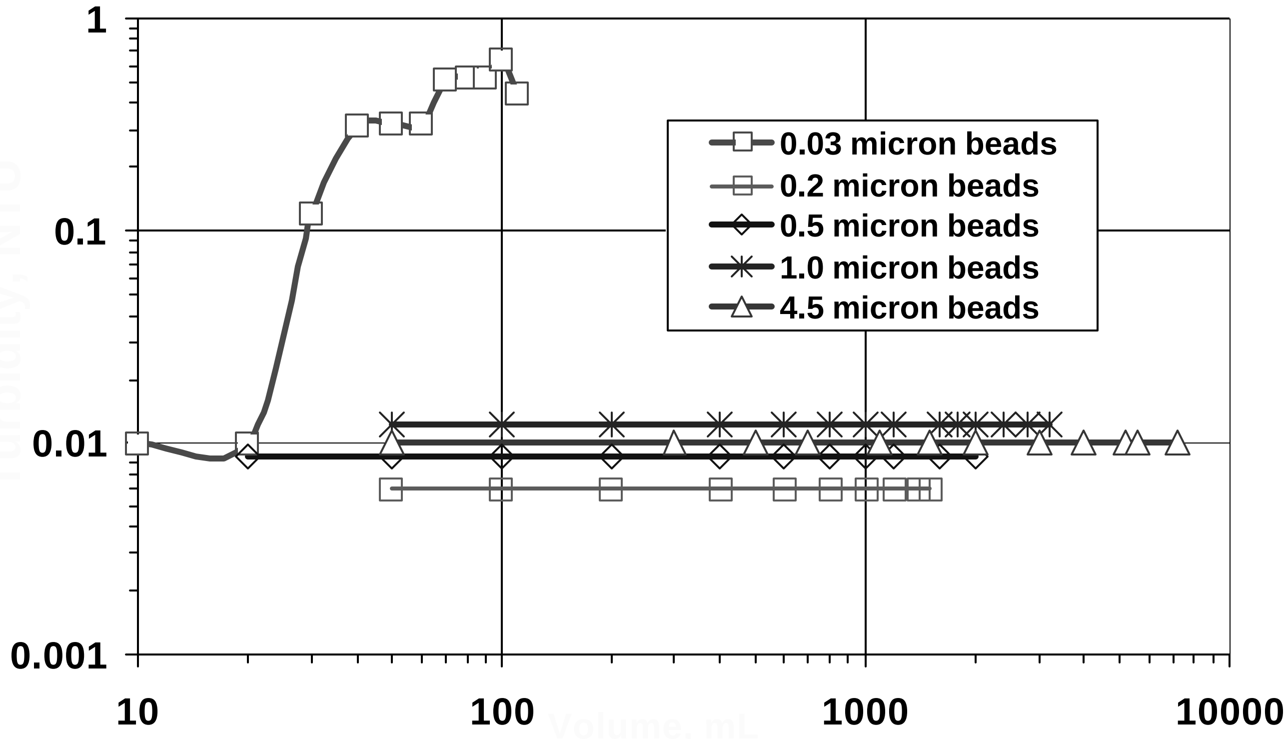
1 - C. Shields, High Performance Microfiltration Media, Presented at American Filtration Meeting, Marriott, Baltimore/Washington Airport, Nov. 16-17, 2004

Dirt holding capacity of a pleated cartridge



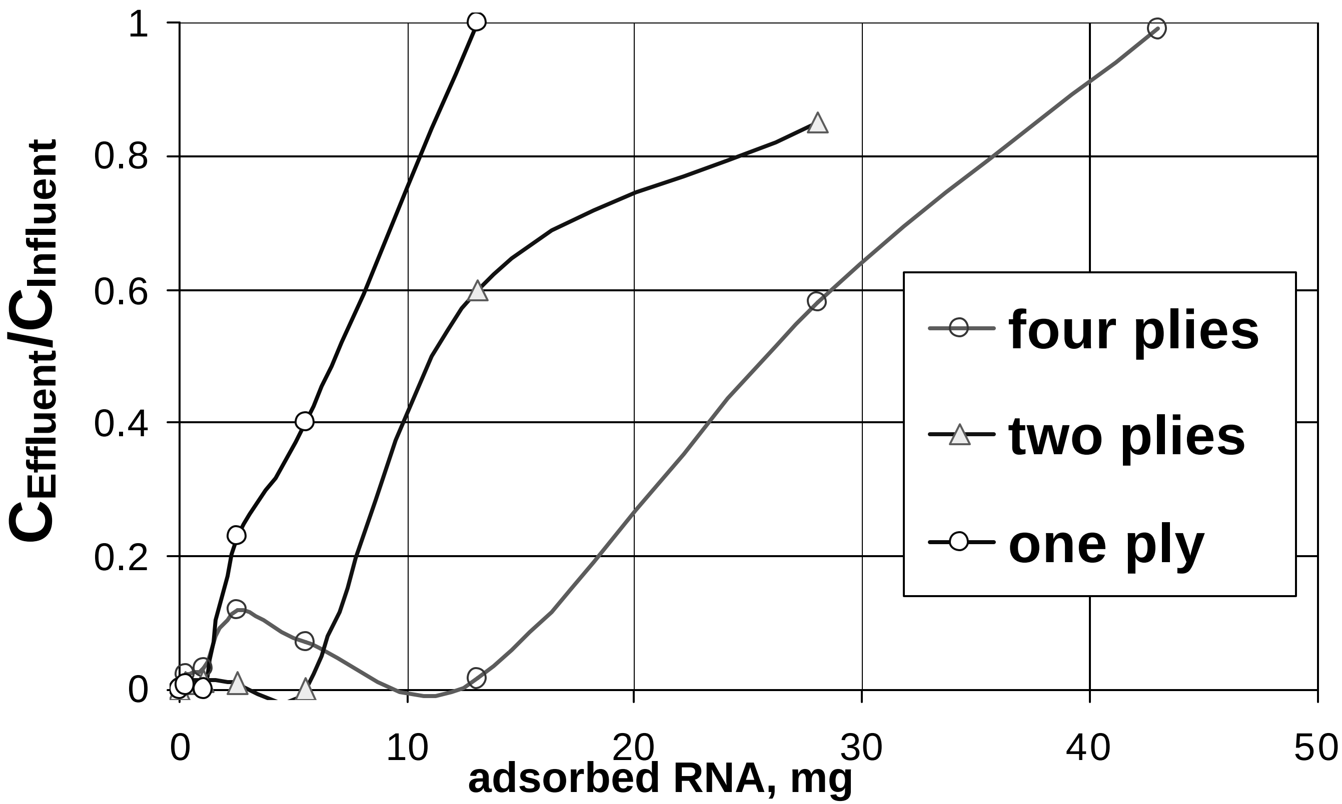
A 2.5” diameter X 5” pleated cartridge was challenged by 250 NTU of A2FTD @ 1.5 gallons/min. At 90 minutes (141 gallons) it had filtered out 119 grams of dust while producing an effluent < 0.01 NTU, at which point the test was terminated because of excessive pressure drop, but without breakthrough. It had removed >99.996% of dirt throughout the test.

Adsorption curves for different size latex beads



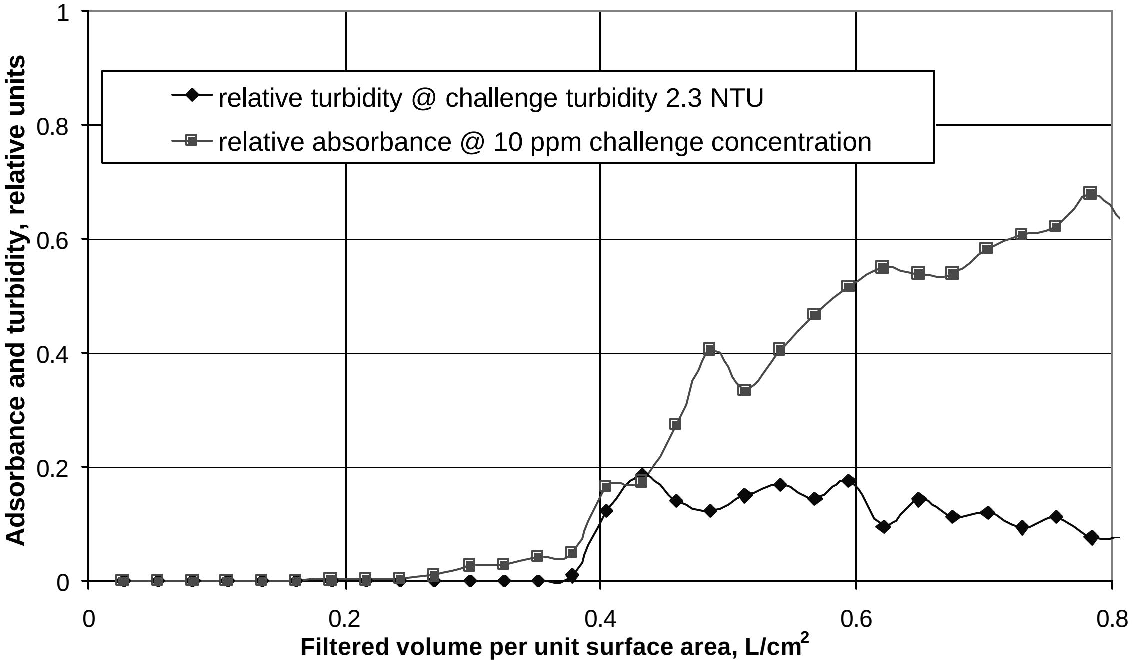
When a single layer of filter 25 mm in diameter was challenged by a continuous stream of latex beads it will eventually clog without exhibiting breakthrough, except with the smallest (0.03 µm) beads. Bacteria size particles (0.2 to 4.5 µm) are intercepted with high efficiency. A small virus would require a thicker filter in order to achieve 4 LRV.

RNA Adsorption Curves as Function of Thickness



DNA and RNA are filtered much like virus. The thicker the filter, the higher is the retention factor.

FILTRATION OF SUB-MICRON ORGANIC PARTICLES



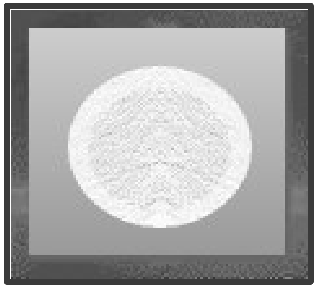
The filter is excellent for adsorbing turbidity. Filters (25 mm diameter) were challenged with humic acid, an organic particle small enough to pass through “Absolute” 0.2 µ filters. Breakthrough was detected by both optical turbidity and spectrophotometric methods. Note the high filtration efficiency until the filter is exhausted at about 0.4 L of fluid/cm^2 of filter area.

Nano ink adsorption – Slide # 1

A pigment ink with a particle size of about 2 nm was diluted until it was transparent.

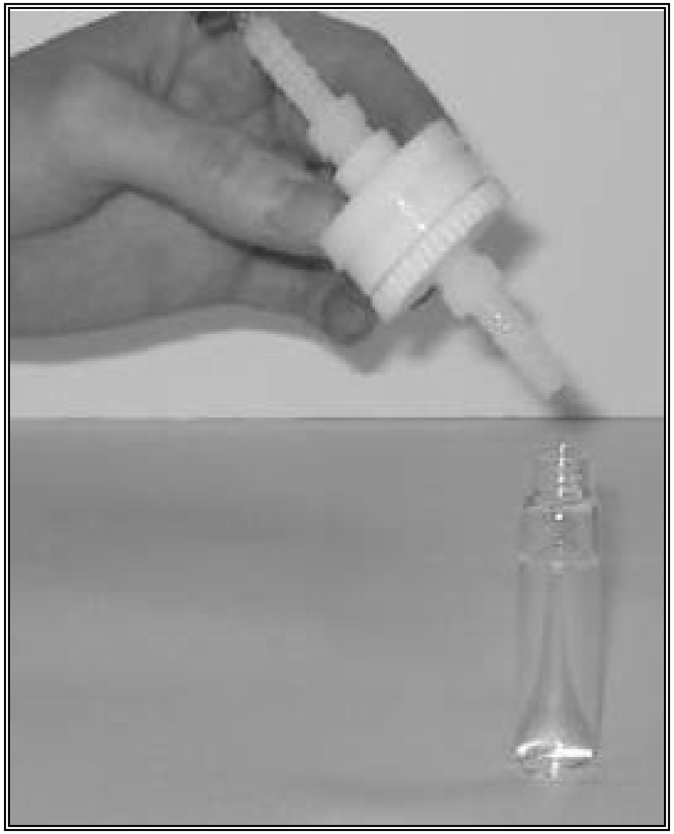
Ink Test - #2

When forced through a 25 nm ultraporous (UP) membrane (Millipore VS), the back pressure was very high. The small amount of ink that passed through the UP membrane was as colored as the influent.



After the test, the UP filter showed very little color change

Ink Test - #3



When the ink was injected through the NanoCeram® filter, the back pressure was low and filtration was easy. Yet the effluent came out water white. The filter (above) was intensely colored, proving that it retained nanometer size particles while the 25 nm UP membrane was ineffective.

Modeling Summary

Experimental data were gathered on the adsorption of 0.03 µm latex beads over a wide variety of conditions of concentration, bed thickness, temperature and pH

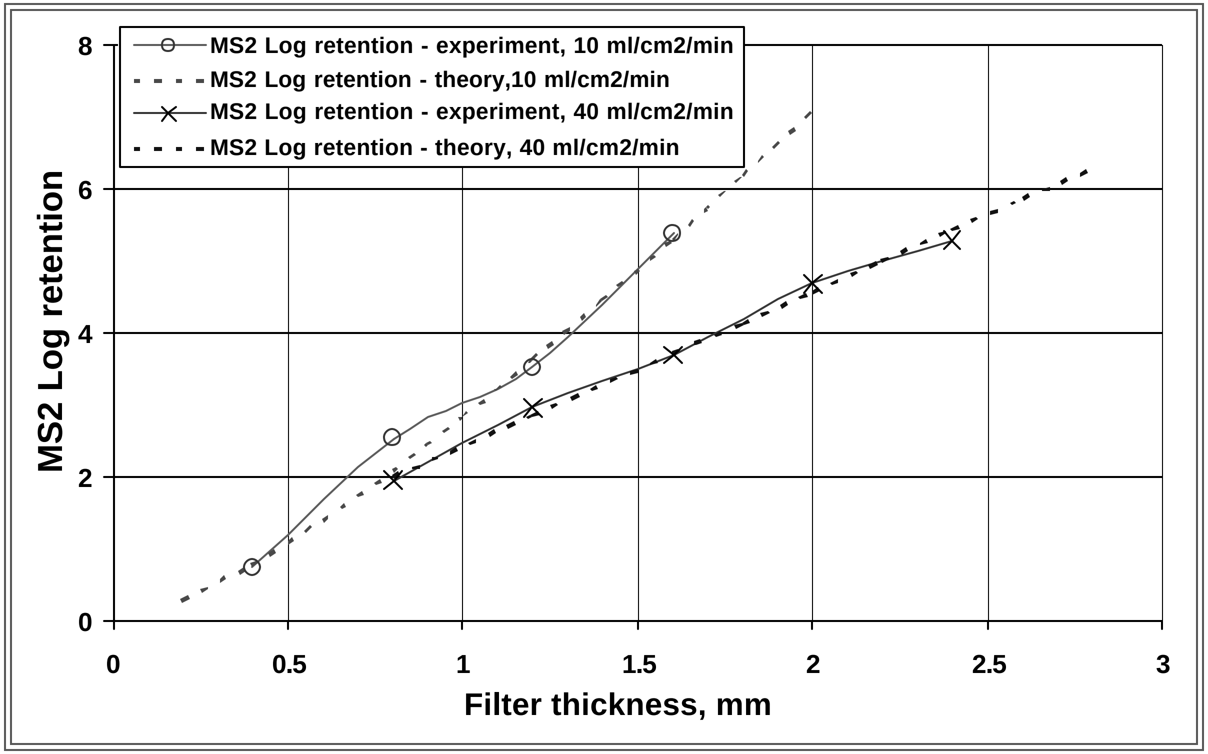
Particle retention was highly repeatable

Filtration efficiency was not measurably affected over pH 5-9, by simulated sea water or temperatures between 4 and 50+º C

A model was developed that predicts performance as a function of particle concentration, flowrate and filter thickness. The model is similar to classical ones describing adsorption of solutes from solvents.

The model has been used to scale the filters to larger area and higher retention efficiency

TESTING THE MODEL WITH VIRUS



The model was tested with MS2 virus (25 µm). This figure shows projected retention as a function of filter thickness for two different flow velocities. Experimental data show a high correlation with the model. Note that virus retention is predictable to at least 6 LRV.

Filter configurations

- Pleated filter – Large area with high flow, high dirt capacity, and high efficiency. Excellent for filtering particles down to about 0.1 microns.

- Thicker depth filters are necessary for achieving >99.99% retention of virus to meet EPA certification as a drinking water filter. Such filters are readily produced by wrapping the media around a mandrel. Flow rate is lower than pleated filters because of the thicker filter and also because pleated filters have higher area.

- Depth filter performance is dependent on a number of parameters. Projections require an assessment of the challenge stream. We invite inquiries about the capability and custom assembly of such filters

THE EPA STANDARD FOR DRINKING WATER FILTERS IS VERY CHALLENGING

- The filter must exceed 6 LRV for Klebsiella terrigena bacteria, 4 LRV for rotavirus and polio and 3 LRV for cysts, at the filters rated flow and capacity
- During test points (last 50% of capacity) when challenged by the above microbes, the challenge water must also contain:

  1 – Total dissolved solids of 1500 mg/l

  2 – Total organic carbon of 10 mg/l as humic acid (30 mg/l of humic acid)

  3 - pH of 9

  4 - A2 Fine test dust to bring the mixture to 15 NTU

  5 - 4º C test temperature

Other than reverse osmosis, there are no filters that have met this challenge! And RO is very slow, requiring storage tanks and cross flow filtration that wastes considerable water

DEVELOPMENT AND MANUFACTURING STATUS

Pleated cartridges 2.5” diameter, 5” and 10” long are currently available and 20” long will be available September 2005

Pleated 4.5”cartridges 10” and 20” long will be available in September

Design and development is underway for a cartridge suitable for certification to the EPA Guide Standard for Drinking Water Filters

Success has been achieved at the laboratory level in integrating an antimicrobial component into the filter media. The antimicrobial acts as a bacteriastatic and virus control, minimizing the potential for leakage of pathogens into the stream.

PLEATED NANOCERAM® CARTRIDGES

2.5” diameter cartridges – 5” and 10” long (P2.5-5 and P2.5-10) are shown.
The small unit is a prototype.
4.5” diameter X 10” long (P4.5-10) is shown.

SUGGESTED APPLICATIONS – PREFILTERS FOR REVERSE OSMOSIS MEMBRANES

RO filters are expensive to replace and are highly sensitive to fouling by sub-micron particles. Ultraporous (UP) membranes are typically used as RO prefilters. They too are subject to fouling, and are used in a cross-filtration mode to minimize fouling. Cross flow results in a waste stream, often 3-10 times greater than the stream being purified. NanoCeram® is used in a dead-end (direct flow) mode and there is no waste stream. Also NanoCeram has a very high capacity for sub-micron particles without needing to be cleaned as in the case of UP membranes

CLEAN-UP OF COOLANTS

- Water base coolants are used in cooling equipment and work space. The coolants can harbor pathogenic bacteria such as Legionella. Turbidity accumulation can occur on cooling surfaces to reduce efficiency. NanoCeram® is effective for removing bacteria and turbidity and because of its higher capacity, the cost of changing out filters is substantially reduced.

- Water base coolants are also used in cutting and grinding machines. The coolants are recycled and cleaned of suspended metals. Ultra-fine metal powders are generally not removed by conventional filters and recirculate, affecting tolerances and causing poor finishes. Many of these suspended particles contain nickel and other metals that are of concern as health hazards if ingested. Bacteria also build up in such coolants. When the fluid re-contacts the working surface the heat aerosolizes these objectionable contaminants into the breathing zone of the machine operator. NanoCeram® was tested and found to convert such blackened coolants to water white.

PHARMACEUTICAL AND BIOTECH

- Removal of contaminants from incoming water supplies is crucial – Prefiltered RO is a typical treatment, and two stage prefiltered RO is used for purifying water for injection (WFI). NanoCeram® prefilters would aid such processing.

- NanoCeram® is effective for retaining endotoxins, bacteria and virus

- Retention of 6 LRV or greater virus is attainable with customized multi-layer filters

- There is concern about endocrine disruptors in pharmaceutical waste streams. NanoCeram would filter many of these contaminants before disposal.

PROTEIN SEPARATION

- One ml of diluted mouse serum was added to 200 mg of NanoCeram® powder samples. After washing off non-adsorbed proteins with 0.1? tris-HCl (pH 6.8) the bound proteins were eluted with 0.5 M Na_2CO_3. Approximately 73% of the protein was recovered. Gel electrophoresis under fully denaturing conditions showed that protein adsorption was nonspecific. Protein adsorption was minimally affected by ionic solutions.

- NanoCeram media could be used to immobilize certain proteins and enzymes – probably those with a net electronegative charge. Such media could separate proteins on the basis of charge (or rather charge density).

FILTERING PROCESS WATER

- The large and growing market for membrane filters is driven mostly by reverse osmosis. However approximately 20% of all membranes sold are used for filtering particulates
- Ultraporous (UP) membranes are used to produce pure and ultrapure water in a number of industries including microelectronics, food and drink and medical device manufacture
- Because NanoCeram filters are less expensive, have a flux tens to hundreds of times greater than membranes, with a higher filtration efficiency and greater dirt holding capacity and wastes no water, they are far superior to UP membrane filters in such applications.

BIOLOGICAL SAMPLING

- Particles may be lifted off NanoCeram® surfaces by mass action using eluents that are also readily adsorbed. A mixture of 2.5% beef serum, 0.5% gylcine @ pH=9 was used to extract virus from a NanoCeram® cartridge. Such filters are being developed as a high volume sampler to assay virus in streams, lakes and seawater.

- Argonide has a contract to develop a concentrator/collector of biological agents for the purpose of detection by a specific bacteria/virus detector being developed by Ciencia Corporation (Hartford, Conn.). We have demonstrated recovery of 95% of MS2 virus where the initial virus (MS2) concentration was 10^3 PFU/ml. (Enrichment factor ~ 2000 times). Projected enrichment by a 2-stage system is projected to be > 1 million. The concentrator will be tested with bacteria and virus deposited via aerosol as well as from contaminated water.

OTHER APPLICATIONS

- Swimming pools and spas (we are tooling up for large (20+ gallons/min) capacity filters for pools and spas
- Processing food and drink- A Chianti wine with an initial sediment of 0.3 NTU was purified to less than 0.01 NTU by passing through one layer of filter. Excess yeast in beer could be removed using NanoCeram®.
- Prefiltration prior to ultraviolet or ozone treatment to minimize the burden on such sterilization schemes
- Filtering turbidity, cysts and bacteria from household well water
- Biological warfare filters for military, hospitals and other critical buildings

ARSENIC ADSORPTION

Under a grant from the U S Environmental Protection Agency, Argonide developed a granular arsenic adsorbent called "Alfox" This sorbent is currently in the laboratory stage but is being scaled up to the multi-kilogram level. The features of the sorbent are:

- Adsorption of As III without previous oxidation
- High dynamic capacity of As III and V – more than twice that of Bayoxide E-33 when measured at pH 8.5 and about four times greater when compared at pH = 6.5
- Bulk density of 1 g/cc as compared to about 0.5 g/cc for Bayoxide
- More resistant to attrition during flow than Bayoxide

Sponsors

2003– Phase I SBIR on Arsenic

2002-04, phase II SBIR on cleaning recycled space cabin water

Phase I on portable purifier (2004)
Phase I for air filter (2005)

Support of Russian WMD scientists on Arsenic, nanomaterials

DTRA

Subcontract from Ciencia 2004-2007

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