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# Specialized Short Course on **MEMBRANE TECHNOLOGY** for Water and Wastewater Treatment

27 – 28 June 2009 (4 -5 Rajab 1430 H)

Prince Khalid bin Sultan Chair for Water Research  
Civil Engineering Department, Hall 1 A 36  
College of Engineering, King Saud University





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# Lecture 4

## Membrane Technology System Design and Fouling Control

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Ph.D, P.Eng. (M), C.Eng. (UK), C.Sci. (UK), C.W.E.M. (UK), MIEM, DNS, PPI

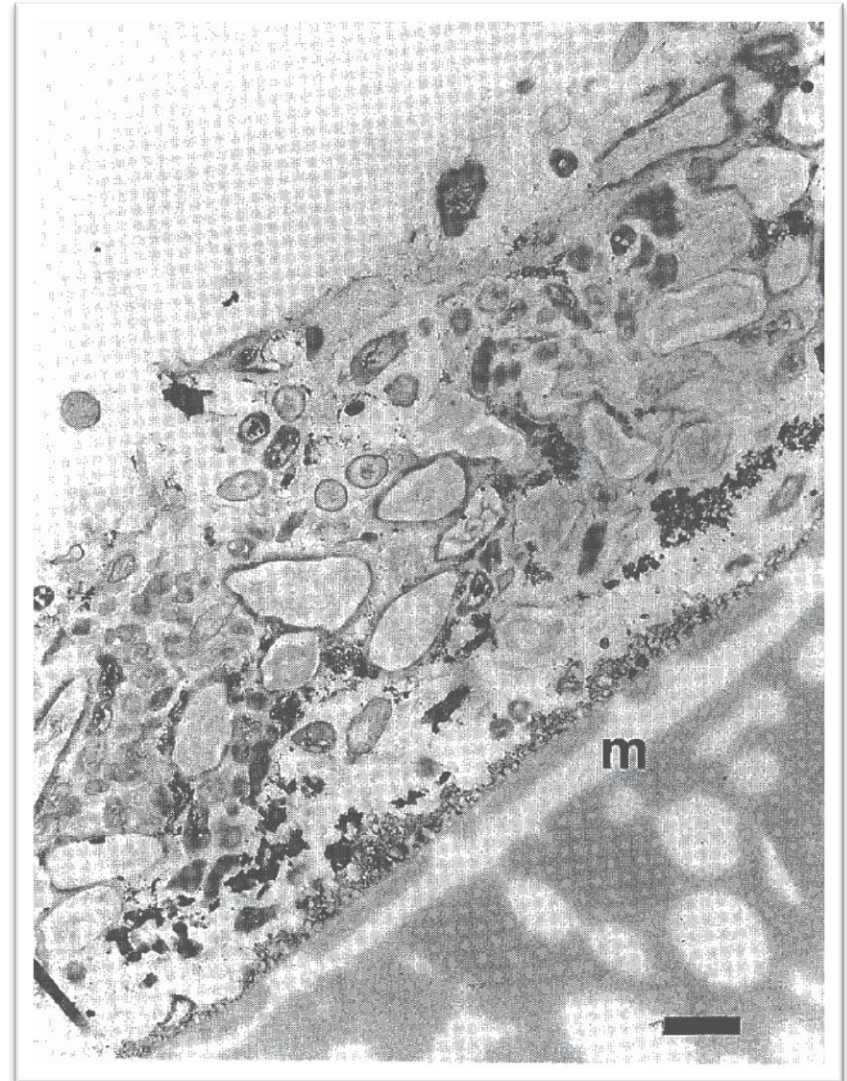
Institute of Environmental and Water Resources Management (IPASA)  
Universiti Teknologi Malaysia



## Fouling is Common

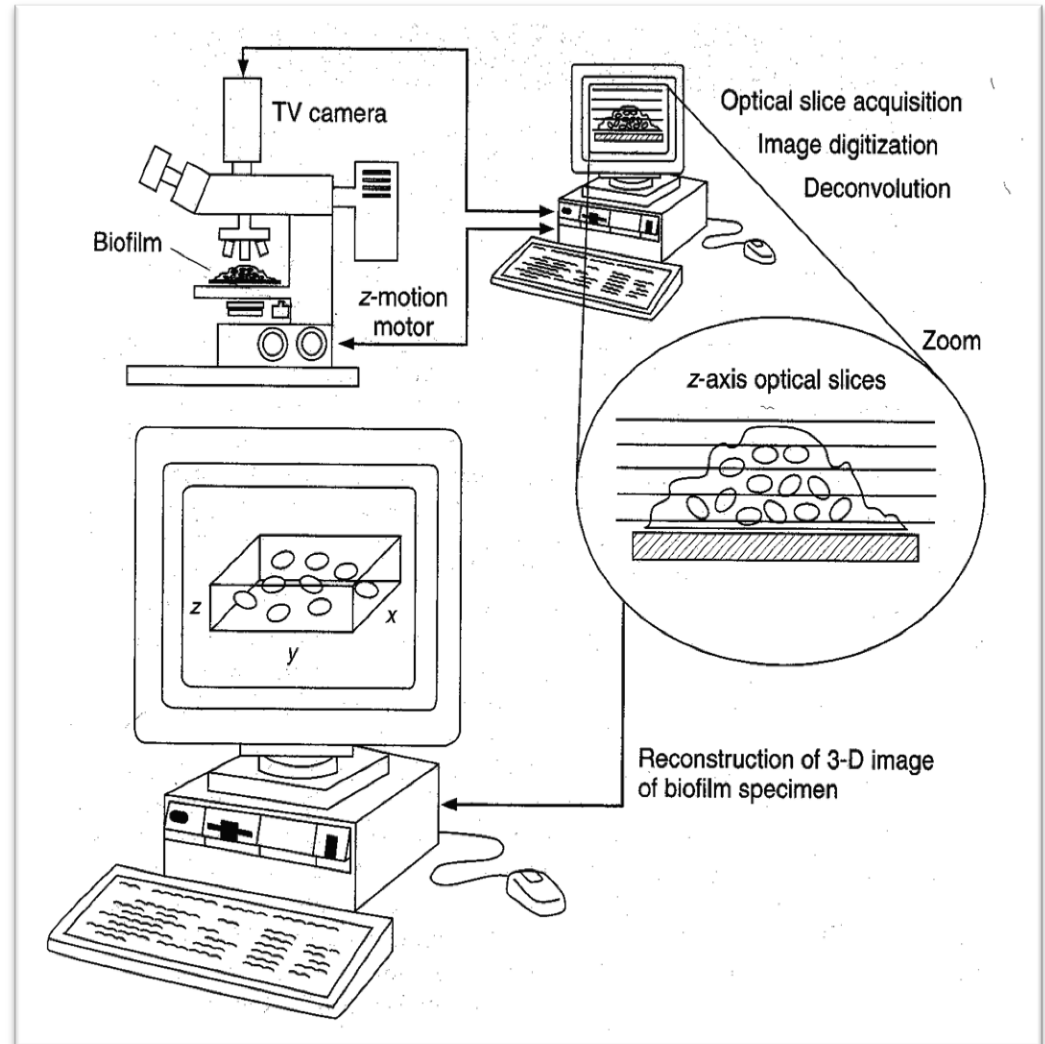
**TEM photomicrograph of cross section through a biofilm formed on a polyetherurea UF membrane (m) fed with domestic tap water showing cells and unidentified electron-dense material embedded in the EPS matrix.**

**Bar = 1µm**



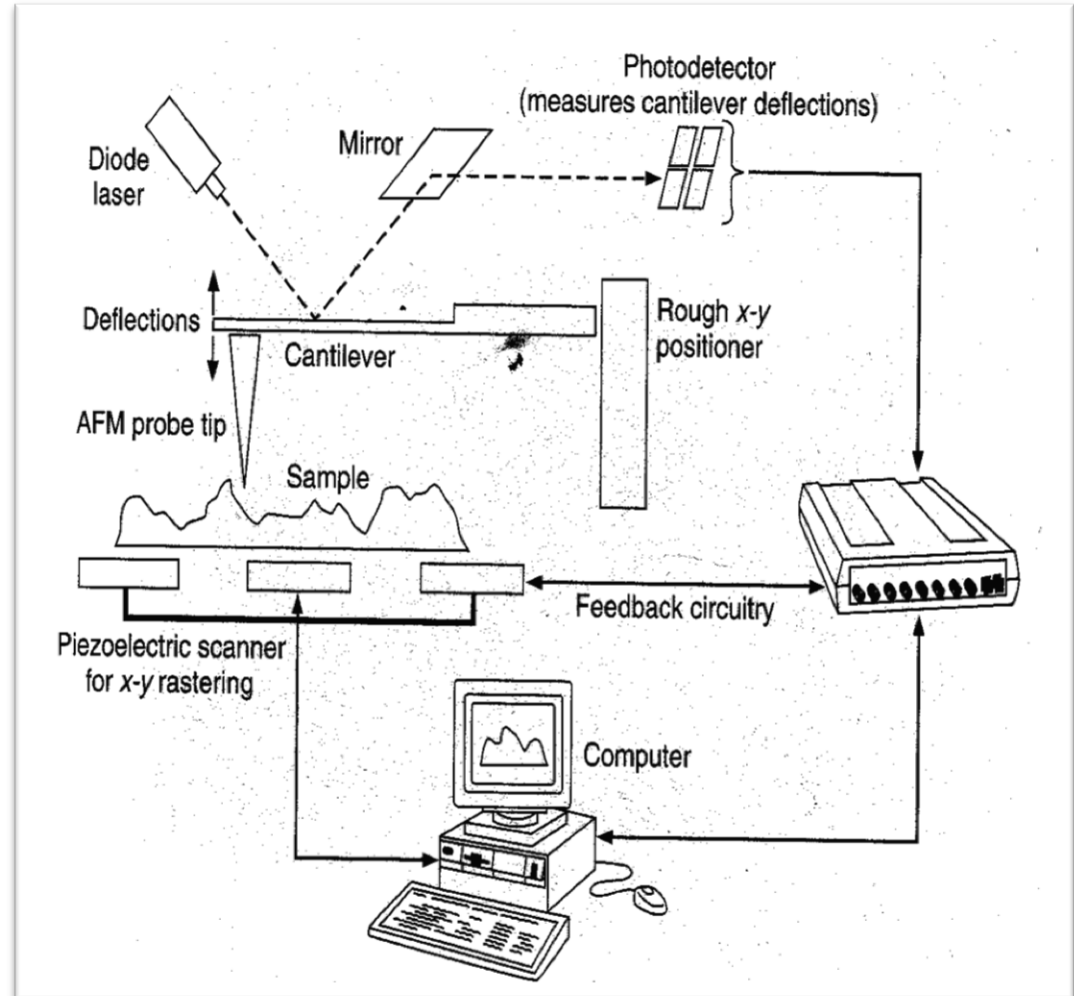
# How to Study Fouling

**Schematic illustration of a DMC system. Optical slices are captured digitally along the specimen z axis. Hazy and defocused information in each section is subsequently removed digitally via deconvolution algorithms. The sections are then digitally recombined to produce a 3-D virtual image of the specimen**



# How to Study Fouling

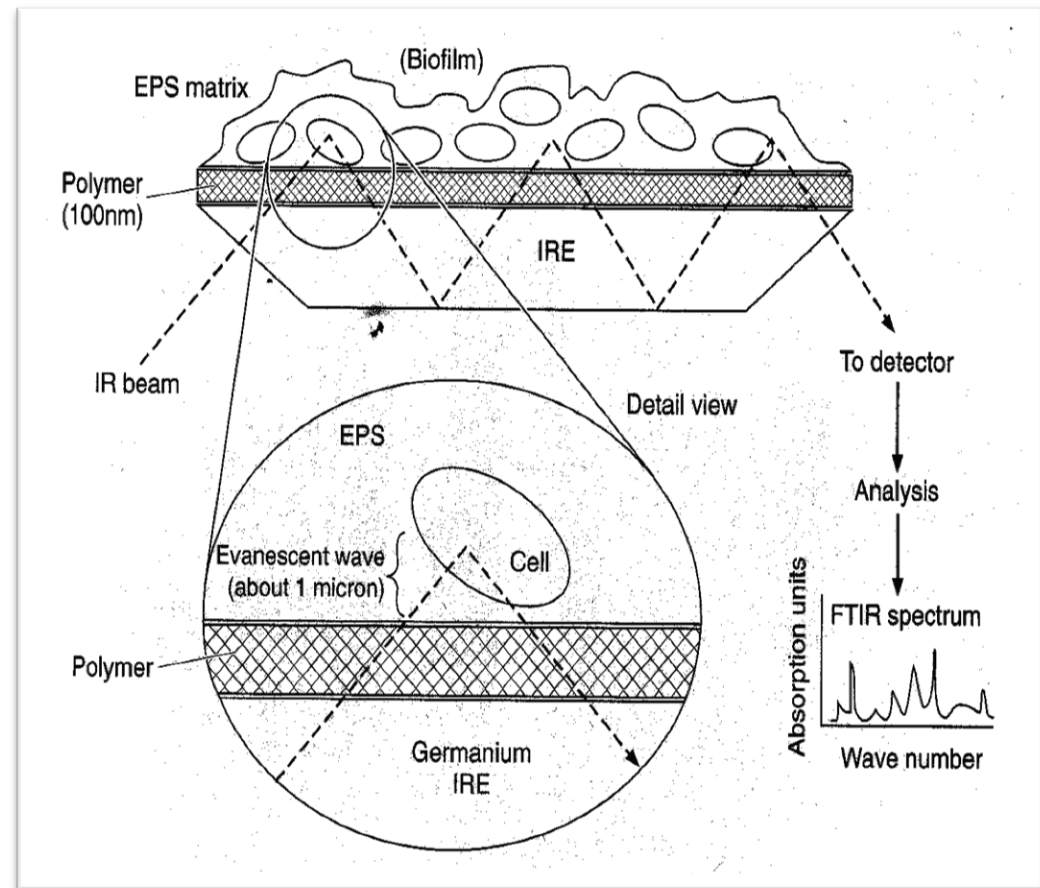
Schematic illustration of AFM system. A silicon nitride probe tip mounted on a cantilever is rastered in the x-y plane over the specimen surface. Z- axis cantilever deflections and sideways torsion ( measured by laser ) signal tip-specimen interactions and surface topographic features. Digital data are recombined to generate a 3-D virtual image of the specimen topographic



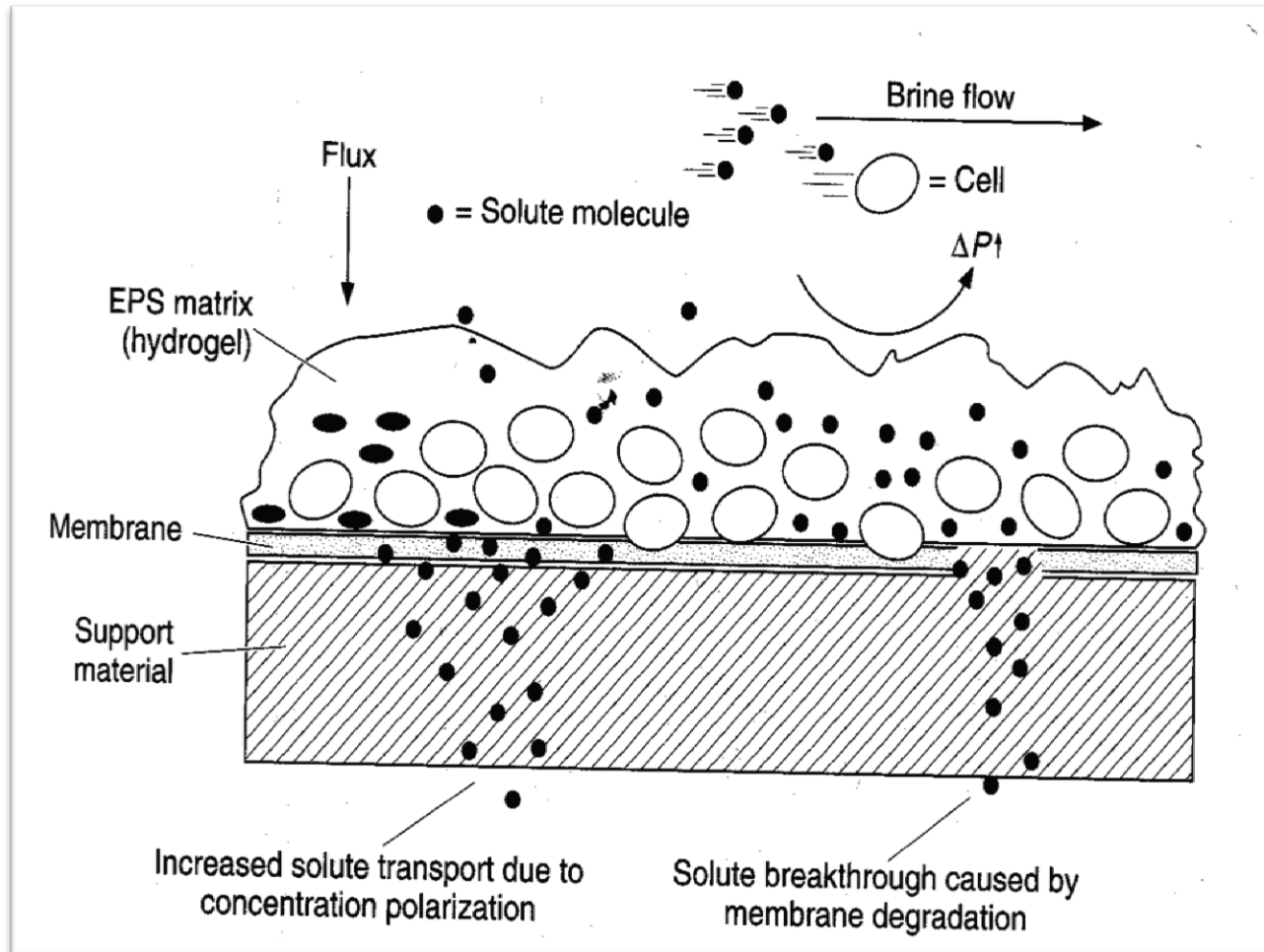


# How to Study Fouling

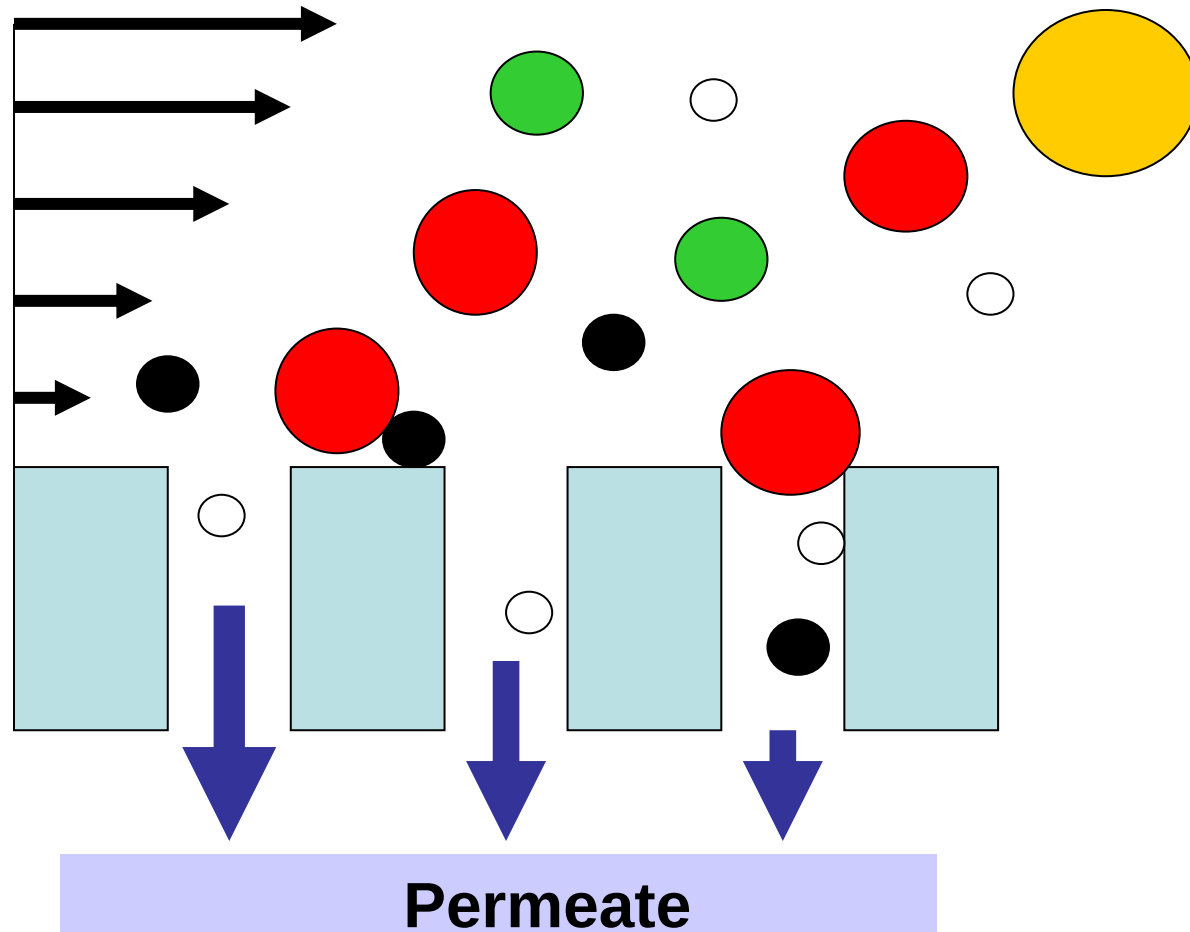
Schematic illustration of ATR-FTIR spectrometry technique for monitoring biofilm formation. The molecular composition of a biofilm growing on the surface of a polymer-coated germanium IRE is quantified by determining the attenuation of an IR signal (evanescent wave) which penetrates a short distance (about 100 nm) to allow biofilm detection



## Major Effects of Fouling



# Fouling on Membrane Surface





| Observed biofouling effect             | Description/comment                                                                                                                                                                                                                                                                                                                               |
|----------------------------------------|---------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| Membrane flux decline                  | Gradual loss of water flux due to biofilm accumulation; results in corresponding increase in system energy costs; flux may be partially restored by periodic chemical cleaning.                                                                                                                                                                   |
| Reduced solute rejection               | Results from enhanced opportunity for concentration polarization within the membrane biofilm, which has a hydrogel consistency; may also result from membrane biodegradation.                                                                                                                                                                     |
| Enhanced mineral scaling               | Biofilm formation enhances opportunity for mineral scaling due to increased concentration polarization or by providing nucleation sites for precipitate growth.                                                                                                                                                                                   |
| Increased module differential pressure | Results from increased fluid frictional drag (energy losses) associated with surface biofilm formation; also caused by physical blockage of feed channel spaces due to biological growth; effect may be severe; effect may or may not be reversible by cleaning or biocide treatment.                                                             |
| Permeate contamination                 | Caused by cellular detachment and/or biomass sloughing from colonized permeate surfaces of membrane, including polyester support fibers, glue lines, permeate collection materials, etc.                                                                                                                                                          |
| Membrane biodegradation                | Caused by direct enzymatic hydrolysis of membrane polymers or by pH extremes associated with microcolonies on the membrane surface; may occur rapidly under physiologically favorable circumstances (e.g., warm temperatures, suitable nutrients); effect is essentially irreversible resulting in permanent loss of solute rejection properties. |
| Module component biodeterioration      | Usually results from glue line biodegradation.                                                                                                                                                                                                                                                                                                    |
| Reduced membrane life                  | Results from a combination of all of the above factors; inappropriate or excessive biocide applications and excessive cleaning frequencies contribute to shortened membrane life.                                                                                                                                                                 |

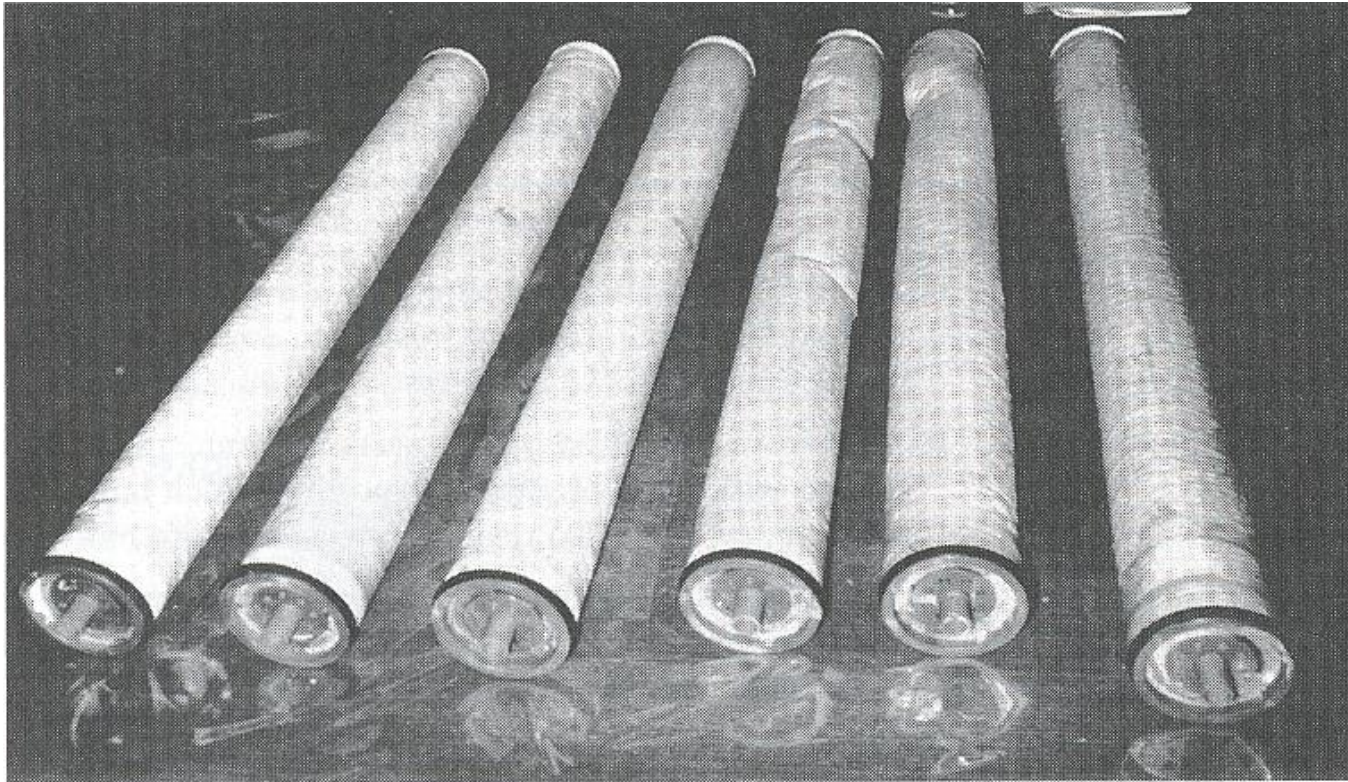
## Principal Adverse Effects of RO Membrane Biofouling



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# Collapse of RO Elements

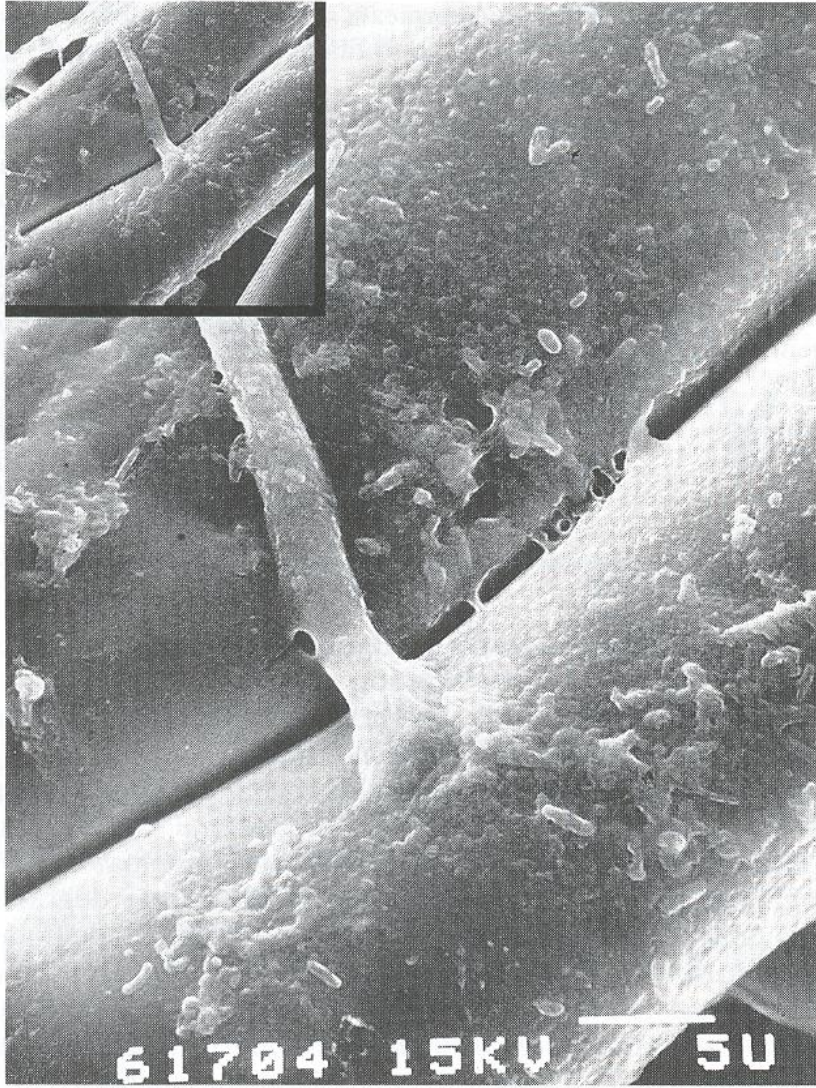


Longitudinal collapse of RO elements operated in excess of manufacturer's specified module differential pressure. Differential pressure increase resulted primarily from the combined effects of colloidal and microbial fouling

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## Biofilm Development

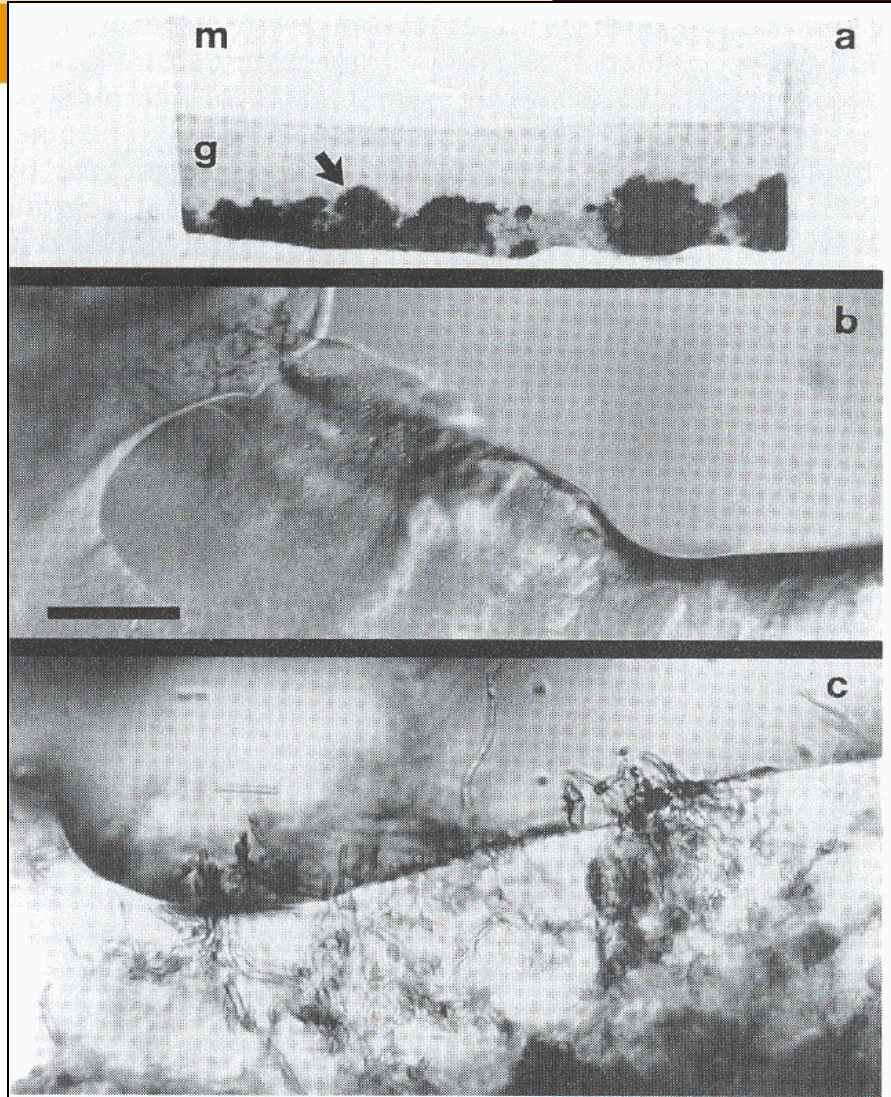


SEM photomicrograph of biofilm development on non-woven polyester support fibers (inset) on permeate surface of RO membrane.

Note partial occlusion of rod-shaped bacteria by EPS. Bar = 5um.



# Biodeterioration



Biodeterioration of polyurethane glue line of an RO membrane element by filamentous fungi: (*panel a*) Macroscopic view of glue line (approximately actual size); dark irregular patches indicate areas of penetration of fungal filaments (hyphae) into glue line (arrow); m = cellulose acetate membrane; g = polyurethane glue line. (*Panel b*) Microscopic view of uninfected (control) region of glue line. (*Panel c*) microscopic view of infected region of glue line showing invasion by fungal hyphae and loss of glue integrity.

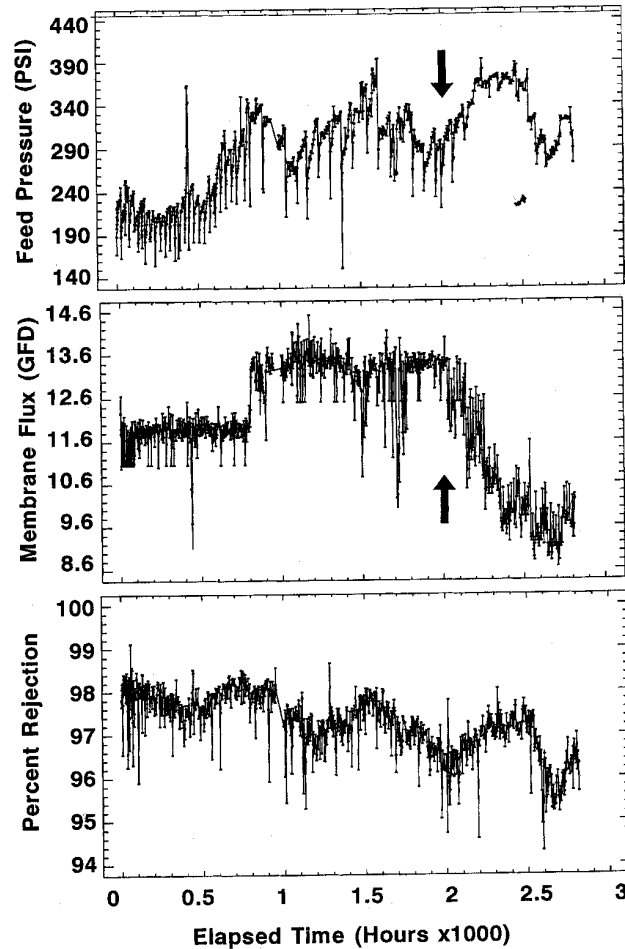
Bar = 100  $\mu$ m.



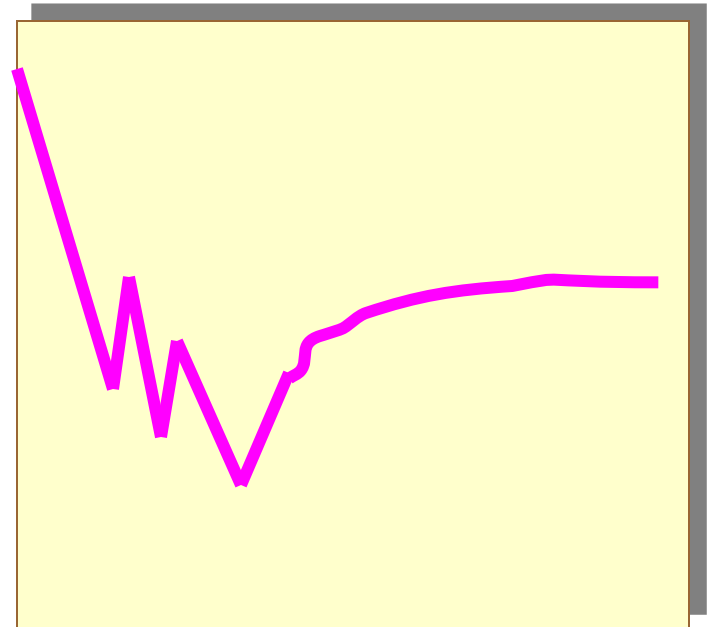
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# Typical Permeate Flow Decline in Membrane Operation



$\text{m}^3/\text{h}$



Days





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# Conventional Solution to Fouling

- **Back washing**
- **Mechanical cleaning**
- **Chemical cleaning**
- **Ultrasonic cleaning**
- **Change membrane module**
- **Engage membrane manufacturer**



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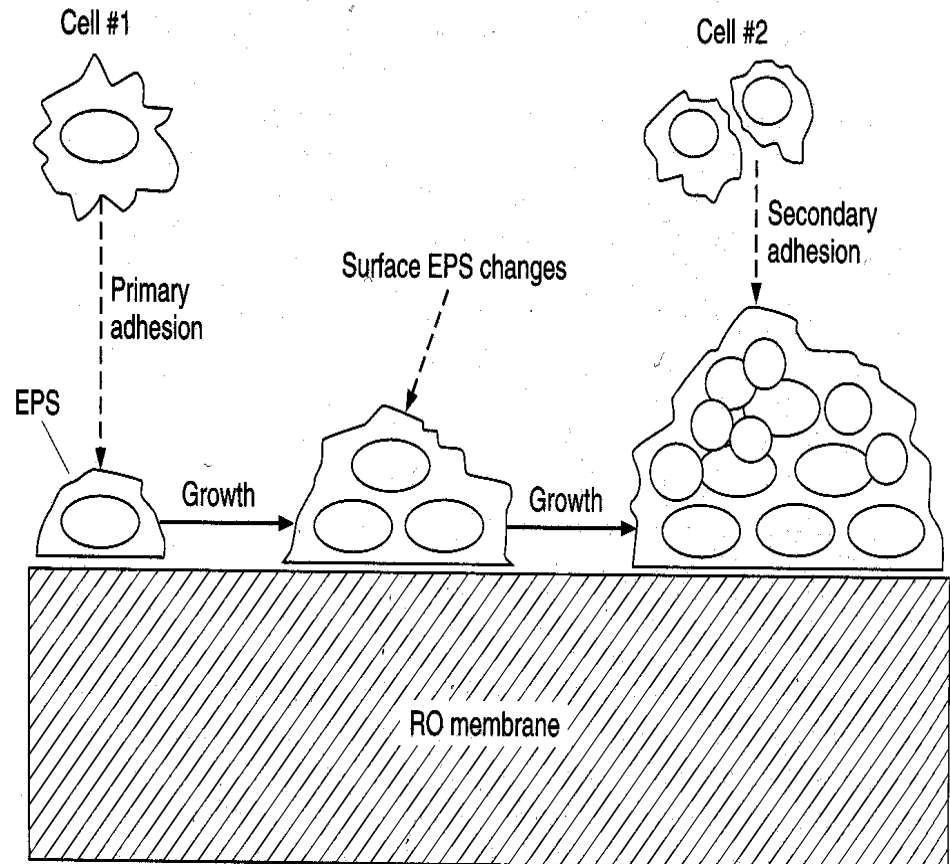
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# New Solutions to BioFouling

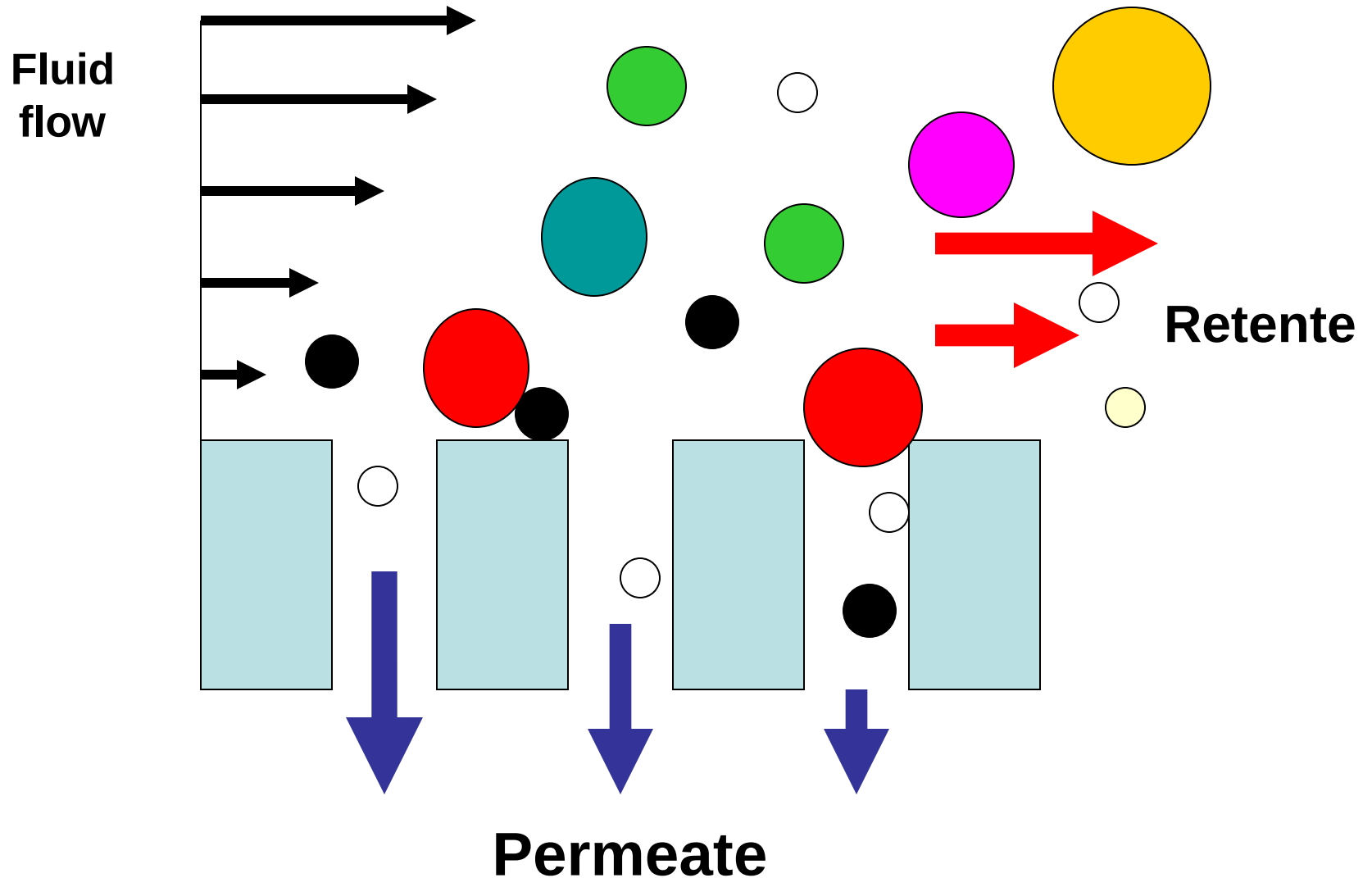
- **Back washing**
- **Chemical cleaning**
- **Change membrane module**
- **Biotech solution**



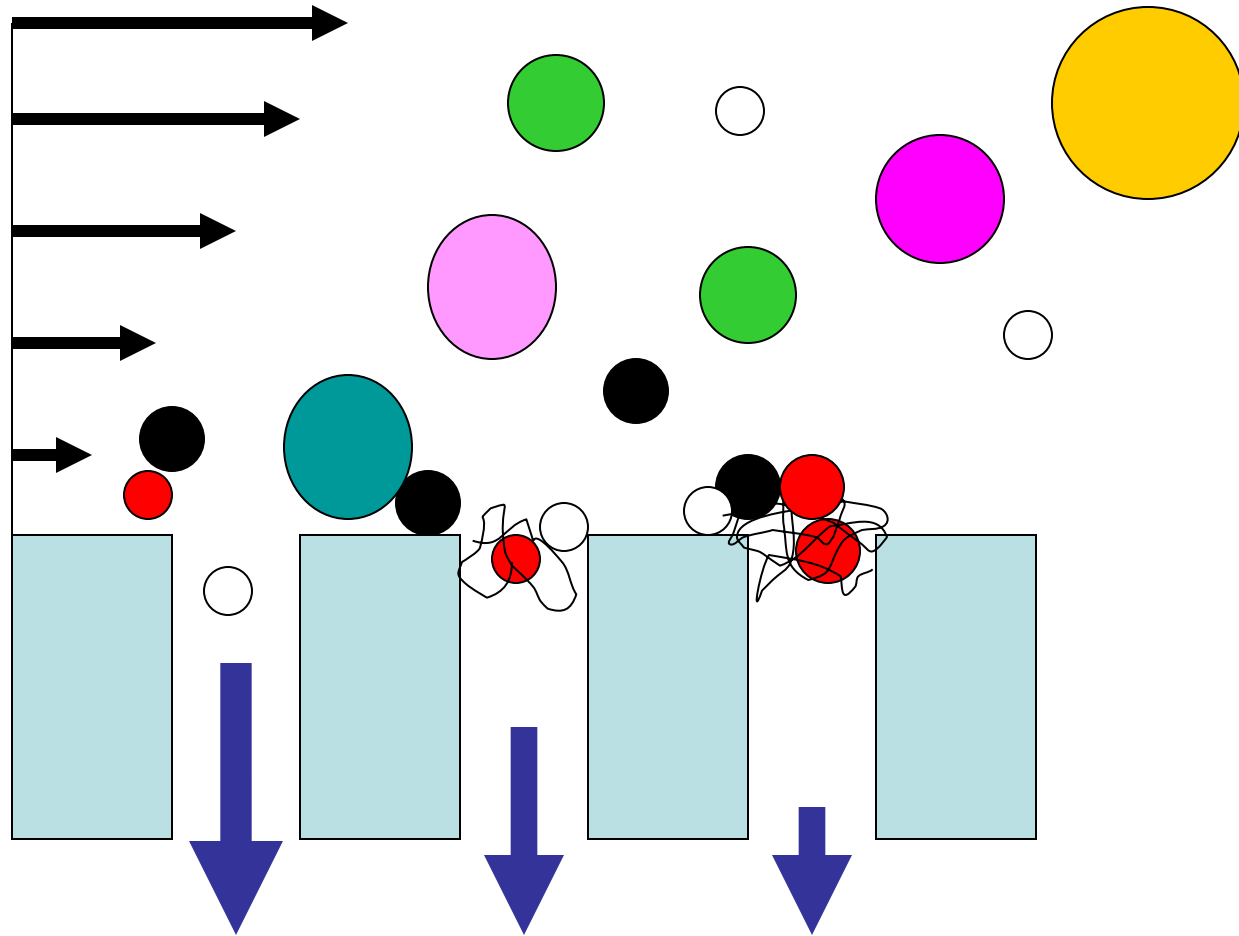
## Schematic illustration of major events in membrane biofouling process



# Fouling on Membrane Surface



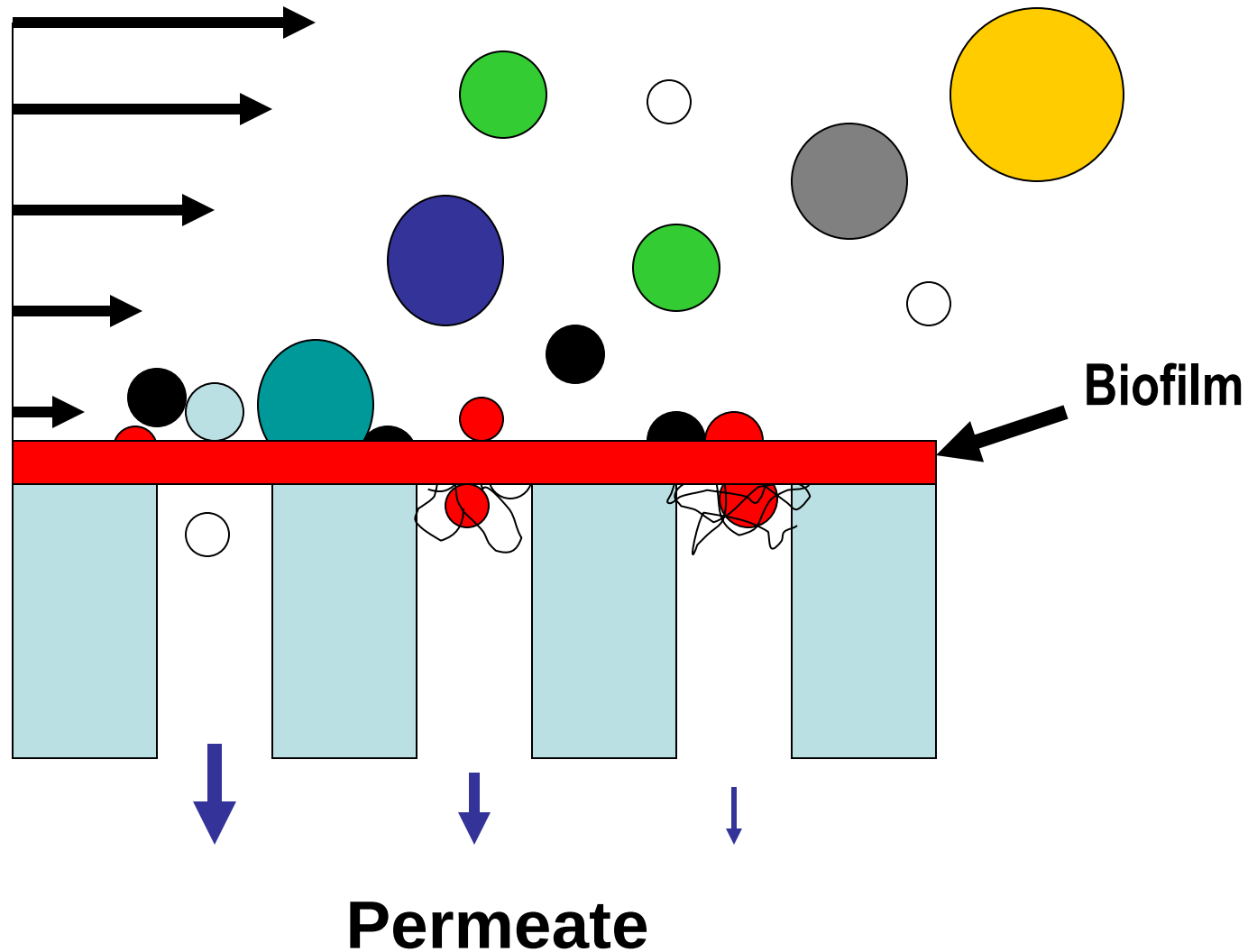
# Biofouling on Membrane Surface



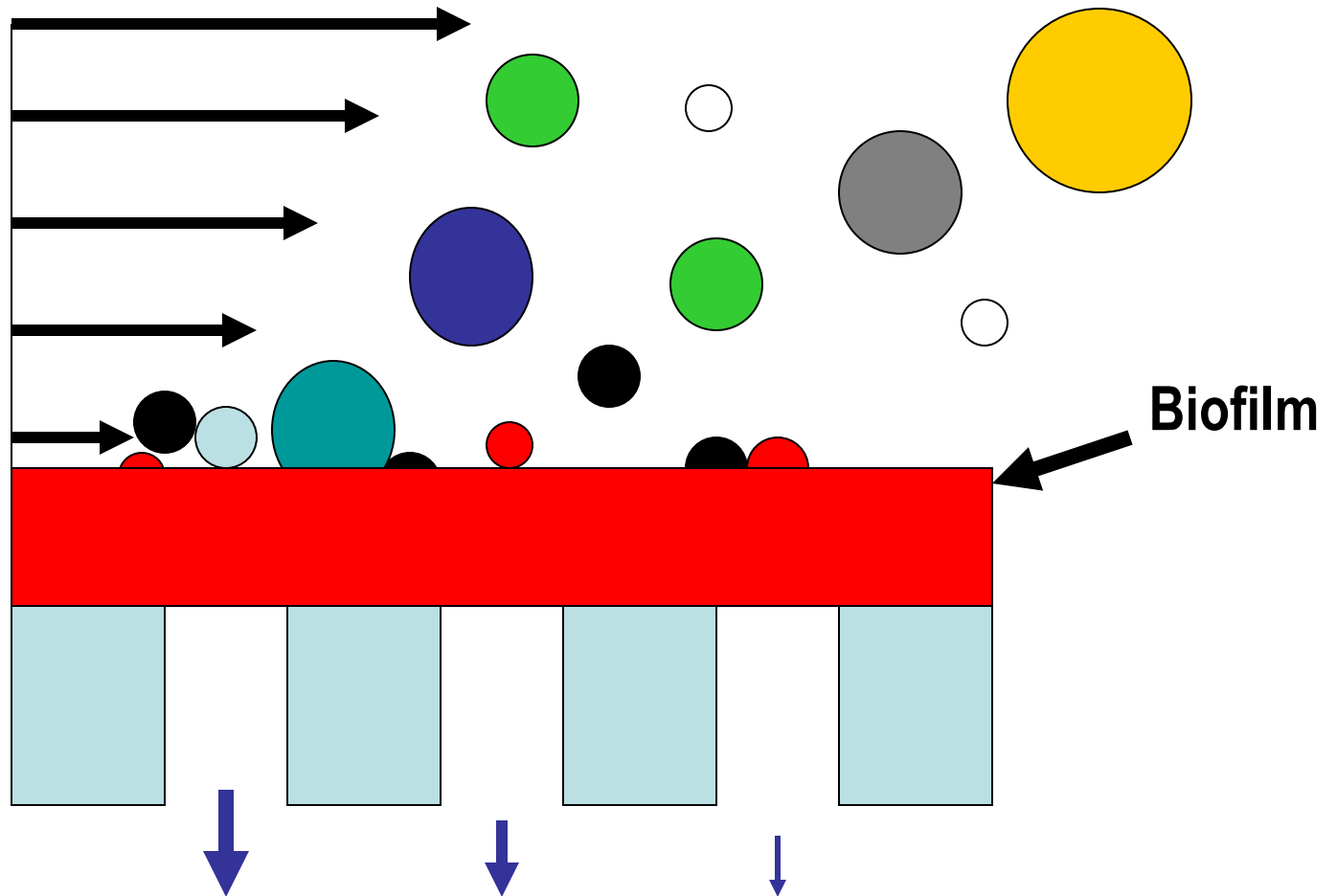
**Permeate**



# Biofouling on Low-Pressure Membrane Surface

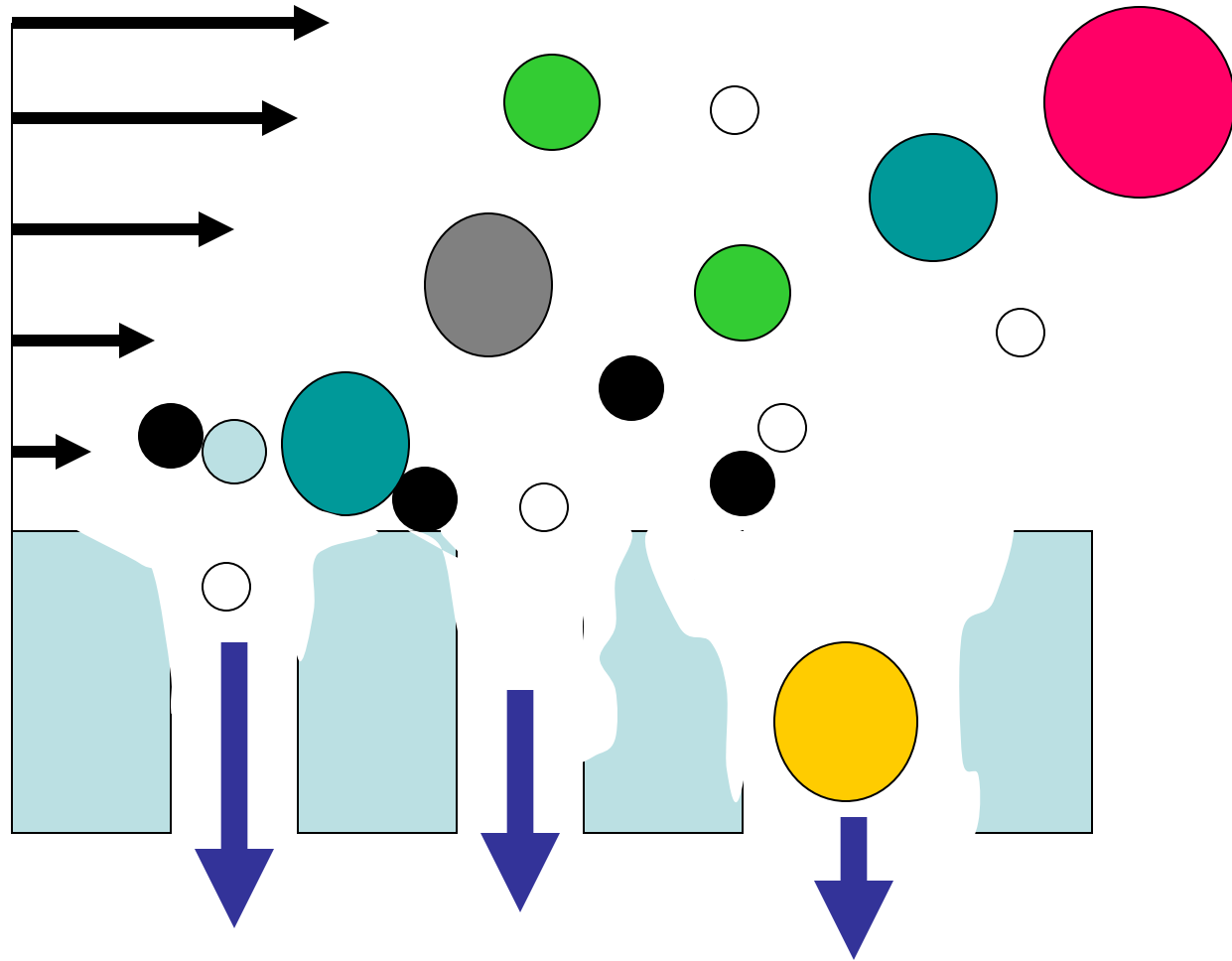


# Biofouling on High-Pressure Membrane



**Permeate**

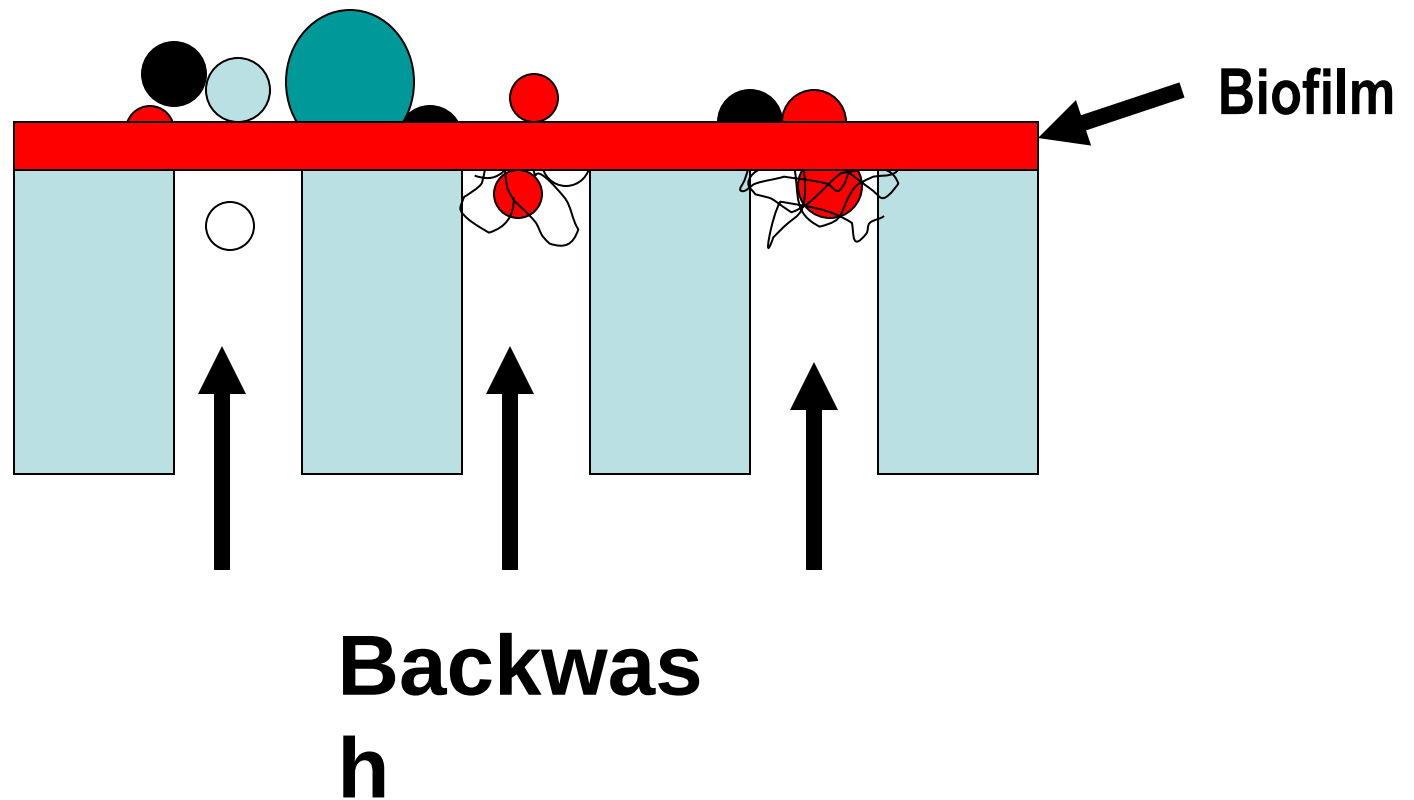
# Chemical Cleaning of Biofoulants



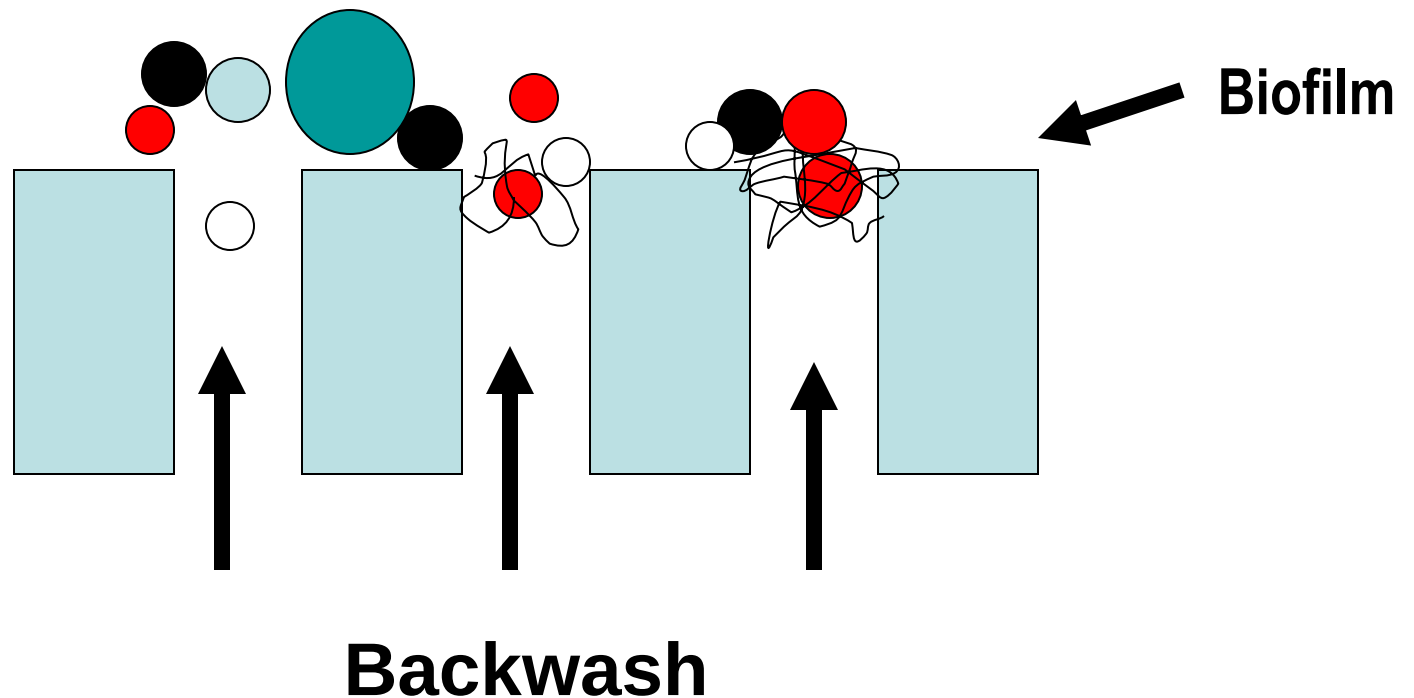
**Permeate**



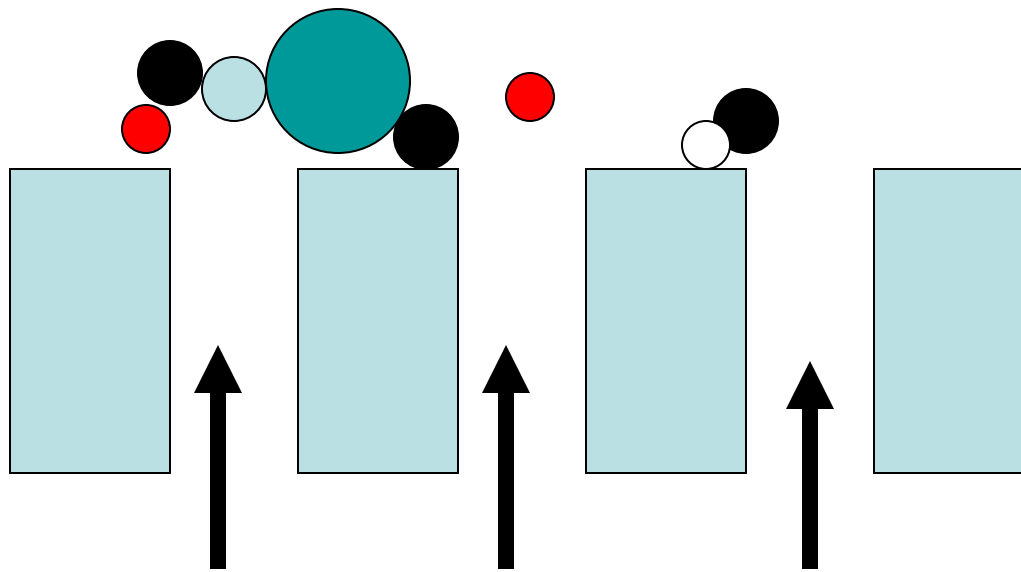
# Backwash on Low-Pressure Membrane



# Backwash on Low-Pressure Membrane



# Backwash on Low-Pressure Membrane

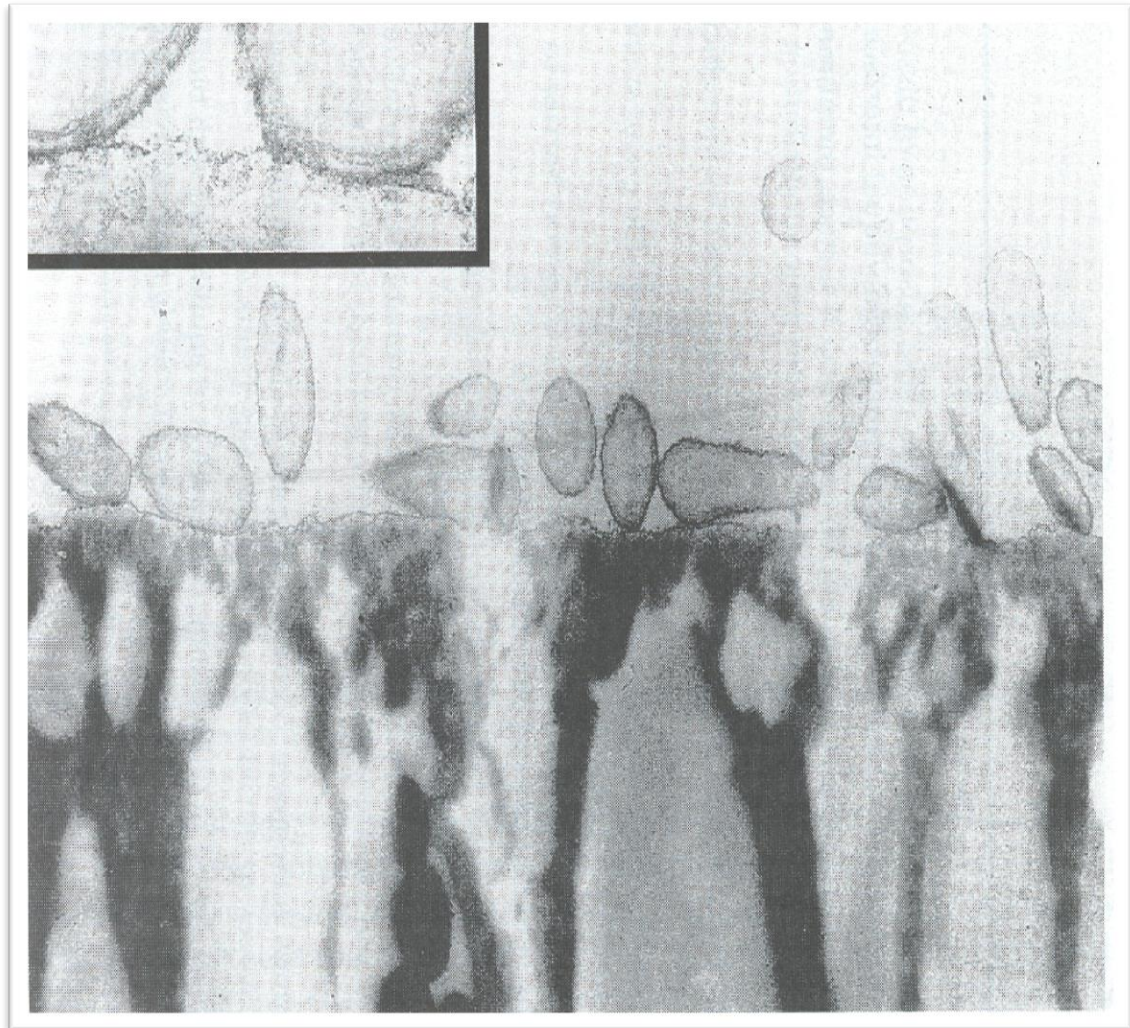


**Backwash (Plus Chemicals)**

## BioFouling

TEM photomicrograph of *Pseudomonas diminuta* cells attached to an aromatic cross-linked polyamide RO membrane. Note intimate association of bacterial surface macromolecules with the synthetic polymer surface (inset). Cells are about 1  $\mu\text{m}$  in diameter.

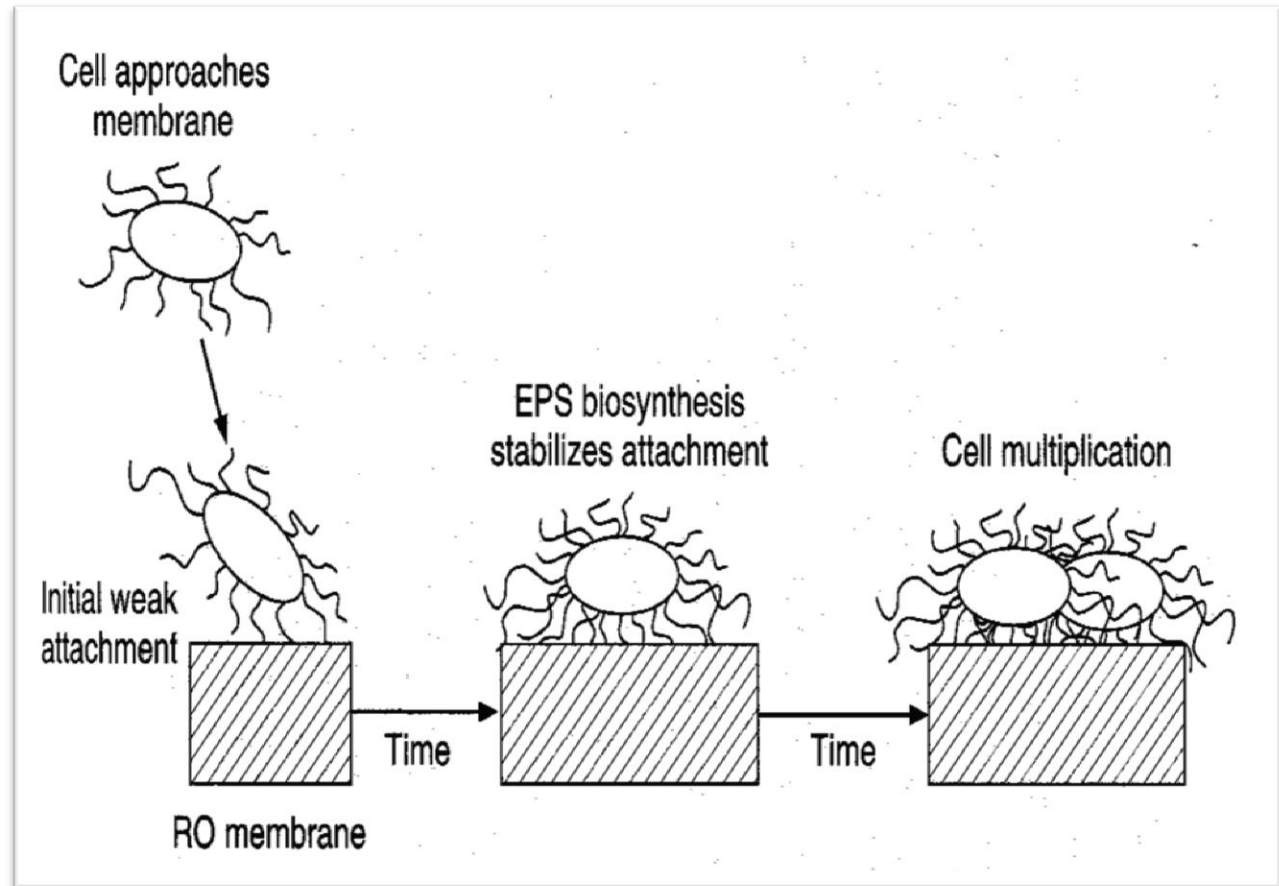
(Courtesy of Gabriella Schaule, University of Stuttgart, Stuttgart Germany)





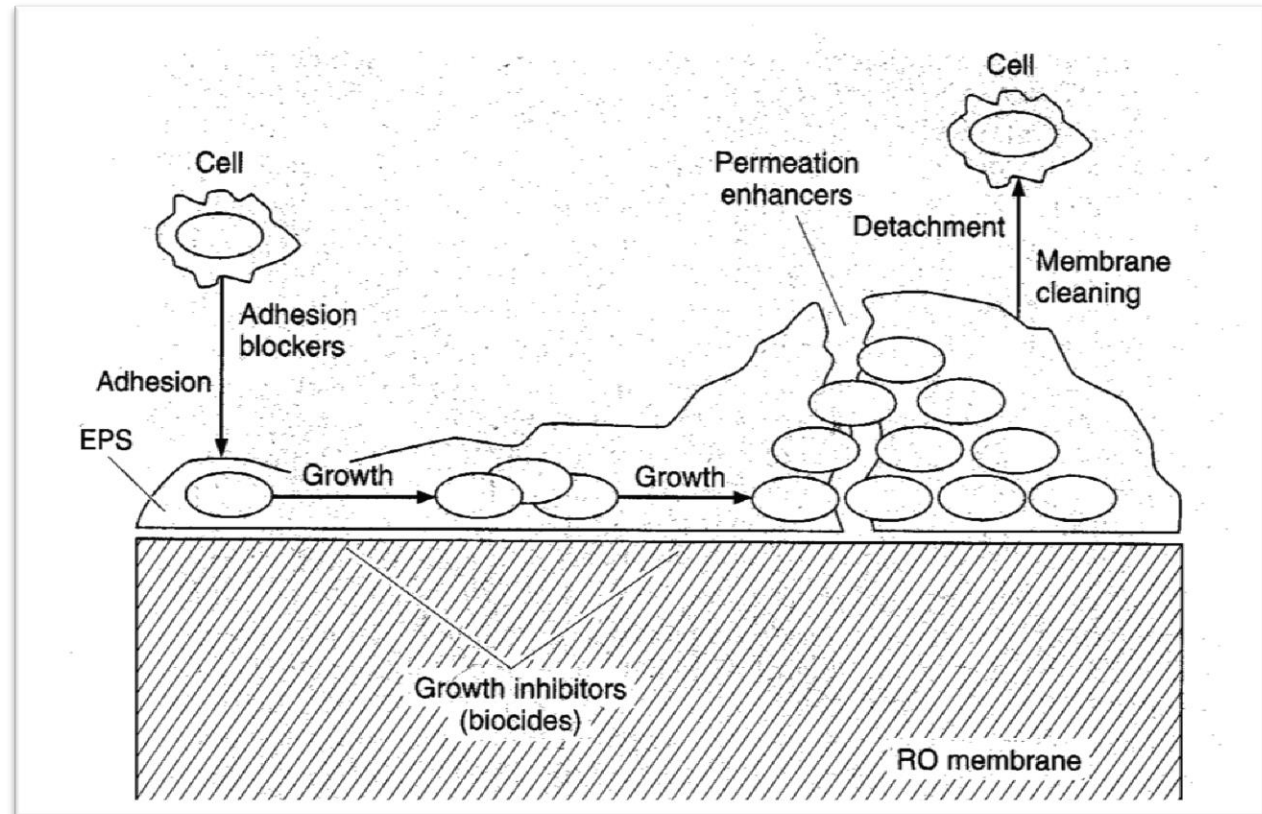
# Bacterial Adhesion Process

Schematic illustration of the bacterial adhesion process. Note that the irreversible adhesion phase is associated with EPS biosynthesis



## BioFouling Process

Schematic diagram showing potential points of intervention in the membrane biofouling process





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## Potential Points of Intervention in the Membrane Biofouling Process

# BioFouling Process

| Intervention point<br>or process | Approach or strategy                                                                                                                                                                                                                              |
|----------------------------------|---------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| Bacterial adhesion               | Addition to feedwater of biodispersants, surfactants, chaotropic agents, chelating agents, biocides, and other chemical compounds or formulations that interfere with bacterial attachment and colonization processes.                            |
| Growth and cell division         | Introduction of nonmetabolizable nutrient analogs, nutrient limitation (starvation), application of antimicrobial agents, biocides, electrical fields, or other conditions or substances that retard biofilm growth.                              |
| Feedwater bacteria               | Use of physicochemical processes such as continuous micro-filtration, affinity columns, biofloculants, etc., that physically remove or precipitate bacteria from the feedwater.                                                                   |
| Biofilm structure                | Treatment of biofilm with physicochemical cleaning agents, chelating agents, chaotropic agents, ultrasound, dispersants, electric fields, air backwashes (in the case of MF), or other agents/conditions which tend to disrupt biofilm integrity. |
| Biofilm permeation               | Addition of physicochemical agents/conditions or application of processes which enhance biofilm permeability to water and solute molecules; avoids need to remove biofilm.                                                                        |
| Membrane polymer                 | Design and synthesis of new membrane polymers with low affinity surfaces, surfaces that can be regenerated in situ, membranes with delayed-release antimetabolites or biocides incorporated, chlorine-resistant membranes, etc.                   |

# Events in Membrane Biofouling Processes

| Event                        | Time to onset*  | Description/explanation                                                                                                                                                                               |
|------------------------------|-----------------|-------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| Primary organic film         | Seconds/minutes | Typically referred to as the <i>conditioning</i> film; defined as the rapid adsorption of dissolved organic macromolecules and inorganic substances at the membrane/liquid interface.                 |
| Primary cell adhesion        | Seconds/minutes | Refers to pioneer bacterial attachment; dependent on nature of cell surface, membrane type, feedwater chemistry, and system hydrodynamics; provides major contribution to early biofilm accumulation. |
| Cellular detachment          | Seconds/minutes | Influences biofilm accumulation rate; detachment is sometimes enhanced by microbicidal agents, dispersants, etc.                                                                                      |
| Cell growth/multiplication   | Minutes/hours   | Occurs at expense of soluble and sorbed feedwater nutrients; may provide greatest contribution to biofilm formation where biocides are not present.                                                   |
| Biopolymer (EPS) synthesis   | Minutes/hours   | Provides for greater biofilm structural integrity; acts as a reactive transport barrier to chemical biocides; promotes nutrient concentration/storage.                                                |
| Particle/colloid entrainment | Seconds/minutes | Secondary effect where suspended particles and colloidal material are passively entrained in the biopolymer matrix or within biofilm void spaces.                                                     |
| Secondary cell adhesion      | Days/weeks      | Commences after primary biofilm formation by pioneer cells; probably strongly influenced by surface properties and physiology of primary biofilm and leads to greater species diversity.              |
| Biofilm sloughing            | Days/weeks      | Refers to cell and biomass detachment; occurs in response to changes in hydrodynamic shear or turbulence forces, or introduction of biocides, dispersants, etc.                                       |
| Biofilm senescence           | Weeks/months    | Refers to accelerated cell die-off in old biofilms; cell death is in equilibrium with biofilm growth in continuous flow systems; may result in release of soluble nutrients via cell lysis.           |

\*

Refer to time a new membrane is placed into operation.





# Design Criteria

| Criteria            | Example                          |
|---------------------|----------------------------------|
| Design flow         | m <sup>3</sup> /day              |
| Membrane modules    | 4UF, 2MF                         |
| Operation systems   | 2 Parallel                       |
| Recovery rate       | 90%                              |
| Pretreatment        | Filter cloth                     |
| Cleaning procedures | Pressurize water & air scrubbing |



## Recovery Rate

The percent of feedwater recovered is given in terms of water recovery:

$$\text{Recovery} = Q_p / Q_f \times 100\%$$

where  $Q_p$  = product water flow, m<sup>3</sup>/d or gpm

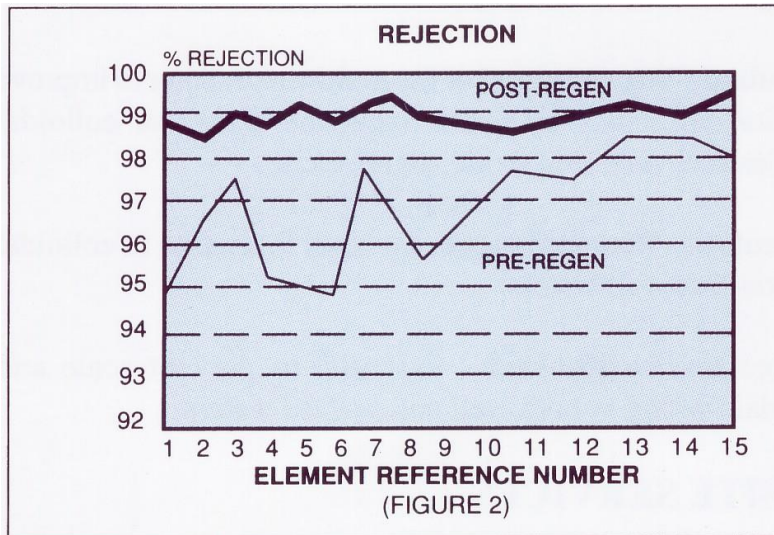
$Q_f$  = feedwater flow, m<sup>3</sup>/d or gpm



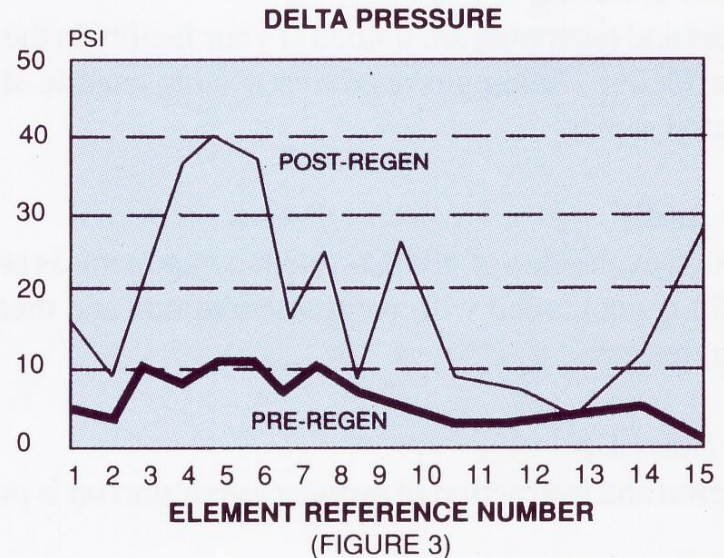
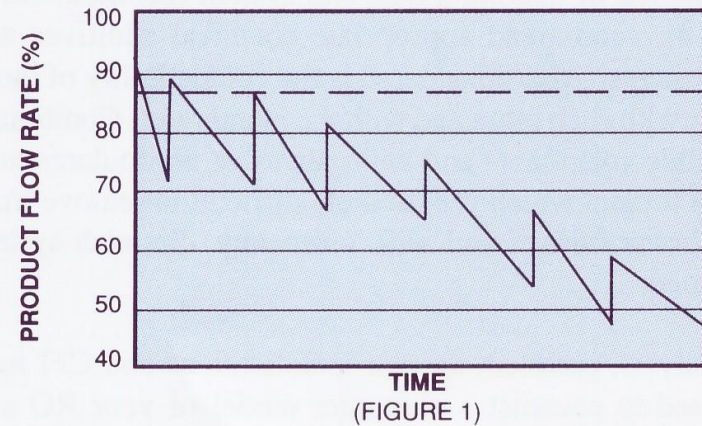
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# Cleaning Effects (1)



**THEORETICAL CLEANING RESPONSE CYCLE**



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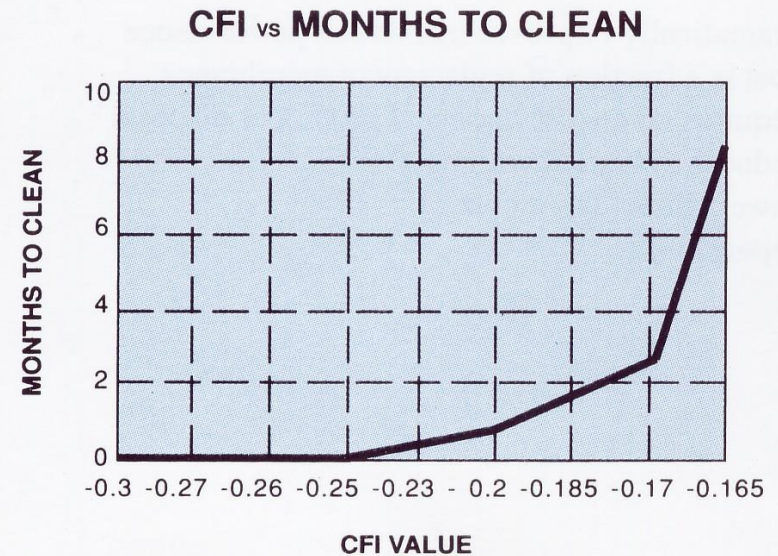
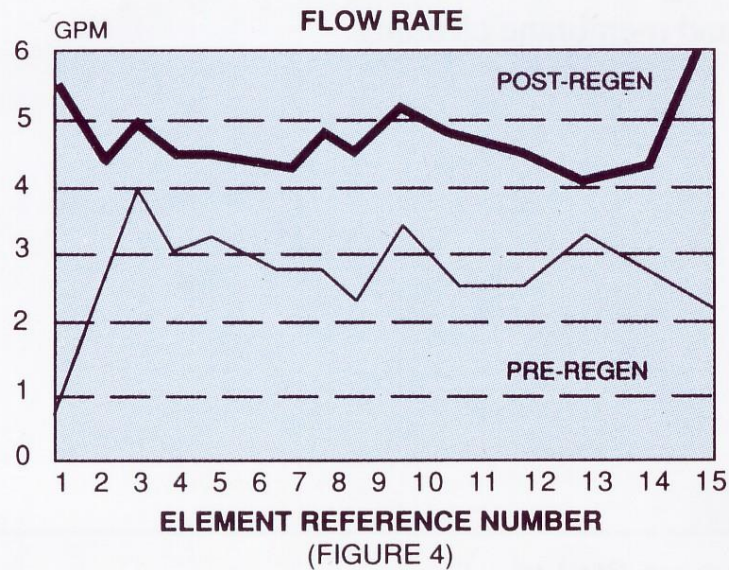




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## Cleaning Effects (2)



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# Some Generic Components in cleaning Solution Designed to Treat Biofouling Separation Membrane

| Component         | Examples                                                                                                                                                           | Membrane compatibility*                                            | Concentration†                                                                                      | Function/comment                                                                                                                                                                                                                                                                                                                                                                                                          |
|-------------------|--------------------------------------------------------------------------------------------------------------------------------------------------------------------|--------------------------------------------------------------------|-----------------------------------------------------------------------------------------------------|---------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| Detergent         | SDS<br>SDBS<br>Triton series<br>Quaternary amines<br>Many others                                                                                                   | CA, PA, PS<br>CA, PA, PS<br>CA, PS, PE<br>CA, PS, PE               | 0.01–2.0%                                                                                           | Detergents function to (1) disrupt hydrophobic bonds involved in bacterial attachment and EPS stabilization, and (2) denature proteins and other macromolecules involved in stabilizing cell envelopes of bacteria. Detergents weaken biofilm structural integrity and exhibit biocidal activity in many instances (e.g., the quats). Compatibility of untested detergents with membranes must be determined empirically. |
| Chaotropic agents | Urea<br>Guanidium HCl                                                                                                                                              | CA, others unknown<br>CA, others unknown                           | 6–8 molar<br>1–2 molar                                                                              | Chaotropic agents disrupt hydrogen bonds involved in stabilizing secondary/tertiary structure of biopolymers. Most effective when used in combination with denaturing agents such as SDS and/or chelating agents such as EDTA.                                                                                                                                                                                            |
| Chelating agent   | Citrate<br>EDTA                                                                                                                                                    | All<br>All                                                         | 0.1–1.0%<br>0.1–1.0%                                                                                | Chelate divalent cations such as calcium and magnesium, which are involved in stabilizing cell envelopes of bacteria. EDTA also exhibits broad spectrum biocidal activity. Chelating agents are especially effective when applied in combination with detergents and other antimicrobial agents and enzymes.                                                                                                              |
| Enzyme(s)         | Proteases<br>Esterases<br>Lipases<br>Polysaccharidases<br>Lysozyme                                                                                                 | All                                                                | 10–100 mg/L<br>10–100 mg/L<br>10–100 mg/L<br>10–100 mg/L                                            | A combination of enzymes may assist in breaking down the biofilm EPS matrix which typically consists of complex heteropolysaccharides, lipopolysaccharides, and proteins.                                                                                                                                                                                                                                                 |
| Biocide           | Sodium bisulfite<br>Quaternary amines<br>Formaldehyde<br>Glutaraldehyde<br>Isothiozolinone<br>(Kathon)<br>Sodium benzoate<br>Monochloramine<br>EDTA<br>Many others | All<br>CA, PS, PE<br>All<br>All<br>All<br>All<br>CA, PA, PS<br>All | 10–100 mg/L<br>0.1–1.0%<br>0.1–5.0%<br>0.1–5.0%<br>0.1–1.0%<br>0.1–1.0%<br>0.5–5.0 mg/L<br>0.1–1.0% | Broad spectrum microbial biocides are useful in inhibiting or slowing regrowth of biofilm microorganisms following chemical cleaning of membranes. Compatibility of untested biocides with membranes must be determined empirically.                                                                                                                                                                                      |

# Disinfecting Agents Commonly Used to control Biofouling in membrane System

| Biocide category | Examples                      | Useful concentration range | Membrane compatibility         | Comments                                                                                                                                                                                                                                                                                          |
|------------------|-------------------------------|----------------------------|--------------------------------|---------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| Oxidizing        | Chlorine                      | 0.1–1.0 mg/L               | CA, PS                         | The oxidizing biocides listed are used primarily as feedwater additives. Because monochloramine exhibits reduced oxidizing activity compared to free chlorine, it does not harm performance of PA membranes. It is an excellent biocide, especially with respect to biofilms (see text).          |
|                  | Monochloramine                | 0.5–5.0 mg/L               | All                            |                                                                                                                                                                                                                                                                                                   |
|                  | Peracetic acid                | 0.1–1.0 mg/L               | CA, PS                         |                                                                                                                                                                                                                                                                                                   |
|                  | Hydrogen peroxide             | 0.1–1.0 mg/L               | All                            |                                                                                                                                                                                                                                                                                                   |
|                  | Iodine                        |                            |                                |                                                                                                                                                                                                                                                                                                   |
| Nonoxidizing     | Formaldehyde                  | 0.5–5.0%                   | All                            | Sodium bisulfite is the only nonoxidizing biocide currently used as a feedwater additive. All others listed are used primarily to preserve membranes from biodegradation/biodeterioration during plant inactivity.                                                                                |
|                  | Glutaraldehyde                | 0.5–5.0%                   | All                            |                                                                                                                                                                                                                                                                                                   |
|                  | Bisulfite                     | 1.0–100 mg/L               | All                            |                                                                                                                                                                                                                                                                                                   |
|                  | 2-methyl-4-isothiazolin-3-one | 0.01–1.0%                  | All                            |                                                                                                                                                                                                                                                                                                   |
|                  | Quaternary amines             | 0.01–1.0%                  | CA, PS                         |                                                                                                                                                                                                                                                                                                   |
|                  | Benzoate                      | 0.1–1.0%                   | All                            |                                                                                                                                                                                                                                                                                                   |
|                  | EDTA                          | 0.01–1.0%                  | All                            |                                                                                                                                                                                                                                                                                                   |
| Irradiation      | Ultraviolet                   | 1–2 MR*                    | All (conditional) <sup>†</sup> | Disinfection by irradiation is very effective but leaves no biocide residual following exposure; thus, surviving bacteria may regrow on membrane surfaces. Gamma irradiation is particularly suited for disinfection of new membrane modules (sealed in plastic wrap) prior to long-term storage. |
|                  | Gamma                         |                            | All (conditional)              |                                                                                                                                                                                                                                                                                                   |

\* MR = megarads.

<sup>†</sup> Compatibility depends on radiation dosage, temperature, pH, redox potential, and other factors.



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## Ex. 1: Reverse Osmosis

A city's water demand is 26.5 metric ton/day (7 mgd). What is the source water (feedwater) flow required for a brackish water RO, if the plant recovery rate is 78 percent?

***Solution 1***



## Ex. 2: Reverse Osmosis

For a brackish water RO treatment plant, the feedwater applied is 53.0 ton/d (14 mgd) to the membrane and the product water yields 42.4 ton/d (11.2 mgd). What is the percentage of brine rate?

*Solution 2*





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## Ex. 3: Reverse Osmosis

At a brackish water RO treatment plant, the total dissolved solids concentrations for the pretreated feed water and the product water are 2860 and 89 mg/L, respectively. Determine the percentages of salt rejection and salt passage.

**Solution 3**



## Ex. 4: Reverse Osmosis

A pretreated feedwater to a brackish water RO process contains 2600 mg/L of TDS. The flow is 0.25 m<sup>3</sup>/s (5.7 mgd). The designed TDS concentration of the product water is no more than 450 mg/L. The net pressure is 40 atm. The membrane manufacturer provides that the membrane has a water flux rate coefficient of  $1.8 \times 10^{-6}$  s/m and solute mass transfer rate of  $1.2 \times 10^{-6}$  m/s. Determine the membrane area required.

**Solution 4**



# Silt Density Index

**The Silt Density Index (SDI) is calculated as:**

$$\text{SDI} = \left( 1 - \frac{t_i}{t_f} \right) \frac{100}{15}$$

**where  $t_i$  = time initially needed to filter 500 mL of sample**  
 **$t_f$  = time needed to filter 500 mL at the end of the 15 min test period**



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## Ex. 5: Silt Density Index

The time initially required to filter 500 mL of a dual-media filter effluent is 14.5 s. The time required to filter 500 mL of the same water sample at the end of the 15 min test period is 48 s. Calculate the SDI.

**Solution 5**



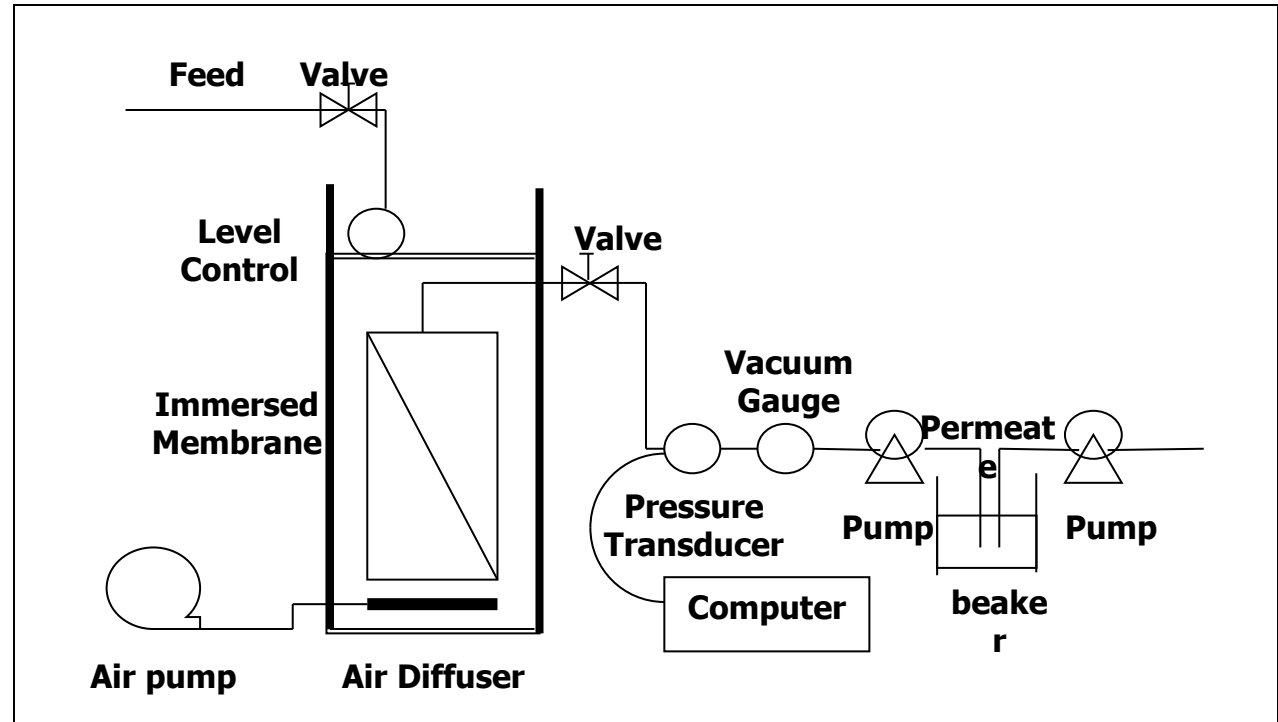


## **Critical Flux of Membrane Bioreactor (MBR) for Palm Oil Mill Effluent (POME) Treatment under Aerobic Conditions**

### **Objectives**

- To determine critical flux of a flat sheet MF membrane for POME treatment
- To determine the optimum operating conditions of flux in POME treatment to optimize the permeate production

# MBR Pilot Plant



## Significance of Study

- Most of the studies conducted on fouling were focused on fouling mechanism in side-stream membrane systems for POME treatment
- This study is focused on fouling control in submerged MF MBR through flux control



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# Operational Stage

- Permeability Test
- Critical Flux Test
- Operating Conditions



# Permeability Test

- Conducted before and after experiment in MBR
  - The pump suction rate was controlled
  - Permeate collected for 1 minute in measuring cylinder to measure volume and record TMP
  - Permeability was calculated
  
- To determine suitability of the MF membranes for POME treatment





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# Critical Flux Concept

- Critical flux is the flux at which colloidal deposition takes place
- Below the critical flux value, the flux is directly proportional to transmembrane pressure (TMP)



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# Importance of Critical Flux

- Essential design parameter
  - Determine membrane area and reactor volume required
- Limit of operation
  - Avoid rapid fouling
  - Avoid frequent cleaning



# Critical Flux Test

- To determine the critical flux value
  - Permeate collected for 1 minute in measuring cylinder to measure volume and record TMP
  - Suction rate increased for every 10 minutes until critical flux exceeded
  - When critical flux exceeded, suction rate decreased for every 10 minutes
- To determine three appropriate fluxes



# Operating Conditions

- Three flux levels were determined

|        | Condition     | Flux, LMH |
|--------|---------------|-----------|
| Flux 1 | Supercritical | 12        |
| Flux 2 | Critical      | 11        |
| Flux 3 | Subcritical   | 10        |

- TMP and permeate quality were monitored



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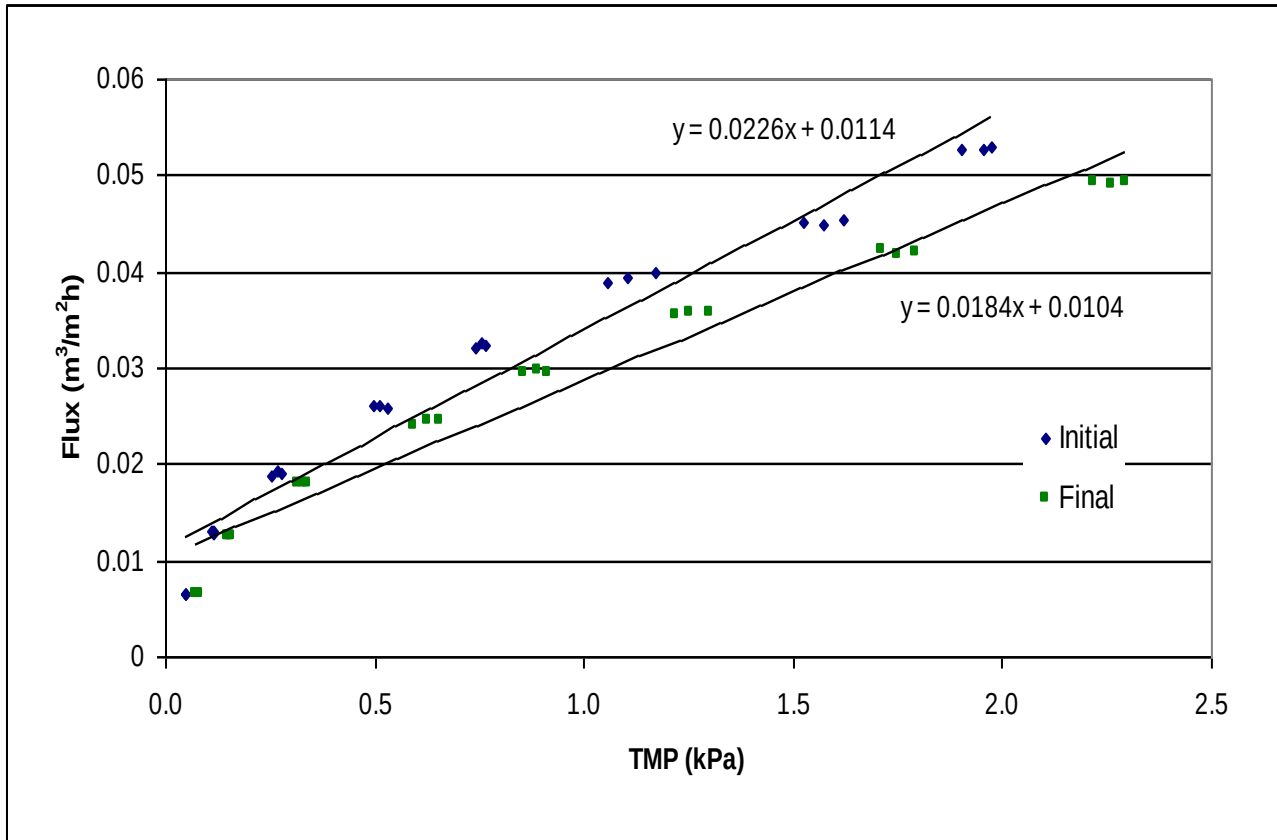
# Results and Discussion

- Permeability of Distilled Water
- Critical Flux
- Effect of Fixed Flux on TMP
- Permeate Quality

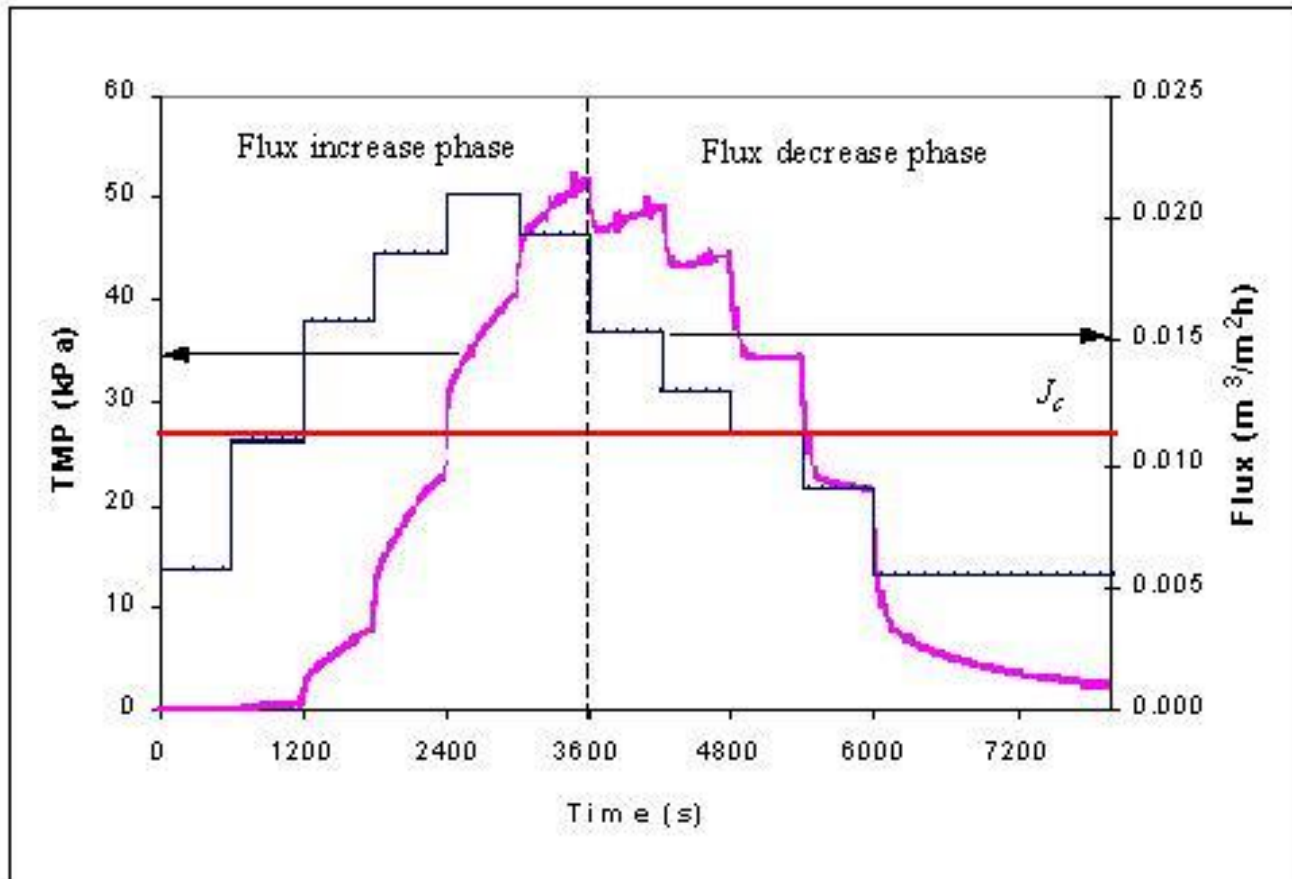




# Permeability of Distilled Water



## Critical Flux, $J_c$

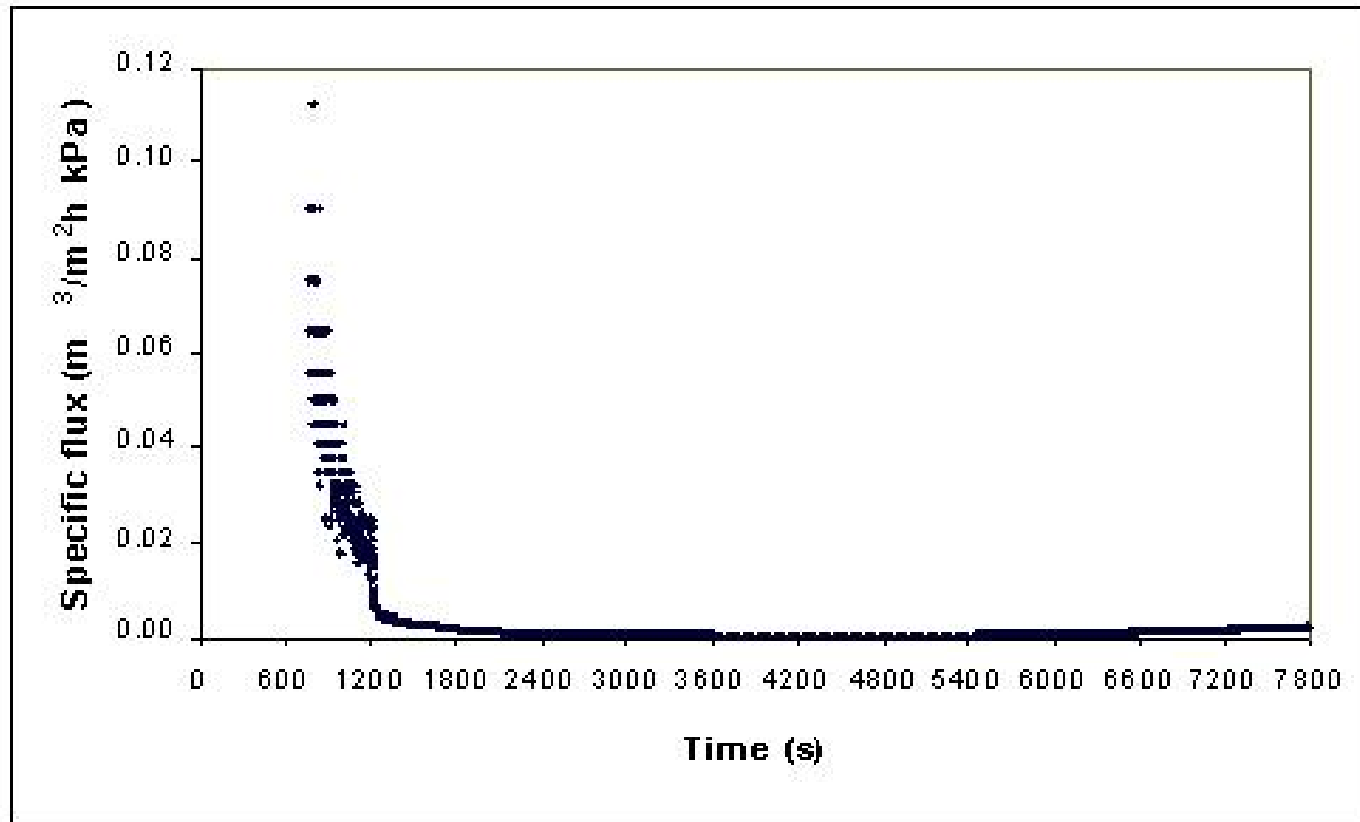




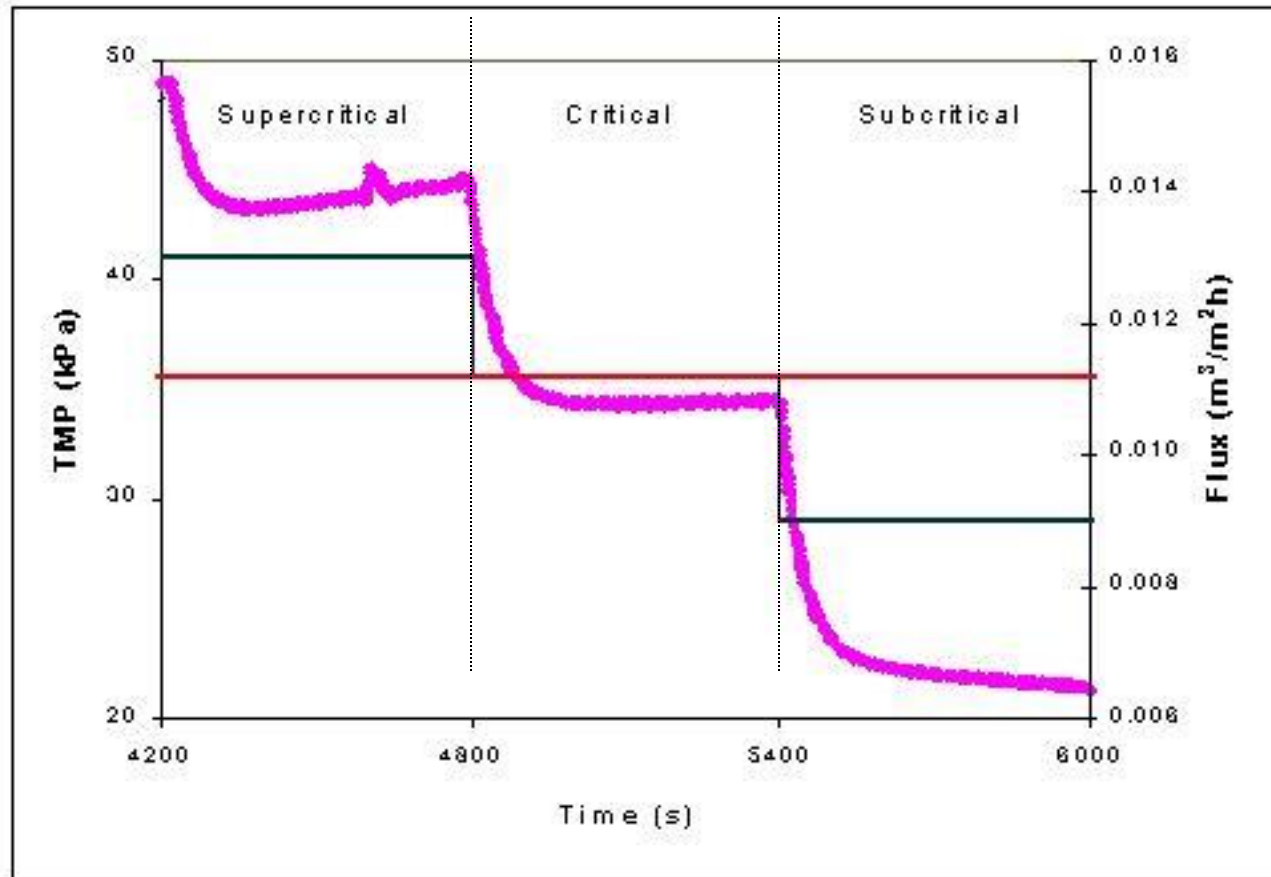
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# Specific Flux from Critical Flux

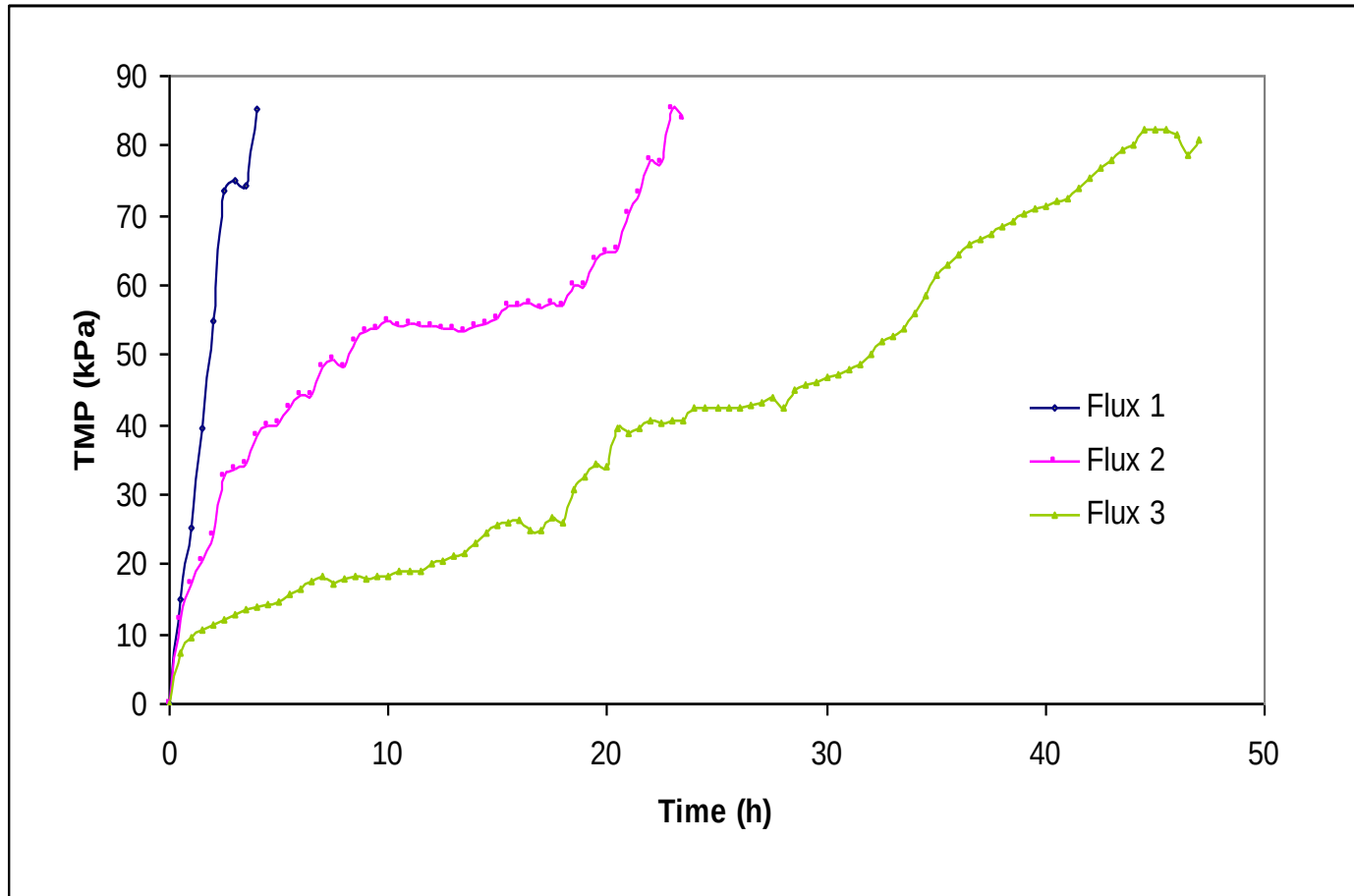


## $J_c$ Determination





# Effect of Fixed Flux on TMP







# Permeate Quality

| Parameter               | Influent   | Effluent   |            |            | Effluent with PAC |
|-------------------------|------------|------------|------------|------------|-------------------|
|                         |            | Flux 1     | Flux 2     | Flux 3     |                   |
| BOD <sub>5</sub> (mg/l) | 180 ± 40*  | 15 ± 5     | 10 ± 5     | 10 ± 5     | -                 |
| COD (mg/l)              | 1015 ± 50  | 150 ± 5    | 110 ± 5    | 100 ± 5    | -                 |
| TKN (mg/l)              | 325 ± 30   | 83.0 ± 9.1 | 79.5 ± 7.5 | 76.5 ± 6.3 | -                 |
| TP (mg/l)               | 212 ± 19   | 65.2 ± 7.3 | 63.6 ± 5.6 | 62.3 ± 5.1 | -                 |
| Turbidity (NTU)         | 45.5 ± 3.5 | 3.0 ± 1.0  |            |            | 1.0 ± 0.0         |
| Colour (ADMI)           | 3120 ± 220 | 2400 ± 100 |            |            | 17.5 ± 0.5        |

\* BOD<sub>3</sub> at 30°C



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## Conclusion

- Critical flux is among the important design consideration
- $J_c$ : 0.0112 m<sup>3</sup>/m<sup>2</sup>h
- Sub-critical flux
  - Less fouling
  - Simple cleaning method
  - Longer membrane lifespan