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Filtration for Ink Jet Ink Formulation

Techniques for Evaluating Filtration in the Laboratory



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Why Pall

Established more than 60 years ago, Pall Corporation has grown to be the largest and most diverse filtration, separations, and purification company in the world. Our global presence is far-reaching, and our product portfolio and technical expertise are extensive.

Pall offers a variety of filtration products and services to help you improve efficiency and results in the lab. Our staff scientists and engineers provide services and conduct research and development, with intensive, broad-based assistance from Pall's worldwide technical support network. Our experts work directly with you to determine how Pall products and technologies can benefit you.







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Optimizing Filtration in the Ink Jet Lab

Selecting the Correct Filtration for Ink Jet Ink Formulation is Critical.

In the lab, cost-effective formulation of high-quality inks can be achieved when proper filtration is selected. Preliminary ink formulations that are filtered correctly will perform better and are more likely to require only minimal modification. Various types of laboratory tests can be effective in determining optimal filtration for ink formulation.

Pall offers products and services specifically designed for the ink jet industry. Our scientists, technical specialists, and sales professionals are knowledgeable about the inks, chemistries, applications, and cleanliness procedures required for digital printing. We manufacture laboratory test filters for pilot-scale filtration testing and will work with you to select filtration that ensures an efficient ink formulation process and excellent results.













Techniques for Evaluating Filtration for Ink Formulation

There are several types of laboratory tests that can be used to determine optimal filtration for ink formulation and for use on-board printers. The tests described below are the most frequently recommended and the most commonly used. (Note: Specific testing procedures for filterability and ink cleanliness can be found on pages 6-9.)

Test filters are used for filterability and other types of testing. They are fabricated of the same filter media and have the same structure as their full-size counterparts, but are much smaller in scale. Data gathered from test filter studies can be extrapolated to the full-scale process. In addition, the quality of the effluent produced will be representative of the quality expected when using a full-size filter.

Filterability testing

Filterability testing during pilot-scale manufacturing will help determine filter usage and allow optimization of the entire filtration process. It is a tool by which the ink jet ink formulator can maximize filter performance and filtration economics while maintaining consistent ink quality.

Filterability testing can provide answers to these and other questions about ink jet ink filtration.

- Do I need a prefilter?
- How much will it cost (per liter) to filter this ink?
- What is the best flow rate?
- When do I change filters?
- How do I prevent gel breakthrough?

A specifically designed apparatus is needed to conduct the testing. In addition to generating valuable analytical data, the rig can be used to produce ink samples for customer testing. (Note: A list of equipment and instructions for setting up the test rig are provided on page 6.)

Filter and ink compatibility testing

As ink with aggressive chemistries becomes more prevalent, testing for filter and ink compatibility has become critical to the ink development process. Though compatibility is a complex issue, the real-time test experience improves one's understanding of a particular filter's compatibility with a given ink chemistry and how it might impact printer performance.

Printhead performance testing

Tests can be conducted to determine how filtration during ink formulation will impact the long-term performance of an ink jet printhead. Small filter discs are used to facilitate laboratory testing with small sample volumes. These discs are generally available in the same variety of media as full-size filters.

Ink cleanliness quality testing

Real-time ink quality checks are an integral part of the ink jet ink formulation process. A robust quality control process ensures that ink production has conformed to quality requirements and that a quality product will be shipped to the customer.

Filterability Testing Procedures

Filterability testing consists of taking differential pressure measurements while passing fluid through a small test filter at a constant flow rate.

The following information is provided to ensure the accuracy of your test results.

- List of required equipment
- Procedure for performing testing and data collection
- Instructions for setting up the equipment
- Method for analyzing the data

Equipment checklist

The equipment listed below is required to build the filterability test rig.

- Laboratory test filter. Small-scale version of full-size filter with identical design, construction, and media.
- Filter housing. Small stainless steel filter housing for the laboratory test filter.
- **Vent valve.** Ball valve (or similar), installed on the upstream side of the filter housing, that allows trapped air to be vented.
- **Pressure gauges.** Pressure gauges, installed on the filter housing, on the inlet and outlet ports of the filter. If the outlet discharges to atmospheric pressure, the outlet pressure gauge is not required. Recommended pressure range is 0-30 psig/2.07 barg.
- **Pump.** Small laboratory pump capable of flow rates from 0.1-2 lpm. A peristaltic pump is most commonly used.
- **Tubing.** Tubing that is compatible with the ink chemistry and pump type.
- **Reservoirs.** Vessels that feed to the pump and receive filtered fluid from the filter housing.

• Other. Glassware (for volumetric measurement), mixing apparatus (for unstable dispersions), stopwatch (for accurate time measurement), various fittings and hand tools.

Equipment set-up

Set up the test rig as indicated in Figure 1.



Figure 1: Equipment set-up for filterability testing

Testing and data collection

To perform filterability testing and plot the data retrieved, follow the steps below.

- 1. Install a new filter, with gaskets, into the filter housing.
- 2. Open the vent valve to allow air to purge from the system.
- 3. Turn on the pump to start the flow; allow ink to fill the tubing and housing.
- 4. As soon as ink starts flowing from the vent, close the vent valve and start the stopwatch.
- 5. When ink starts flowing from the filter outlet tubing, record the clean differential pressure value.
- 6. Using volumetric glassware, measure the flow rate and record the value.
- 7. Continue to record differential pressure values at set time intervals.
- 8. Once the filter has achieved maximum differential pressure (usually 25 psid/1.70 bard), turn off the pump and allow the system pressure to subside.
- 9. Remove and discard the filter; clean the housing and equipment.
- 10. Plot the data using the sample graph in Figure 2 as a guide.



Figure 2: Type of graph used to plot data

Analysis

To analyze the data, refer to the following instructions.

- After the data is plotted, determine the point at which filter life is exhausted (typically 25 psid/1.70 bard). This is the expected filter life for the specific test flux rate, filter type, and ink formulation.
- 2. Calculate the total throughput for the full-size filter using the following formula and the scale-up factors listed below.

(Test Flow Rate) x (Scale-Up Factor) x (Expected Filter Life [in minutes]) = Total Throughput

Filter	Scale-up factor
Nexis® A filters	7.1
Poly-Fine® XLD filters	6.2
Profile [®] Star filters	8.4
Profile® II filters	9.8
Ultipor [®] GF filters	8.4
Ultipor® N66	7.8
Water-Fine filters	6.2

- 3. If the expected filter life (in minutes) is too short, repeat the test with a prefilter. Save the effluent from the prefilter test to use when repeating the filterability test on the final filter.
- 4. To optimize to a given ink chemistry, repeat testing with different flow rates.
- 5. Once optimized, keep the same flow rate for each formulation to assure continuity of data from batch to batch.

Ink Cleanliness Quality Testing Procedures

Ink cleanliness quality testing involves determining the cleanliness factor of an ink sample. The cleanliness factor is defined as the ratio of membrane plugging between two distinct intervals. Since cleanliness factors vary depending on ink type, you will need to correlate the resulting cleanliness factor back to printer performance.

The following information is provided to guide you through the testing process.

- Instructions for setting up the equipment
- Procedure for performing testing and data collection
- Process for troubleshooting

Equipment checklist

The equipment listed below is required to conduct ink cleanliness quality testing.

- Vacuum flask. Flask must have a 1 liter minimum capacity.
- Vacuum source with valve. Source must be able to maintain constant vacuum at 200 torr or greater.
- Filter disc holder and funnel. To start with, a holder that can accommodate a 47 mm-diameter disc and a glass funnel with a capacity of at least 100 ml.
- Analysis filter discs. For dye-based inks, Pall Ultipor N66 disc filters rated at 0.45 μm; for pigmented inks, Pall Ultipor GF Plus disc filters rated at 1 μm.
- Other. Tubing (for vacuum connection), glassware (for volumetric measurement), stopwatch (for accurate time measurement), clean sample containers.

Equipment set-up

Set up the test equipment as indicated in Figure 3.



Figure 3: Equipment set-up for ink cleanliness quality testing

Testing and data collection

To perform ink cleanliness testing and chart the data retrieved, follow the steps below.

- 1. Install analysis filter disc in the filter holder.
- 2. From the same ink batch, measure out three samples of equal size. (A 100 ml sample is recommended to start.)
- 3. Start the vacuum source; pour the sample into the funnel.
- 4. Open the vacuum source valve while simultaneously starting the stopwatch.
- As soon as the sample has passed through the analysis membrane, stop the stopwatch, close the vacuum source valve, and record the time (t_j).

- 6. Repeat the procedure for the second sample, but do not record the time.
- Repeat the procedure for the third sample, and record the time (t_f).
- 8. Determine the cleanliness factor **(CF)** using the following formula.

CF = t_i /t_f (cleanliness factor = time initial/time final)

Sample Test Data Chart

Filter	t _i	t _f	CF
Cyan	8	10	.80
Magenta	10	12	.83
Yellow	9	11	.82
Black	11	14	.79

Troubleshooting

This section provides descriptions of the problems most commonly encountered during ink cleanliness quality testing, their probable causes, and corrective actions. The likelihood of experiencing any of these problems depends on the formulation and cleanliness of the ink.

Problem	Problem Cause	Solution	
For each sample, the CF values for the filtered and unfiltered inks are very similar.	The sample size is not large large enough.	Increase the sample size, and repeat the test.	
The analysis membrane plugs quickly, even when a small sample is used.	The solids loading or viscosity of the ink is too high.	Dilute the sample in a 1:1 ratio with very clean carrier fluid and repeat the test.	
	The ink cleanliness needs to be improved.	Refilter at a higher efficiency, and repeat the test.	
The accuracy of the data is being compromised because test times are less than five seconds.	The sample size is not large enough.	Increase the sample size, and repeat the test.	

Laboratory Test Filter Selection

The table below provides a sampling of Pall's test filters. For information about our complete line of test filters, please contact your local Pall office or distributor.

Filter Family	Removal Rating	Test Filter Part No.	
Nexis A filters	0.5 µm	NXA 0.5-TFU-DOEE 047	
	1 µm	NXA 1-TFU-DOEE 047	
	3 µm	NXA 3-TFU-DOEE 047	
	5 µm	NXA 5-TFU-DOEE 047	
	10 µm	NXA 10-TFU-DOEE 047	
Poly-Fine XLD filters	1.5 µm	XLD 1.5-TFUE 147	
	3 µm	XLD 3-TFUE 147	
	4.5 µm	XLD 4.5-TFUE 147	
	10 µm	XLD 10-TFUE 147	
Profile Star filters	0.8 µm	H1A1A0081J	
	1.5 µm	H1A1A0151J	
	3 µm	H1A1A0301J	
	5 µm	H1A1A0501J	
Profile II filters	0.2 µm	5EC4888-389-002J	
	0.3 µm	5EC4888-389-003J	
	0.5 µm	5EC4888-389-005J	
	1 µm	5EC4888-389-010J	
	3 µm	5EC4888-389-030J	
Profile Nano filters	0.1 µm	NP100-TFU-DOEE	
Ultipor GF filters	0.1 µm	PUY01ZU001ZJ	
	0.2 µm	PUY01ZU002ZJ	
	0.45 µm	PUY01ZU0045J	
	0.7 μm	PUY01ZU007ZJ	
	1 µm	PUY01ZU010ZJ	
	2 µm	PUY01ZU220ZJ	
	3 µm	PUY01ZU030ZJ	
Ultipor N66 filters	0.2 µm	PUY01NAEYJ	
Water-Fine filters	0.1 µm	WFN 0.1-TFUE 147	
	0.2 µm	WFN 0.2-TFUE 147	
	0.45 µm	WFN 0.45-TFUE 147	









Note: We offer the Pall Low Volume Test Filter Housing (M01SS-01GN4JV-BE) for use with our test filters.

Pall Filtration Products for Digital Printers

Pall has a wide range of filters that are designed with added features to accommodate ink jet printers. When used on-board digital printing systems, these filters provide maximum printhead protection and excellent printer performance.

Please visit www.pall.com for more details.



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