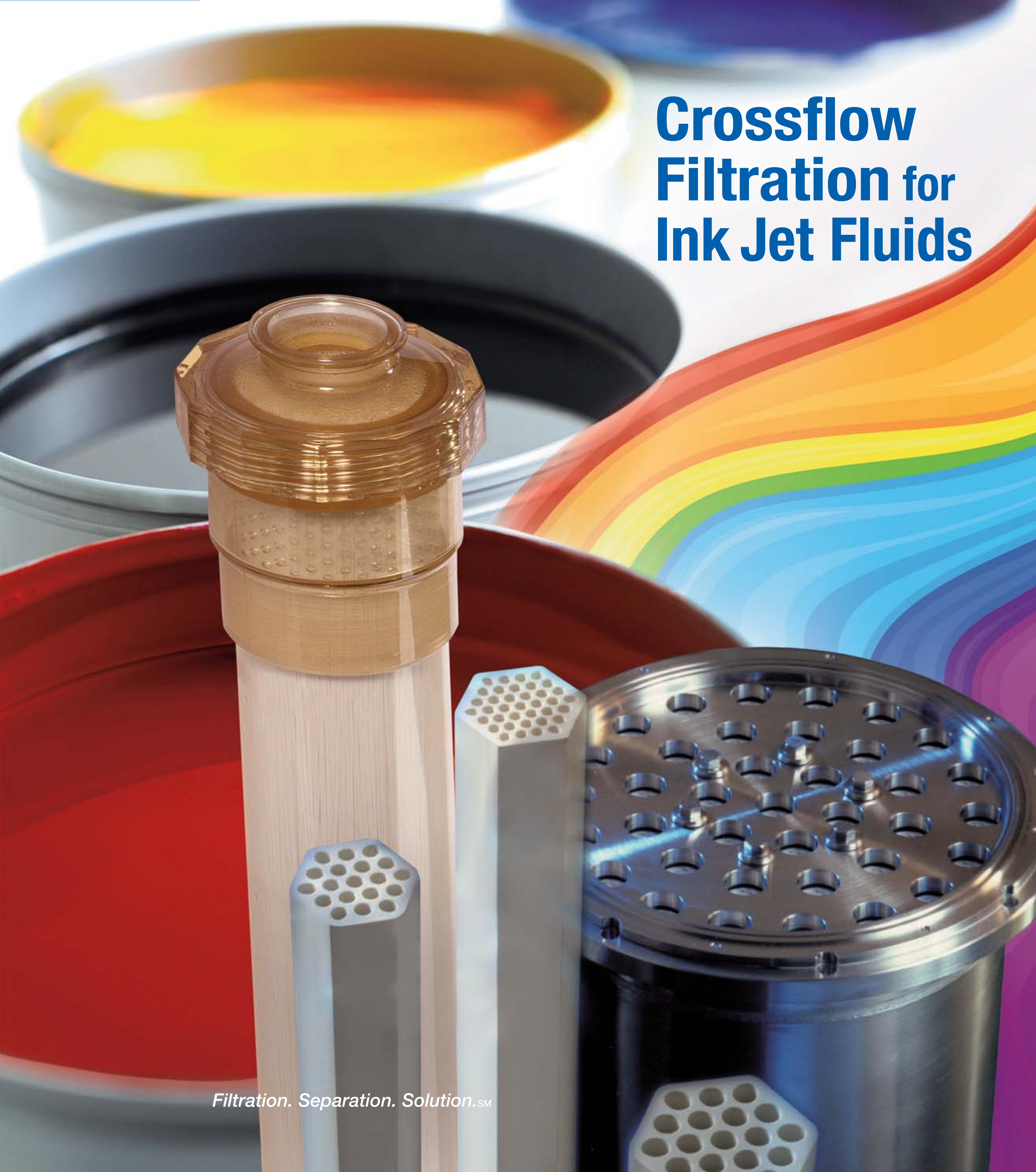
Pall Corporation

Crossflow Filtration for Ink Jet Fluids



Filtration. Separation. Solution.SM

For ink jet ink and colorant formulation, efficient filtration makes all the difference.

Improve your process and product with crossflow technology.

In recent years, ink jet ink and colorant formulators have faced an increasing number of challenges. The improved pigmented dispersions that are being developed have more complex chemistries, which cause the formation of micro-sized contaminant byproducts. Global competition has forced many ink jet ink and colorant formulators to reevaluate their processes and investigate new filtration and separation technologies to drive down processing costs. In addition, the move toward greener practices in industry has placed emphasis on water recovery and closed-loop regenerable processes.

Crossflow technology successfully addresses challenges for which traditional direct cartridge filtration is not feasible or practical. Pall Corporation is in a unique position as a supplier of a number of different crossflow technologies, among them Microza[1] hollow fiber and Membralox® ceramic membranes. Having applied a number of crossflow technologies to a variety of applications in diverse industries, Pall Corporation has developed expertise in crossflow products and supporting system design.

[1] Microza is a trademark of Asahi Kasei Chemicals Corporation.

Why Pall?

Established more than 60 years ago, Pall Corporation is recognized as a worldwide leader in filtration, separation, and purification. Our global presence is far reaching, and our product portfolio and technical expertise are extensive. Pall's engineered products enable process and product innovation while minimizing emissions and waste.

Pall Corporation is one of the leading suppliers to ink jet ink formulators, offering products and services specifically designed for the ink jet industry. Our staff scientists and engineers conduct research and development and provide technical support with intensive, broad-based assistance from Pall's worldwide network.They are knowledgeable about the chemistries, applications, and cleanliness procedures required for ink jet ink formulation. As part of a customized Total Fluid Management℠ (TFM) solution, our experts work directly with customers to select filtration products and services that ensure an efficient formulation process and excellent results.

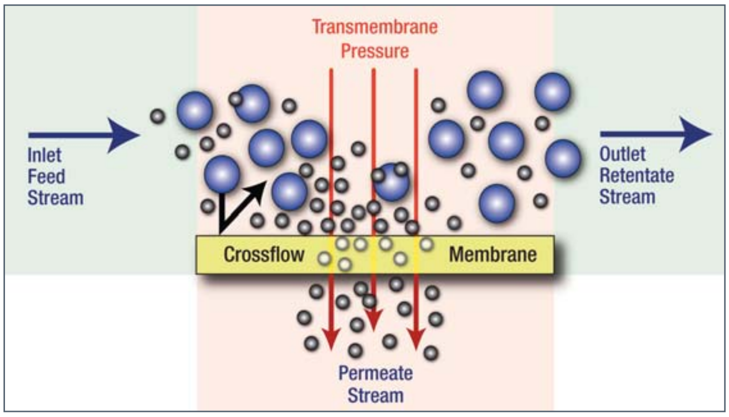
What is Total Fluid Management?

Pall's Total Fluid Management program involves the integration of properly selected filtration and separation equipment and services into a production process to yield the highest efficiency at the lowest cost. This program consists of a wide range of filtration products, advanced technologies, and services to improve system operation and increase productivity.

How Crossflow Filtration Works

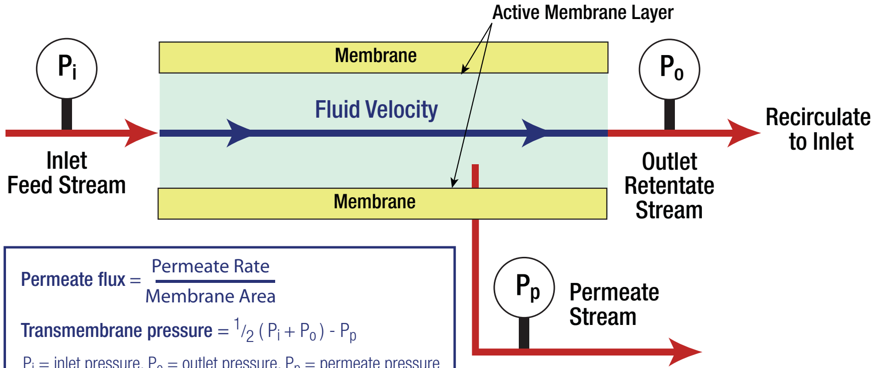
In crossflow filtration, an incoming feed stream passes across the surface of a crossflow membrane, and two exiting streams are generated. The permeate stream is the portion of the fluid that passes through the membrane. This filtered fluid will contain some percentage of soluble and/or insoluble components from the initial feed stream that are smaller than the membrane removal rating. The remainder of the feed stream, which does not pass through the crossflow membrane, is known as the retentate stream. (See Figure 1.)

Figure 1: The mechanics of crossflow filtration



As the feed stream passes across the membrane, the transmembrane pressure (TMP) drives the separation of the fluid.[2] The rapid crossflow at the active membrane layer continually sweeps away particles and other material, preventing buildup on the membrane surface.[3] As a result, crossflow membranes can delay fouling and maintain stable permeate flux rates.[4] (See Figure 2.)

Figure 2: Key crossflow parameters



$$\text{Permeate flux} = \frac{\text{Permeate Rate}}{\text{Membrane Area}}$$

$$\text{Transmembrane pressure} = \tfrac{1}{2}(P_i + P_o) - P_p$$

P_i = inlet pressure, P_o = outlet pressure, P_p = permeate pressure

---

[2] Increasing the transmembrane pressure typically increases the permeate rate up to a certain threshold value; however, this increase is not always proportional.

[3] Fluid velocity, the speed at which the fluid moves across the membrane, impacts both the fouling of the membrane and the permeate rate through the membrane. With increased velocity, the fouling rate decreases and the permeate rate increases.

[4] Permeate flux rate is affected by the membrane rating, fluid velocity, viscosity, suspended solids concentration, transmembrane pressure, temperature, and membrane fouling. Permeate rates decay as membranes foul from particulate and gels that do not fully pass through the membrane.

Advantages of Crossflow Filtration

Crossflow filtration has a number of distinctive features that are associated with significant benefits. These benefits make crossflow the preferred filtration method for certain applications involving ink jet fluids.

Key Feature	Benefit
Generates two outlet streams (retentate and permeate) from one inlet feed stream	Allows separation of contaminants sized below or above the membrane rating at a relatively sharp particle-size cutoff
Only a small percentage of the feed stream passes through the membrane on each pass	Prevents immediate plugging of the membrane by fluids with a relatively high percentage of suspended solids
Membranes are available in ultrafiltration (UF) molecular weight ratings	Allows removal or separation of low-molecular-weight impurities
Membranes can be regenerated via backwash or reverse flow in process	Increases permeate volume, improving filtration economics
Membrane flux can be restored with chemical cleaning	Extends membrane life significantly, reducing waste and associated disposal costs

Filtration that respects and protects the environment

Pall crossflow membranes help ink jet ink and colorant formulators protect the environment while reducing expenses. These membranes can be cleaned and reused, providing a regenerable filtration solution that can last for years. This translates into reduced waste and associated disposal costs. Crossflow membranes allow formulators to conserve water through the recovery of waste-water produced during formulation processes. Crossflow is among the many filtration technologies that Pall provides to help customers preserve profits while protecting the environment.

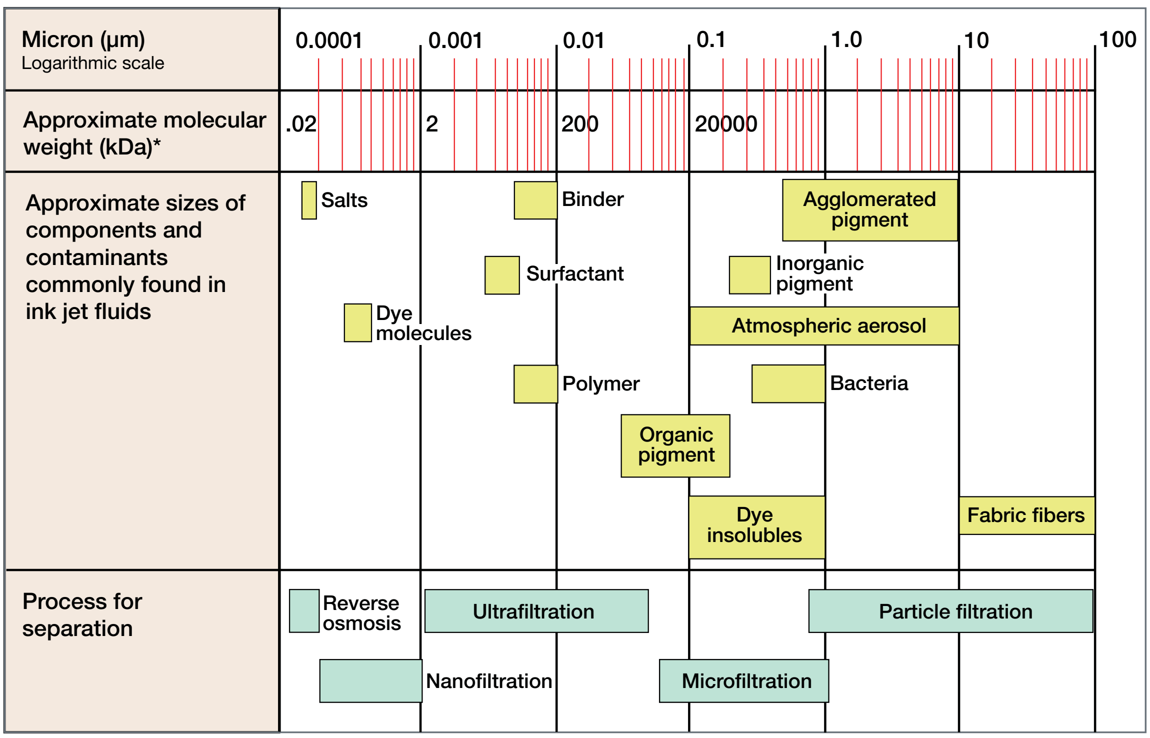
Customers worldwide look to us to help them purify and conserve water, consume less energy, and minimize waste.

ENABLING A GREENER FUTURE

Crossflow Technology for Ink Jet Ink and Colorant Applications

Ink jet ink fluids are comprised of components and contaminants of various sizes. They range from 100 Da to more than 100 µm. (See Figure 3.)

Figure 3: Relative sizes of components and contaminants commonly found in ink jet fluids



* Based on the molecular structure of Dextran

Crossflow technology can be used to separate these components and contaminants in the following applications.

- Removal of impurities from pigmented aqueous colorants
- Classification of pigmented dispersions for inks and colorants
- Dye purification
- Waste stream management

Removal of Impurities from Pigmented Aqueous Colorants

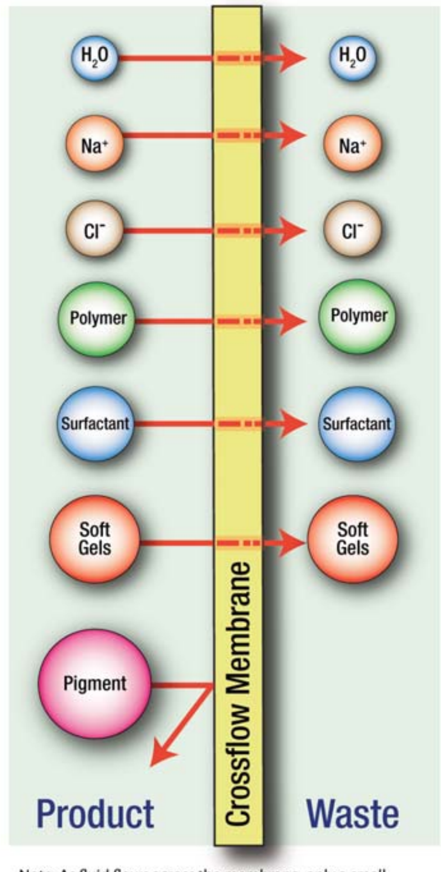
The process of formulating pigmented ink jet colorants includes removing low-molecular-weight impurities such as salts, excess polymer, and surfactant. Crossflow in diafiltration mode can be used to accomplish this. A membrane with the correct rating allows the impurities and carrier fluid to pass through, while retaining the pigment. (See Figure 4.) The ink colorant recirculates through the crossflow module. A portion of the impurities and fluid pass through the membrane, exiting the module as the permeate stream.

In diafiltration mode, deionized (DI) water is added to replace the volume of the exiting permeate stream, and the impurities are effectively washed out of the pigmented colorant. The amount of water added is typically expressed as a multiple of the initial starting batch volume, also known as diafiltration volume. As the number of diafiltration volumes increases, the salt concentration in the retentate decreases. (Refer to Table 1.)

Table 1: Relationship between diafiltration and salt concentration

Diafiltration Volume	Retentate Salt Concentration (%)
0	100.0
1	36.8
2	13.5
3	5.0
4	1.8
5	0.7
6	0.2
7	0.1

Figure 4: Separation of low-molecular-weight impurities from a pigmented dispersion



Note: As fluid flows across the membrane, only a small percentage of upstream components pass through.

Classification of Pigmented Dispersions for Inks and Colorants

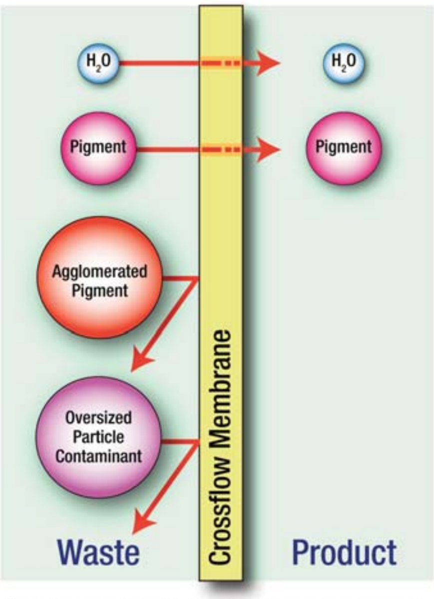
Crossflow microfiltration (MF) membranes can successfully remove larger contaminants and agglomerates from pigmented dispersions. The particles that are specifically targeted are those larger than the desirable particle size distribution of the pigmented dispersion.

The pigmented dispersion passes through the membrane, and any larger contaminants and agglomerates remain in the retentate fluid. (See Figure 5.) Water is continuously added to the system to maintain the concentration of the retentate fluid stream and minimize flux decay. The ink or colorant, which is the desirable product, gets diluted by the water. An additional step is then necessary to return the permeate to the initial concentration.

Dye Purification

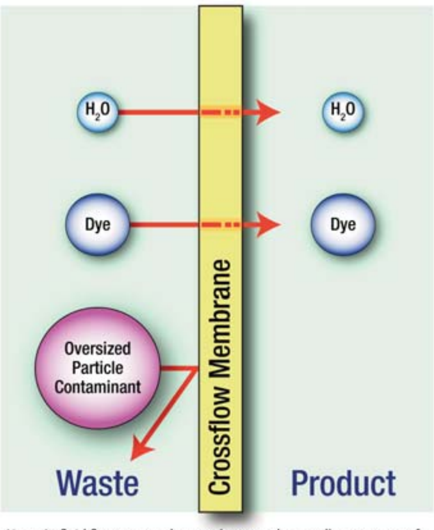
The filtration of dyes is a necessary step in the formulation of dye-based ink jet inks and colorants. Dyes that have not been filtered may contain insoluble hard or soft particles that result from dye synthesis and mixing processes. The dye molecules, which are a low molecular weight and size, easily pass through the membrane into the permeate stream, while the larger insoluble particles become part of the retentate stream. (See Figure 6.) As the dye-based ink or colorant is recirculated across the membrane, the permeate stream is collected in the form of the purified dye product. The retentate stream is concentrated into a sludge-like waste, which is disposed of.

Figure 5: Separation of larger contaminants and agglomerates from a pigmented dispersion



Note: As fluid flows across the membrane, only a small percentage of upstream components pass through.

Figure 6: Separation of oversized contaminants from dye

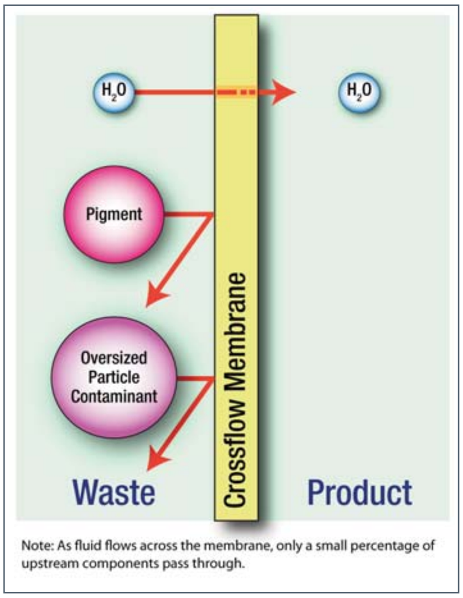


Note: As fluid flows across the membrane, only a small percentage of upstream components pass through.

Waste Stream Management

Wash water used in processing pigmented ink and colorant can be reclaimed by filtering it through a crossflow ultrafiltration membrane. The water passes through the membrane, and the undesirable particulate, including pigment and environmental debris, is retained and concentrated over multiple passes through the crossflow module. (See Figure 7.) The filtered water can be reused as wash water or purified for use in ink or colorant formulation.

Figure 7: Separation of pigment and oversized contaminant from process water



Membralox production system *(left)*; Microza production system *(below)*

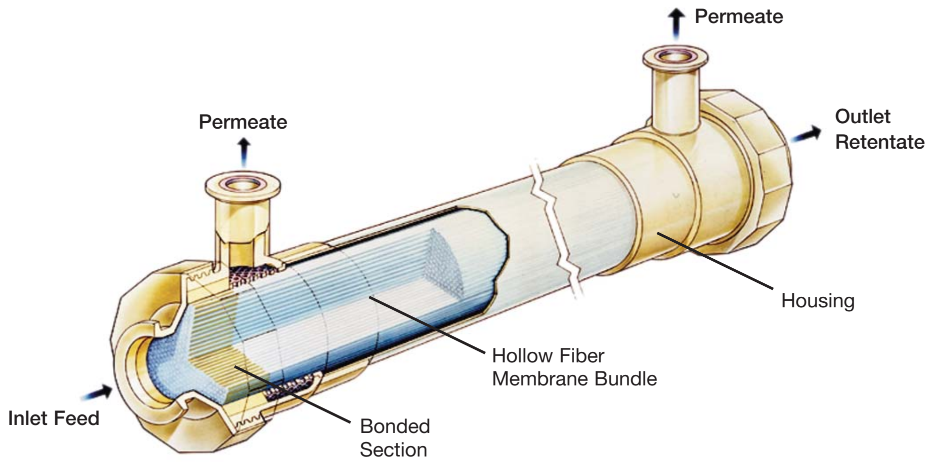
Recommended Products for Crossflow Filtration

Pall offers crossflow products to optimize filtration of ink jet fluids. Microza hollow fiber membranes and Membralox ceramic membranes are both excellent crossflow options. Each has characteristics that are well suited to specific applications and conditions.

Microza Membranes and Modules

Microza crossflow products consist of a bundle of hollow fiber membranes, with inner diameters ranging from 0.7 mm (0.03 in) to 2.6 mm (0.10 in), potted and encapsulated in a modular configuration. (See Figure 8.) The many fibers provide a highly effective membrane in a relatively small area. Microza membranes are available for both ultrafiltration and microfiltration.

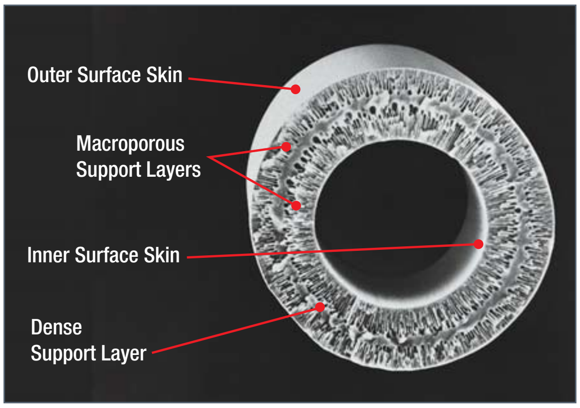
Figure 8: Full-length Microza crossflow module



Ultrafiltration

Microza ultrafiltration hollow fibers consist of a unique, double-skinned proprietary membrane surface over a dense, highly porous internal support layer. This provides for excellent flow characteristics and outstanding fiber durability. (See Figure 9.) The membranes are available in a variety of ratings from 3,000 Da to 500,000 Da. Membrane materials include polysulfone (PS), polyacrylinitrile (PAN) and polyvinyldiflouride (PVDF).

Figure 9: Microza hollow fiber ultrafiltration membrane



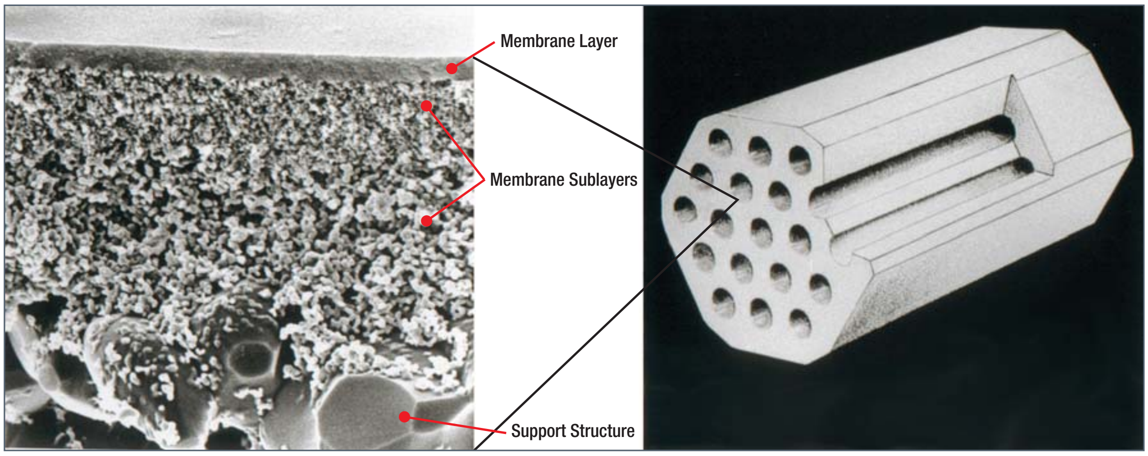
Microfiltration

Microza microfiltration hollow fibers have smooth inner and outer membrane skins and a highly porous symmetrical support structure, allowing for high flow rates. Ratings range from 0.1 µm to 0.65 µm. Membrane materials include polyethylene (PE) and PVDF.

Membralox Ceramic Membranes

Membralox ceramic membranes are asymmetric multichannel elements comprised of a porous alumina support structure and a filtering layer of alumina, zirconia, or titania. (See Figure 10.) They are available in ratings ranging from 0.005 µm (5 nm) to 1.4 µm.

Figure 10: Layered construction of an ultrafiltration Membralox membrane within a channel



The Membralox product can be configured as a single element or group of 3-60 elements installed in a module housing. The ends of the elements are sealed in the module housing with gaskets to prevent the retentate from passing through to the permeate side. Membralox technology is also available in small-scale, single-channel tubes for feasibility testing.

In-process cleaning involves periodically reinjecting a small volume of permeate back through the membrane at high pressure for a very short time. The back pressure (backpulse) shock breaks up the fouling layer on the membrane surface. The remnants of the fouling layer are then washed away by the retentate stream. The frequent destruction of the fouling layer helps to sustain the permeate rate.

Microza and Membralox Membrane Specifications

Characteristic	Membrane	
	Microza	**Membralox**
Materials of construction	PS, PAN, PVDF, PE	Alpha alumina, zirconia, titania
Solvent compatibility	Dependent upon membrane type	Limited by gasket material
pH range	1-14	0-14
Maximum operating temperature	80 °C / 176 °F	95 °C[5] / 203 °F
Operating pressure (TMP)	1-3 bar / 14.5-43.5 psi	1-10 bar / 14.5-145 psi
Fiber ID/channel ID	0.7-2.6 mm / 0.03-0.10 in	3-6 mm / 0.12-0.24 in
Maximum membrane area per module	50 m^2 / 538 ft^2	24.8 m^2 / 267 ft^2
Permeate flux	Medium	High
Method of flux restoration	Backwash	Backpulse

[5] Temperature limitation is due to the housing design/gasket material.

Membralox ceramic membranes *(left, shown with housings)*; Microza hollow fiber membranes *(below)*

Crossflow Bench and Pilot Testing

A crossflow system is complex: It consists of a supporting skid complete with pumps, piping, and valves. These are necessary to drive adequate flow through the membranes and to generate sufficient transmembrane pressure. The skid is typically equipped with a programmable logic controller (PLC) that interfaces with gauges and sensors to monitor membrane performance over time. Automation is necessary to conduct the periodic in-process cleaning of the membranes. Because of the many components involved, crossflow systems require a substantial investment. To reduce the risk of error in membrane selection and system sizing, Pall strongly recommends preliminary bench and pilot testing.

Bench Testing

Bench testing is an efficient means of evaluating multiple membranes in a small-scale lab environment.

The main objectives of crossflow bench testing include:

- establishing the feasibility of crossflow technology for the target application.
- generating small amounts of retentate and/or permeate for further analysis.
- identifying membrane ratings that may be appropriate for the target application.

The Pall VariMem bench system is a complete system designed to test Microza and Membralox modules in a small-scale lab environment. Like other crossflow systems, it requires supporting equipment to drive flow through the modules and generate transmembrane pressure. The unit has a pump with a throttling valve at the retentate outlet, as well as gauges to read inlet, outlet, and permeate pressures. Data can be recorded from a digital display.

Pall VariMem crossflow system

Pilot Testing

After feasibility has been determined, the next step is pilot testing. At this stage, critical data is obtained to correctly size and operate a production system.

The main objectives of pilot testing include:

- establishing optimum process parameters (TMP, flow vs. permeate) for scale-up to production.
- determining membrane area and flow rate required to achieve target permeate rate.
- calculating filtration economics (capital cost vs. amount of product generated).
- establishing in-process and post-batch cleaning protocols.

The Pall SBC crossflow system is a fully functional pilot system that can also serve as a small batch production system. The unit has the pumping capacity needed to run both Microza and Membralox full-length modules in a number of different modes of operation. The use of full-length modules for pilot testing is important because the data generated is the most accurate predictor of production system size and required in-process cleaning protocol. The pilot unit consists of a pump with a throttling valve at the retentate outlet; gauges to read inlet, outlet, and permeate pressures; and a digital data display. In addition, it has automatic in-process cleaning controls to adjust the frequency and duration of membrane cleaning.

Pall SBC crossflow pilot system

Pall Corporation

Microelectronics

25 Harbor Park Drive
Port Washington, New York 11050
+ 1 516 484 3600 telephone
+ 1 800 360 7255 toll free US

*Filtration. Separation. Solution.*SM

Visit us on the Web at www.pall.com/micro

Pall Corporation has offices and plants throughout the world. For Pall representatives in your area, please go to www.pall.com/contact

Because of technological developments related to the products, systems, and/or services described herein, the data and procedures are subject to change without notice. Please consult your Pall representative or visit www.pall.com to verify that this information remains valid. Products in this document may be covered by the following patent number: EP 0 442 410

MECFIJEN Produced in the USA February 2011