WaterSep

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Hollow Fiber Caustic Stability Study

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February 10, 2009

Purpose

Evaluate the stability of the membrane properties of the WaterSep PES hollow fiber for permeability and retention when exposed to repeated sodium hydroxide cycles.

Background

Sodium hydroxide is the most commonly used agent for cleaning, sanitizing and depyrogenating membrane elements. For tangential flow filtration membrane processes used either in re-use or single-use applications, stability of membrane properties before and after caustic cycles is vital for membrane performance to maintain consistency from batch to batch.

To evaluate the stability of membrane properties of WaterSep's hollow fiber elements, a study was conducted exposing a WaterSep 50kD m-PES membrane hollow fiber to ten (10) repeated non-fouling one hour caustic cycles of 0.5N sodium hydroxide at 35 – 40^{o}C. Water permeability (NWP) was measured before the first caustic cycle and after each subsequent cycle. After completing ten (10) caustic cycles, the element was challenged with a single-solute intermediate marker to measure the retention property. Unlike most studies that use a solute marker with either "complete" transmission or retentive properties that expose only major changes, this study uses an "intermediate" marker positioned on the steep selectivity profile thus providing a high resolution transparency of any change. "Test" element results were compared to the "control" element results with the same membrane lot but not exposed to any caustic cycles.

Summary of Results

After 10 repeated 1 hour caustic cycles of 0.5N NaOH at 35- 40^{o}C, the permeability and retention membrane properties remained stable.

The membrane permeability (NWP) measured showed a maximum decrease of 19% between the initial and final test cycle. This change is well within the acceptable standard deviation of the membrane specification.

The retention properties as measured by this intermediate marker increased 18% indicating a minor shift towards a tighter NMWL of the membrane. This observed retention change for a marker on the steep selectivity slope denotes less than a 2% shift of the membrane R90 value. This is well within the membrane specification range.

Conclusion:

WaterSep m-PES hollow fiber membranes showed stable permeability and retention properties after repeated exposures to hot caustic cycles.

Test Procedure

Two hollow fiber test elements were made with the same membrane lot.   Test included:

Control Element

1. Measure the water flux rate at 10 psig after 1 minute (before IPA)
2. Flush with 35% IPA for 3 minutes at 10 psig
3. Measure the water flux rate at 10 psig after 1 minute (after IPA)
4. Continue to flush permeate until refractometer reading baseline to 0.000
5. Replace feed with PVP K30 and measure % passage of PVP at 1, 3, 5 minutes at 10 psig and 4200 sec-1 shear.

Test Element

1. Measure the water flux rate at 10 psig after 1 minute (NWP_o before IPA)
2. Flush with 35% IPA for 3 minutes at 10 psig
3. Measure the water flux rate at 10 psig after 1 minute (NWP_o after IPA)
4. Replace feed with caustic solution and recirculate for 1 hour at 9000 sec-1, retentate open.
5. Replace feed with water and measure the water flux rate at 10 psig after 1 minute (NWP_i before IPA).
6. Flush with 35% IPA for 3 minutes at 10 psig
7. Measure the water flux rate at 10 psig after 1 minute (NWP_i after IPA)
8. Repeat steps 4 to 7 until 10 cycles are completed
9. Continue to flush permeate until refractometer reading baseline to 0.000
10. Replace feed with PVP K30 and measure % passage of PVP at 1, 3, 5 minutes at 10 psig and 4200 sec-1 shear.
11. Calculate % passage (Cp/Cf) using refractometer.

Materials and Equipment

"Control" Element and "Test" Element:

<u>Type</u>: m-PES hollow fiber
<u>ID/OD:</u>   1.0 mm / 1.4 mm OD
<u>Area:</u> 59 cm^2
<u>Cutoff:</u> 50 K Dalton MWCO
<u>Fiber Number:</u> 8
<u>Fiber Length:</u> 7.0 inches.

System:

A bench top tangential filtration system was used.   Key components included:

- Pump – Micropump – (Cole-Parmer R-07001-40)
- Retentate Flow Rate – Rotometer with needle valve –(Cole-Parmer R-32005-38)
- Refractometer – Atago (RX-007alpha)

Figure 1: Caustic & IPA Cycle Process Diagram

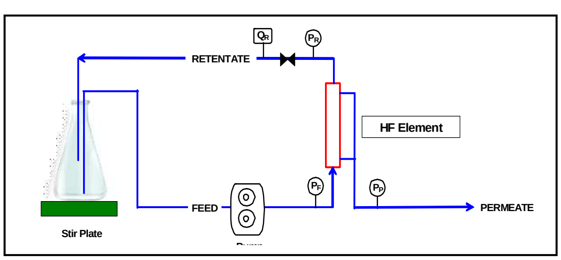
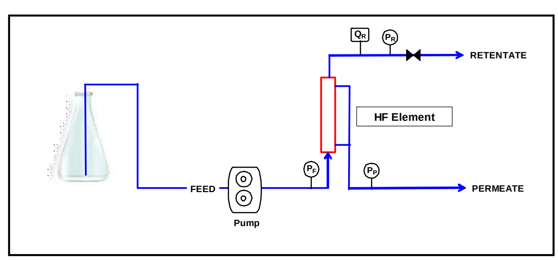


Figure 2: Water NWP Process Diagram



Test Results and Summary:

NWP Summary

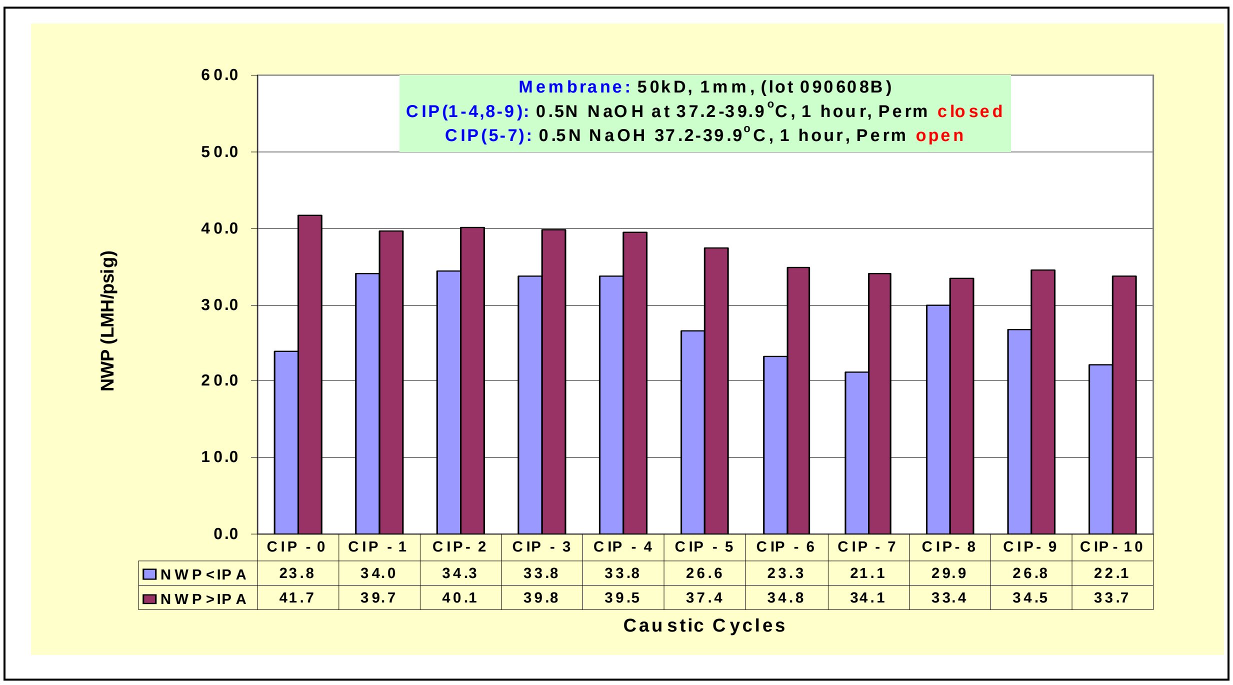
Asymmetric polymeric microporous structures offer excellent robust and mechanical structural strength due to the dense and uniform morphology. As a result, complete water wetting of pores structures is often compromised due to the high surface tension differential between water and the membrane surface. Hence, NWP measurements were performed both before and after flushing with 35% isopropyl alcohol (IPA). Results are presented for each.

Table 1: Summary of the normalized water permeability (NWP) measured on the "control" and "test" elements before and after the 10 caustic cycles.

Test Step		Element: Control	Element: Test	% Change
NWPo	Before IPA	23.4	23.8	100%
	After IPA	42.0	41.7	100%
NWP_{10}	Before IPA		22.1	94%
	After IPA		33.7	81%
PVP K30 Passage	Before Caustic	77.1		100%
	After 10 cycles		63.1	82%

With the same membrane lot used in the "control" and "test" element, the normalized water permeability results presented in Table 1 were identical, as expected, when comparing the corresponding before IPA results and corresponding after IPA results.

Figure 1: Water filtrate flux rates were measured before and after IPA flushing at each caustic cycle for a total of 10 caustic cycles.



In Figure 1, the NWP profile is presented for the "test" element measured before and after each caustic cycle. NWP results measured after IPA exposure showed a stable profile that leveled off to a maximum NWP reduction of 19%. This change is still well within the permeability specification. NWP results measured before IPA showed a smaller decrease (6%) from initial to final cycle but with a higher expected standard deviation. Thus, more weight is always given to the NWP after IPA.

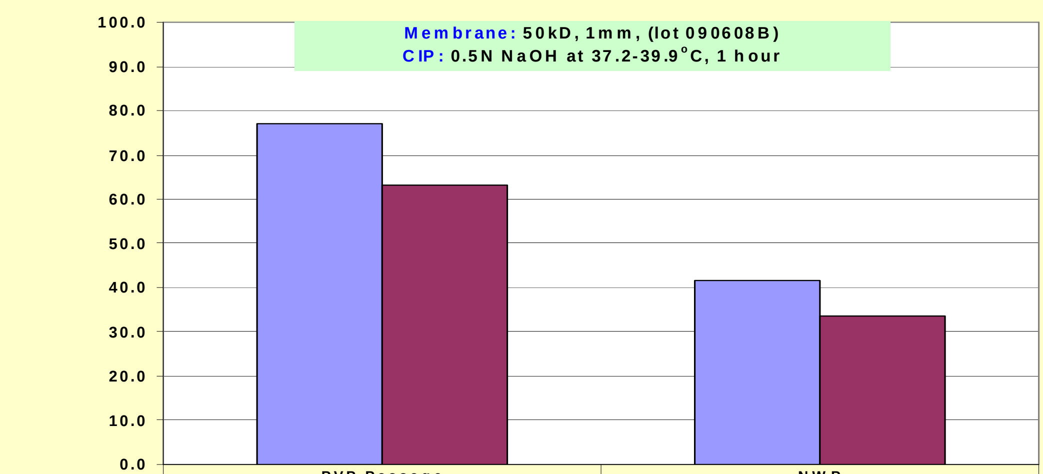
Retention Summary

Table 1 and Figure 2 presents the single solute test result performed on the "control" element and "test" element. The "control" element had a single-solute retention result for PVP K30 of 77.1% passage represented the retention property for this membrane lot before caustic exposure. The solute retention measured on the "test" element after exposure to ten repeated hot caustic cycles was 63.1% passage. Comparing the retention results presented between the two elements, an 18% reduction was observed to the membrane lot due to exposure to hot caustic cycles.

Since the single-solute marker used to measure retentivity of the membrane is on the steep selectivity slope, this observed change denotes less than a 2% shift of the membrane R90 value, thus well within the membrane specification range.

In conclusion, for UF applications where the product is concentrated and lies on the upper end of the retention selectivity profile, this 2% observed R90 change will have no effect on product recovery. For those applications that demand a fractionation with biological species located on the steep selectivity slope from a retentive product, the maximum change of 18% would be observed.

Figure 2: Comparing the water permeability (NWP) and retention of the membrane before and after 10 caustic cycles.



	PVP Passage	NWP
Before CIP	77.1	41.7
After 10 CIP	63.1	33.7