



vito
vision on technology

29/03/2012

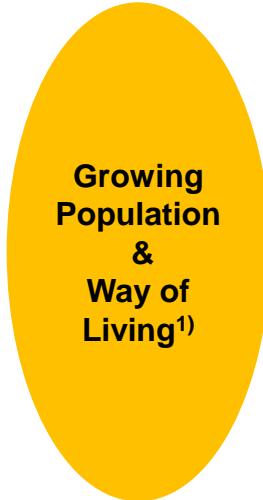
Membranen en nieuwe technologie

Wilrijk, 27 februari, 2012

Ludo Diels

DPSIR-analysis of global problems

DRIVERS lead to..

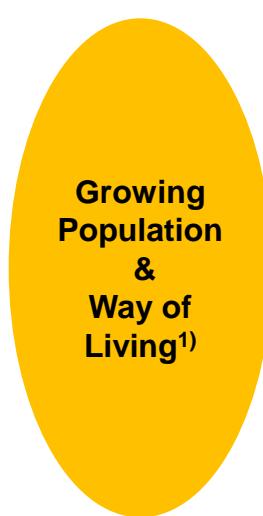


PRESSURES on..

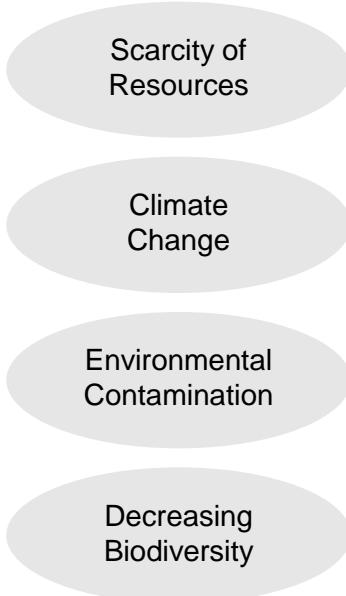


Successful structural changes need radical transitions in central societal systems

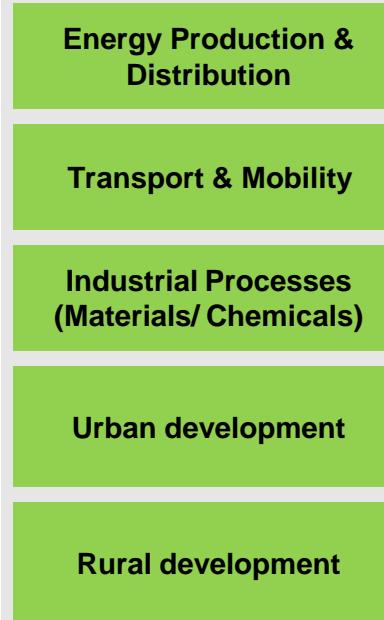
DRIVERS lead to..



PRESSURES on..



..STATE of
Key Societal systems



..ultimately leading to respective visible
IMPACTS (illustrative examples)

- Security of Supply issues
- High Fossile Fuel prices
- Inefficient distribution and storage
- Traffic Jams
- Car/ship/plane emissions
- Water pollution; Waste
- Inefficient usage of materials
- Production inefficiencies
- Low energy efficiency in housing
- Waste and wastewater
- Deforestation
- Water scarcity and wastewater
- Food supply

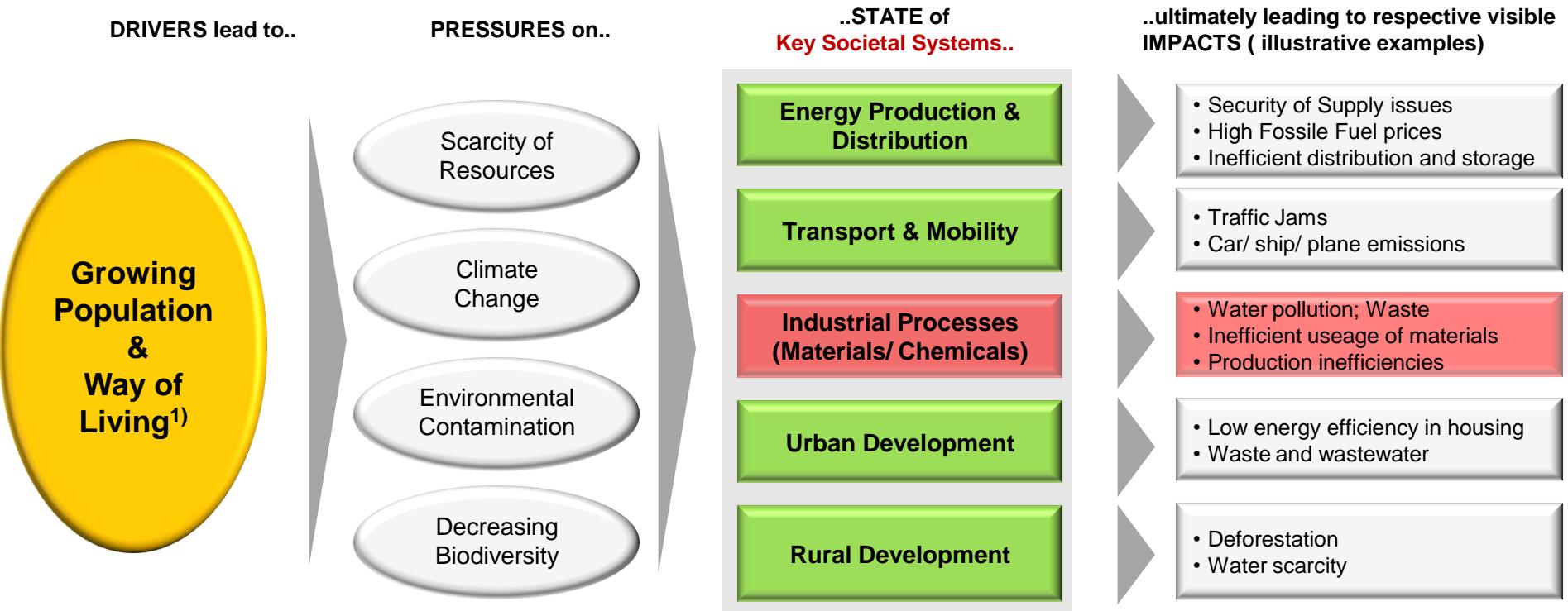
While Solutions or "Responses" are at hand at each stage, a transition will really be successful when the need of acting end-of-pipe disappears

RESPONSES



Notes: 1) Way of Living is directly related to degree of Development

REAL CHANGE = TRANSITIONS, NOT OPTIMISATIONS



While Solutions or "Responses" are at hand at each stage, a transition will really be successful when the need of acting end-of-pipe disappears

RESPONSES



Lifestyle Changes



Policy



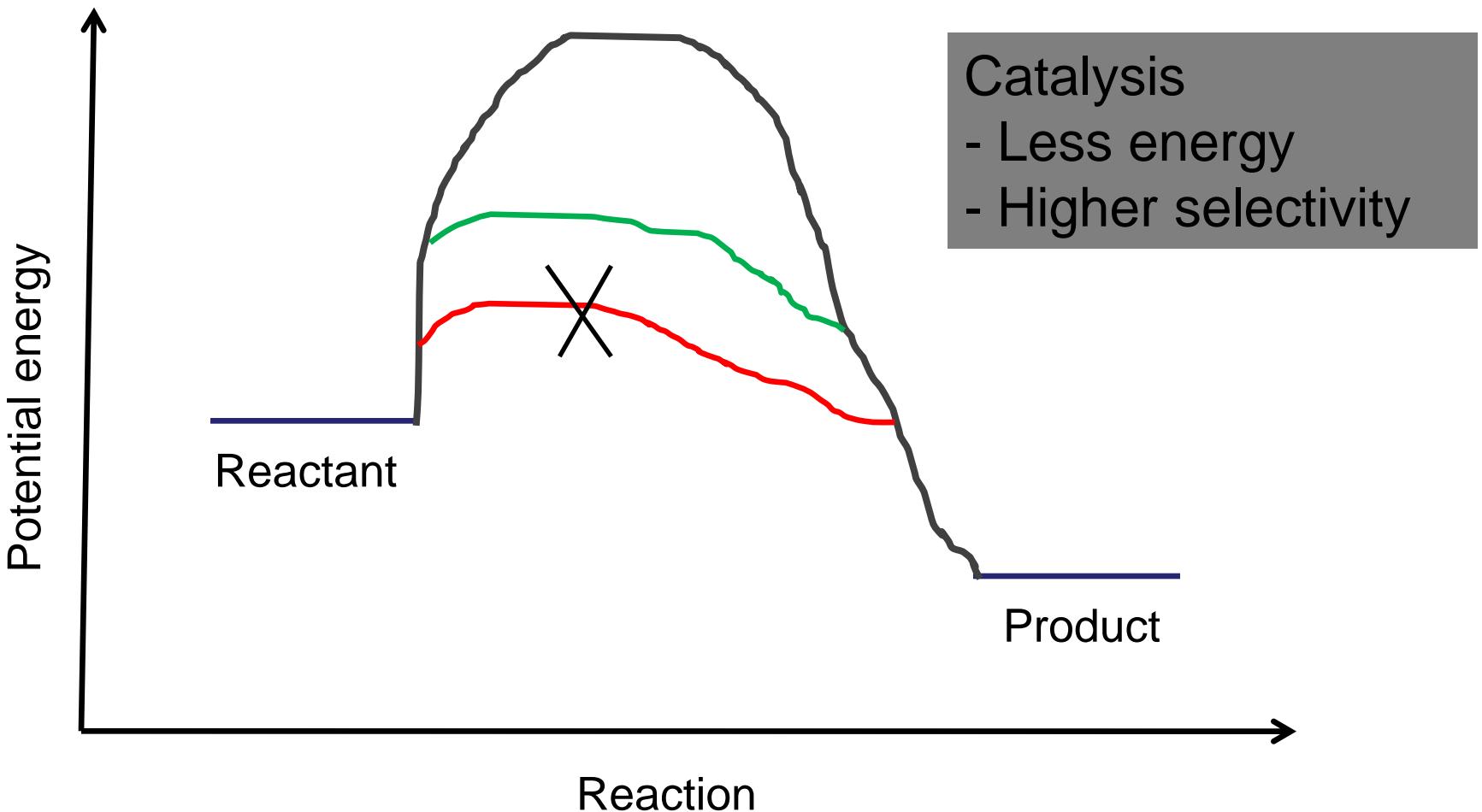
Cleantech solutions



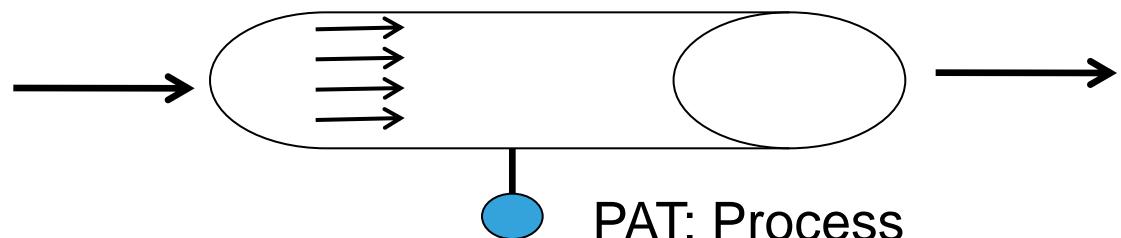
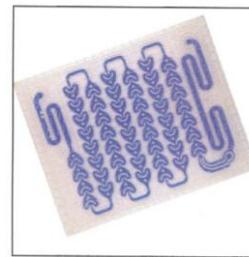
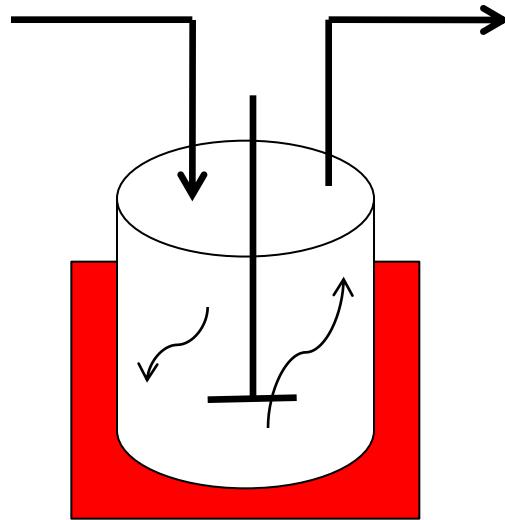
Remedial Cleantech/
Cleaning solutions

Notes: 1) Way of Living is directly related to degree of Development

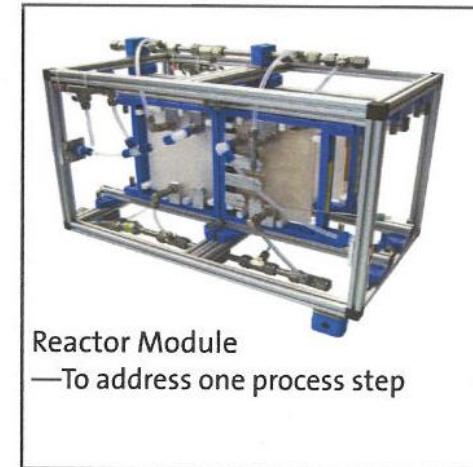
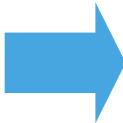
Maximize the effectiveness of inter and intramolecular reactions



Give each molecule the same processing history

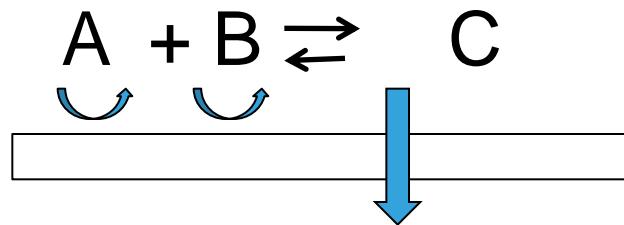


PAT: Process
Analytics Technology

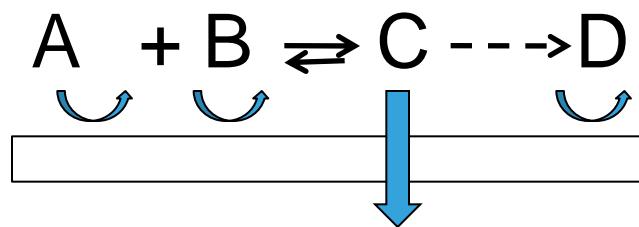


Reactor Module
—To address one process step

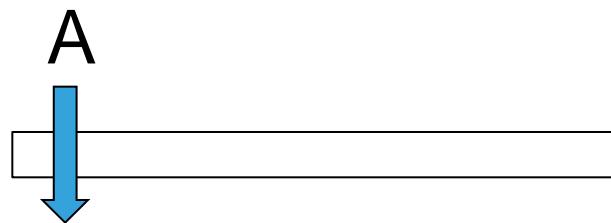
Process intensification: In situ product recovery



Change in
equilibrium

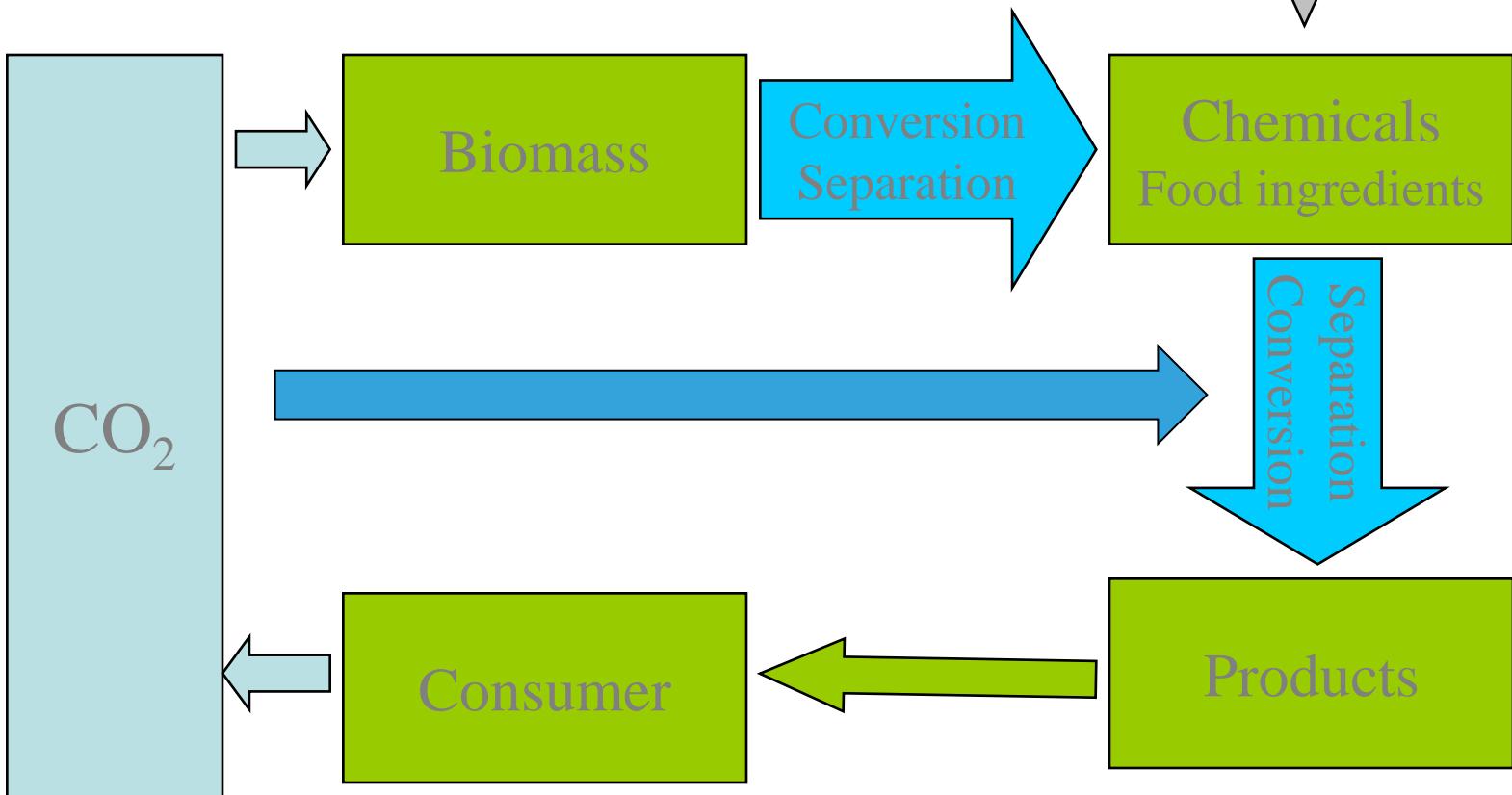
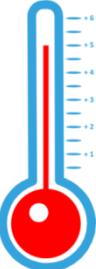


Avoiding further
reactions



Dosing chemicals
to control the right
reaction

Objective 2: From fossil-based to biomass-based chemistry

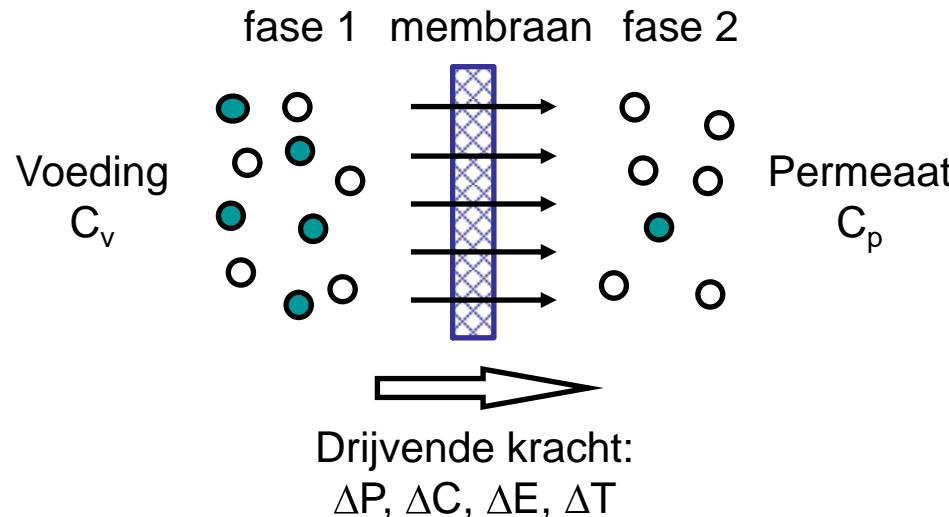


OUTLINE

1. Inleiding
2. Membranen / Modules en ontwerp
3. Procesparameters en fouling
4. Overzicht waterige membraantoepassingen
5. Case-studies membranen voor waterzuiveringstoepassingen
6. Overzicht en case-studies niet-waterige membraantoepassingen
7. Conclusies

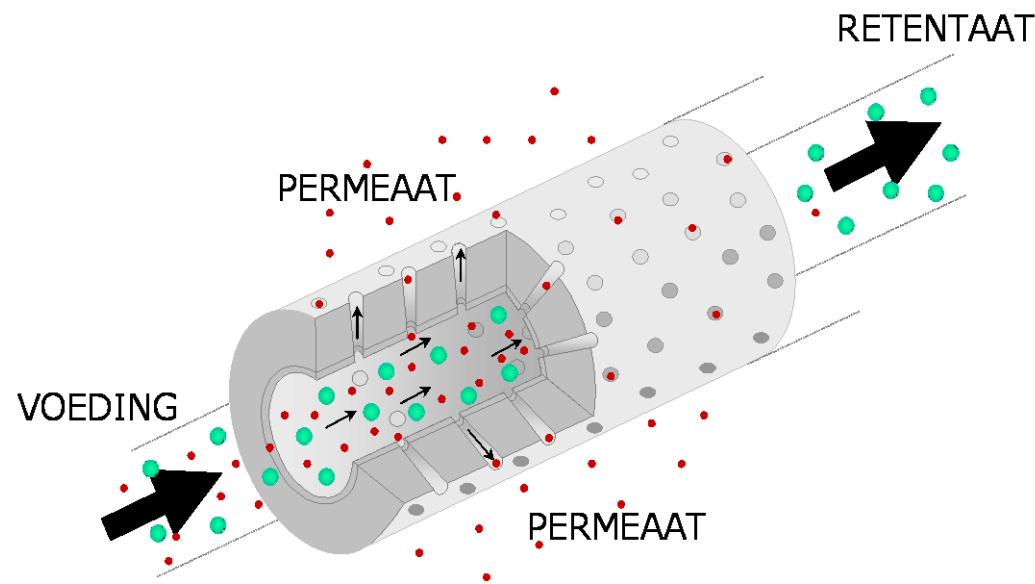
DEFINITIE MEMBRAAN

- ✓ Semi-selectieve barrière tussen twee fasen



- ✓ Efficiëntie bepaald door
 - Permeabiliteit of flux
 - Selectiviteit of retentie

DEFINITIE MEMBRAAN



PORIËNBEREIK

Proces	Soort deeltje	Dimensies (nm)
MF	gist en fungi	1000 - 10000
	bacteriën	300 - 10000
	olie emulsies	100 - 10000
	colloidale deeltjes	100 - 1000
	virussen	30 - 300
UF	proteïnen/polysacchariden (10^4 - 10^6 Da)	2 - 10
	enzymes (10^4 - 10^5 Da)	2 - 5
NF	klassieke antibiotica (300 – 1000 Da)	0.6 - 1.2
RO	organische moleculen (30 – 500 Da)	0.3 - 0.8
	anorganische ionen (10 – 100 Da)	0.2 - 0.4
	water (18 Da)	0.2

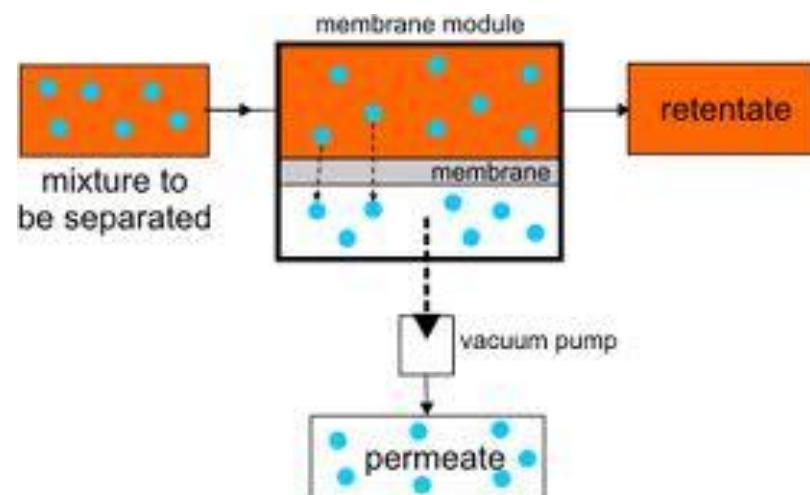
DRUKGEDREVEN MEMBRAANPROCESSEN

Proces	Tegengehouden componenten	Druk	Poriegrootte
RO	99% voor meeste ionen Meeste organische componenten > 150 Da	10 – 100 bar	Dens
NF	95% voor divalente ionen 40% voor monovalente ionen Organische componenten > 300 Da	5 – 20 bar	~1 nm
UF	Meeste organische componenten > 1000 Da	1 – 5 bar	2 – 100 nm
MF	Gesuspendeerde deeltjes > 0.1 µm	0.1 – 3 bar	0.1 – 20 µm

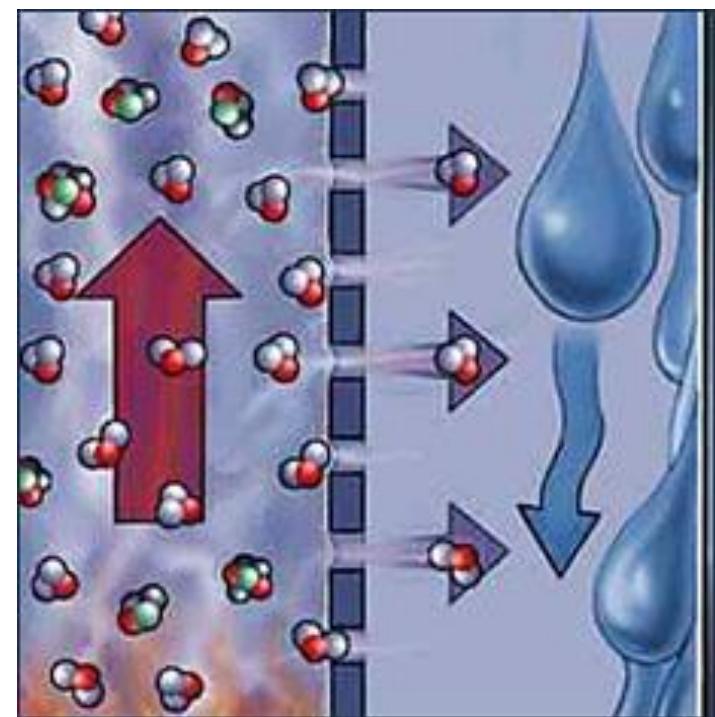
MEMBRAANFILTRATIEPROCESSEN

Proces	Drijvende kracht	Scheiding	Toepassing
Microfiltratie (MF)	ΔP	grootte	bacteriënenfilter (sterilisatie)
Ultrafiltratie (UF)	ΔP	grootte	eiwitconcentratie, olie/water scheiding, klaring
Nanofiltratie (NF)	ΔP	grootte + lading	ontharden water, verwijderen micropolluenten
Omgekeerde osmose (RO)	ΔP	oplos/diffusie	ontzoutting, productie ultrapuur water
Elektrodialyse (ED)	ΔE	lading	verwijdering ionen uit water, scheiden AZ
Gasscheiding (GS)	ΔP	oplos/diffusie	O_2/N_2 , CO_2/H_2 scheiding, verwijderen NO_x
Pervaporatie (PV)	Δp (activiteit)	oplos/diffusie	verwijderen vluchtlige componenten
Membraandistillatie (MD)	$\Delta T/\Delta P$	damp-vloeistof evenwicht	productie puur water, verwijderen VOCs
Vloeibare membranen (LM)	activiteit	oplosbaarheid/ affiniteit	verwijderen metaalionen uit water, zure gassen uit lucht

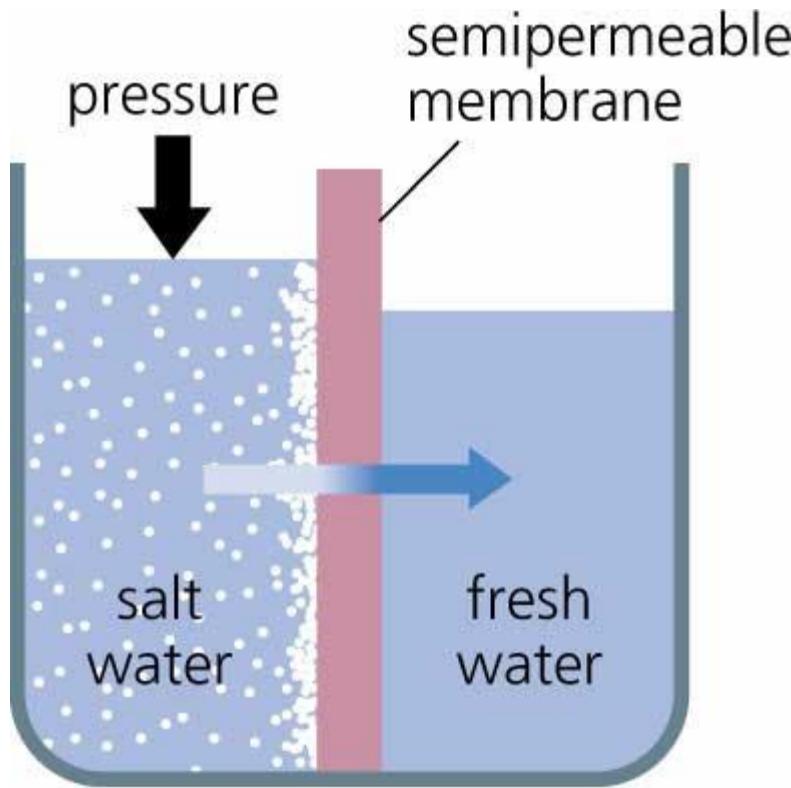
Pervaporation



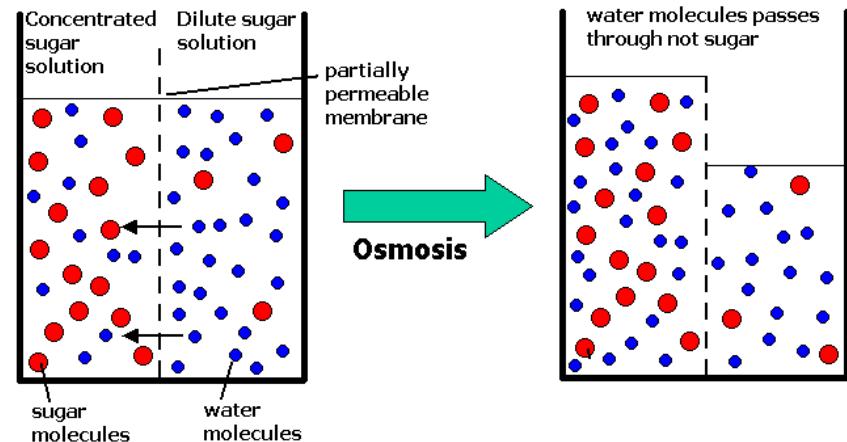
Membrane distillation



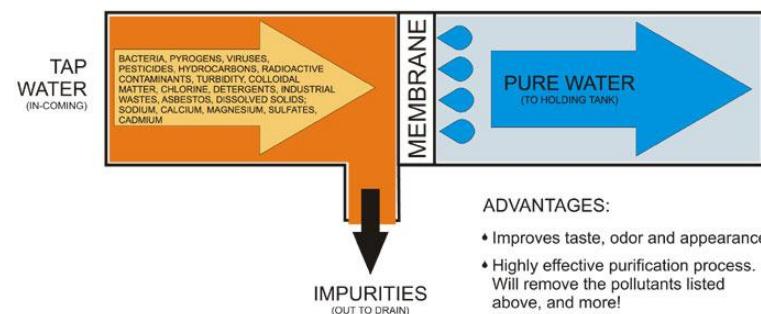
Reverse osmosis



(Forward) Osmosis



FROM TAP WATER TO PURE WATER



ADVANTAGES:

- Improves taste, odor and appearance
- Highly effective purification process. Will remove the pollutants listed above, and more!
- Consumes no energy
- Very convenient
- Flushes away pollutants, does not collect them
- Easy to keep clean
- Low production cost - gives you water of a guaranteed quality for pennies per gallon

ED Principles

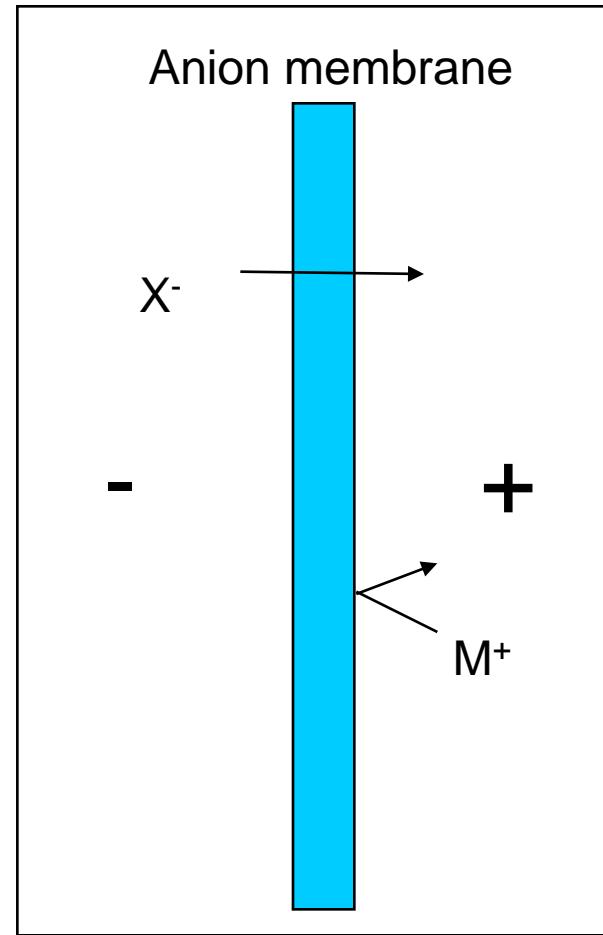
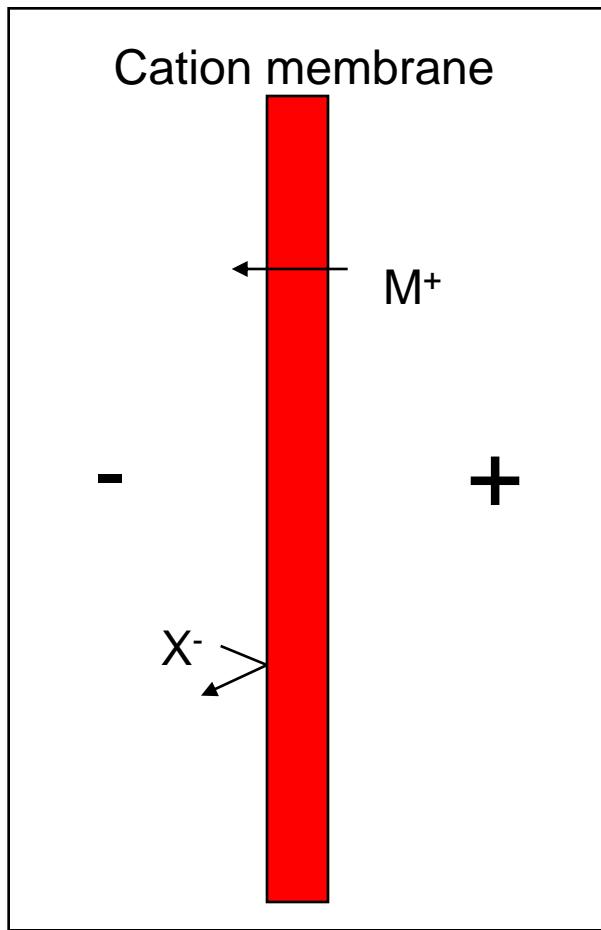
- » Dialysis: process of solute transport through a membrane when solute activity (concentration) is higher on one side of the membrane
= solute diffusion from a concentration gradient
- » Electrodialysis (ED): process of ion transport through a membrane as a result of an electrical field across the membrane
= ion transport is possible **against a concentration gradient (desalination)**
- » Remark:
 - osmosis = desalination with transport of water through the membrane
 - electrodialysis = desalination with transport of solute through the membrane

ED Membranes

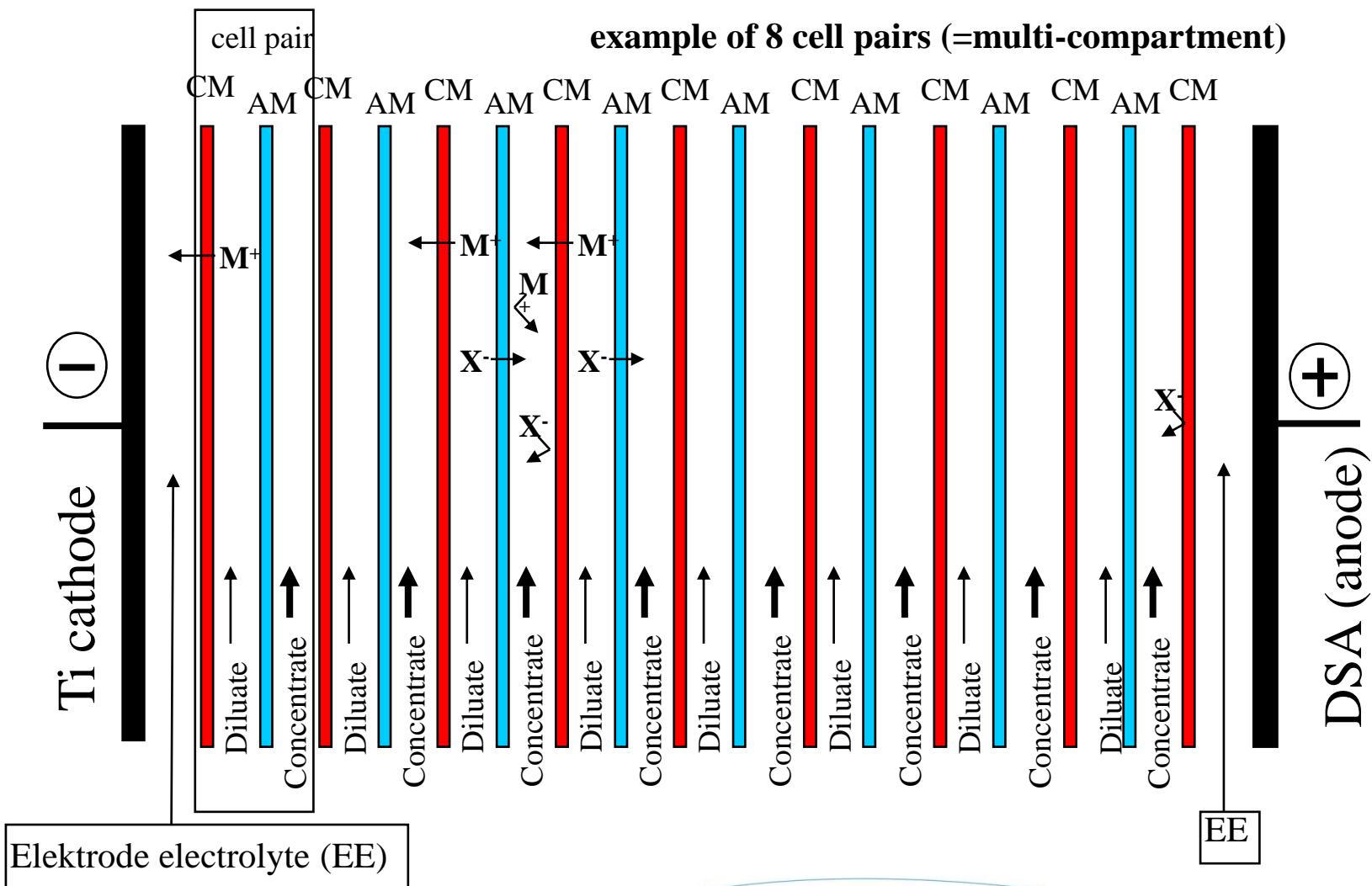
- » Cation selective membranes (CM)
 - » polymer chains with fixed negative groups (SO^{3-} , COO^- , PO_3^{2-} , HPO^{2-} , AsO_3^{2-} , SeO^{3-})
 - » allow cations to diffuse through the CM membrane
- » Anion selective membranes (AM)
 - » polymer chains with fixed positive groups (NH^{3+} , RNH_2^+ , R_3N^+ , R_3P^+ , R_2S^+)
 - » allow anions to diffuse through the AM membrane



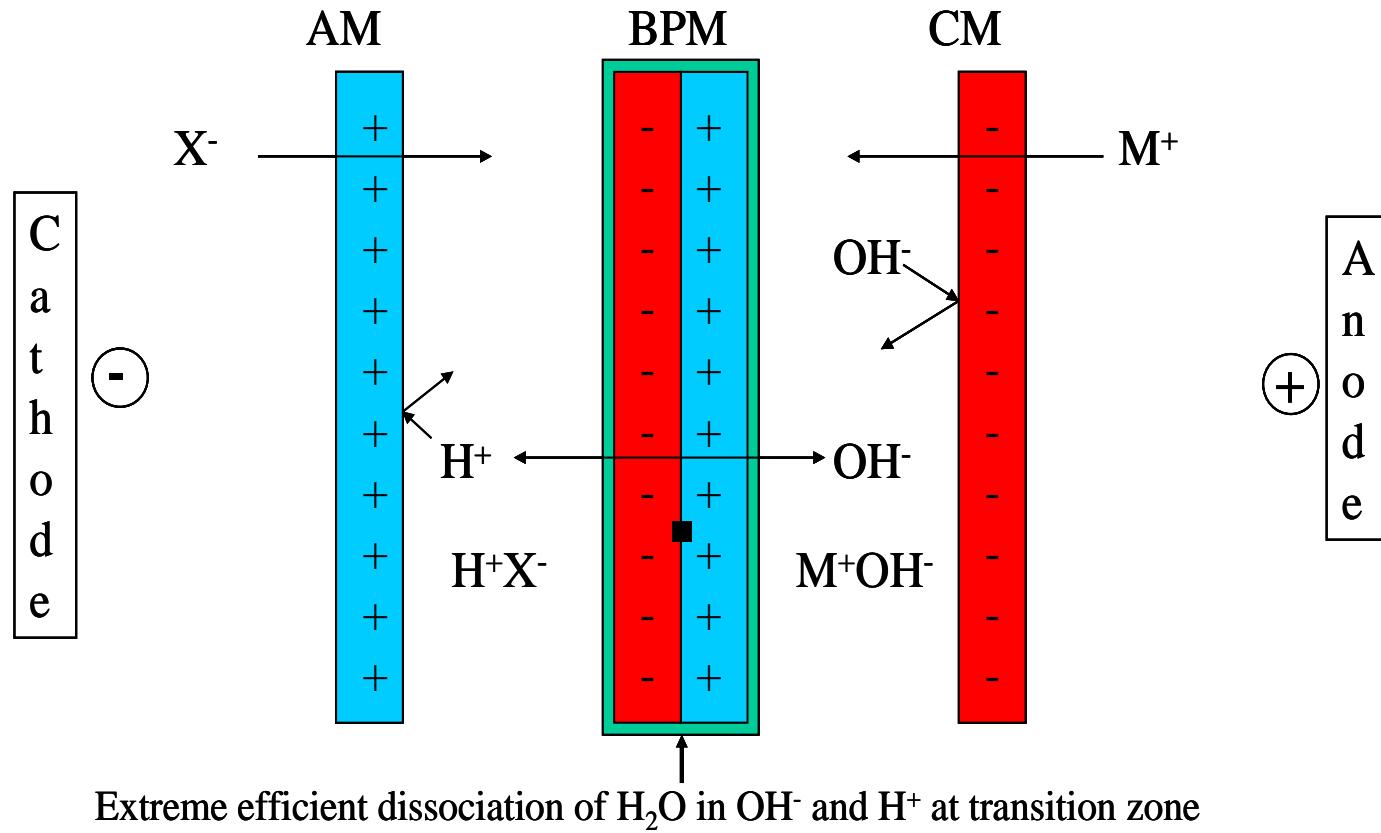
ED Principles



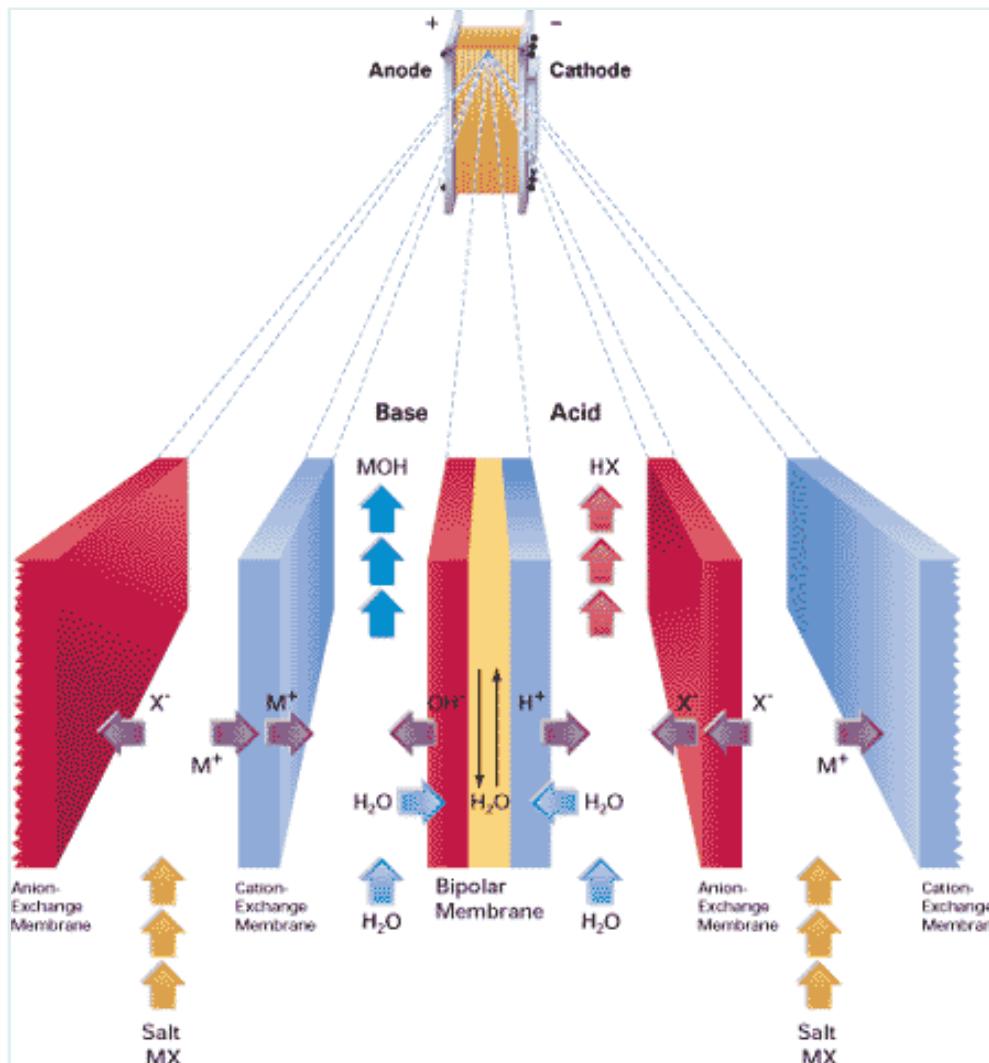
ED Stack



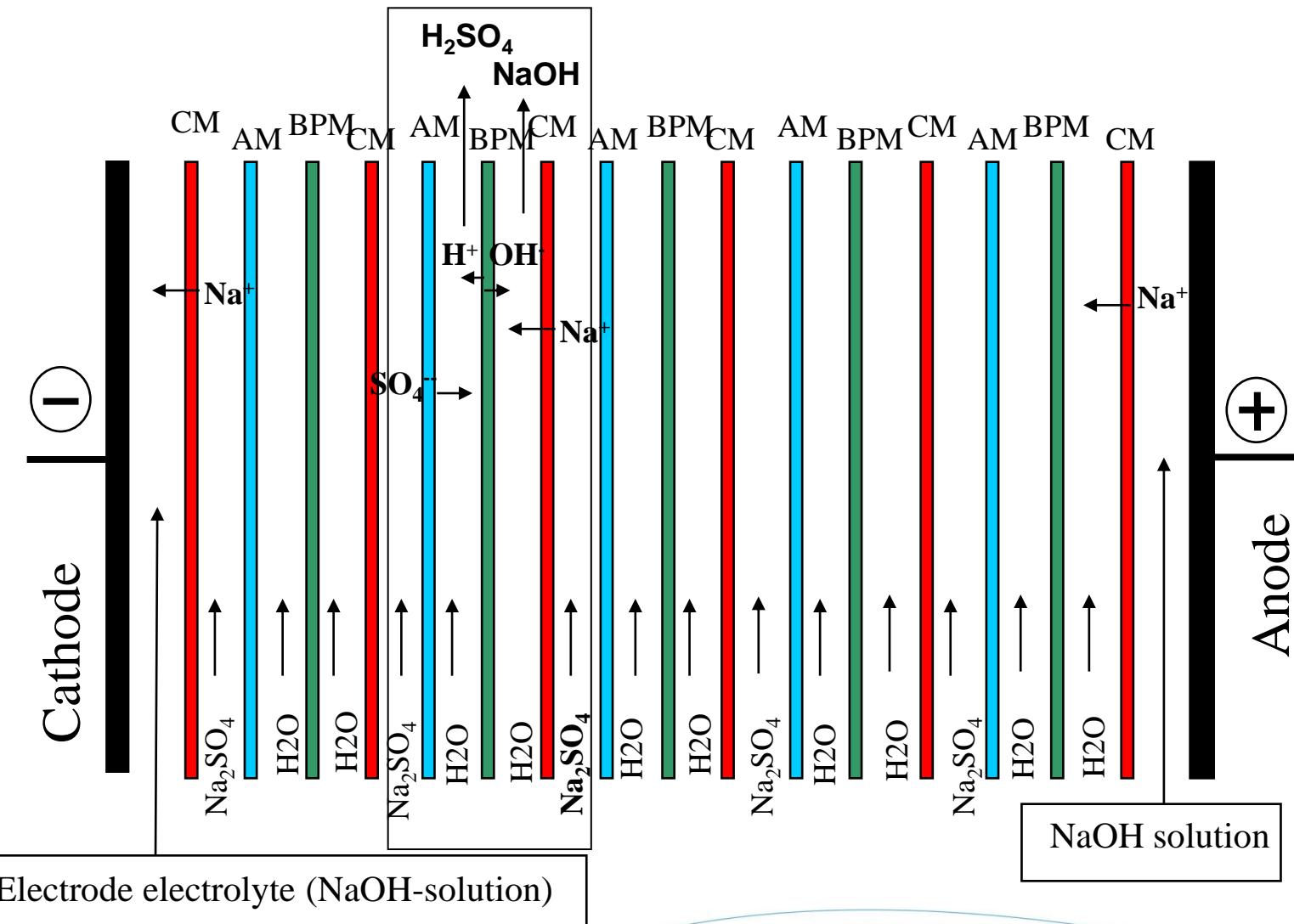
Bipolar membranes



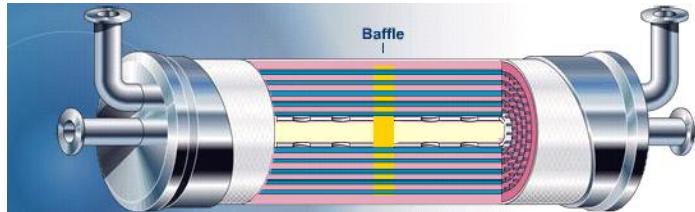
Bipolar membranes



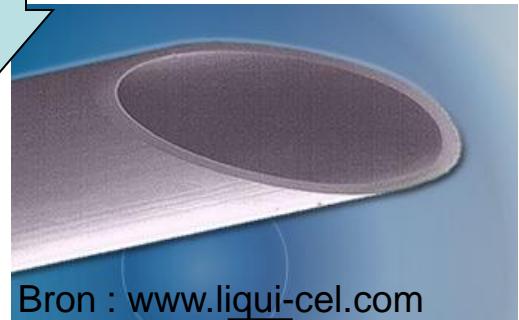
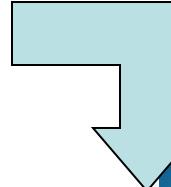
ED BPM of sodium sulphate



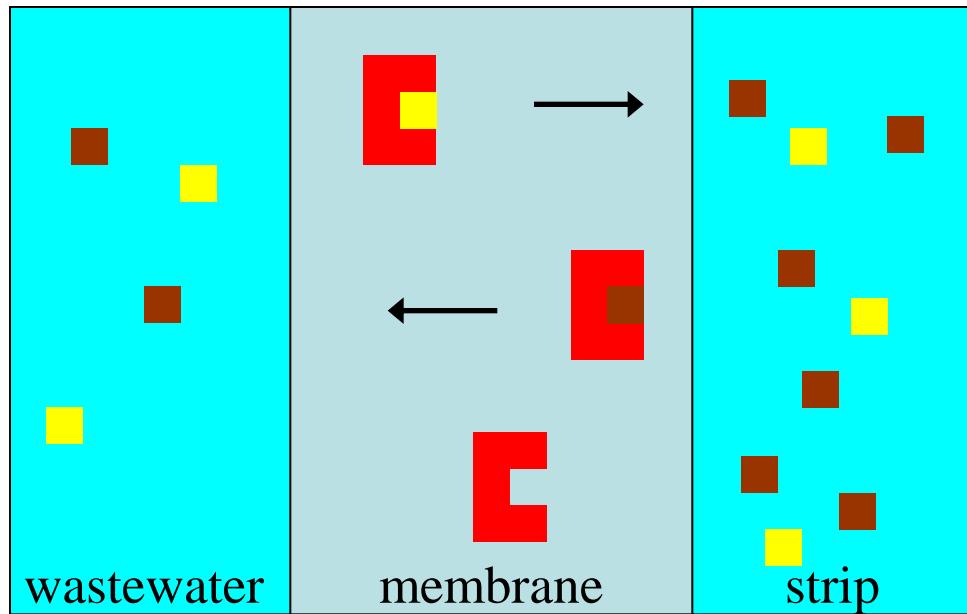
Supported Liquid Membranes (SLM)



Bron : www.liqui-cel.com



Bron : www.liqui-cel.com



■ Metal ion
■ Proton

■ Extractant



VISION ON TECHNOLOGY

Academia
Meets

10 oktober 2006

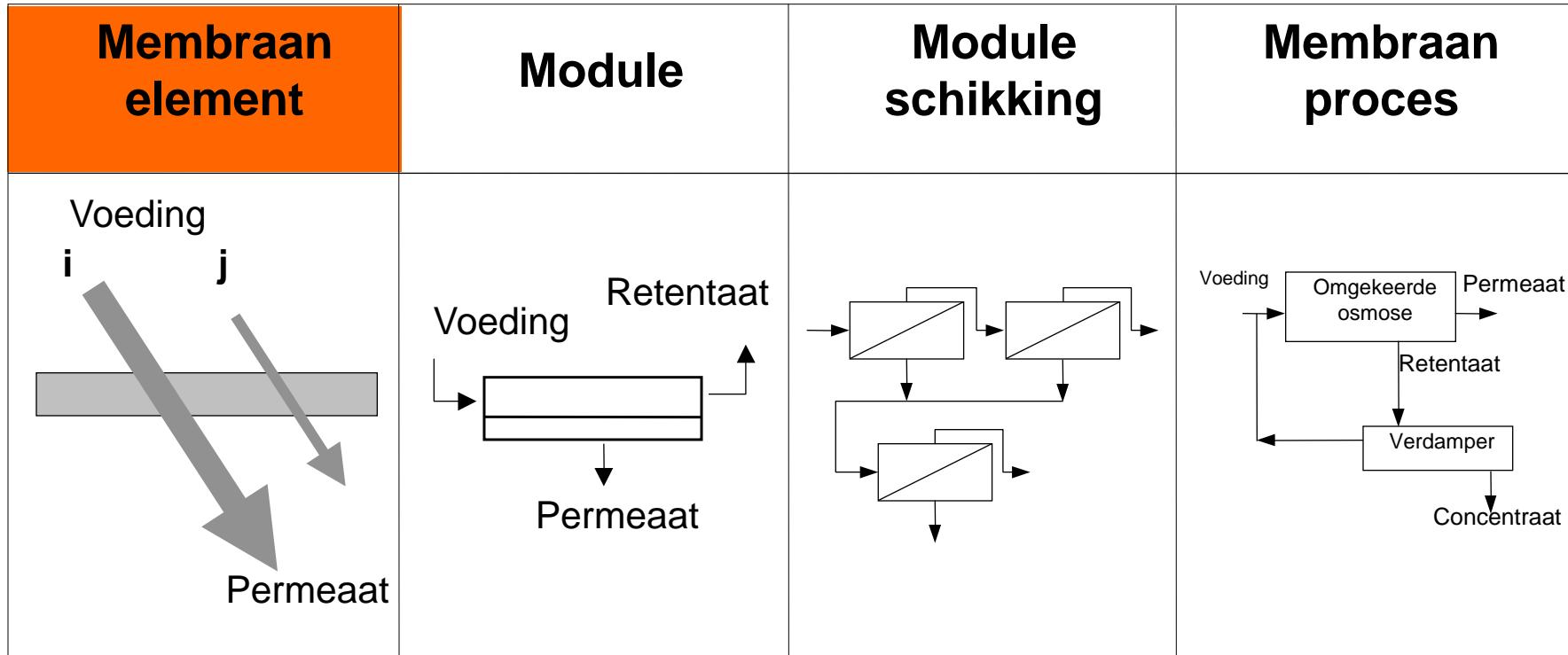
Metals - © 2009, VITO NV – All rights reserved

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PROCESONTWERP



R. Rautenbach, Membranverfahren, Grundlagen der Modul- und Anlagenauslegung

MEMBRAANKLASSIFICATIE

✓ Materiaal

- Organisch: polymeren
- Anorganisch: keramisch, koolstof, metaal, glas
- Mixed matrix, hybride: polymeer + anorganische vullers

✓ Morfologie

- Poreus vs dens
- Homogeen vs heterogeen: alle poriën gelijk vs niet gelijk
- Symmetrisch vs asymmetrisch: alle poriën aan één zijde groter dan aan de andere

✓ Poriegrootte

- MF, UF, NF, RO

✓ Affiniteit

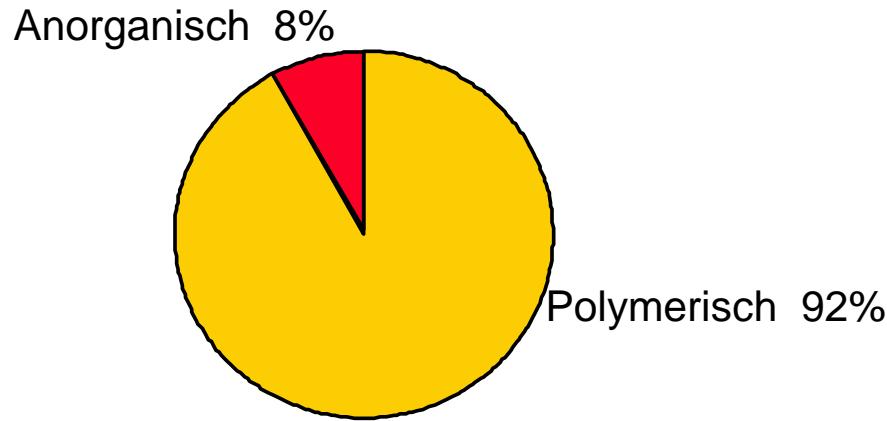
- Hydrofiliciteit/hydrofobiciteit
- Lading
- Functionele groepen

✓ Uitvoeringsvorm

- Vlakke membranen: plate-and-frame, spiraalgewonden module
- Tubulaire membranen: tubulaire en capillaire modules, holle vezels

MEMBRAANMATERIALEN

Verdeling West-Europese markt naar type membraanmateriaal



Membraansynthesetekniken

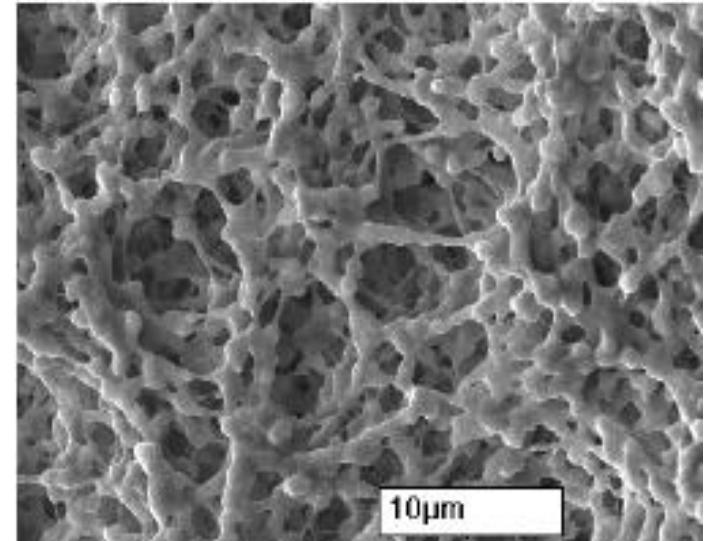
- ✓ Fase-inversie
- ✓ (Stretching)
- ✓ (Track-etching)
- ✓ Coating
- ✓ (Sol-gel synthese)

HOE MAAK JE EEN MEMBRAAN?

1. Fase – inversie techniek

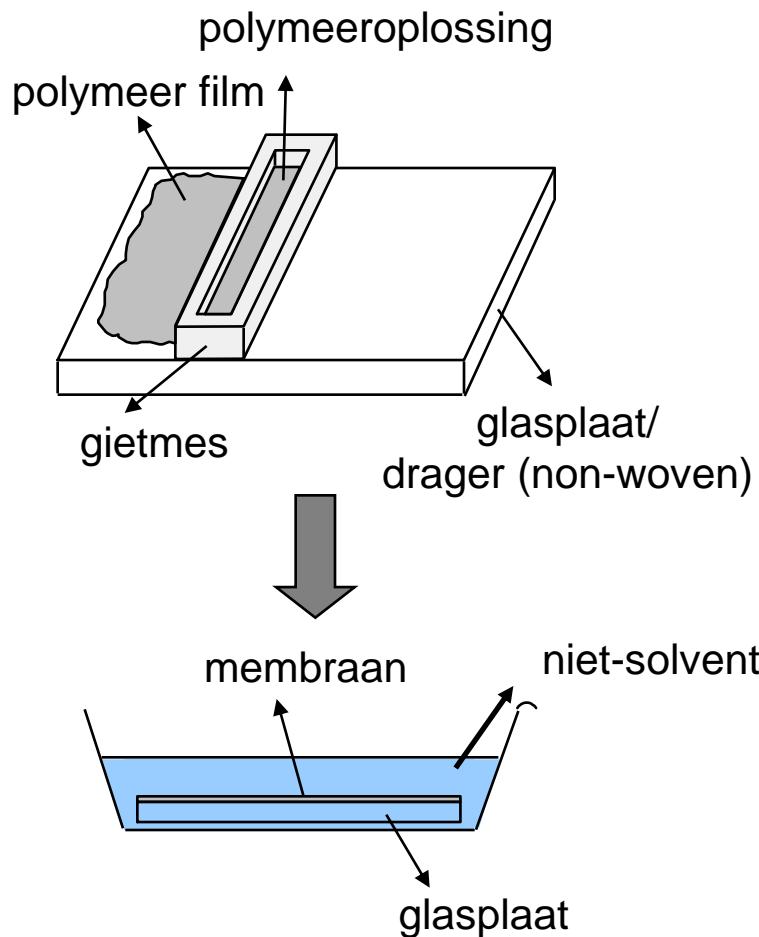
- ✓ Eenvoudig, veelzijdig proces
- ✓ Gecontroleerd door thermodynamische en kinetische parameters
- ✓ Geïnduceerd door niet-solvent, damp of temperatuur
- ✓ Grote variatie aan membraan morfologieën

→ *Meest gebruikte, flexibele techniek*

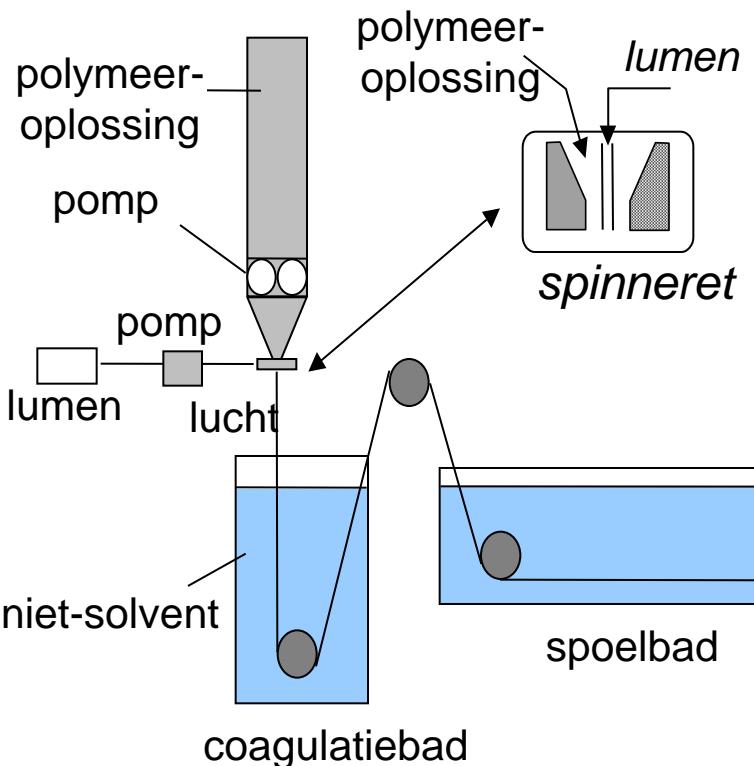


MF fase-inversie membraan

GIETEN VAN VLAKKE MEMBRANEN

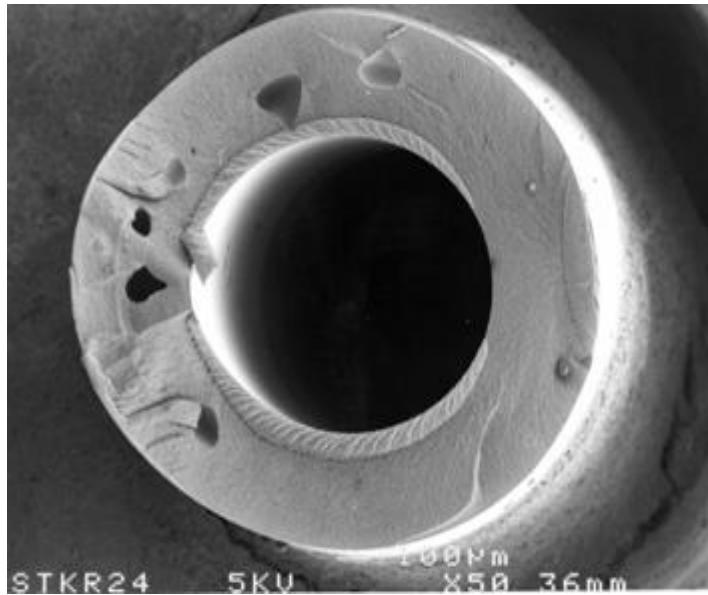


SPINNEN VAN CAPILLAIRE MEMBRANEN

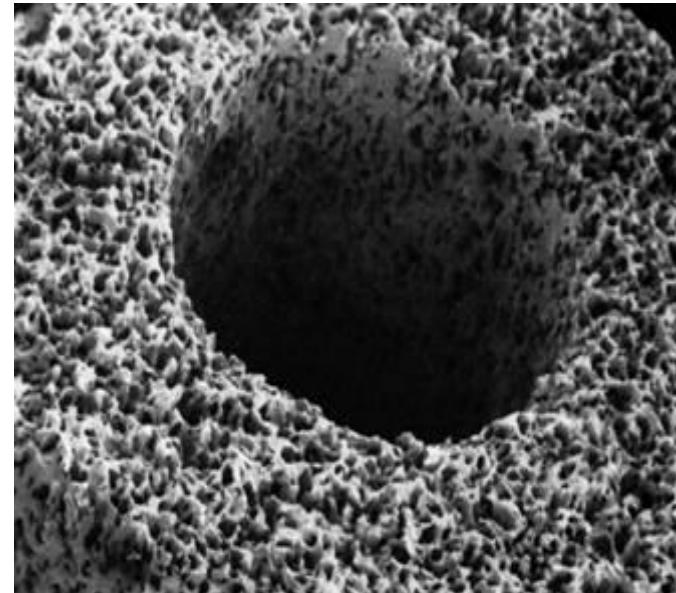


SYMMETRISCHE MEMBRAANSTRUCTUREN

Poreuze structuur, meestal MF



Polymeer holle vezel membraan (Aquasource)

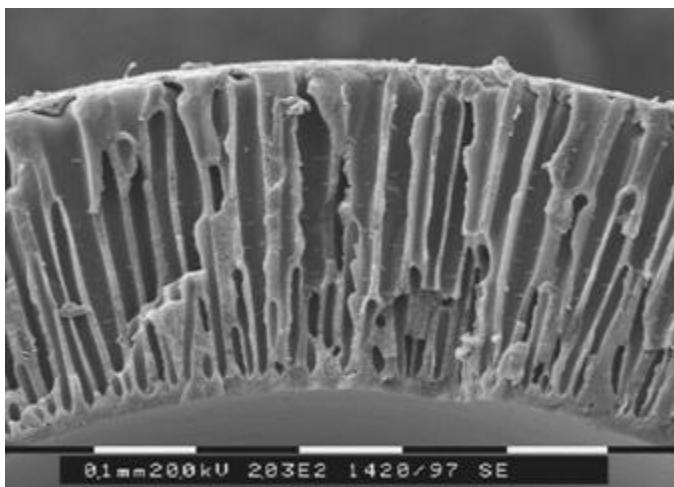


Glazen holle vezel membraan (Schott)

ASYMMETRISCHE MEMBRAANSTRUCTUREN

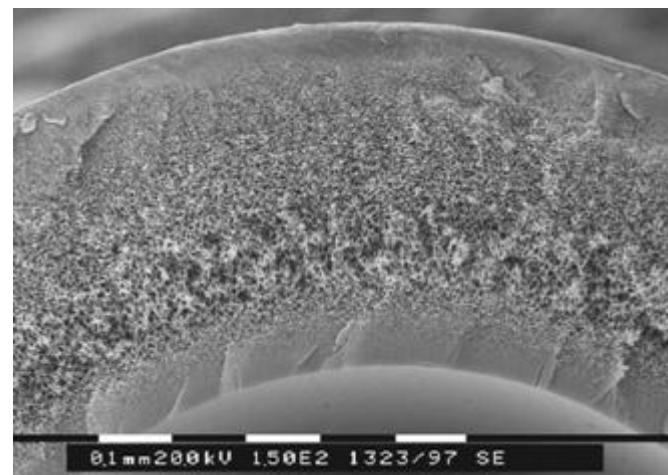
alle dichtere membranen (UF, NF, RO)

ASYMMETRISCH, vingerstructuur



Polymeer UF membraan (Koch)

ASYMMETRISCH, sponsstructuur



Polymeer UF membraan (X-Flow)

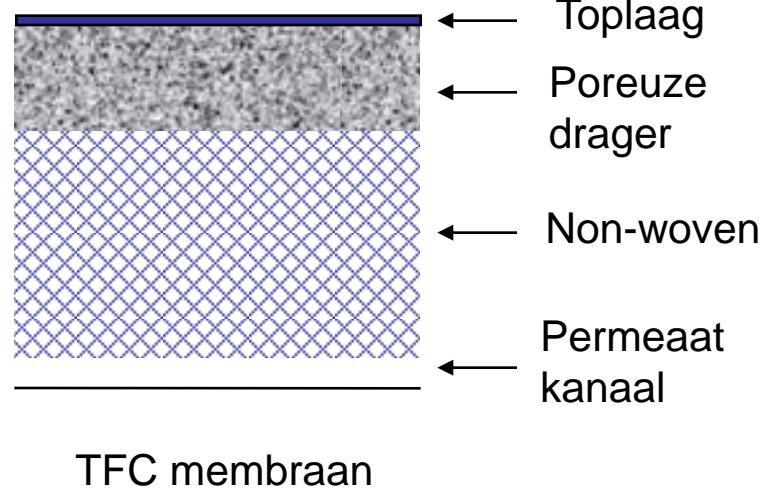
→ Combinatie hoge flux en selectiviteit

HOE MAAK JE EEN MEMBRAAN?

4. Coating techniek

- ✓ Drager = UF membraan
- ✓ Aanbrengen van dunne, selectieve toplaag
- ✓ Procedure: dip-coating, plasma polymerisatie, interfaciale polymerisatie (2 verschillende monomeren reagen aan een interfase), spin coating...
- ✓ Bruikbaar als NF/RO membraan

➔ *Thin Film Composite (TFC) membranen*



MEMBRAANMATERIALEN

✓ MF

- Hydrofobe polymeren: PVDF, PP, PTFE
- Synthese via stretching of fase-inversie
- Goede chemische stabiliteit
- Gevoelig aan fouling

✓ UF

- 'Hydrofiel': cellulose ester, gemodificeerd PSf/PES, PI, PAN, PEEK
- Synthese via fase-inversie

✓ NF/RO

- 'Klassiek' asymmetrische CA of TFC membranen (PA toplaag op PSf drager)
- NF: polybenzimidazolen, polyamidehydrazide, polyimiden
- Toplaag evt. geladen: electrostatische repulsie



Polymeerselectie: Afweging tussen stabiliteit en verwerkbaarheid

POLYMEREN VOOR MF/UF

Polymeer	Verwerkbaarheid	Chemische stabiliteit	Warmtebestendigheid
Polysulfon (UDEL)	Goed	Middelmatig	Middelmatig
Polyethersulfon (VICTREX)	Goed	Middelmatig	Middelmatig
Polytetrafluoroethyleen (Teflon)	Slecht	Uitstekend	Uitstekend
Polyetherketon	Slecht	Uitstekend	Uitstekend
Polyetheretherketon	Slecht	Uitstekend	Uitstekend
Polyimide	Goed	Uitstekend	Uitstekend
Polyacrylonitril	Goed	Middelmatig	Middelmatig

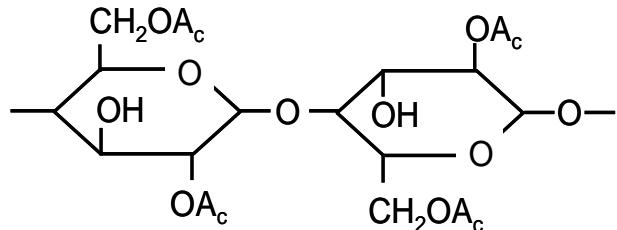
POLYMEERMODIFICATIE

DOEL: verhoging flux en verminderen fouling van hydrofobe membranen

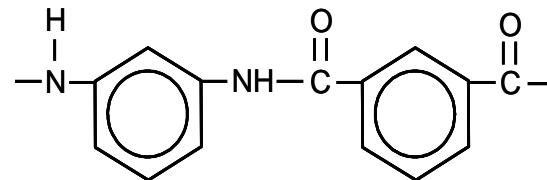
- ✓ Inbouw hydrofiele groepen in polymeer vóór verwerking
 - e.g. sulfonering van PSf
- ✓ Toevoeging tweede momomeer vóór verwerking
 - e.g. PAN + methylmethacrylaat
- ✓ Blending: toevoeging tweede polymeer voor gieten
 - e.g. PES/PVP
- ✓ Oppervlaktemodificatie
 - e.g. coating PVDF met vinylacetaat-vinylalcohol copolymeer, nitrering van PA en PSf

POLYMEREN VOOR NF/RO

Cellulose acetate (CA)



Polyamide (PA)



- 'Integrally skinned' asymmetrische membranen
- Oppervlak niet geladen: minder fouling
- Glad oppervlak: minder fouling
- Matige pH and T stabiliteit
- Gevoelig aan biodegradatie
- Matig bestand tegen chloor

- TFC membranen: PA/PSf
- Variabele oppervlaktelading
- Hogere flux, minder energie vereist
- Hogere pH and temperatuurstabiliteit
- Gevoeliger aan biofouling
- Zeer gevoelig aan vrije chloor!

NIET-POLYMERISCHE MEMBRANEN

Keramische membranen: gelaagde structuur

✓ Drager

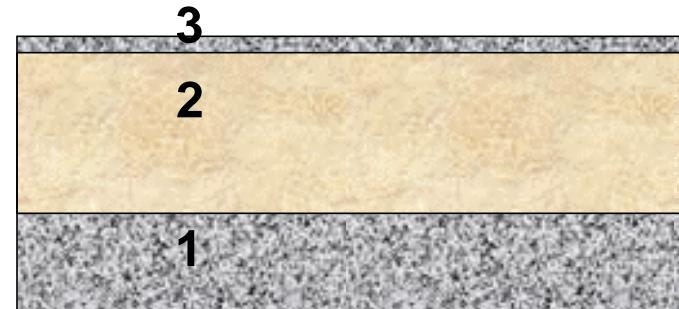
- Poriën > 50nm
- Al_2O_3

✓ Mesoporeuze tussenlaag

- 2 nm < poriën < 50 nm
- γ - Al_2O_3

✓ Toplaag

- Microporeus (2 nm): gepilaarde kleien, SiO_2
- Dicht: protongeleiders (CaZrO_3), O₂-geleiders (LaSrO_3)

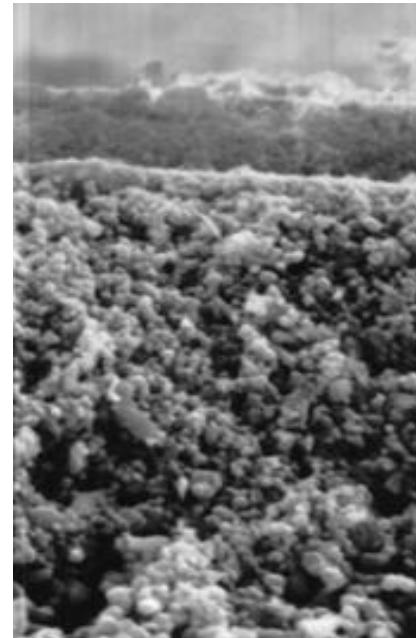


Voordelen: hoge stabiliteit (chemisch, thermisch, mechanisch)

Nadeel: hoge kost, brosheid

KERAMISCHE MEMBRAANSTRUCTUREN

Asymmetrische meerlagenstructuur



Keramische UF/NF membranen

TOEPASSINGSGEBIED MEMBRAANMATERIALEN

Membraanmateriaal (Scheidende laag)	MF	UF	NF	GS
Al_2O_3	X	X	X	
ZrO_2	X	X	X	
SiC	X			
Koolstof	X	X		X
TiO_2	X	X	X	
SiO_2				X
Zeolieten				X
Micro-, mesoporeus glas		X		X
Pd-legering op keramische drager				X
Pt/Pd op Al_2O_3				X
Verschillende metaalsoorten	X			

MEMBRAANSELECTIE

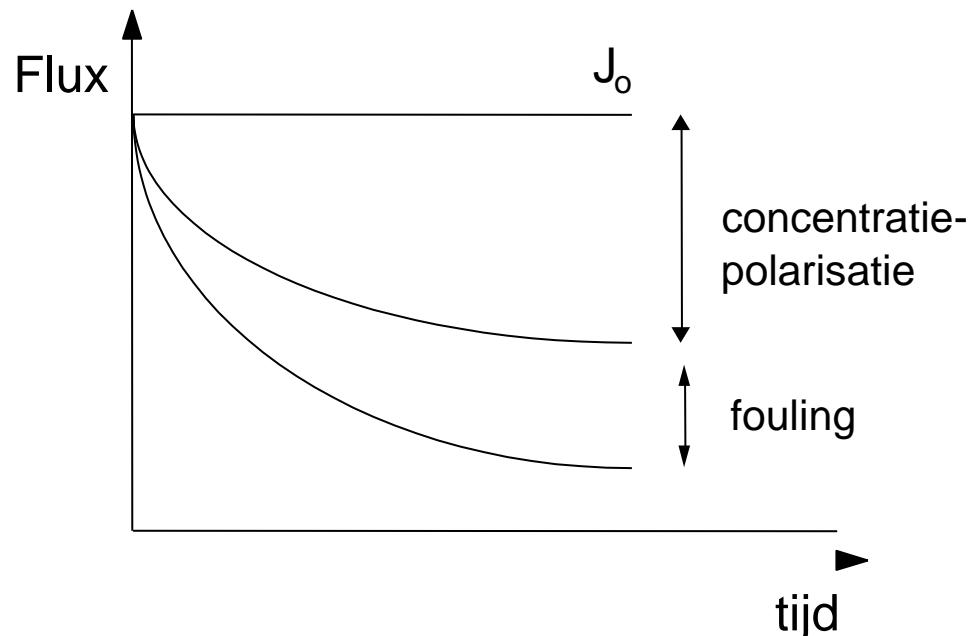
✓ Materiaalkeuze

- Voeding specificaties (pH, T, samenstelling)
- Hydrofiel/hydrofoob
- Reinigingsmethode
- Kostprijs
- Lading

✓ Membraaneigenschappen

- Flux
- Retentie eigenschappen
- Vervuiling

✓ Moduleconfiguratie



MEMBRAANKARAKTERISERING

✓ **Doel:**

Relatie tussen structurele membraaneigenschappen en scheidingsgedrag

✓ **Vraag:**

Welke informatie is nodig om de prestaties van een membraan in een bepaalde toepassing te voorspellen?

Intrinsieke membraaneigenschappen ≠ Actuele membraaneigenschappen

→ B.v. reële flux UF < 10 % van zuiverwaterflux

Oorzaak: Concentratie-polarisatie en fouling

PORIËNBEREIK

- ✓ Membranen reiken van poreus tot niet-poreus, afhankelijk van het scheidingsproces
 - ➔ verschillende karakterisatietechnieken nodig
- ✓ Drie types membranen:
 - *Poreus* (MF, UF): poriën worden gekarakteriseerd (zeefwerking)
 - *Niet-poreus* (RO, PV, GS): materiaal bepaalt performantie (affiniteit solvent/opgeloste stof voor membraan)
 - *Geladen* (ED, NF): extra scheidingsparameter (electrostatische repulsie)

POREUZE MEMBRANEN

Twee types karakteriseringstechnieken

✓ Statische karakteriseringstechnieken

- Microscopie en beeldanalyse
- Kwik porosimetrie
- Gas adsorptie/desorptie

STRUCTUUR

✓ Dynamische karakteriseringstechnieken

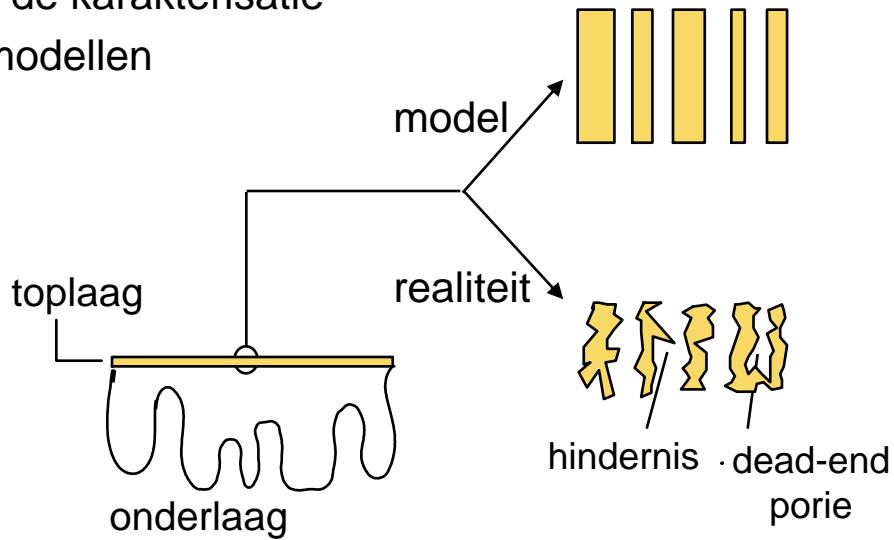
- Bubble point
- Permeabiliteit/flux metingen
- Retentie metingen (cut-off)

TRANSPORT

→ Moeilijk met mekaar in verband te brengen !

POREUZE MEMBRANEN

- ✓ Moeilijke interpretatie omwille van:
 - poriën hebben geen uniforme verdeling
 - storingsfenomen gedurende de karakterisatie
 - gebruik van té eenvoudige modellen

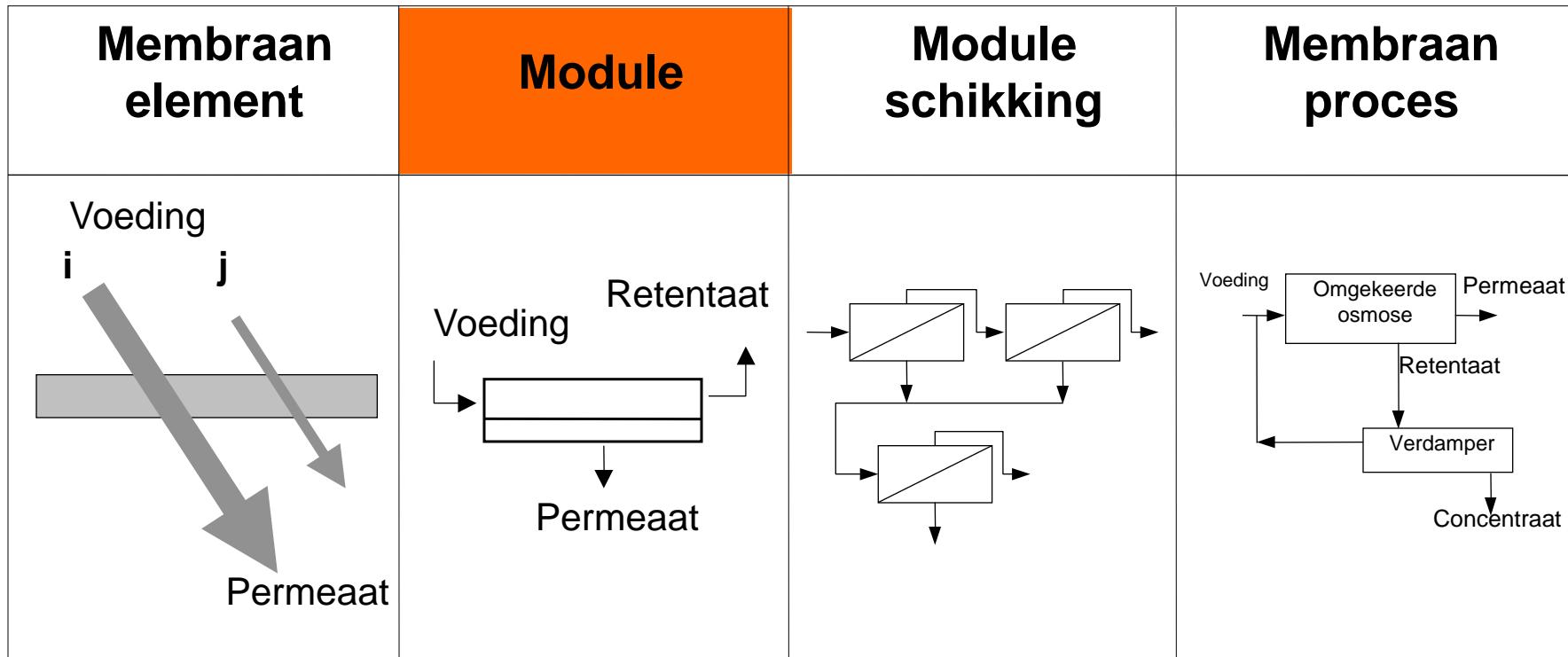


→ Combinatie verschillende technieken nodig!

NIET-POREUZE MEMBRANEN

- ✓ Scheiding op moleculair niveau: oplos/diffusie mechanisme
- ✓ **Belangrijke parameters:**
 - Chemische aard polymeer
 - Polymeermorfologie
 - Interactie polymeer en permeaat
- ✓ **Karakteriseringstechnieken:**
 - Permeabiliteit (gas en vloeistof)
 - Meting fysische polymeereigenschappen (T_g , kristalliniteit, dichtheid)
 - Plasma etching (dikte toplaag)
 - Oppervlakte-analyse methodes

PROCESONTWERP

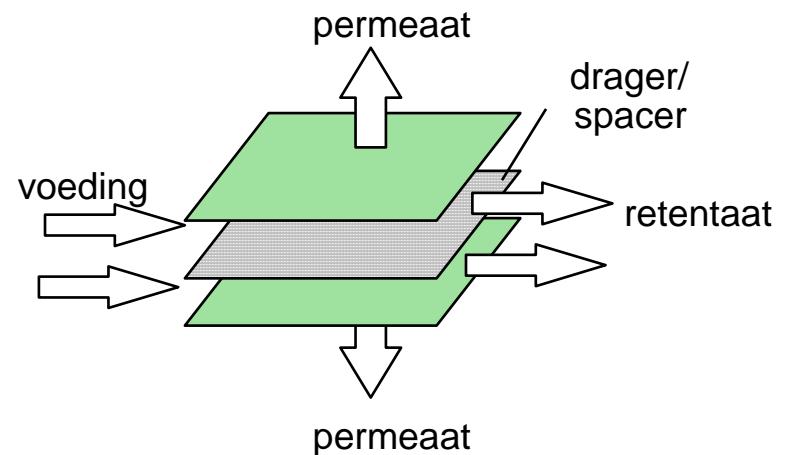


R. Rautenbach, Membranverfahren, Grundlagen der Modul- und Anlagenauslegung

MODULE / UITVOERINGSVORM

Vlakke membranen

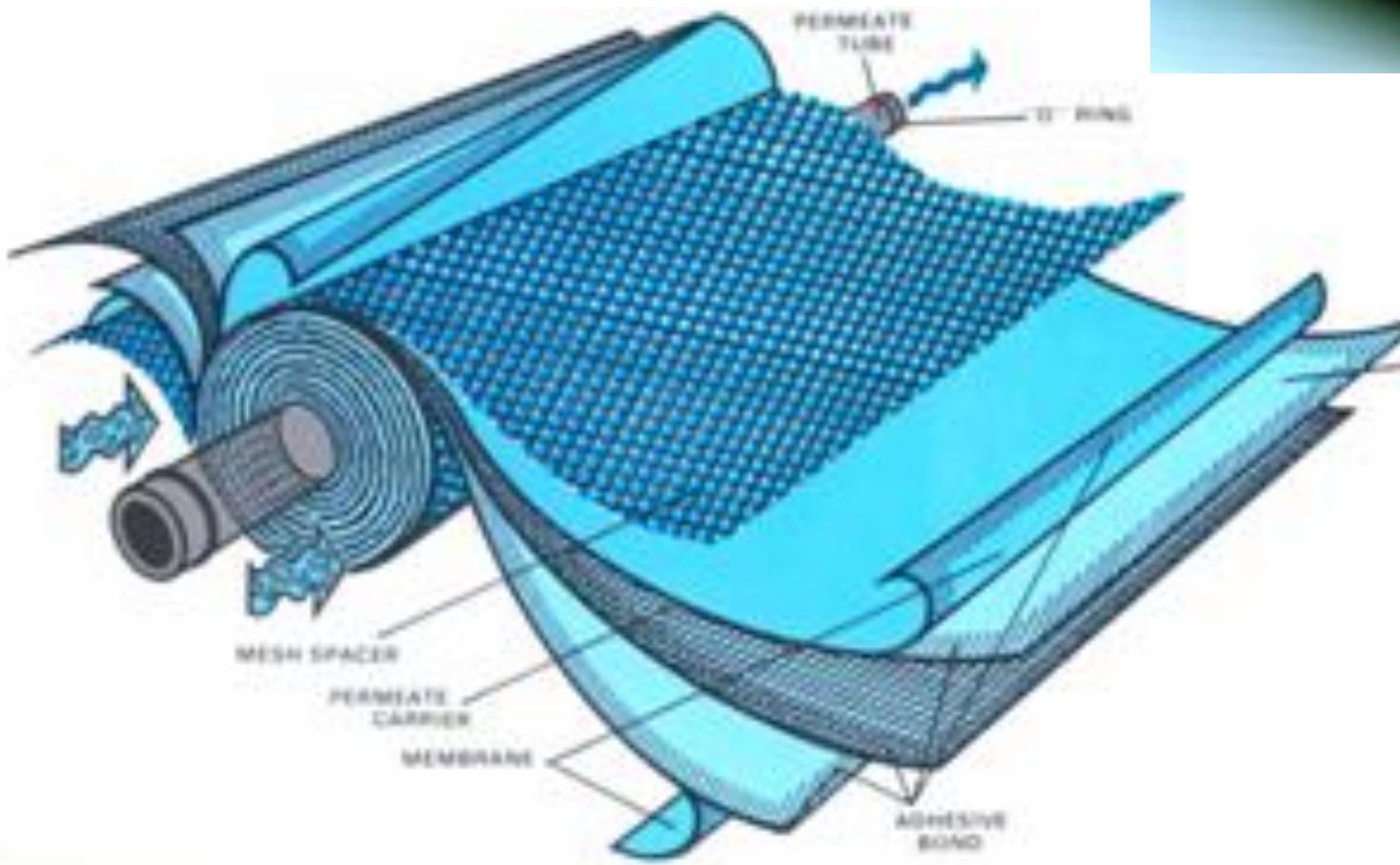
- ✓ Plate-and-frame module



MODULE / UITVOERINGSVORM

Vlakke membranen

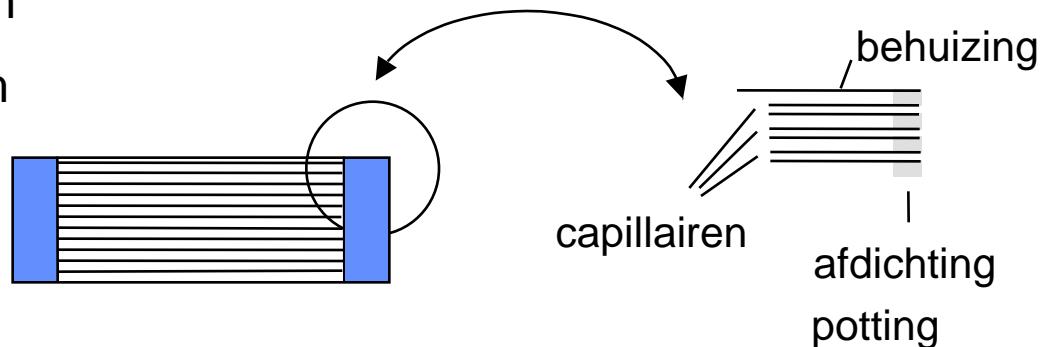
- ✓ Spiraalgewonden module (SW)



MODULE / UITVOERINGSVORM

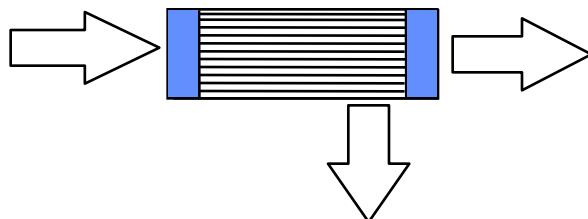
Tubulaire membranen

- ✓ Tubulaire module: $d > 5 \text{ mm}$, + drager
- ✓ Capillaire module: $d: 0.5 - 5 \text{ mm}$
- ✓ Holle vezel module: $d < 0.5 \text{ mm}$

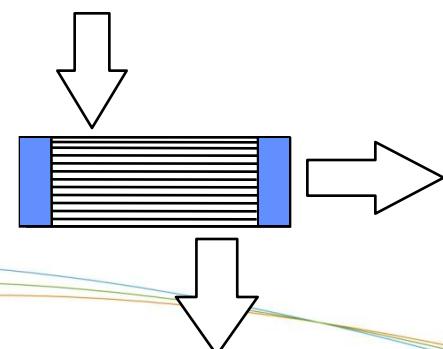


2 types

“inside out”



“outside in”



MODULES: EIGENSCHAPPEN

Eigenschap	Tubulair	Plate-and frame	Spiraal-gewonden	Capillair	Holle vezel
Pakkingsdichtheid	laag	-----→	zeer hoog		
Investering	(hoog)	-----→	laag		
Vervuiling	laag	-----→	zeer hoog		
Reiniging	goed	-----→	slecht		
Operationele kost	hoog	-----→	laag		
Membraanvervanging	ja/neen	ja	neen	neen	neen

MODULES: PAKKINGSDICHTHEID

Configuratie	Opp./volume (m ² /m ³)
Tubulair r = 5 mm	100 - 500
Capillair r = 0.5 mm	500 - 4000
Holle vezel r = 0.05 mm	4000 - 30000
Plate-and-frame	100 - 200
Spiraalgewonden	700 - 1000

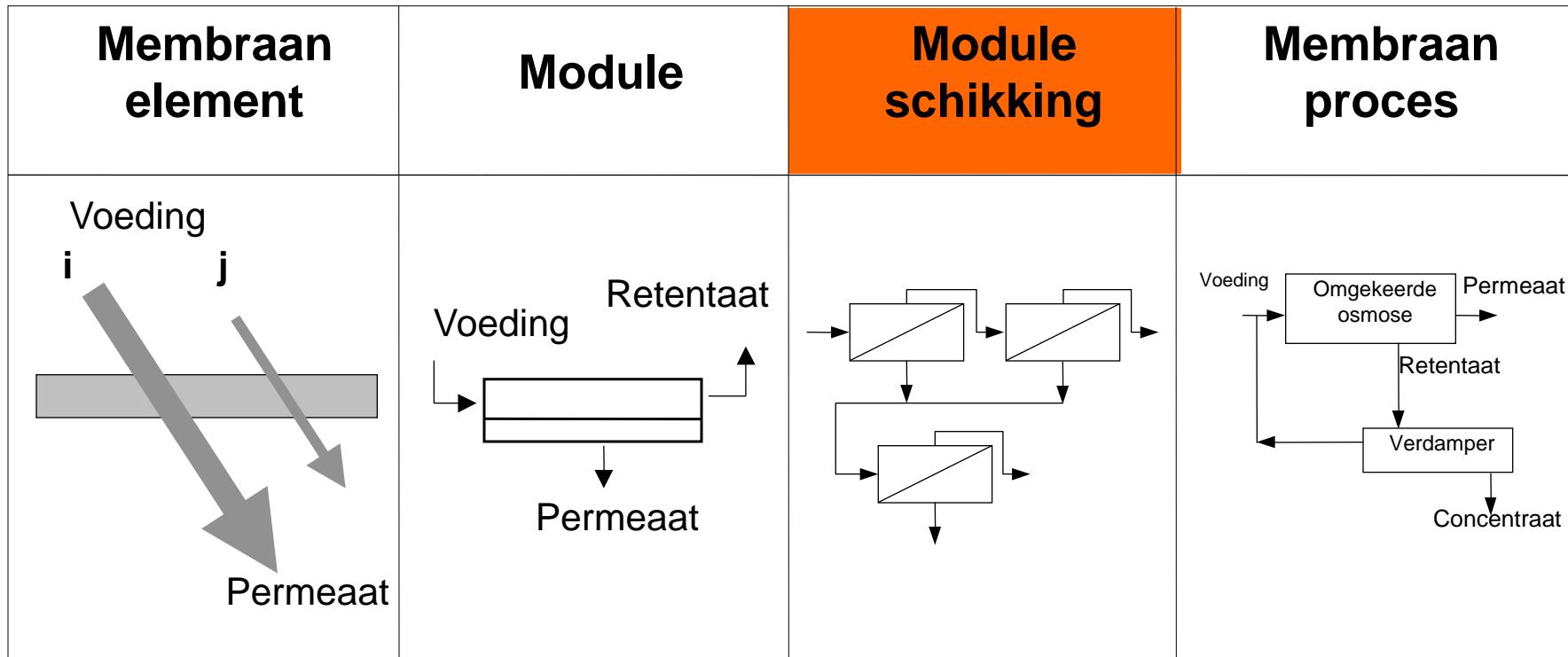
MODULES: KEUZE I.F.V. TOEPASSING

Membraanproces	Tubulair	Plate-and frame	Spiraal-gewonden	Capillair	Holle vezel
MF	++	+		+	+
UF	++	+	+	+	++
NF	++	+	++	++	+
RO	(+)	(+)	++		+
GS			++	+	++
PV		++	+	+	

+ mogelijke configuratie

++ zeer geschikte configuratie

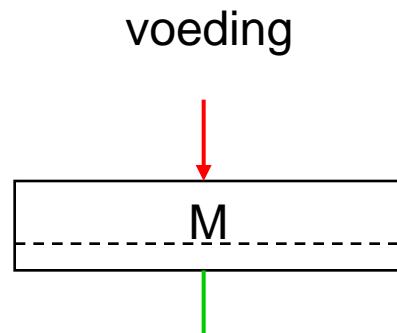
PROCESONTWERP



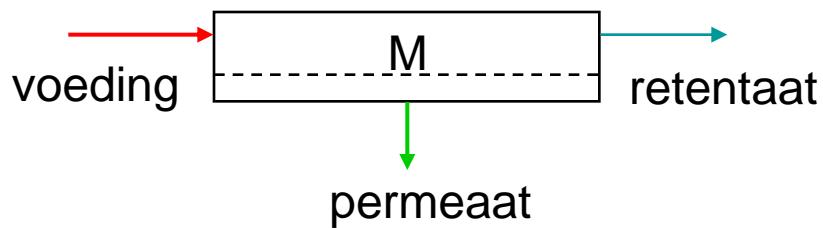
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BEDRIJFSVOERING

✓ Dead-end



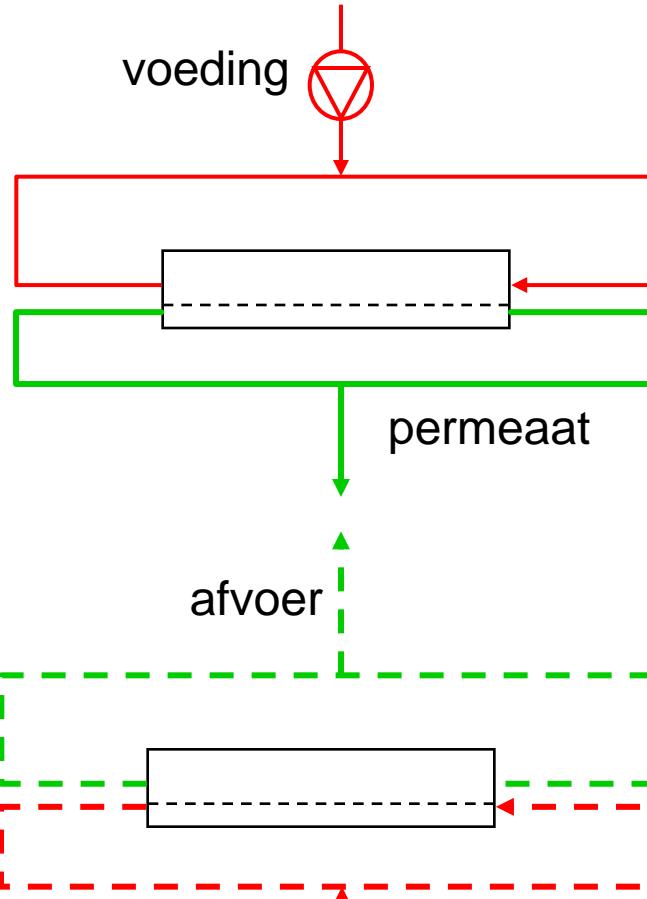
✓ Cross-flow



BEDRIJFSVOERING

✓ Semi dead-end

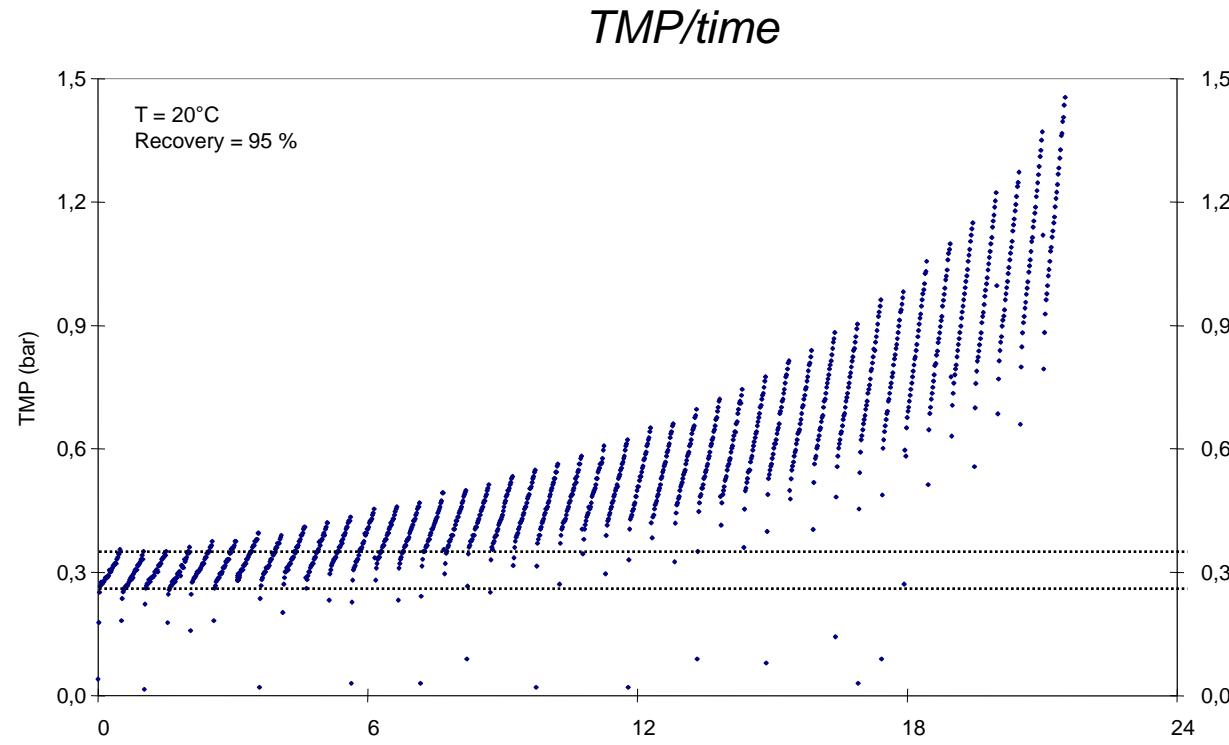
1. Filtratie
2. Terugspoeling of back-flush



PROCESSING

■ Typical parameters of SDE-O

Alterning filtration and washing cycles (backflush/BF)



PROCESSING

- Calculation of net flux/recovery and waste water volume of SDE-O

Example of alternating filtration- and washing cycles for installation with **1200 m²**

	Flux (l/hm ²)	Duration (min)	Volume (l)
Filtration	80	30	48.000
Washing	250	0.75	3.750
Cycle time			Net perm. production

→ Net flux:

$$\frac{44.250 * 60}{1200 * 30.75} = 72 \text{ l/hm}^2$$

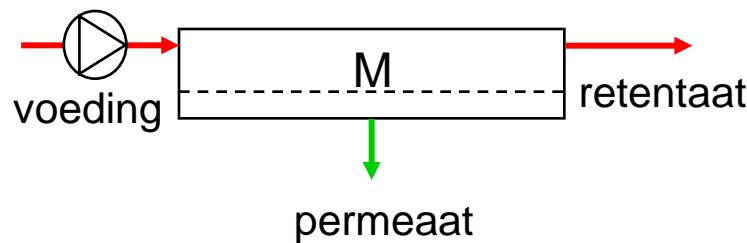
→ Water recovery:

$$\frac{44.250 * 100}{48.000} = 92.2 \%$$

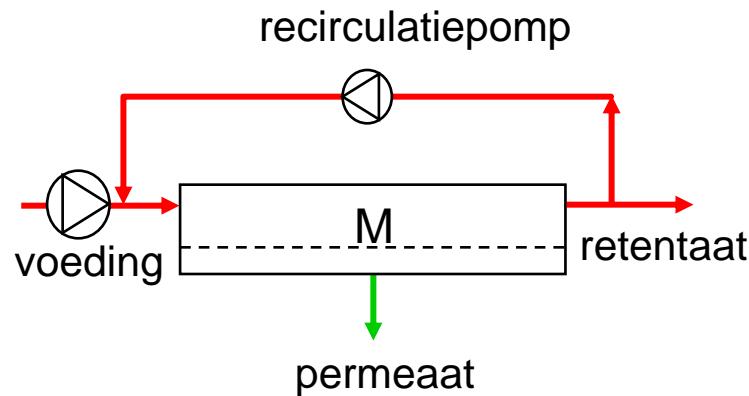
→ Volume of waste water:
3.750 l = 7.8 %

BEDRIJFSVOERING

- ✓ **Enkele doorstroming (“single pass”)**

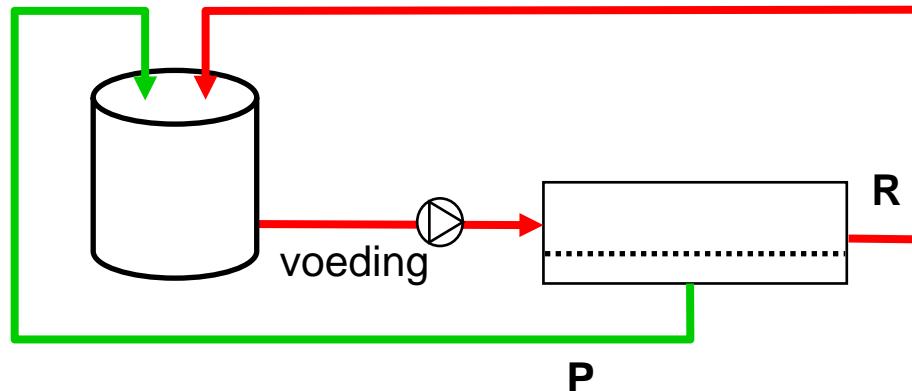


- ✓ **Recirculatie**

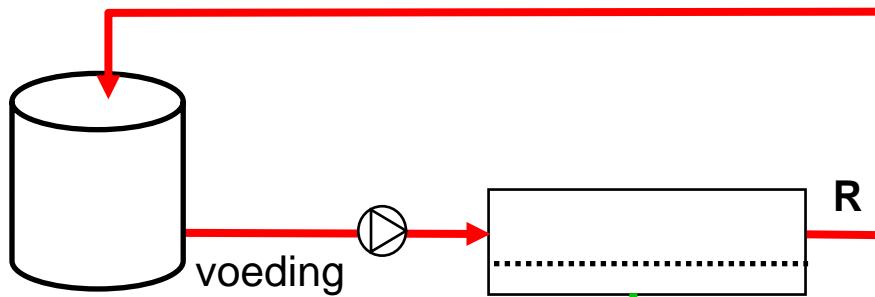


BEDRIJFSVOERING

- ✓ **Volledige recycling**

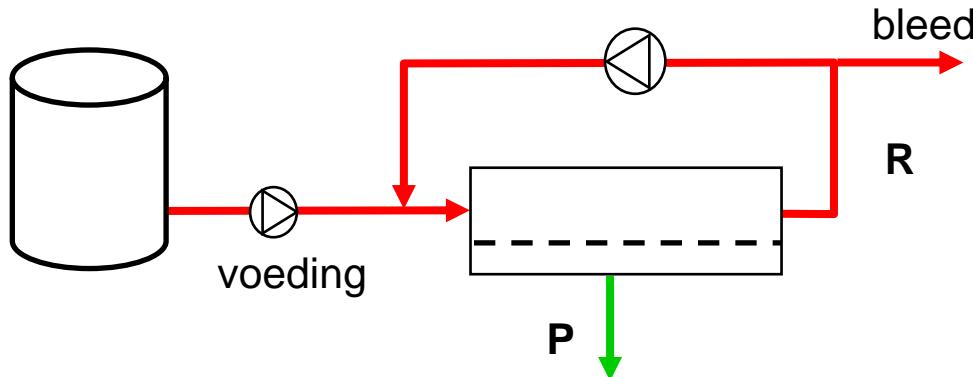


- ✓ **Batch concentratie**

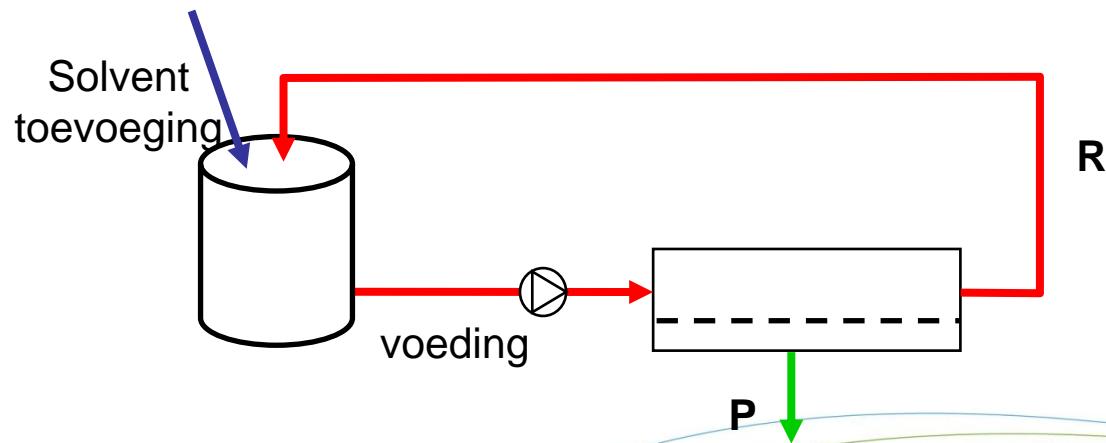


BEDRIJFSVOERING

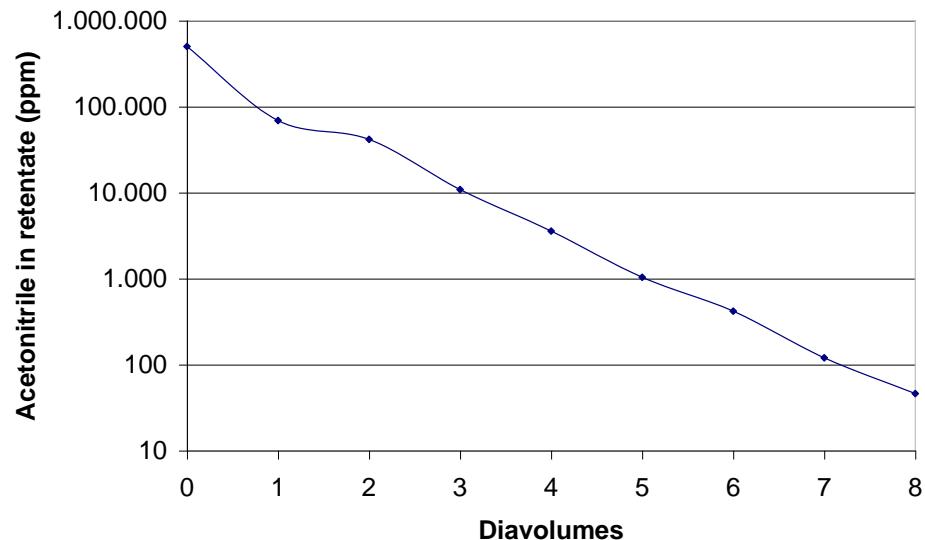
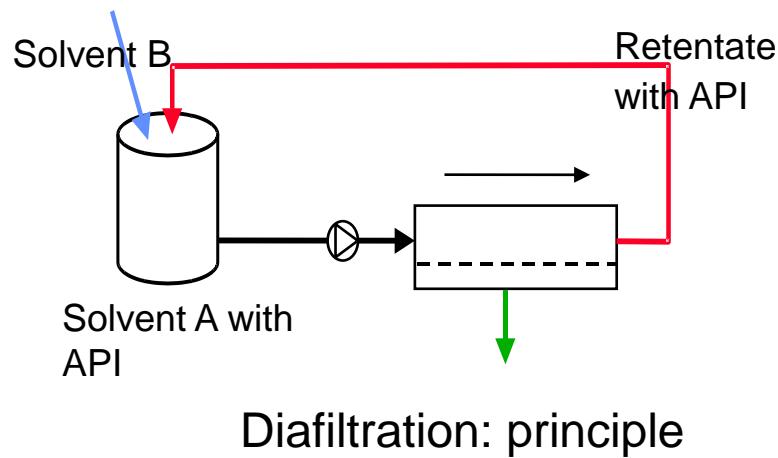
✓ Feed and bleed



✓ Diafiltratie

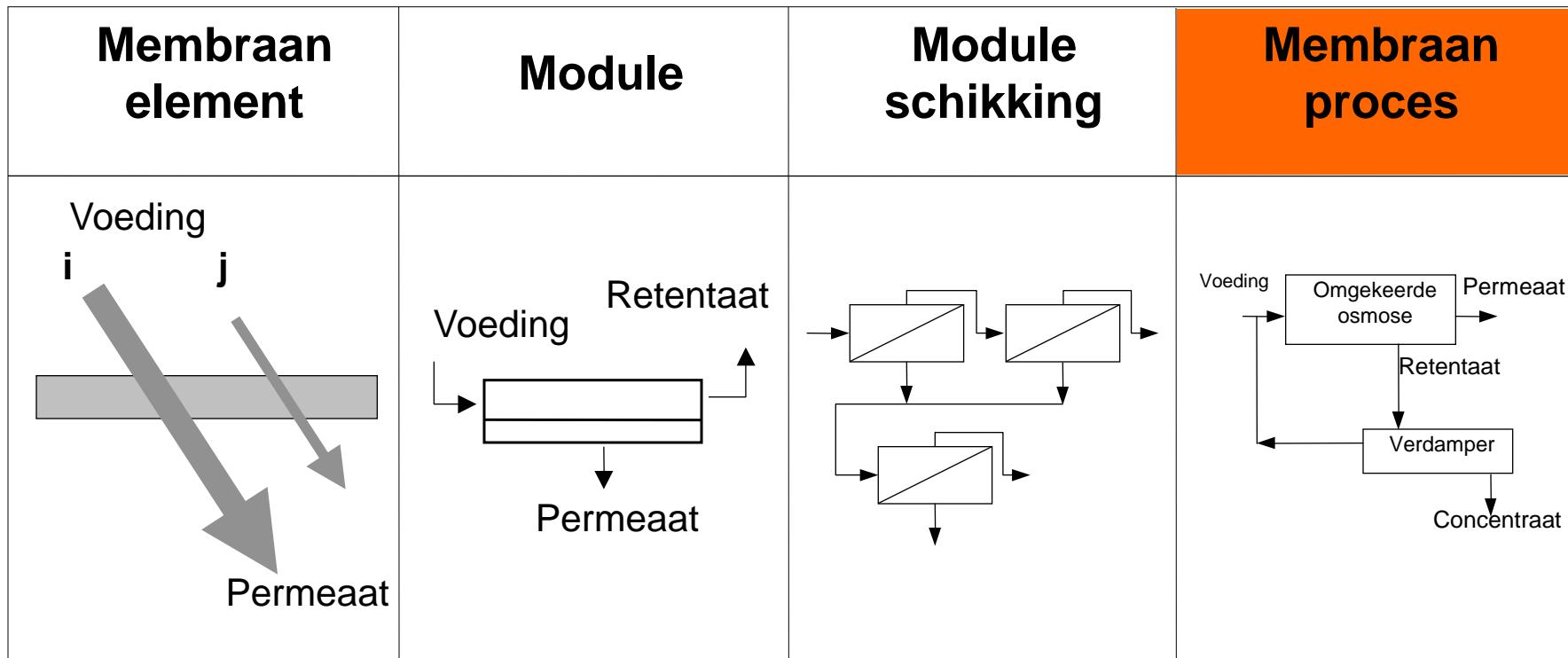


Classical example removal of acetonitrile by diafiltration



Evolution of acetonitrile concentration
in the retentate

PROCESONTWERP



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ONTWERP INSTALLATIE

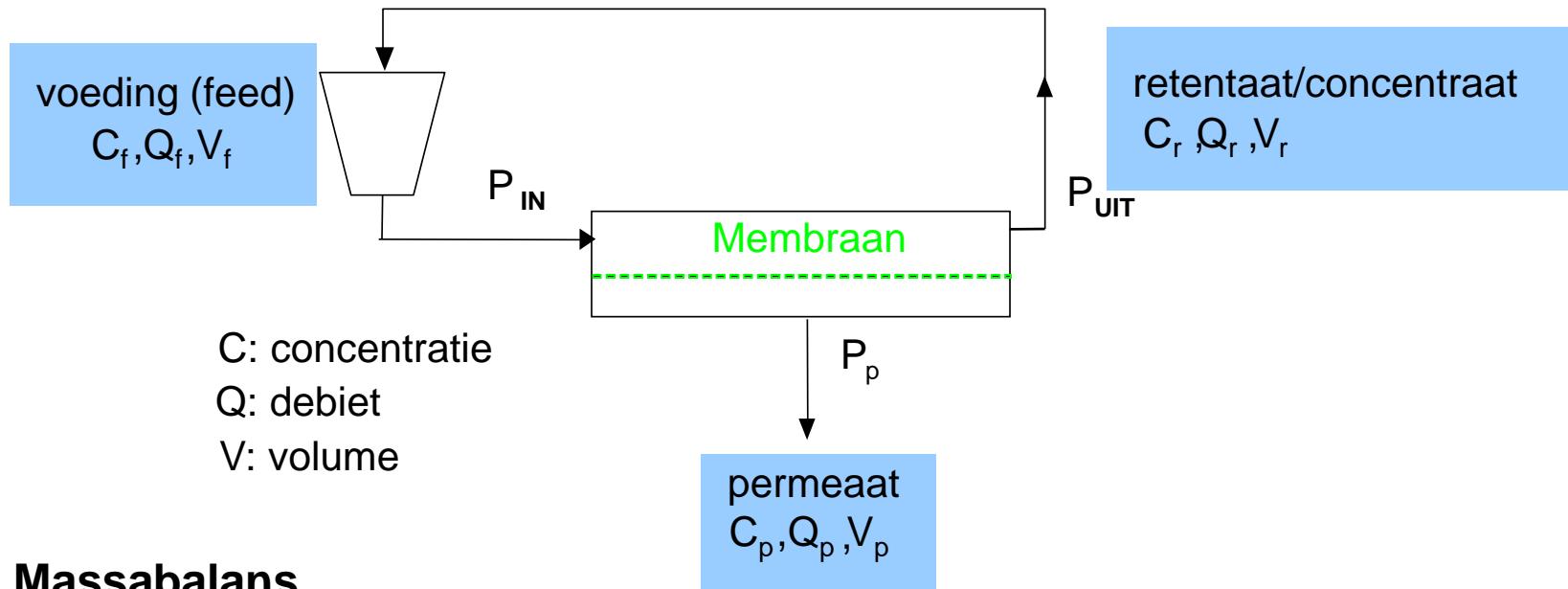
- ✓ **Targets:** debiet en/of permeaatkwaliteit
- ✓ **Membraankeuze:**
 - Scheidingskarakteristieken
 - Kostprijs: 15-40% van installatiekosten
 - Interactie membraanmateriaal - voeding
 - Stabiliteit: T, chemisch, pH → reinigingsmogelijkheden
 - berekening benodigd membraanoppervlak
- ✓ **Configureren van de installatie:**
 - Eén- of meertraps, serie of parallel, recirculatie...
 - Rekening houden met specificaties van membraanmodules

OUTLINE

1. Inleiding
2. Membranen / Modules en ontwerp
3. Procesparameters en fouling
4. Overzicht waterige membraantoepassingen
5. Case-studies membranen voor waterzuiveringstoepassingen
6. Overzicht en case-studies niet-waterige membraantoepassingen
7. Conclusies

TERMINOLOGIE

✓ Membraanproces



✓ Massabalans

- voor één component

$$Q_f \times C_f = Q_r \times C_r + Q_p \times C_p \text{ (massa/t)}$$

$$V_f \times C_f = V_r \times C_r + V_p \times C_p \text{ (massa)} \quad \rightarrow \text{ogenblikkelijke balans}$$

- totaal $Q_f = Q_r + Q_p \text{ (massa/t)}$

$$V_f = V_r + V_p \text{ (volume !)} \quad \rightarrow \text{globale balans}$$

TERMINOLOGIE

Specifieke terminologie afhankelijk van type toepassing (2 soorten)

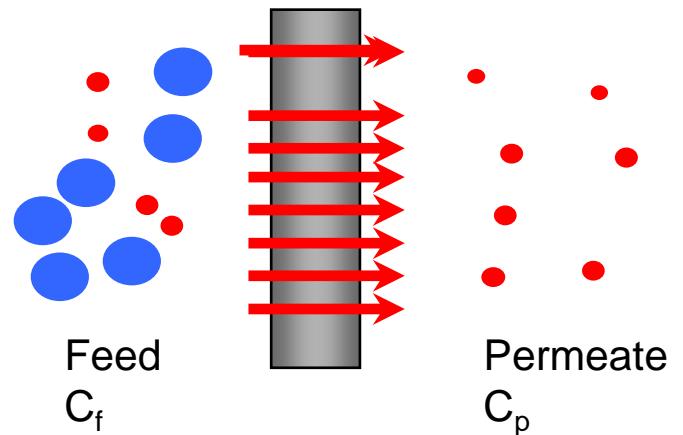
Retentaat/concentraat toepassingen

→ opconcentrering - terugwinning of recuperatie

Permeaat toepassingen

→ klaring/stabilisatie - zuivering - afvalwaterbehandeling

RETENTIE



Verdunde waterige oplossingen:

✓ **Retentie** van component i: $R_i = \frac{C_{v,i} - C_{p,i}}{C_{v,i}}$

C_v = concentratie opgeloste stof in de voeding

C_p = concentratie opgeloste stof in het permeaat

✓ R varieert tussen 1 (ideaal membraan) en 0, of tussen 100 en 0 %

» **Selectiviteit: scheidingsfactor**

» $\alpha(a/b) = (Y_a/Y_b)/(X_a/X_b)$

» X: gewichtsfractie in feed; Y: gewichtsfractie in permeaat

TERMINOLOGIE

Retentaat/concentraattoepassingen

- ✓ Volumetrische concentratiefactor (VCF)

$$VCF = V_f / V_r \text{ (batch)}$$

- ✓ Volume reductie (VR)

$$VR = Q_f / Q_r \text{ (continu)}$$

- ✓ Yield (Y, opbrengst/verlies aan product)

$$Y_c = \frac{\text{massa } C \text{ in retentaat}}{\text{massa } C \text{ in voeding}}$$

$$Y = \frac{C_R \times V_R}{C_f \times V_f} = (VCF)^{(R-1)}$$

TERMINOLOGIE

Retentaat/concentraattoepassingen

- ✓ Concentratie in retentaat (C_r)

$$C_r = C_f VCF^R$$

- ✓ Gemiddelde permeaatconcentratie (\bar{C}_p)

$$\bar{C}_p = C_f \frac{1 - VCF^{(R-1)}}{1 - VCF^{-1}}$$

TERMINOLOGIE

Permeaattoepassingen

- ✓ Recovery (S, %)

$$S = Q_p / Q_f$$

- ✓ Concentratie in retentaat (C_r)

$$C_r = C_f (1-S)^{1-R}$$

- ✓ Gemiddelde permeaatconcentratie (\bar{C}_p)

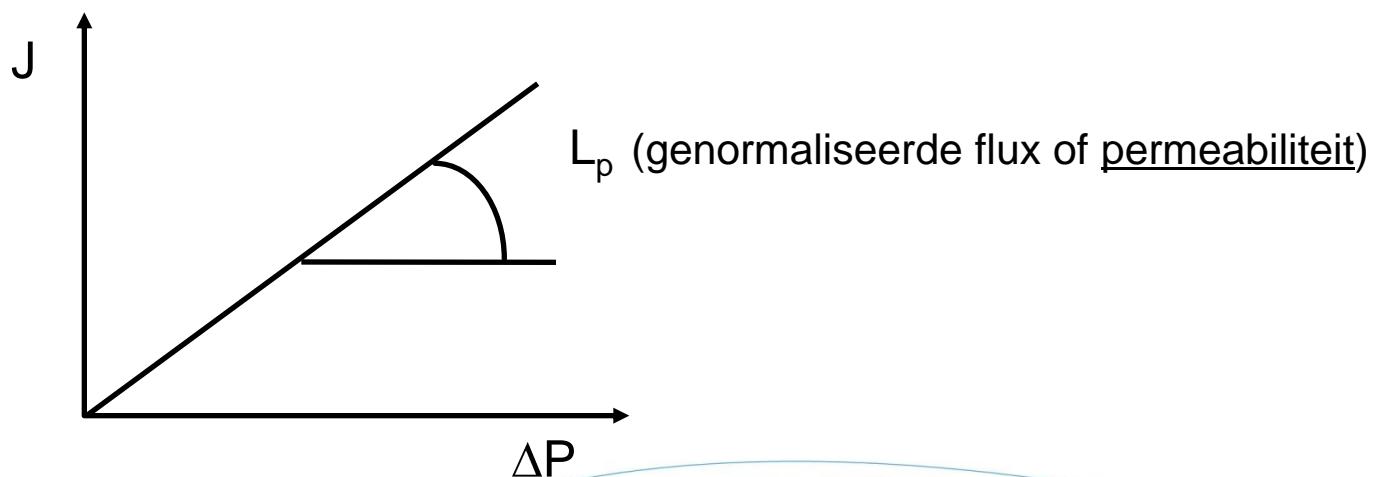
$$\bar{C}_p = \frac{C_f}{S} [1 - (1-S)^{1-R}]$$

FLUX/PERMEABILITEIT

- ✓ Definitie **flux** (J, meestal in l/hm²)

$$J = \frac{(hoeveelheid)}{(oppervlakte) \times (tijd)} = \frac{\text{drijvende kracht}}{\text{viscositeit} \cdot \text{totale weerstand}}$$

- ✓ Zuiver water : lineair verband flux-druk



SELECTIVITEIT: PRAKTIJK

- ✓ **MF:** opgegeven poriëngrootte is absoluut (100 % retentie)
Typische waarden: 0.2 µm, 0.45 µm
- ✓ **UF en NF:** conventie: begrip cut-off waarde o.w.v. poriëndistributie

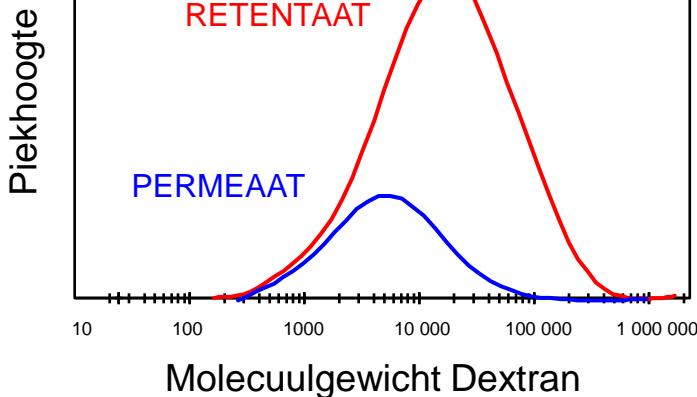
“Molecular weight cut-off” (MWCO) = molecuulgewicht dat voor 90 % door membraan wordt tegengehouden

- MWCO NF: tussen 150 -1.000 Da
- MWCO UF: tussen 1.000 -1.000.000 Da
- ✓ **RO en NF:** zoutretentie voor zowel mono- als bivalente ionen opgegeven
vb. RO: 98.5 % voor NaCl, 99.98 % voor Na₂SO₄

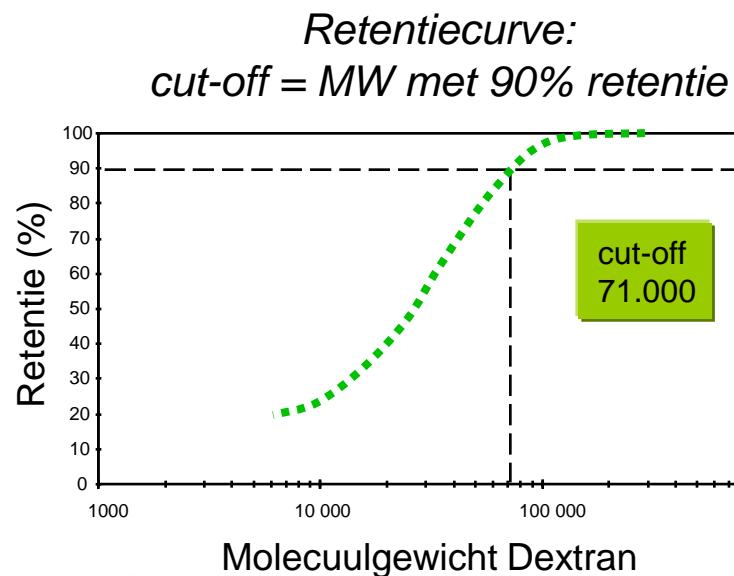
BEPALING MWCO

- ✓ Filtratie met mengsel van testmoleculen (Dextranen en/of PEG)
 - Instellen parameters om concentratie-polarisatie te minimaliseren
 - Staatnaam retentiaat en permeaat
- ✓ Analyse MW verdeling stalen

GPC chromatogram



$$R_i = \left(1 - \frac{C_{pi}}{C_{vi}}\right) \times 100\%$$

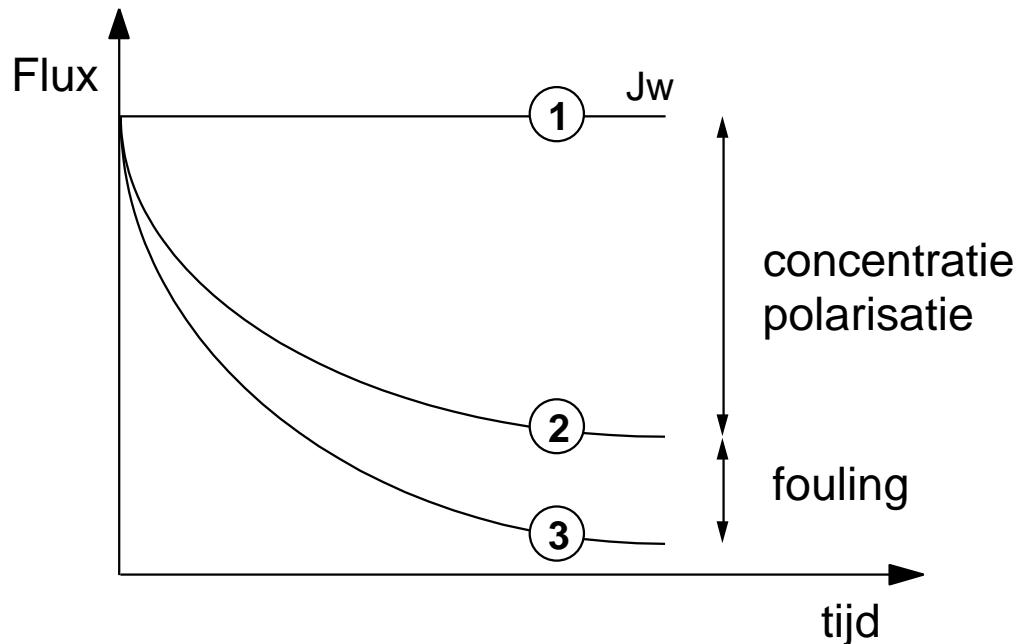


FLUXDALING

Praktijk: bij overgang van zuiver water naar proces/afval water

→ Fluxdaling:

- UF: tot 5% van initiële waterflux
- MF: tot 1% van initiële waterflux



$$\textcircled{1} \quad \text{flux} = \frac{\text{drijvende kracht}}{R_m}$$

$$\textcircled{2} \quad \text{flux} = \frac{\text{drijvende kracht}}{R_m + R_{cp}}$$

$$\textcircled{3} \quad \text{flux} = \frac{\text{drijvende kracht}}{R_m + R_{cp} + R_f}$$

WEERSTANDEN

✓ Theoretisch:

→ J i.f.v. ΔP geeft een rechte : $J = L_p \frac{\Delta P}{\Delta x}$

✓ Realiteit:

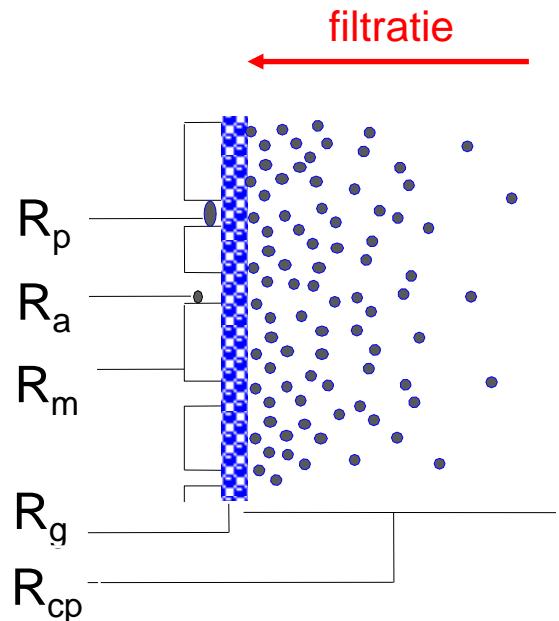
→ grensflux : $J \approx \frac{\Delta P}{R_{tot}}$

→ opbouw weerstanden aan membraanoppervlak

- membraan (m)
 - adsorptie (a)
 - porieblokkering (p)
 - gellaagvorming (g)
 - concentratiepolarisatie (cp)

} Fouling

} Concentratie
Polarisatie



OORZAKEN FLUXDALING

✓ Concentratie-polarisatie

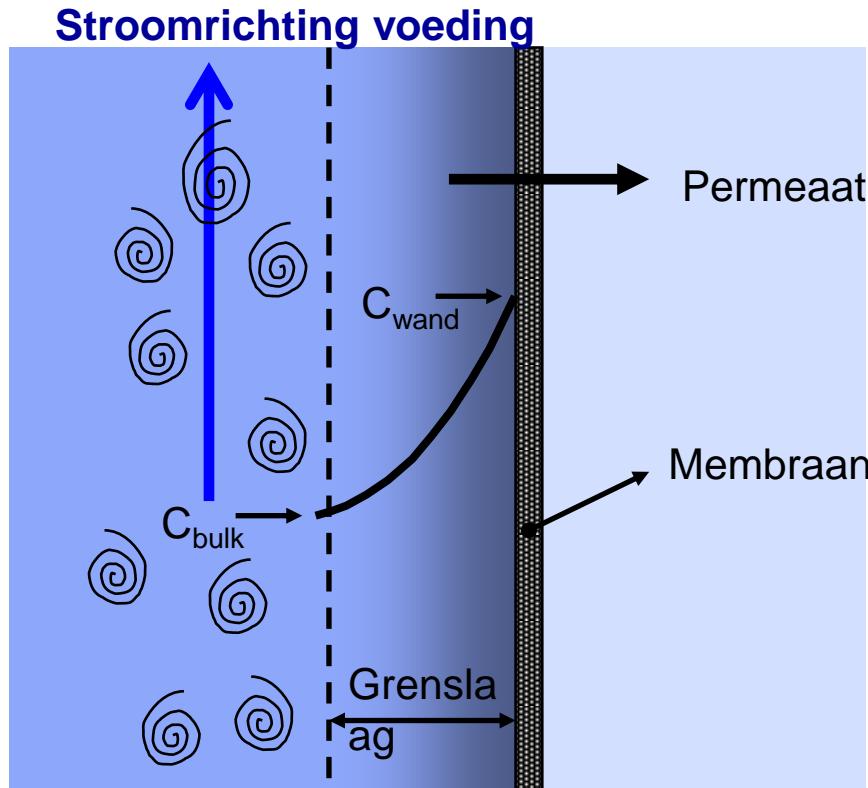
- wordt veroorzaakt door het weerhouden van de stof
- treedt snel op
- reversibel

✓ Membraanvervuiling (“fouling”)

- wordt veroorzaakt door:
 - poriënverstopping (vnl. bij MF)
 - adsorptie op de poriënwanden
 - afzetting van de opgeloste stof op membraanoppervlak
- treedt pas op na lange duur
- irreversibel

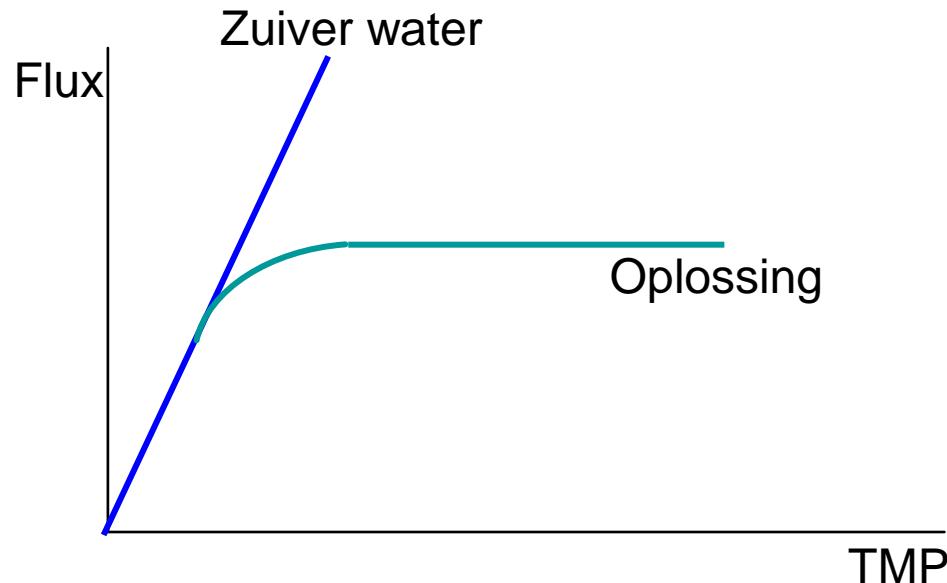
CONCENTRATIE-POLARISATIE

- ✓ wordt veroorzaakt door het weerhouden van de stof
- ✓ treedt snel op
- ✓ reversibel



CONCENTRATIE-POLARISATIE

Ontstaan van een **limietflux** of **plateauflux**



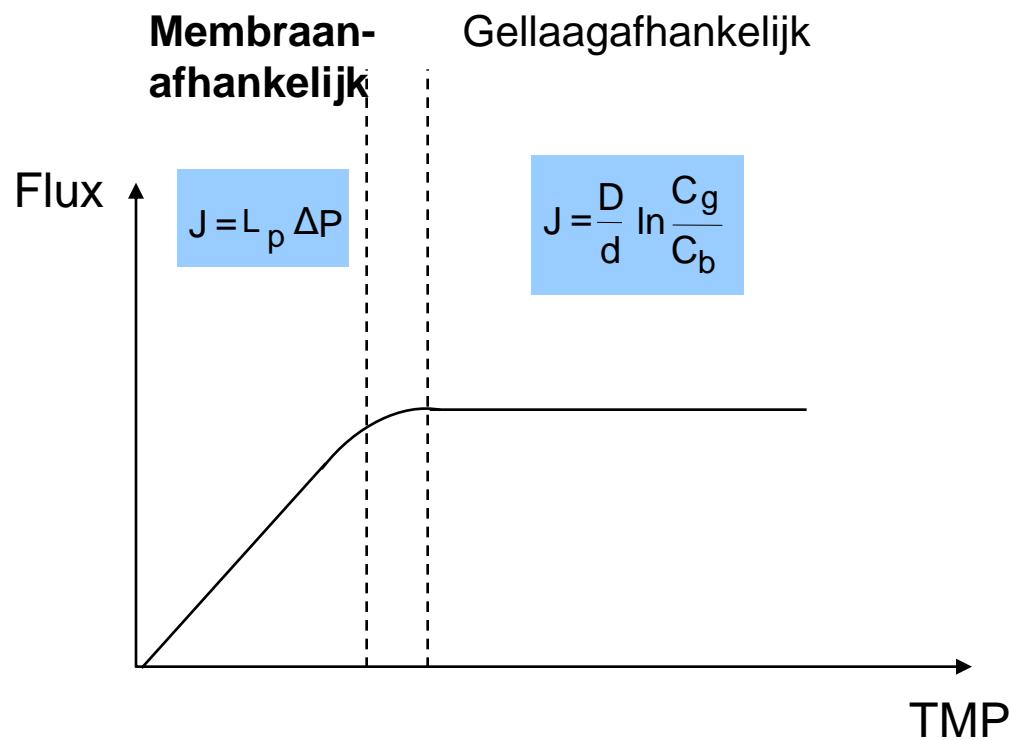
»

VOORNAAMSTE PROCESPARAMETERS

- ✓ Flux/TMP
- ✓ Bulkconcentratie (VCF/S)
- ✓ Temperatuur
- ✓ Tangentiële snelheid

FLUX/TMP

Bij cross-flow: twee verschillende gebieden



BULKCONCENTRATIE

$$J = \frac{D}{d} \cdot \ln \frac{C_g}{C_b}$$

Dus: bij hogere bulkconcentratie \rightarrow lagere limietflux

Limietflux

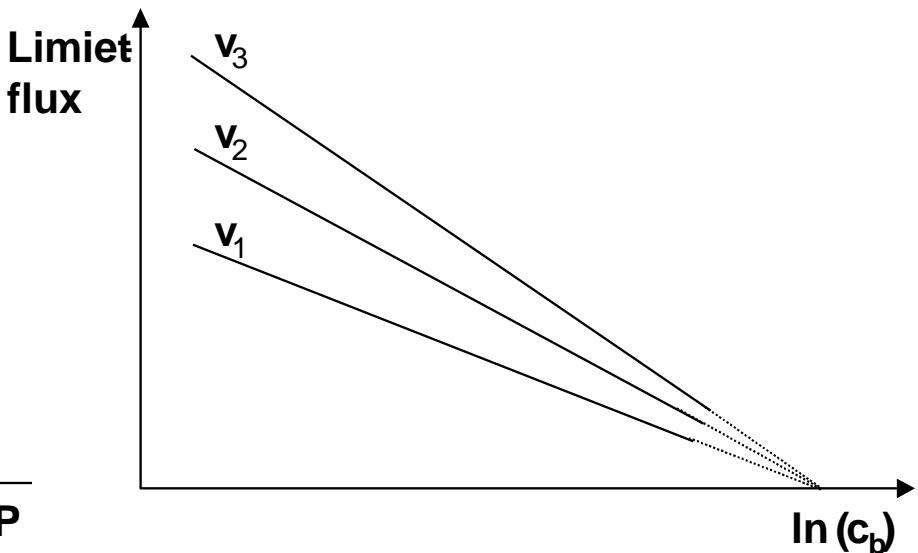
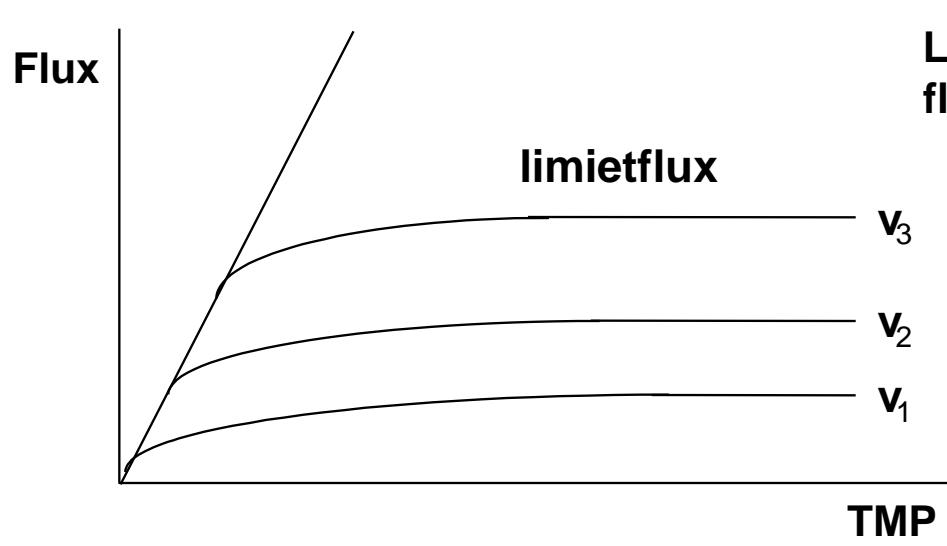
$$\frac{C_g}{C_b} = 20$$

$$\ln (c_b)$$

$$\frac{C_g}{C_b} = 1$$

TANGENTIËLE SNELHEID

$$v_3 > v_2 > v_1$$



TEMPERATUUR

Flux in functie van de temperatuur

$$\ln \frac{J_2}{J_1} = \frac{2}{3} \left[\ln \frac{T_2}{T_1} - \frac{\Delta E_0}{R} \left(\frac{1}{T_2} - \frac{1}{T_1} \right) \right]$$

Experiment : Olie/water emulsie (3 gew %)

T_1 (°C)	J_1 (l/h. m²)	Exp. $\ln J_2/J_1$	Berek. $\ln J_2/J_1$
21	240		
30	288	0.18	0.17
40	348	0.19	0.18
50	414	0.18	0.17
60	492	0.18	0.16

→ Flux neemt met ongeveer 2.5 - 2.75 % toe per 1°C-stijging

FOULING

Afzetting uit de voeding van allerhande materialen op het membraanoppervlak:

- ✓ “deeltjes” in dispersie (anorganische, bacteriën, virussen, algen, ...)
 - ✓ **opgeloste stoffen** concentreren op in retentaat en precipiteren op membraanoppervlak (“scaling”) indien de oplosbaarheidslimiet wordt overschreden
 - ✓ **moleculen** welke aan het membraanoppervlak hechten
 - ✓ **biofouling**: kolonisatie van bacteriën in biofilm
- **Irreversibel**: reiniging nodig!

MINIMALISATIE FOULING

✓ **Membraankeuze:**

- Bepaalt in sterke mate vervuiling en reinigbaarheid
- Minimale interactie tussen opgeloste stof en membraan

✓ **Voorbehandeling voeding**

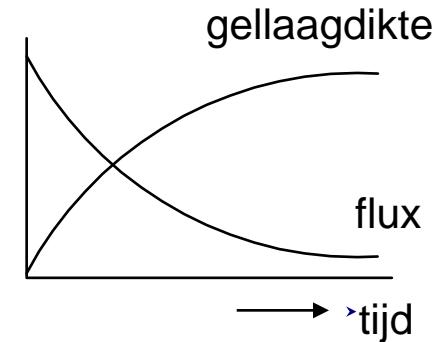
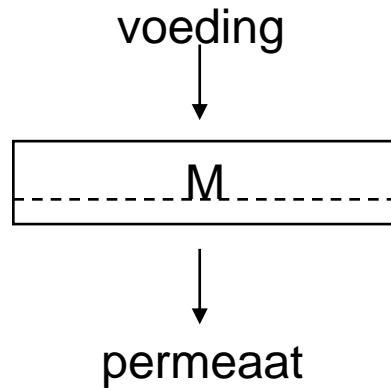
✓ **Optimalisering bedrijfsvoering:**

- Cross-flow i.p.v. dead-end filtratie
- Semi dead-end filtratie
- Backflush/backshock
- Inductie van secundaire stromingspatronen:
 - Luchtspoeling (capillaire membranen)
 - Spacers (spiraal gewonden membranen)
- Chemische reiniging

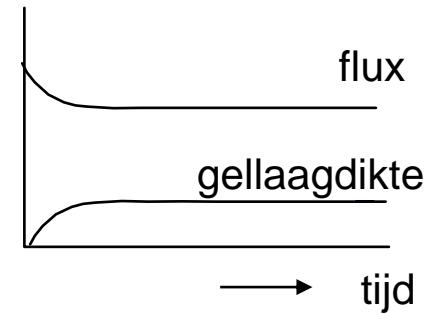
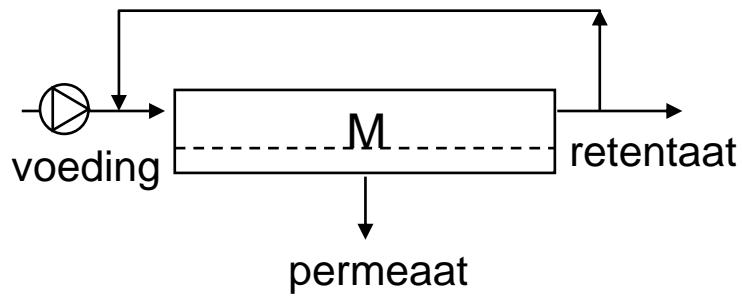
MINIMALISATIE FOULING

Bedrijfsvoering: dead-end versus cross-flow

✓ Dead-end



✓ Cross-flow

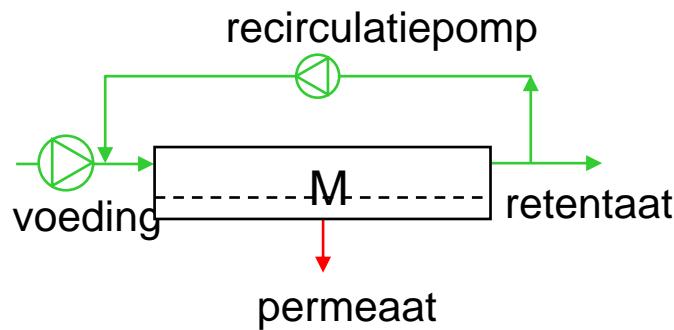


MINIMALISATIE FOULING

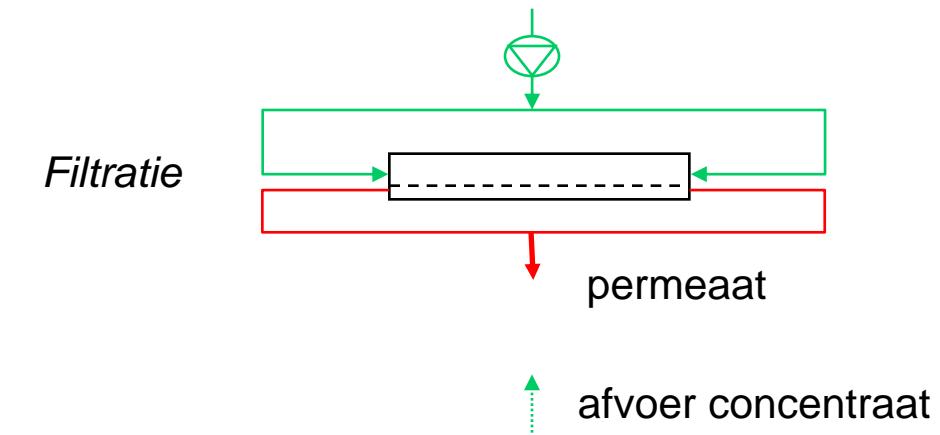
Semi dead-end bedrijfsvoering:

→ reductie energiekost t.o.v. crossflow bedrijfsvoering voor MF/UF

Cross-flow : 4 kWh/m³

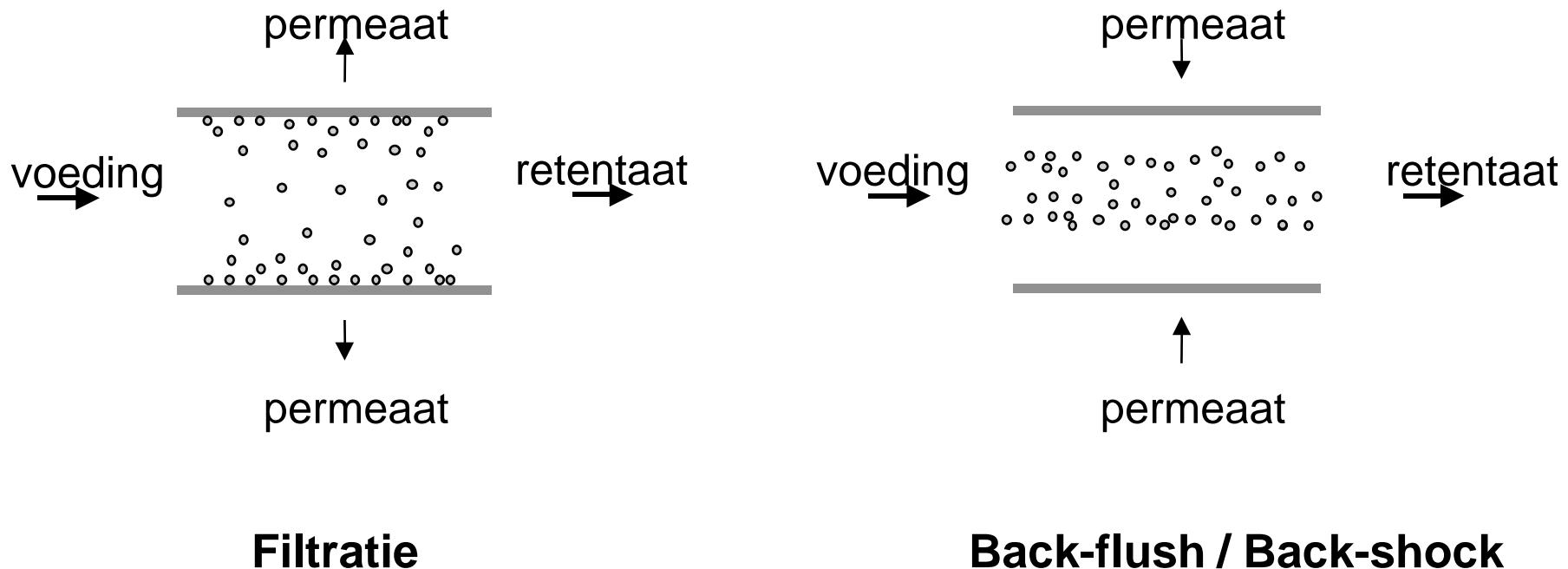


Semi dead-end: 0.1-0.3 kWh/m³



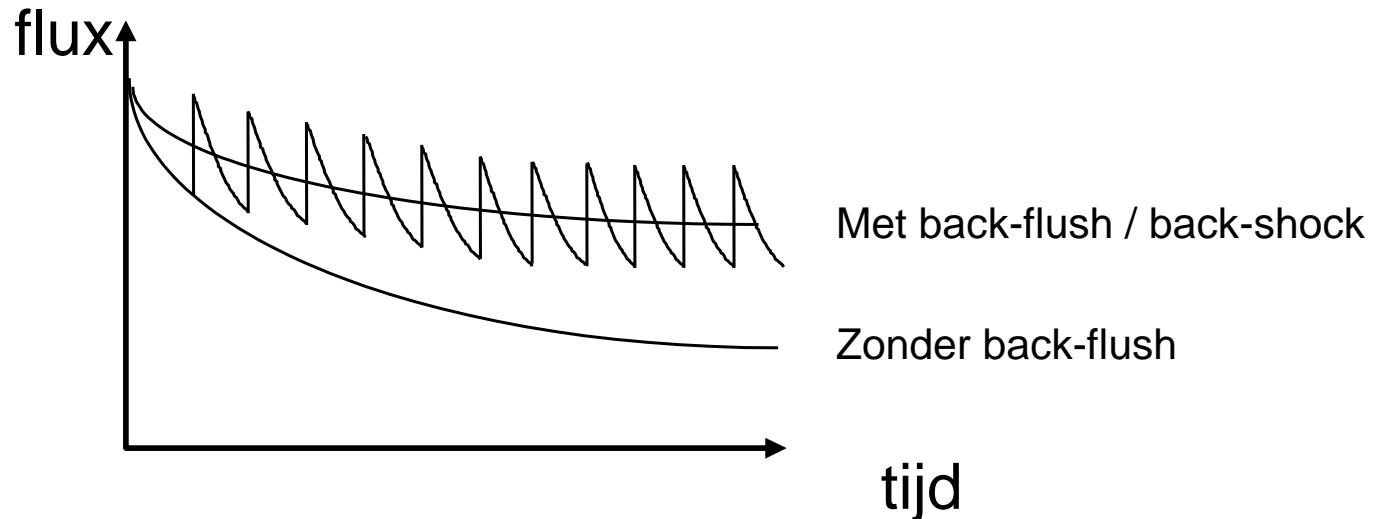
MINIMALISATIE FOULING

Back-flush / Back-shock (vnl. bij MF)



MINIMALISATIE FOULING

Back-flush / back-shock



	Frequentie	% neg. flux	Duur
Back-flush:	10 - 30'	30 - 10	0.5 - 5'
Back-shock:	1 - 10"	5 - 0.5	0.05 - 0.5"

MINIMALISATIE FOULING

Geschikte **voorbehandeling**: bepalend voor levensduur en performantie membraan!

- ✓ Voorfiltratie
- ✓ Chemische klaring of toevoeging van complexeermiddelen
- ✓ pH aanpassing
- ✓ Warmtebehandeling
- ✓ Gebruik van enzymes en preadsorptie op actieve kool

VOORBEHANDELING

Scaling (anorganisch)

Koud / warm (ontkalking)
Zeoliet ontharding
Aanzuring
Antiscalants

Fe, Ba,
silica

Vervuiling (organisch)

Coagulatie
Clarificatie / uitvlokking
Filtratie
Oxidatie
Zandfiltratie
Chlorering
Actieve kool
 H_2S verwijdering
Biocide additie

Biofouling

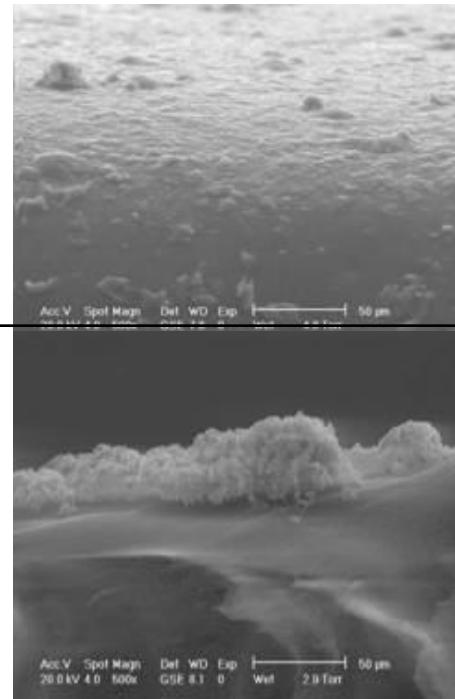
PROCESSING

■ SDE-O / IN-LINE DOSING OF FLOCCULANT

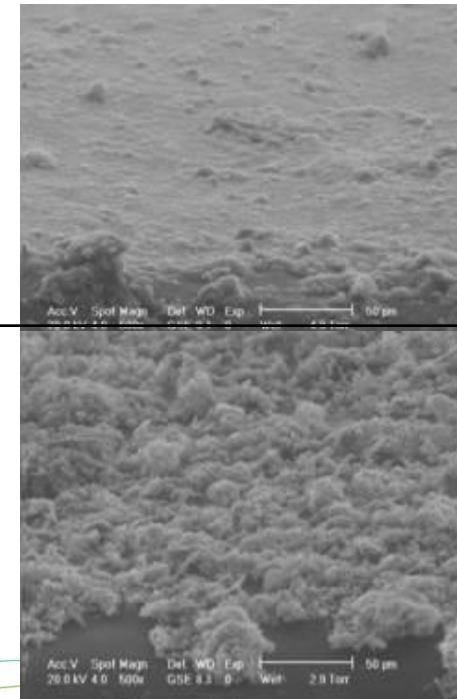
Dosing of flocculant (e.g.: FeCl_3) generates Fe(OH)_3 containing filtration cakes. These are much more permeable (see ESEM).

→ Hence filtration becomes much more stable

Without FeCl_3

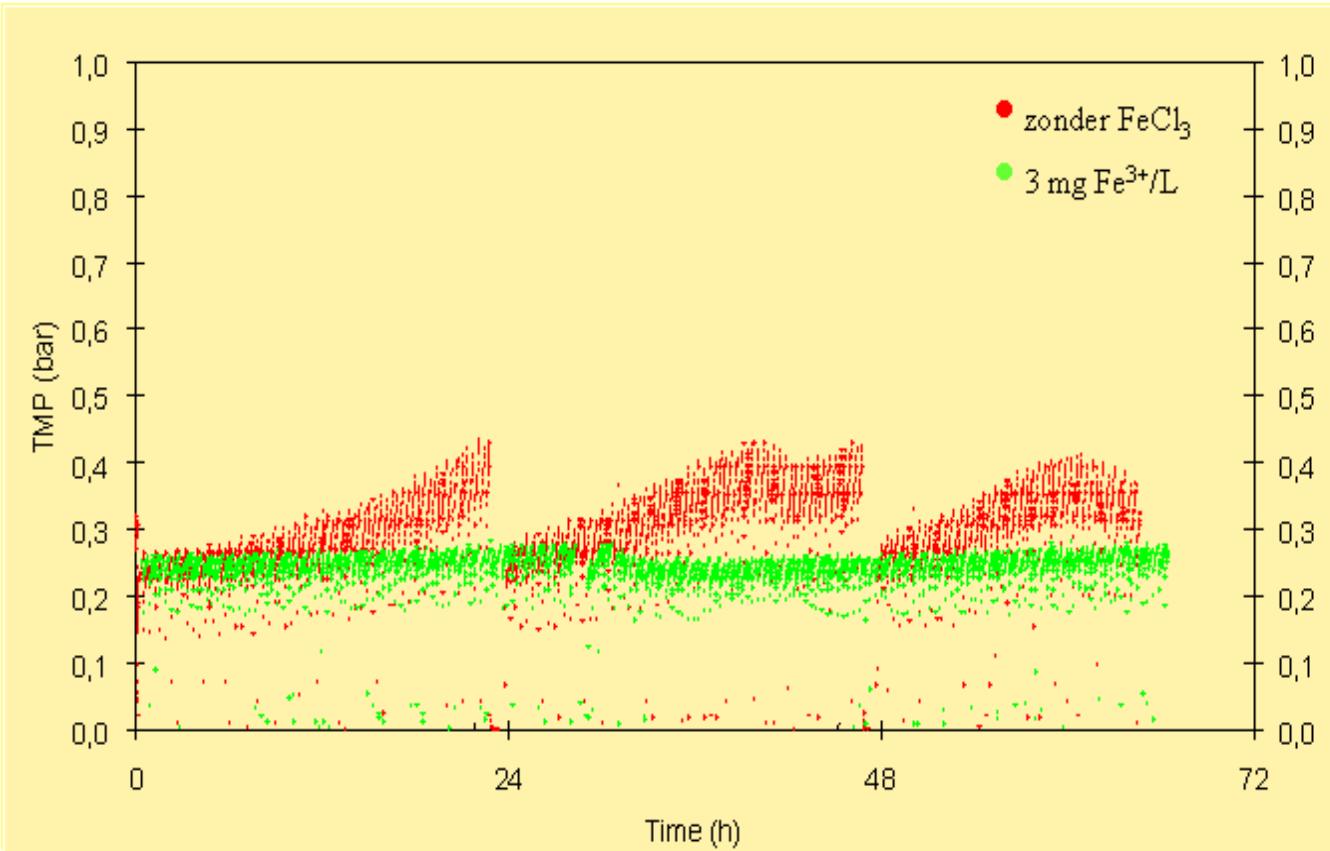


With FeCl_3
(0.7 mg Fe /l)



Calculated
cake porosity:
37 % (without)
and 93 %
(with)

Vb. VOORBEHANDELING: effect dosering flocculant



OUTLINE

1. Inleiding
2. Membranen / Modules en ontwerp
3. Procesparameters en fouling
- 4. Overzicht waterige membraantoepassingen**
5. Case-studies membranen voor waterzuiveringstoepassingen
6. Overzicht en case-studies niet-waterige membraantoepassingen
7. Conclusies

MF / UF - TOEPASSINGEN

- ✓ Concentratie
 - Eiwitten (melk), polymeren, gelatine, ...
- ✓ Terugwinning of recuperatie
 - Proteïnen (kaaswei), sterkpolymeren, indigo, lignine, ...
- ✓ Klaring of stabilisatie
 - Appelsap, wijn, bier, sterilisatie ...
- ✓ (Afval)waterbehandeling
 - Verwijdering zwevende stof (voorzuivering RO), o/w emulsies, bleekeffluenten, ontvettingsbaden, ...
- ✓ Zuivering
 - Bloedfractionering (hemofiltratie), diafiltratie, ...

NF - TOEPASSINGEN

- ✓ Waterbereiding
 - Partiële ontharding
 - Verwijdering opgeloste organische componenten: pesticiden, humuszuren, ...
 - Aanmaak ultrapuur water (NF + IEX)
- ✓ Sulfaatverwijdering uit zeewater
- ✓ Ontzouting van kaaswei (diafiltratie)
 - Permeatie zout, concentratie organische stoffen
- ✓ Zuivering effluenten/spoelwaters
 - Concentratie van Ni, Fe, Cu, Zn... (metaalindustrie)
 - Kleurstofverwijdering (textielindustrie)
 - Ligninesultaat + derivaten (papierindustrie)

RO - TOEPASSINGEN

- ✓ Waterbereiding
 - Ontzouting van brak en zeewater tot drinkwater
 - Aanmaak ultrapuur water
- ✓ Concentratie van productstromen
 - Opconcentreren van melk en wei
 - Voorbehandeling evaporatie
- ✓ Afvalwaterbehandeling
 - Procesindustrie

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TOEPASSING WATERZUIVERING

1) Productie proceswater:

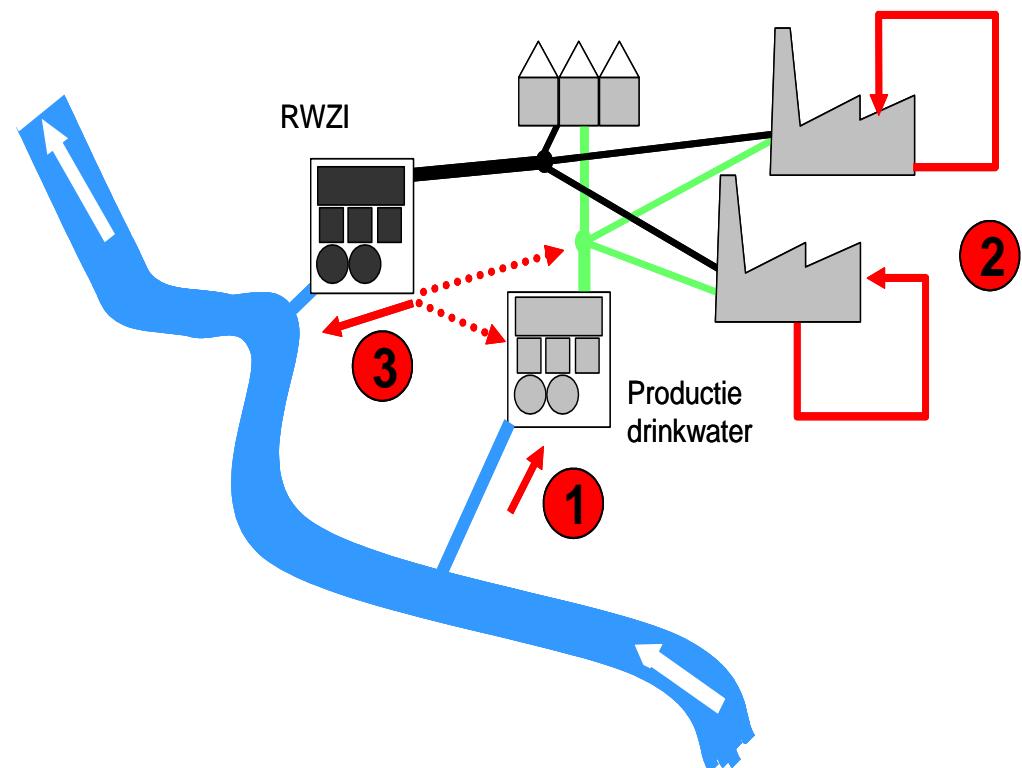
- Alternatieve waterbronnen

2) In-process toepassingen:

- Waterzuivering
- Product recuperatie

3) End-of-pipe toepassingen:

- Zuivering tot hergebruik



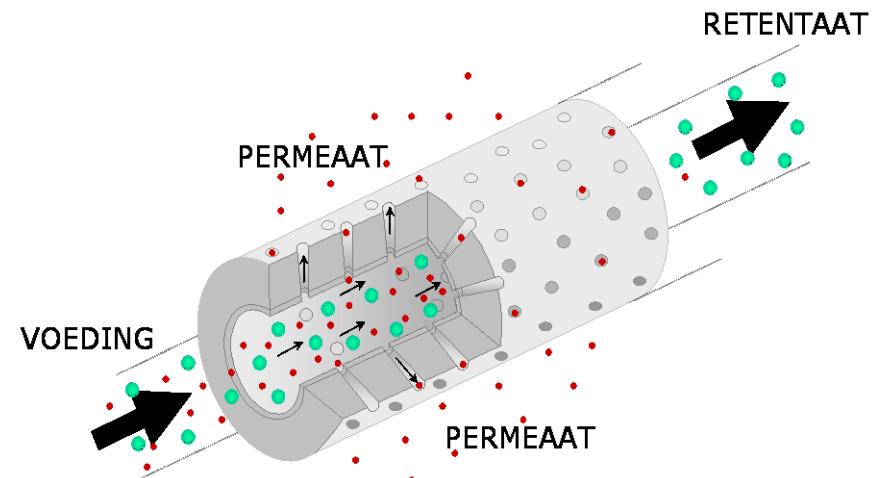
MEMBRAANFILTRATIE IN WATERZUIVERING

✓ Membraanfiltratie:

- MF / UF
- NF
- RO

✓ Implementatiemogelijkheden:

- 1) Voorbehandeling + MF/UF + NF/RO
- 2) Membraanbioreactoren



Concentraat !!

MEMBRAANFILTRATIE IN WATERZUIVERING

- ✓ Bereiding van drink- of proceswater: aaneenschakeling van technieken
 - Voorbehandeling: verwijdering van zwevende delen
 - Nazuivering: verwijdering opgeloste stoffen
- ✓ Klassieke technieken of membraanteknologie

Conventionele voorbehandeling

+

NF/RO

MF/UF

+

NF/RO

MEMBRAANFILTRATIE IN WATERZUIVERING

Voordelen voorbehandeling via MF/UF

- ✓ Membranen zijn absolute fysische barrière:
 - Bacteriën: log 5-6, virussen log 4-5 reductie
- ✓ 30 - 40 % ruimtebesparing en modulair
- ✓ Goedkopere nabehandeling
 - RO 20 % minder investering + lagere operationele kost
- ✓ Eenvoudige bedrijfsvoering
- ✓ Relatief ongevoelig aan wijzigingen ruw water kwaliteit, seizoensschommelingen, procescondities
- ✓ Constante permeaatkwaliteit (betrouwbaar proces)

PROCESEIGENSCHAPPEN

✓ UF:

- Toepassing: Verwijdering SS + bacteriën
- Eisen voedingswater: SS < 50 mg/l, voorfiltratie \pm 200 μm
- Permeaatproductie: 90-95 % van voeding

✓ NF:

- Toepassing: verwijdering TOC en ontharding
- Eisen voedingswater: geen SS, SDI < 5
- Permeaatproductie: 60-90 % (twee- of drietraps)

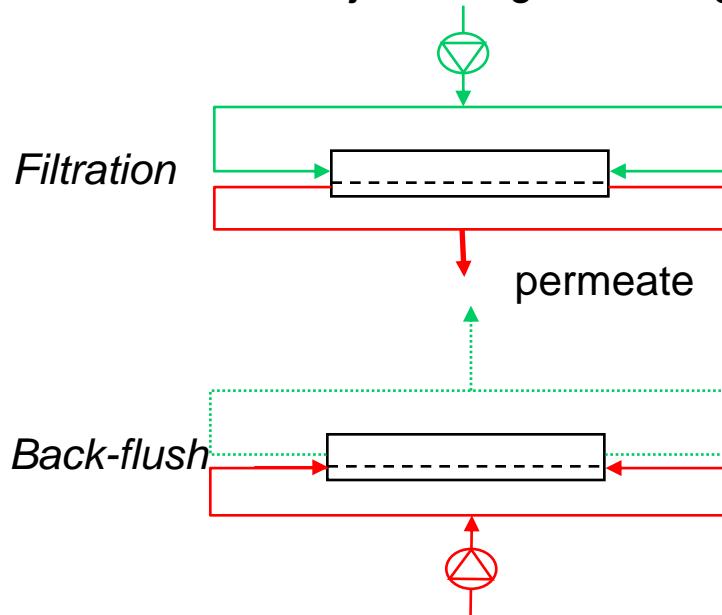
✓ RO:

- Toepassing: ontzoutning
- Idem NF

MF/UF MEMBRANEN

2 types van bedrijfsvoering:

- ✓ Semi dead-end bedrijfsvoering voor de “gemakkelijke” waters



- ✓ Cross-flow bedrijfsvoering voor de “moeilijke” waters



Toepassing: Waterzuivering

- ✓ Bedrijf: IWVA (drinkwaterbedrijf)
- ✓ Target: Verminderen hoeveelheid natuurlijk grondwater
- ✓ Concept: Artificiële opvulling van freatisch watervoerende laag (infiltratie) in duinen
 - Zuivering van riool effluent, duurzame kringsluiting
 - Voorkeur voor membraantechnologie (MF+RO)
- ✓ Capaciteit: max. 4 Mm³/y (285 m³/u)
- ✓ Totale investeringen: 6.35 miljoen euro
- ✓ Projectnaam: “Torreele”

Toepassing: Waterzuivering

Membraankeuze:

- ✓ MF: → Zeeweek (Zenon)
 - kan grote variaties in voedingssamenstelling aan
 - outside-in, luchtspoeling
 - submerged

- ✓ RO: → Dow Filmtec, laag energieverbruik
 - recovery (max. 75 %)
 - membraantype: laag energieverbruik?, lage foulingstendens?
 - reinigingsstrategie

Optimale flux:

- max. 40 l/m² voor MF
- max. 20 l/m² voor RO

Toepassing: Waterzuivering

Behandelingsprocedure:

- ✓ Pre-screening van WWTP Aquafin (1 µm) effluent
- ✓ NaOCl dosering (pre-chlorinatie)
- ✓ MF Zenon Zeeweed
- ✓ NaOCl dosering (post-chlorinatie)
- ✓ Dosering anti-scalant, zuur en bisulfiet
- ✓ MF kandelfilter (5 µm)
- ✓ RO (Dow Filmtec), 2/1 design
- ✓ UV behandeling



→ Infiltratie: 90 % van RO permeaat en 10 % van MF permeaat

Wastewater treatment into drinking water



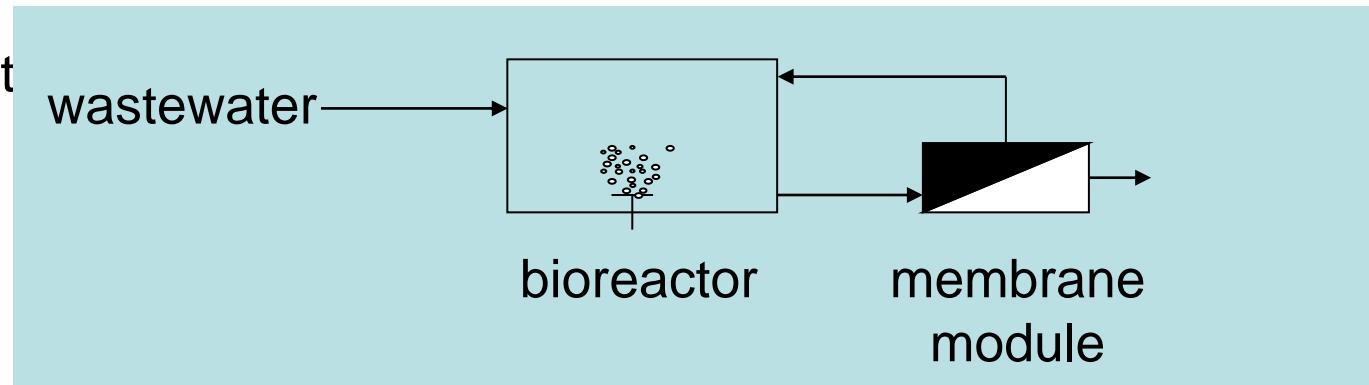
Microfiltration –
two step reverse osmosis



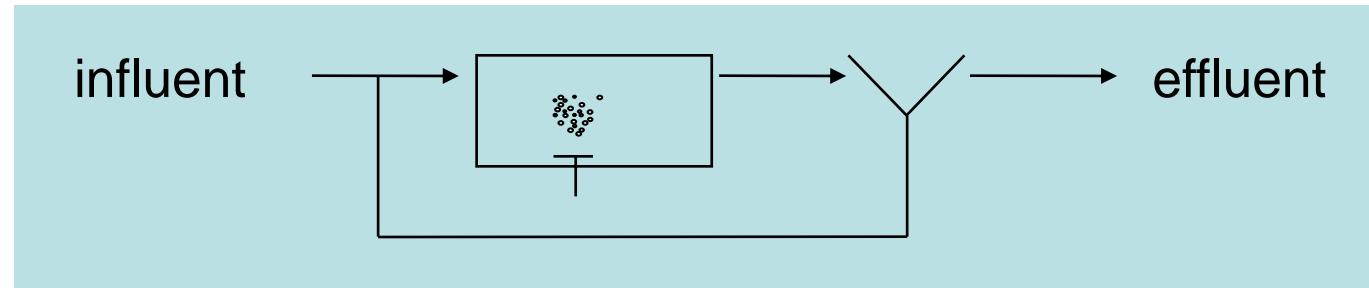
WWTP Wulpfen (Belgium)
IWVA

Waste water: CAS vs MBR

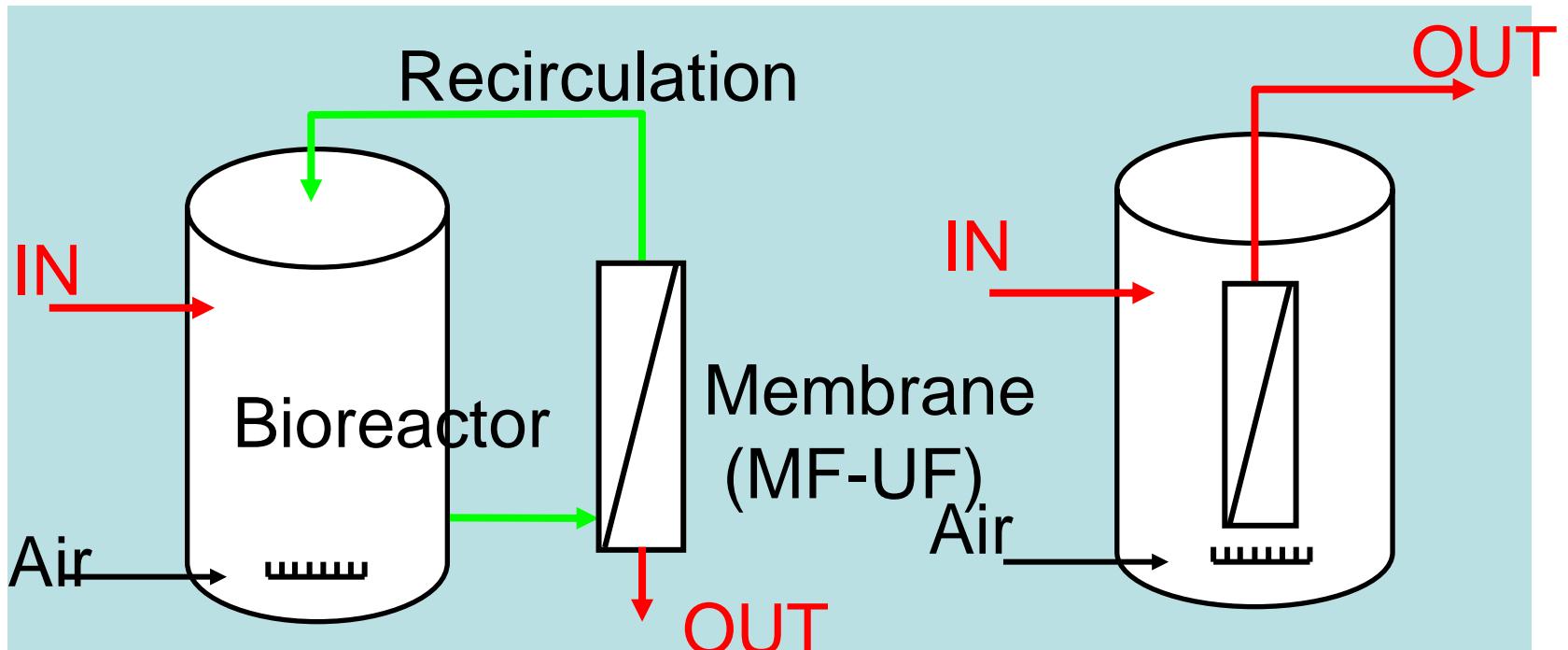
- » MBR concept



- » Conventional activated sludge system (CAS)



Membrane bioreactors: ≠ systems



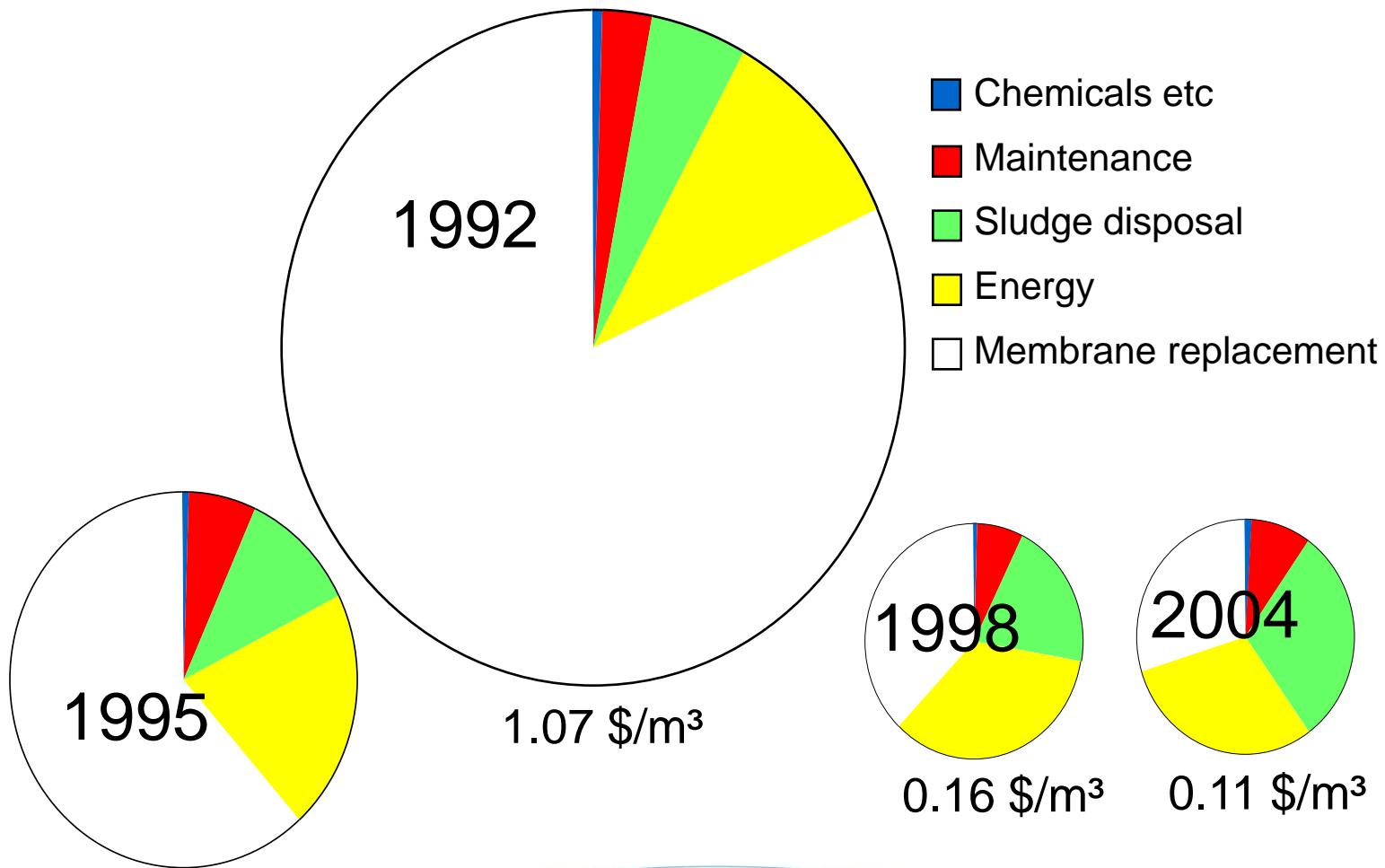
EXTERNAL

- E cost: 5 kWh/m³
- Higher fluxes

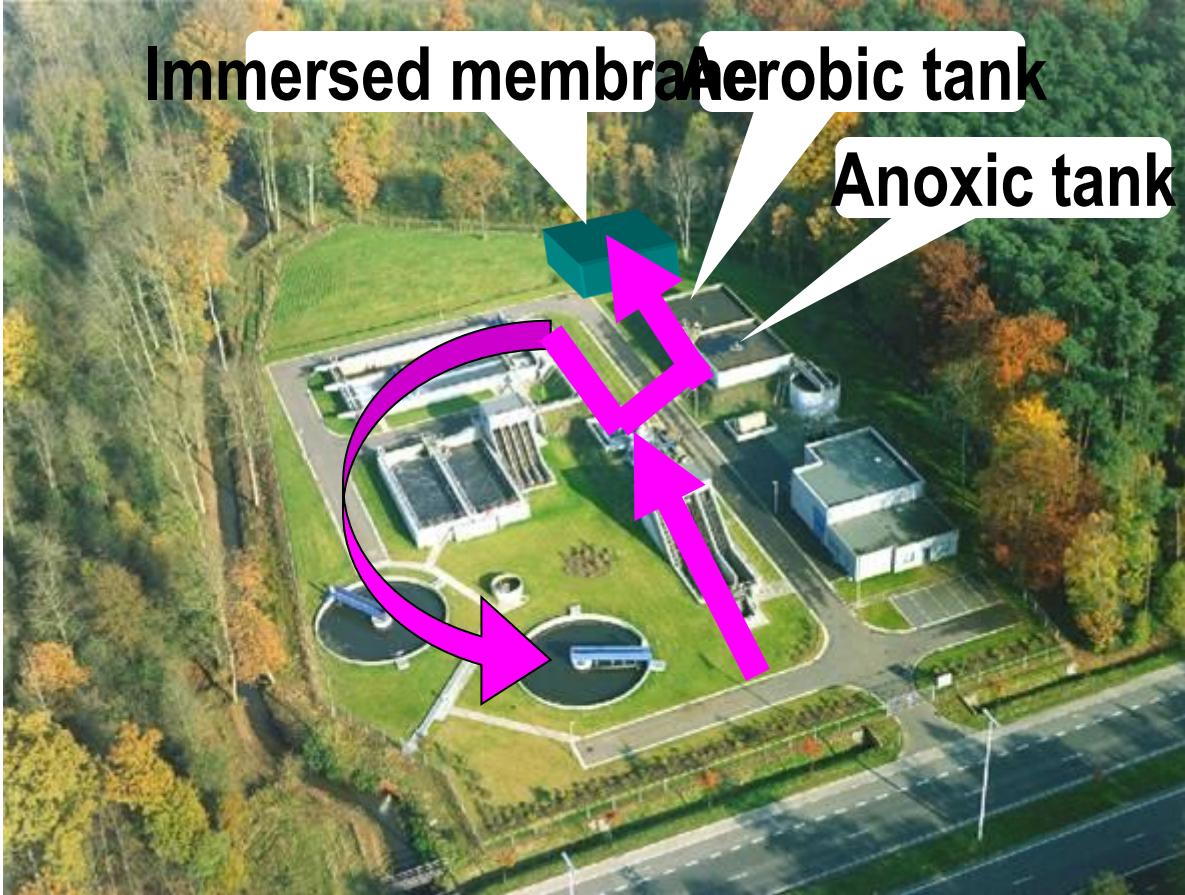
SUBMERGED

- E cost: 0.2-0.5 kWh/m³
- Lower fluxes

SUBMERGED MBR: Evolution of maintenance costs



MBR at WWTP Schilder, ref. Aquafin

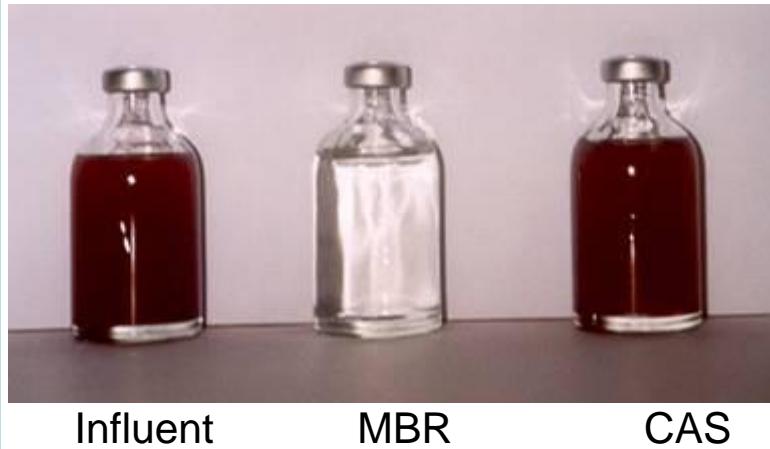
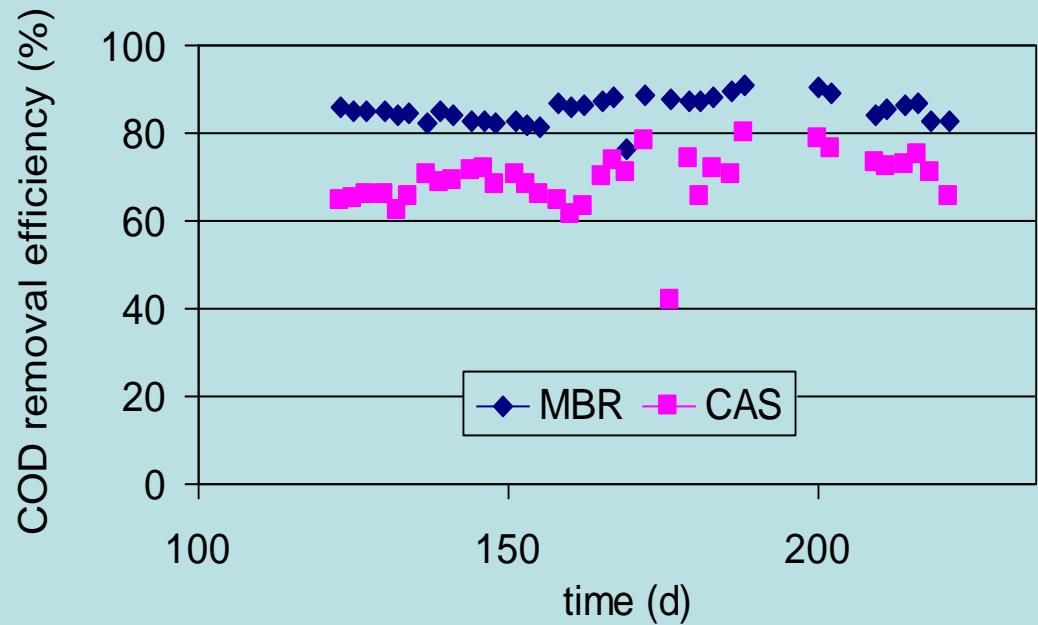


MBR at WWTP Schilder, ref. Aquafin

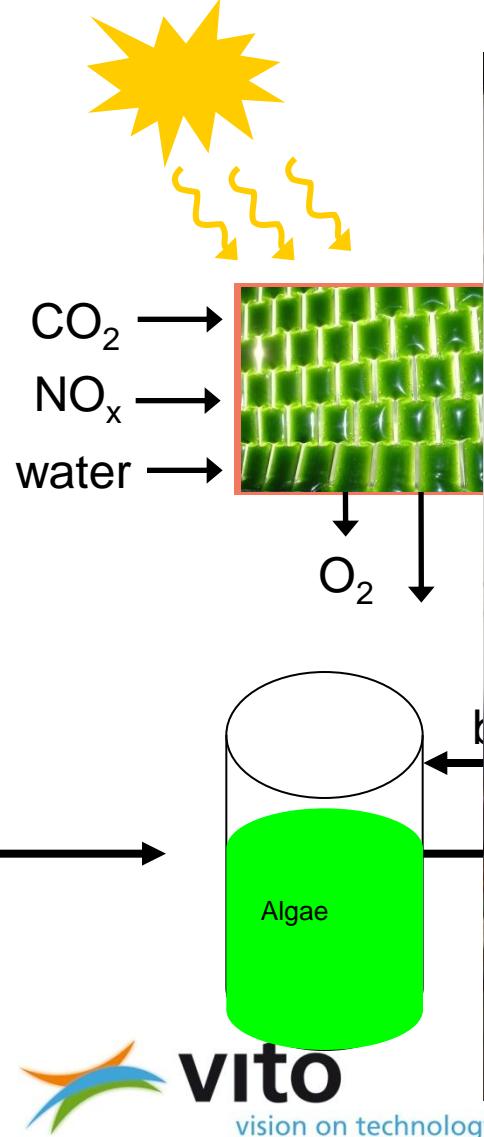


Submerged
membrane bioreactor

Printing & dyeing wastewater



CO_2 / NO_x conversion in algae biomass: downstream processing



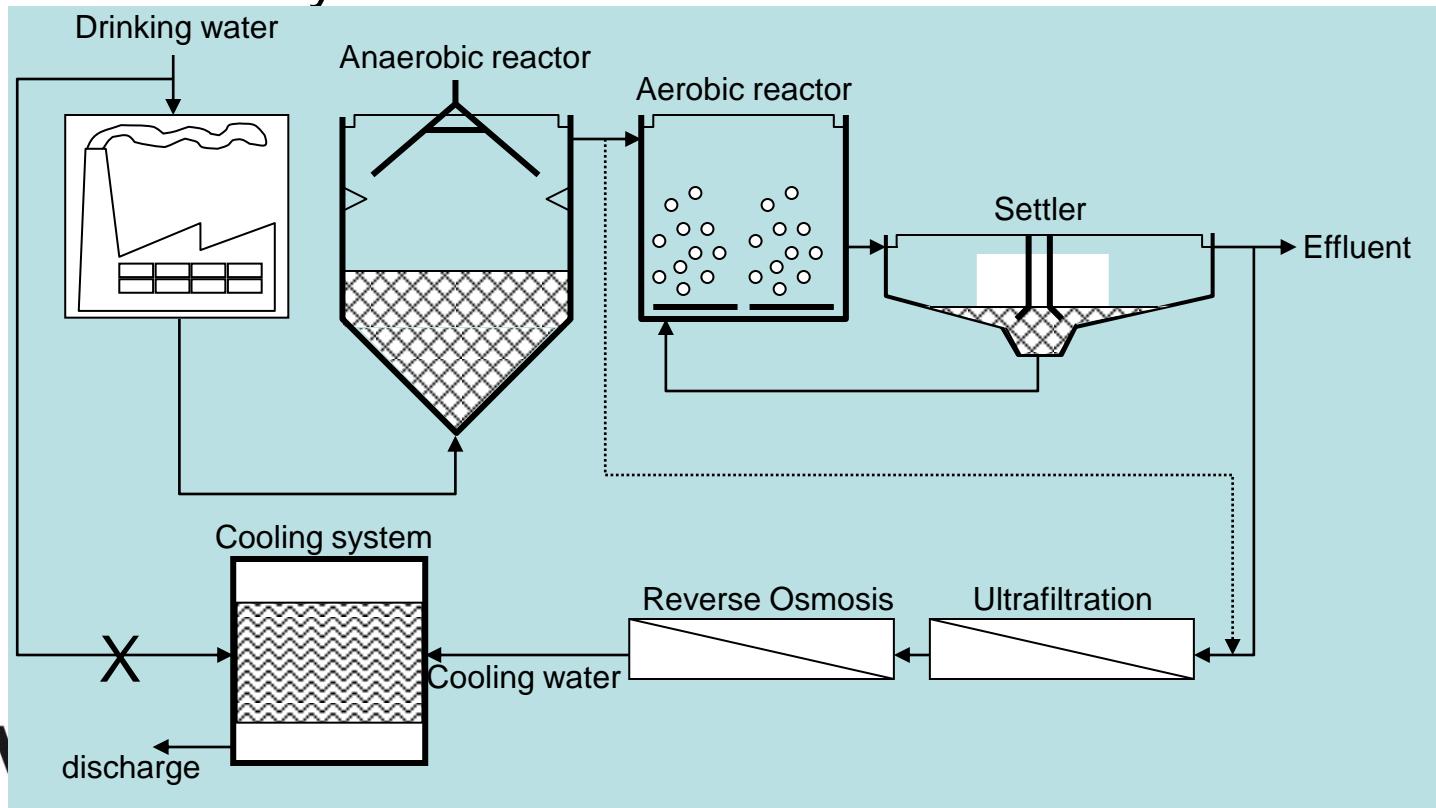
29/03/2012

Confidential – © 2009, \

Cell disruption

CASES: Food industry

- » Objective: reuse of effluent as cooling water
 - » Flow rate: 40 m³/h
 - » Effluent: SS: 200 mg/l + high salt conc. → RO necessary

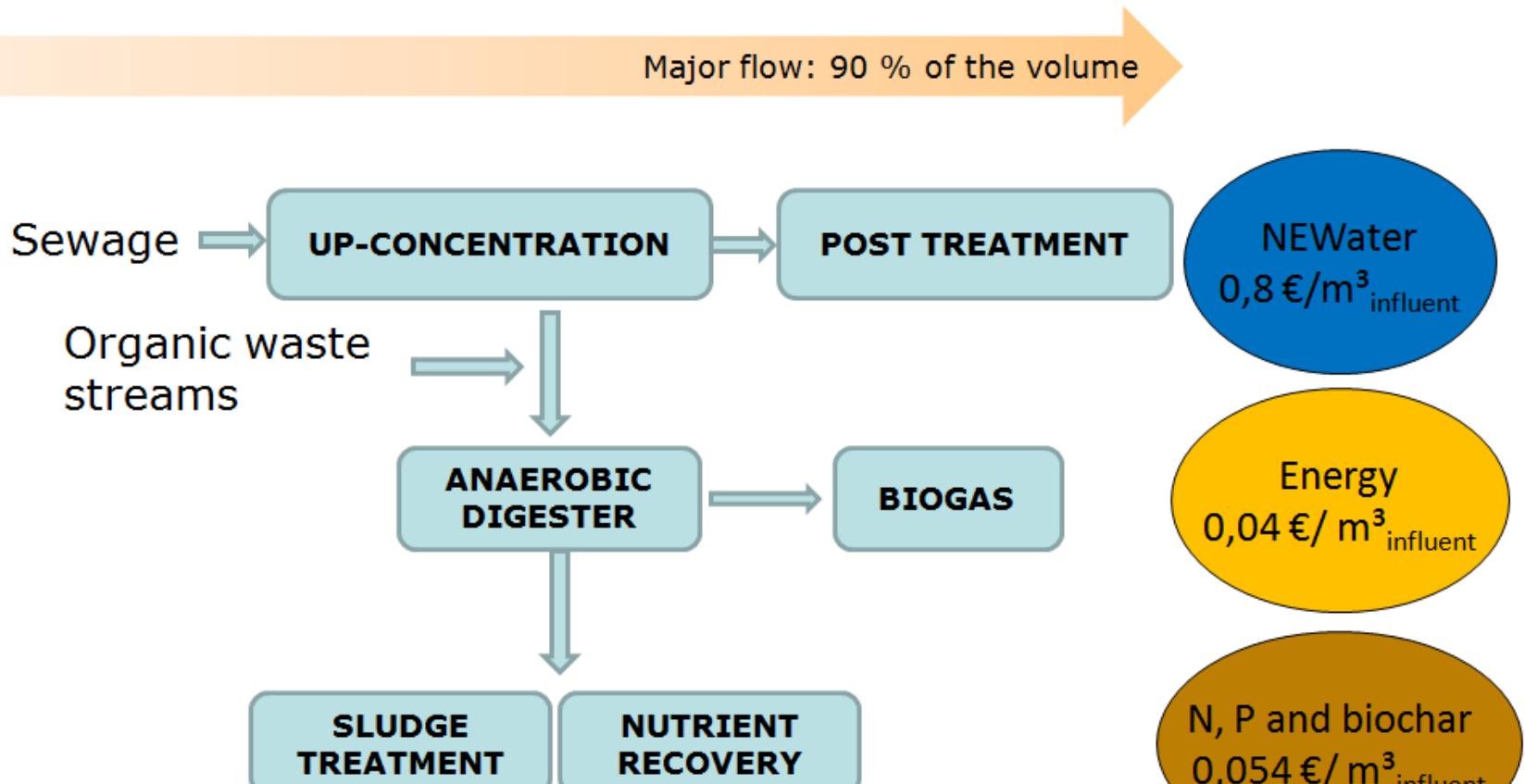


Energy recovery from the integration of wastewater and waste

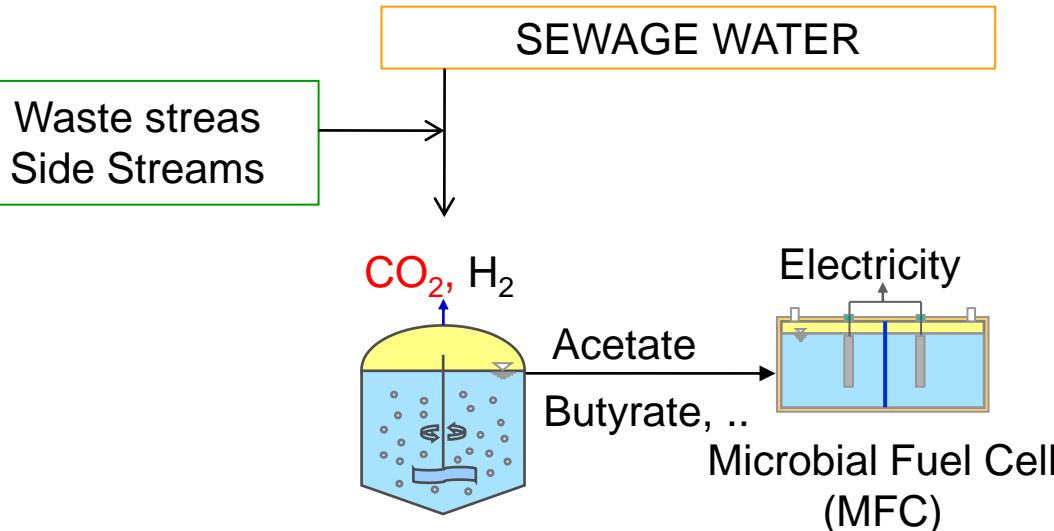


*From energy consumption
to energy production*

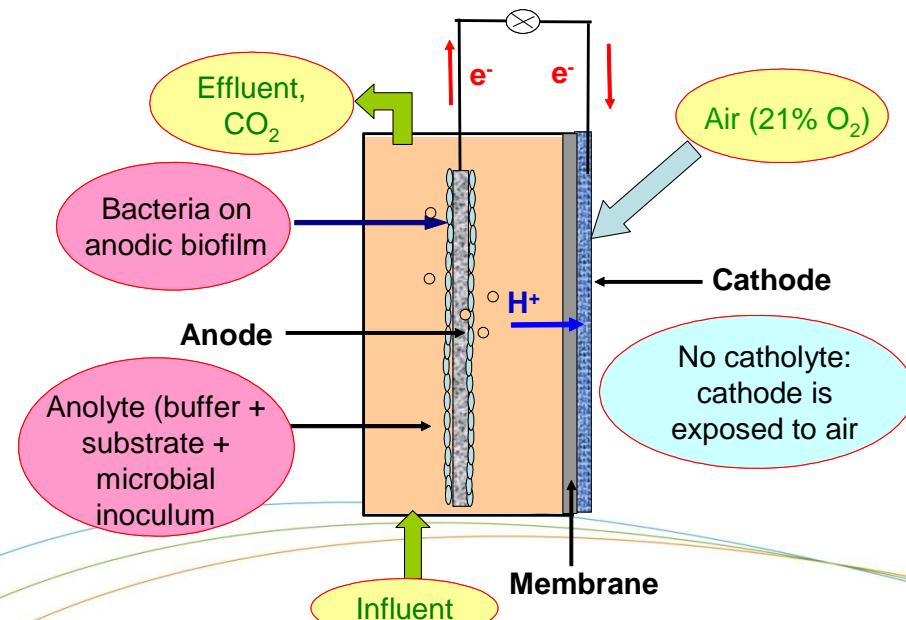
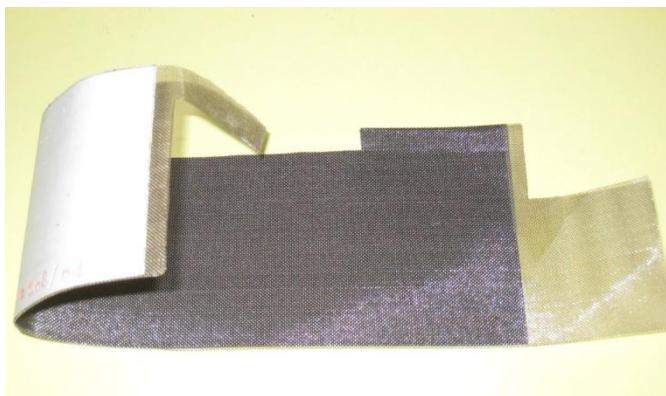
SewagePlus: general concept



SewagePlus: alternative



- Gas porous C-electrode with integrated PTFE-layer (air cathode)
- Zirfon® layer (ion permeable membrane) reduced ionic resistance and oxygen diffusion

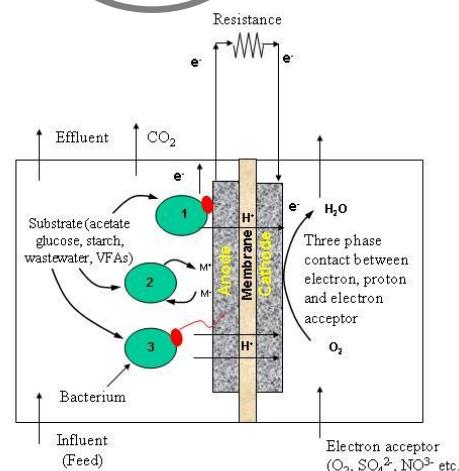
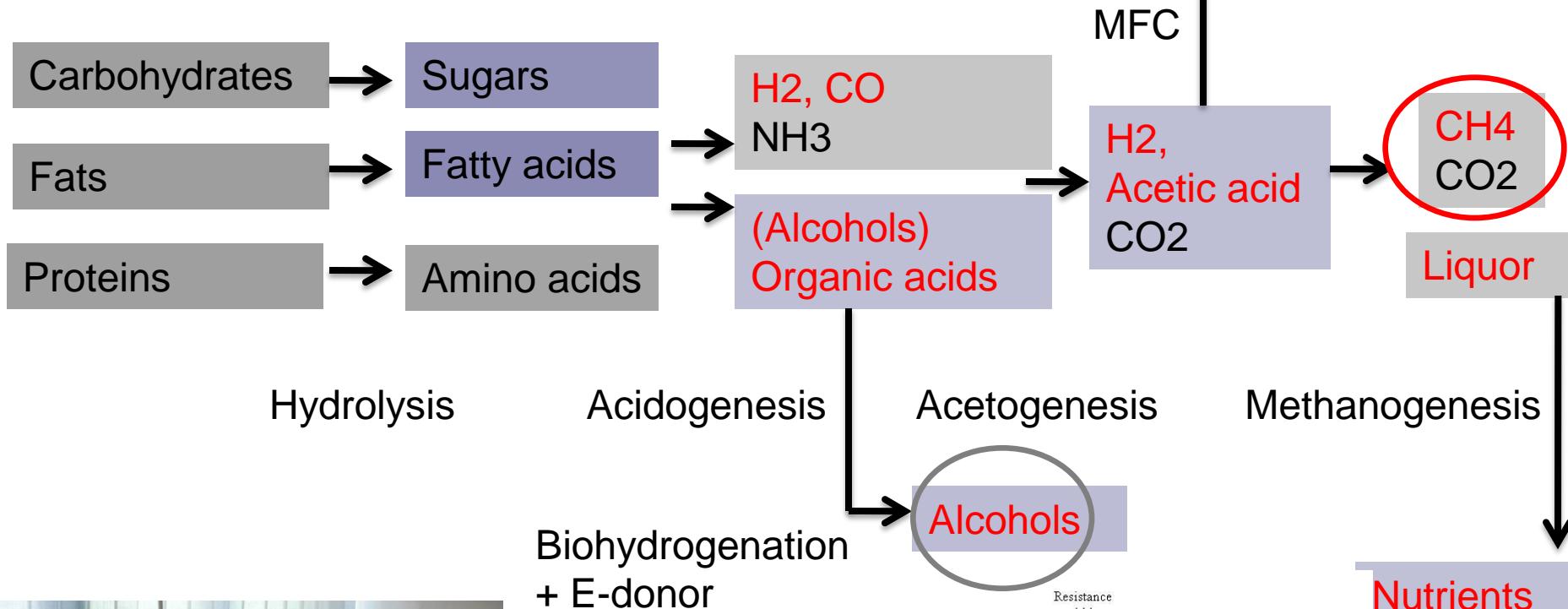


Biobased economy and waste water



Turn the threat in a challenge

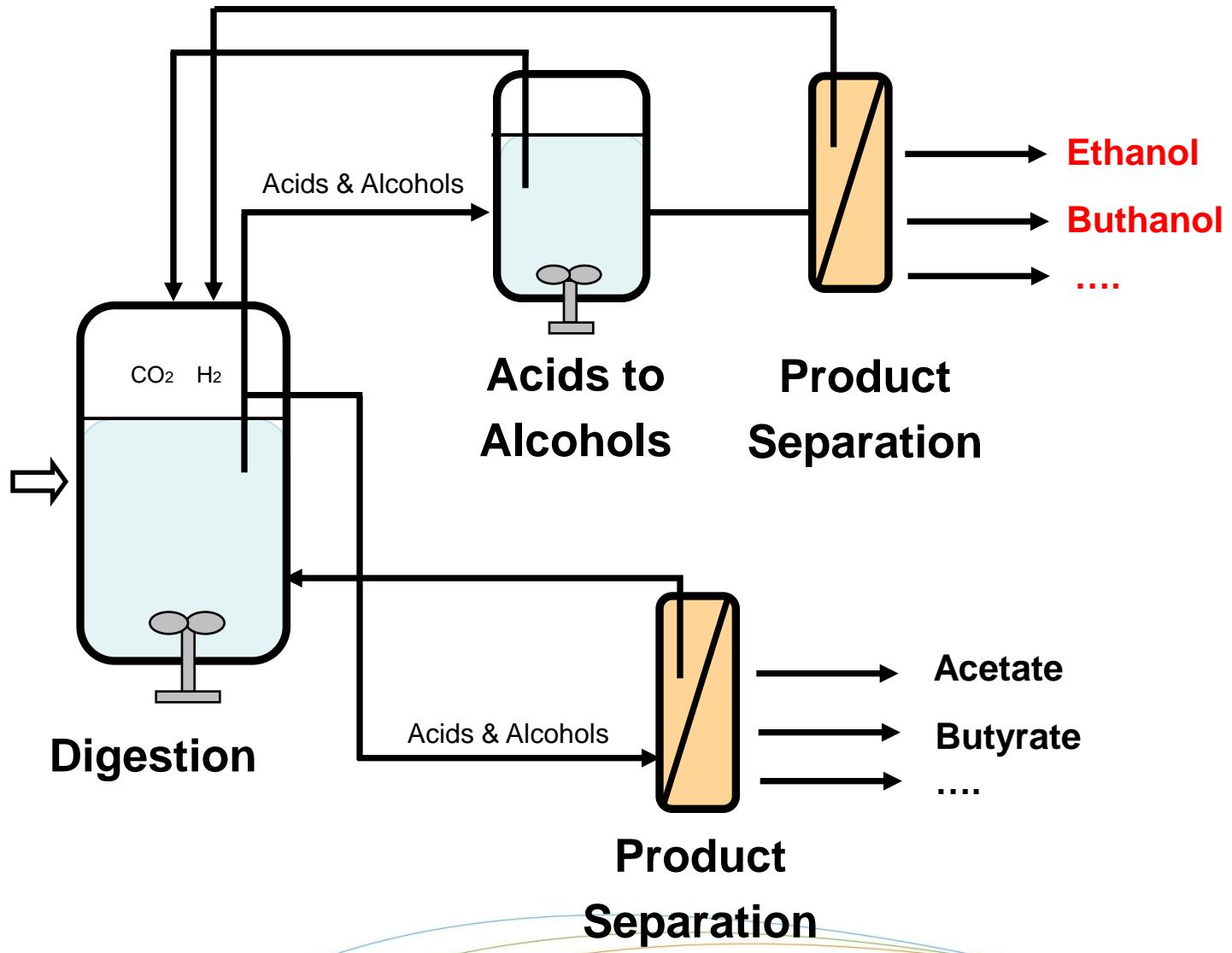
Biomass – chemicals - energy



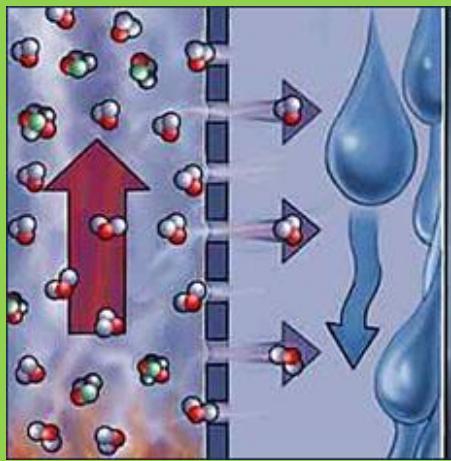
Waste and residues in biorefineries



Biorefinery
wastewater



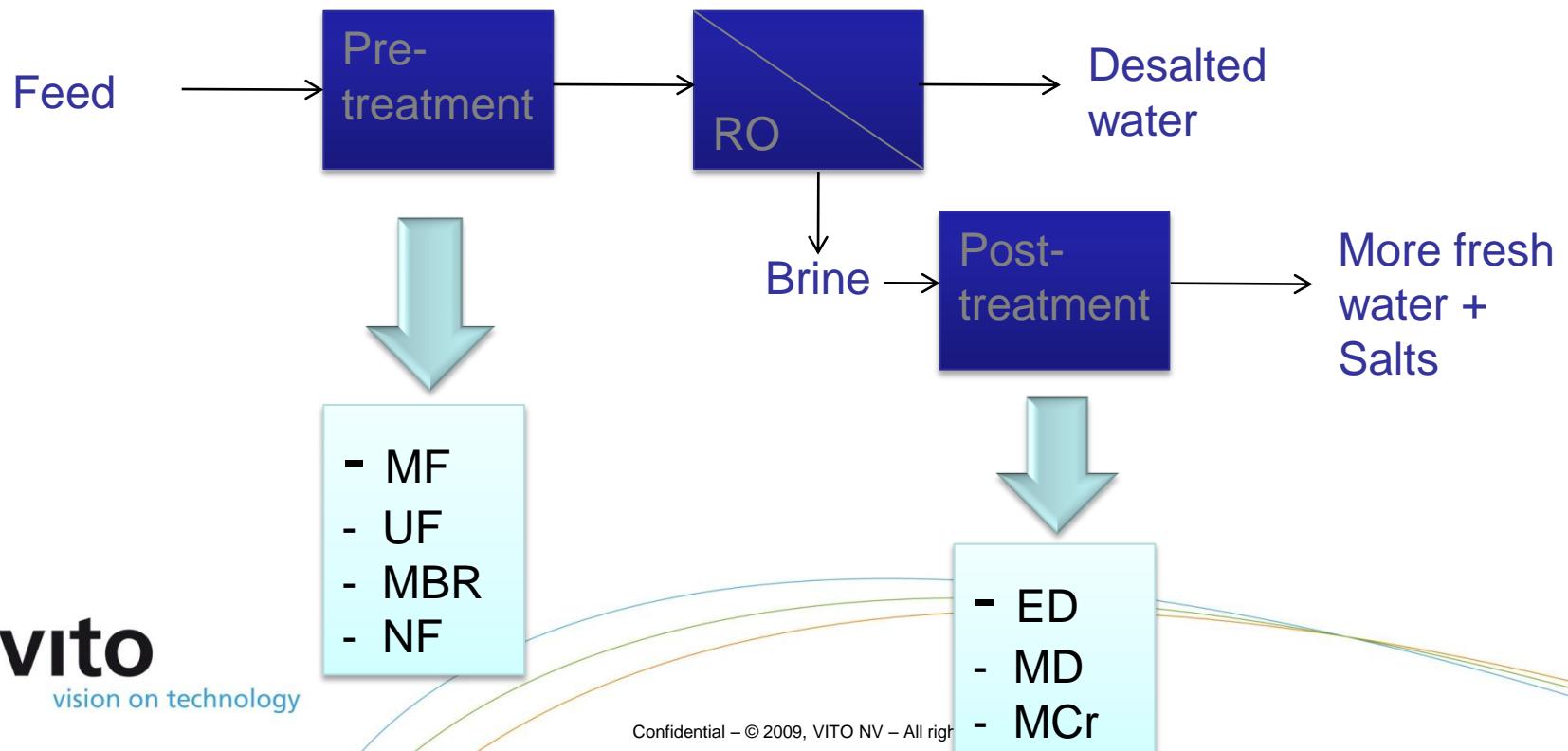
Novel concentrate treatment



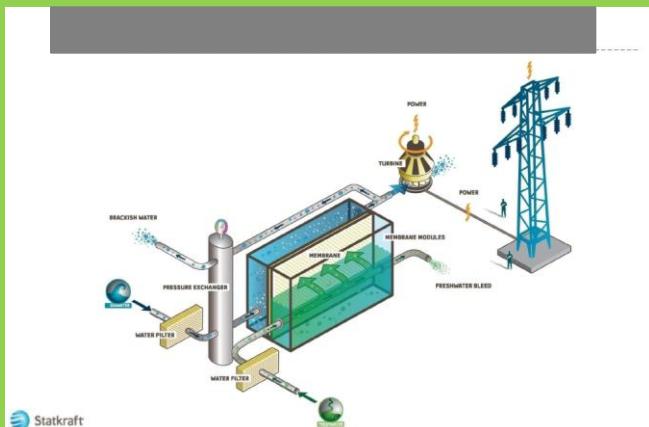
*Towards zero discharge,
recovery of salts, or energy*

Potentialities of an integrated membrane based desalination system

- » Enhancement of water recovery factor
- » Improvement of water quality
- » New brine disposal strategy
- » Cost reduction

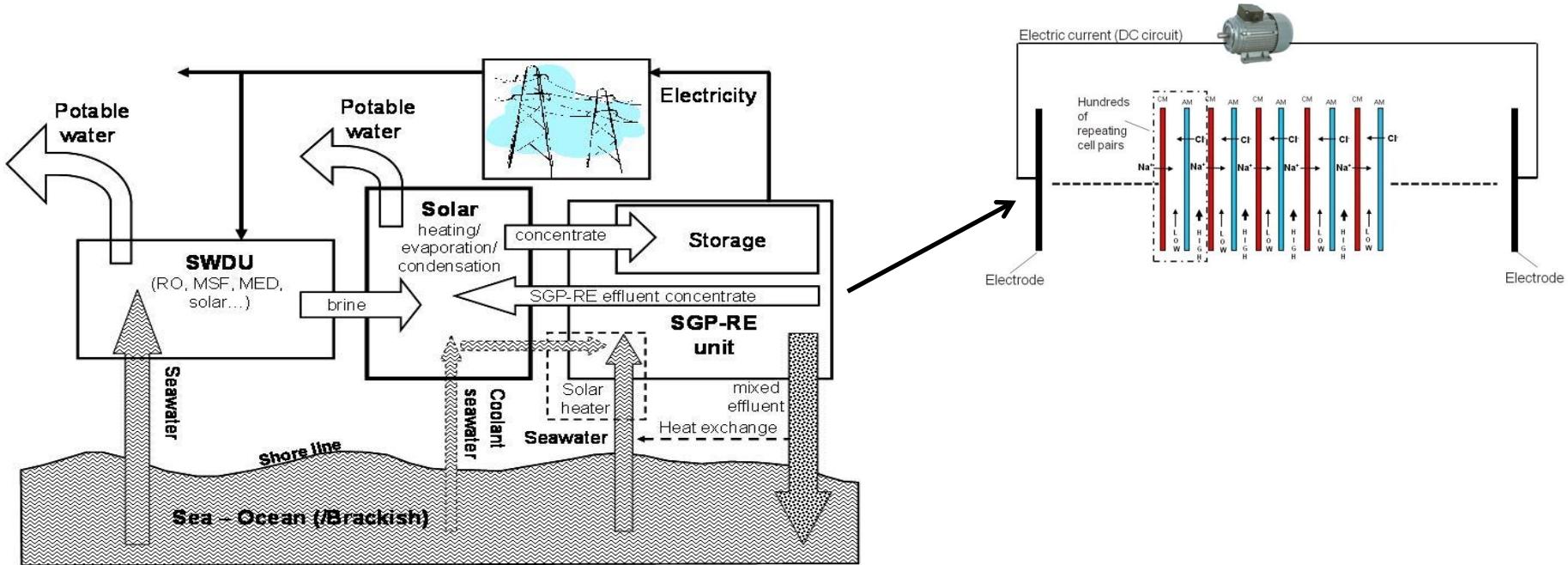


Technologies to generate power from salinity gradients

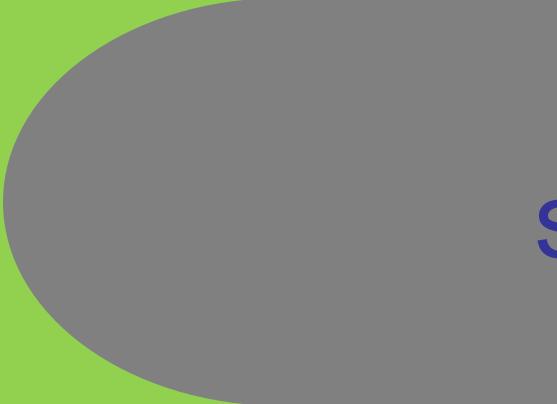


When fresh water meets salt water..

SGP-RE – application concept



Reapower High concentration = brine
Low concentration = sea or brakisch water
Power density ? (target $\geq 8 \text{ W/m}^2$)

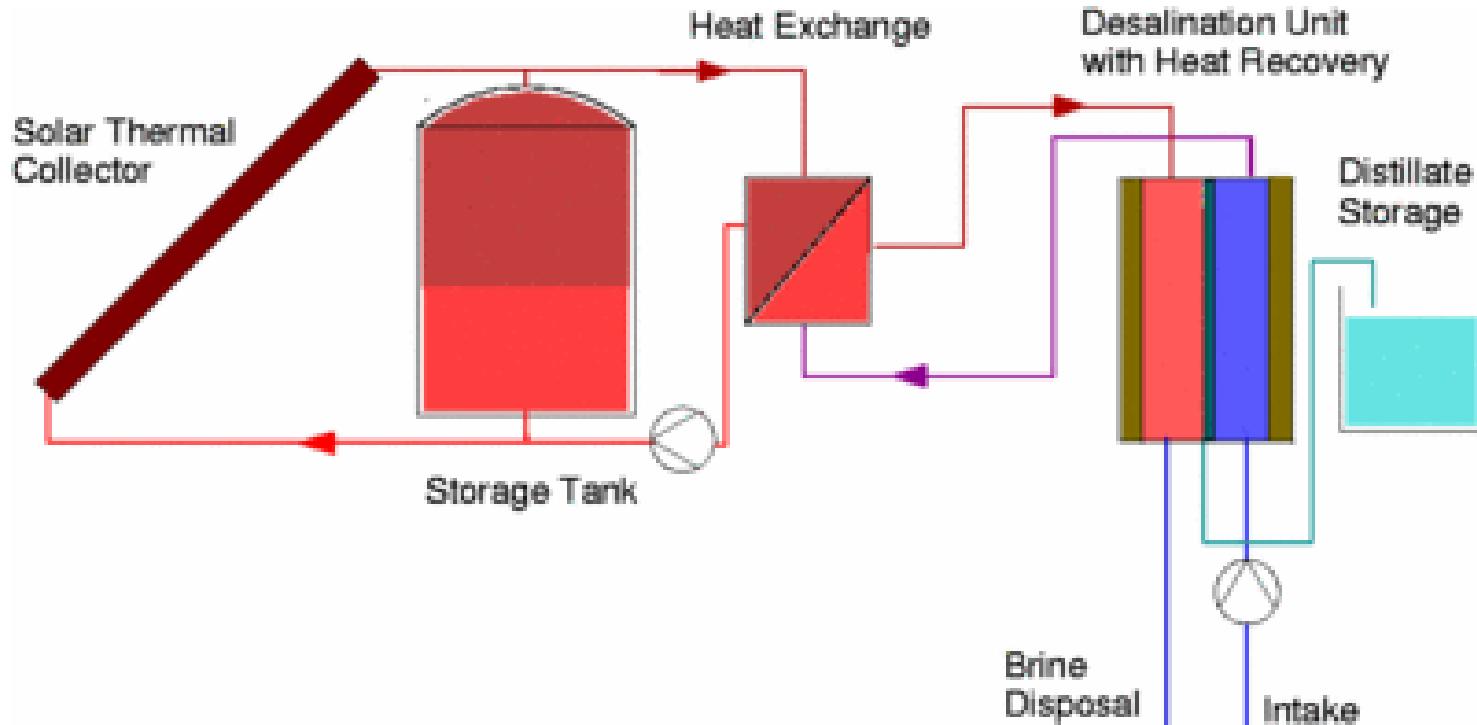


Water treatment systems coupled with renewable energy sources



Reduction of carbon footprint

Solar MD (Solarsprings)



Desalination

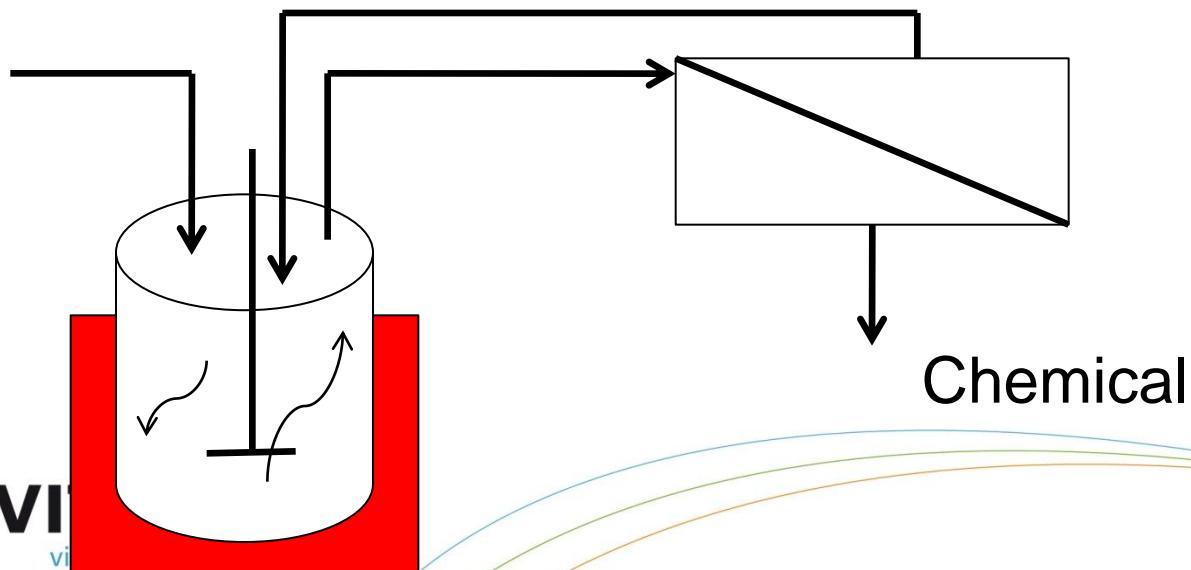
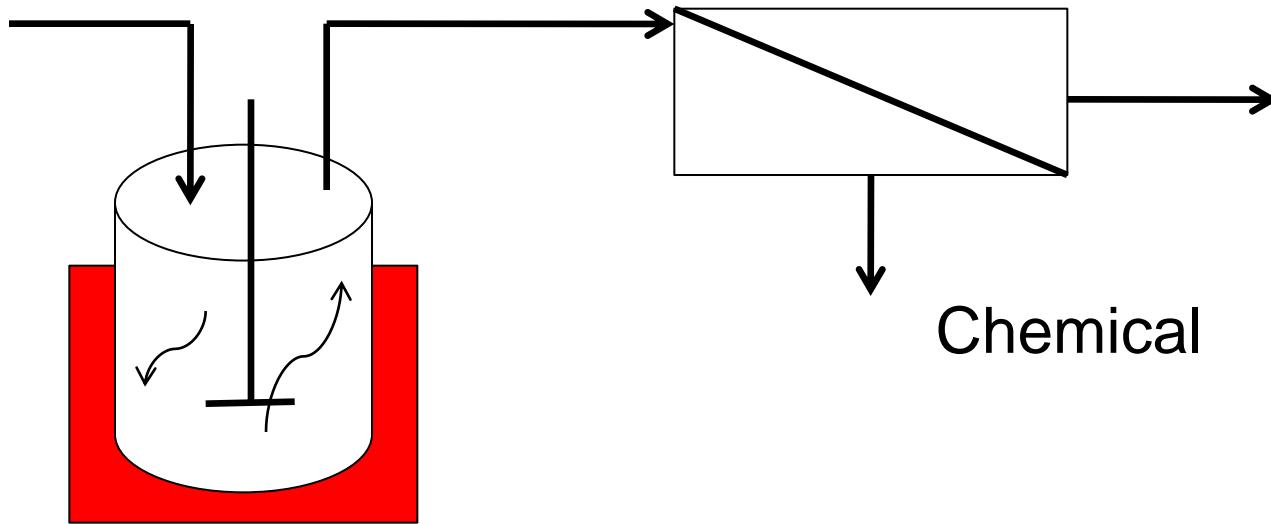
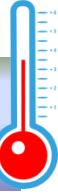


Ashkelon desalination plant (250 million \$) in Israel
produces 100 million m³/yr of desalinated water

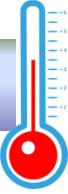
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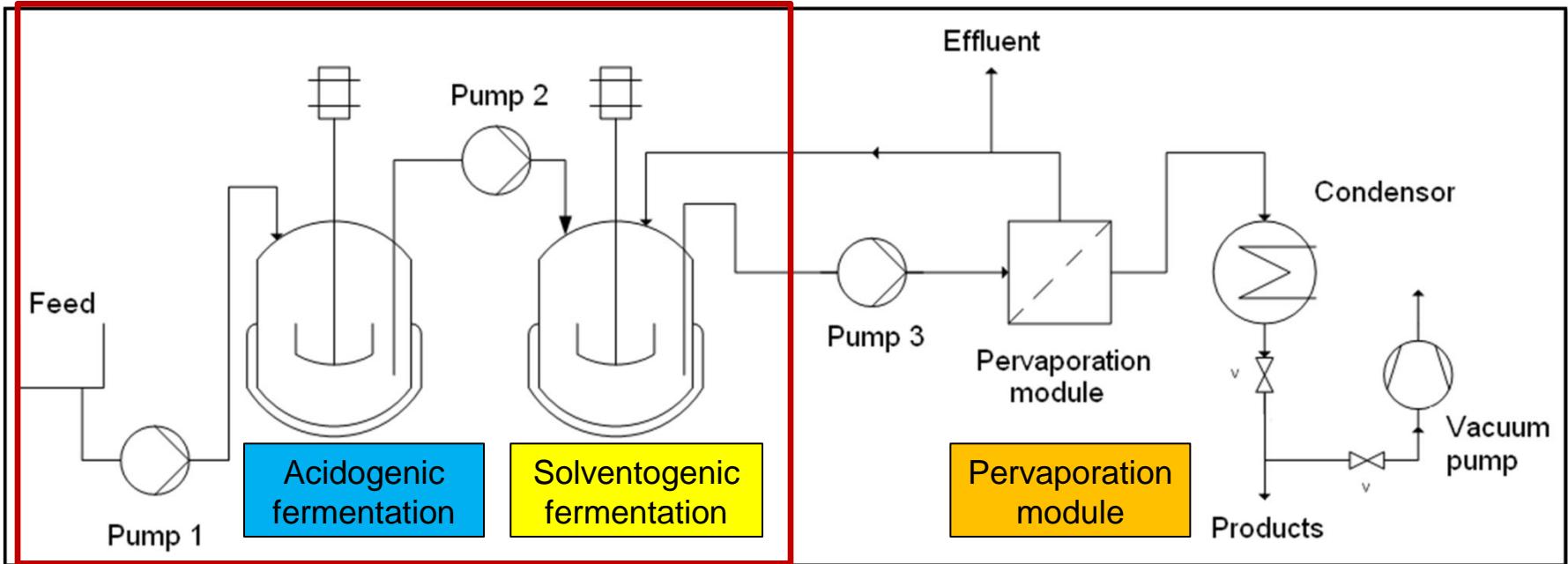
Integrate conversion with downstream processing



In situ alcohol recovery by organophilic pervaporation

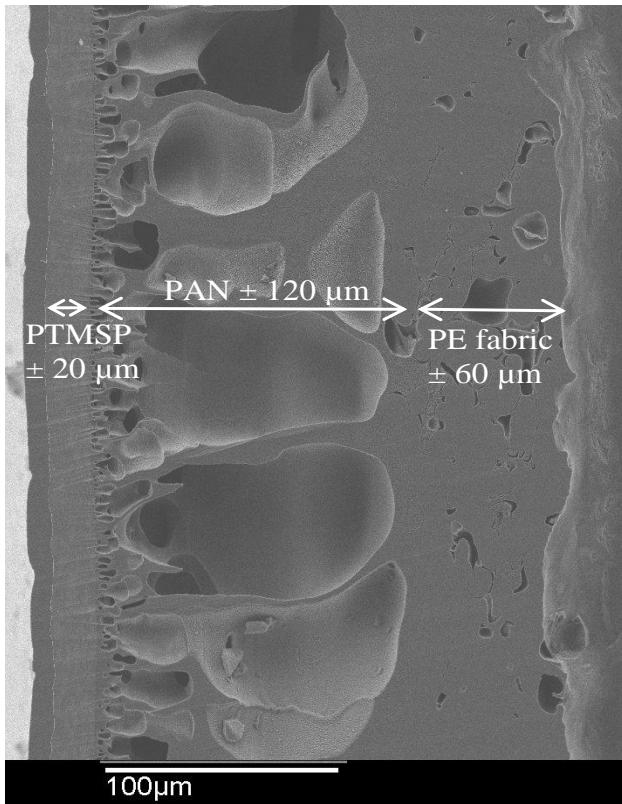


Integration of conversion and membrane separation to improve overall efficiency



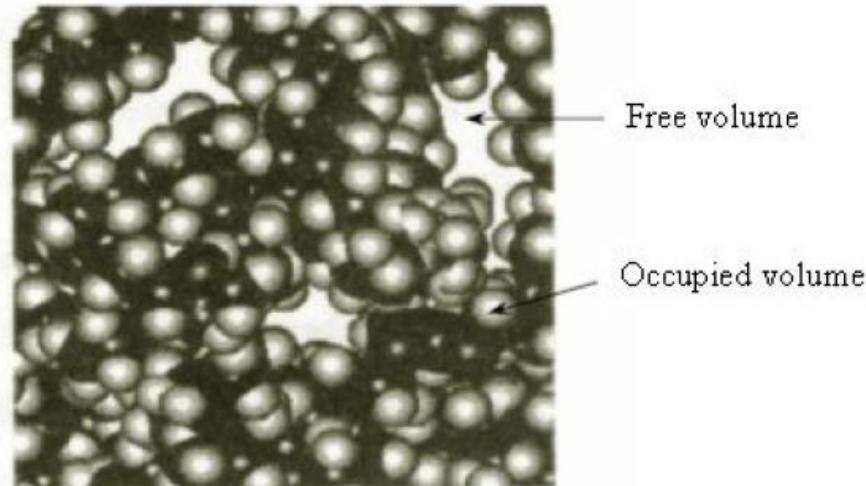
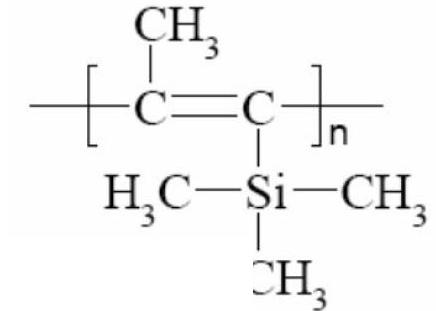
- » Pervaporate concentration from 60 – 180 g/L
- » Productivity enhancement of 87%
- » Use of concentrated feedstocks
- » Development of special membranes!!!

Development of novel OPV membrane for alcohol/water separations



» Mixed matrix membranes:

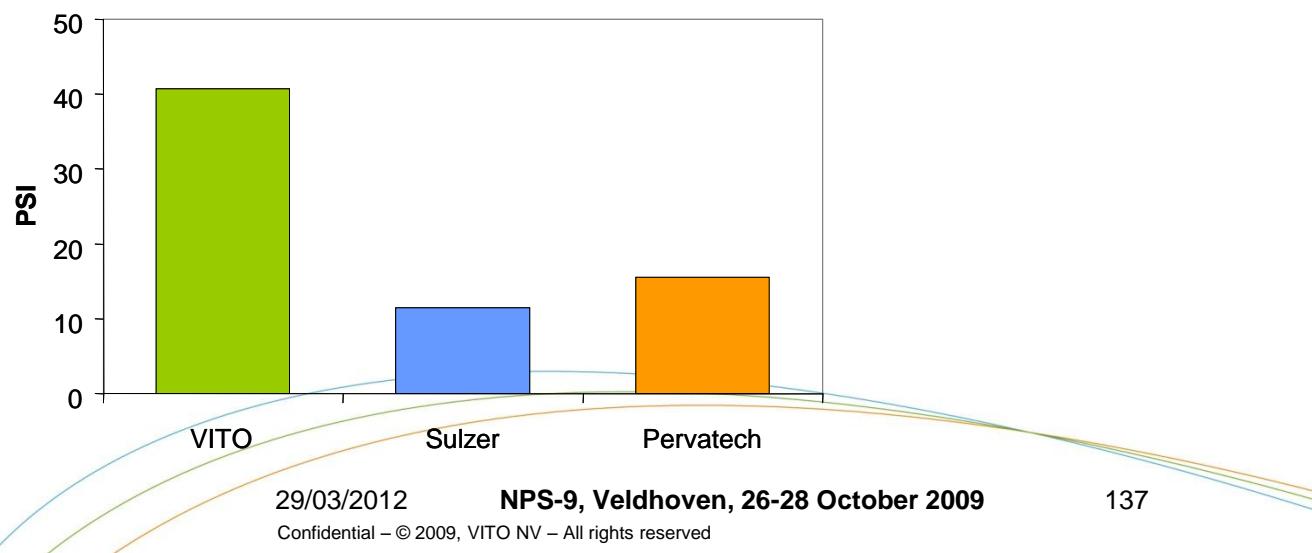
- ✓ PVDF support membrane on non-woven
- ✓ Thin layer of silica-filled PTMSP



OPV on EtOH/H₂O

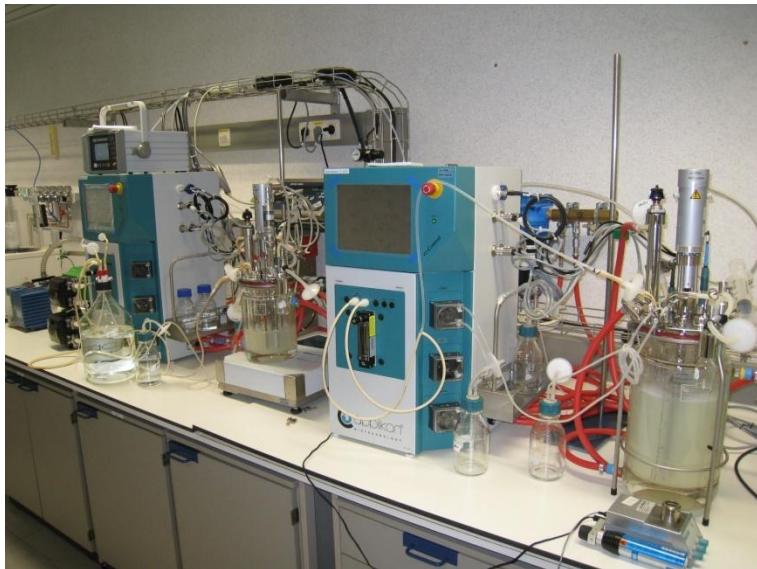
EtOH/H₂O (10 wt%); 50°C

Sample	Thickness selective layer (μm)	Flux (kg.m ⁻² .h ⁻¹)	α (EtOH/H ₂ O)	PSI
VITO	± 10	3.7 ± 1.3	12 ± 2	41
PERVAP 4060	± 2	1.9 ± 0.5	7 ± 2	11
Pervatech	± 2	3.3 ± 0.3	6 ± 1	15



In situ alcohol recuperatie door organofiele pervaporatie

2 staps butanol fermentatie

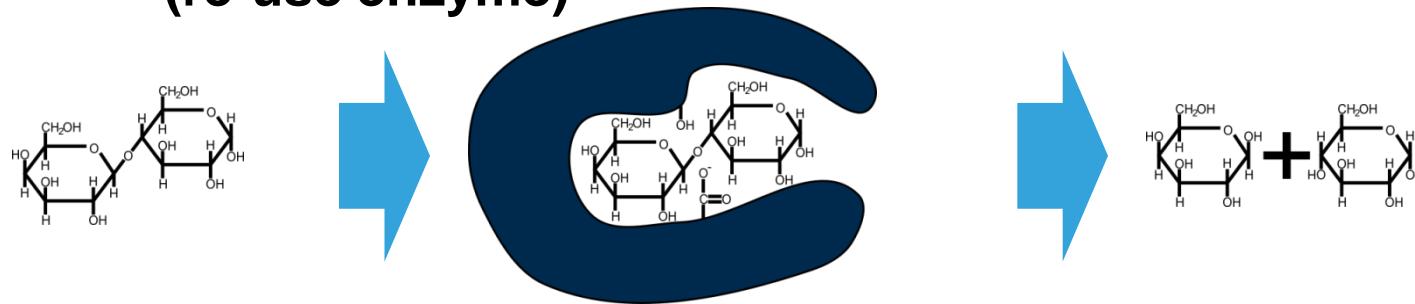


Geïntegreerde downstreamprocessing

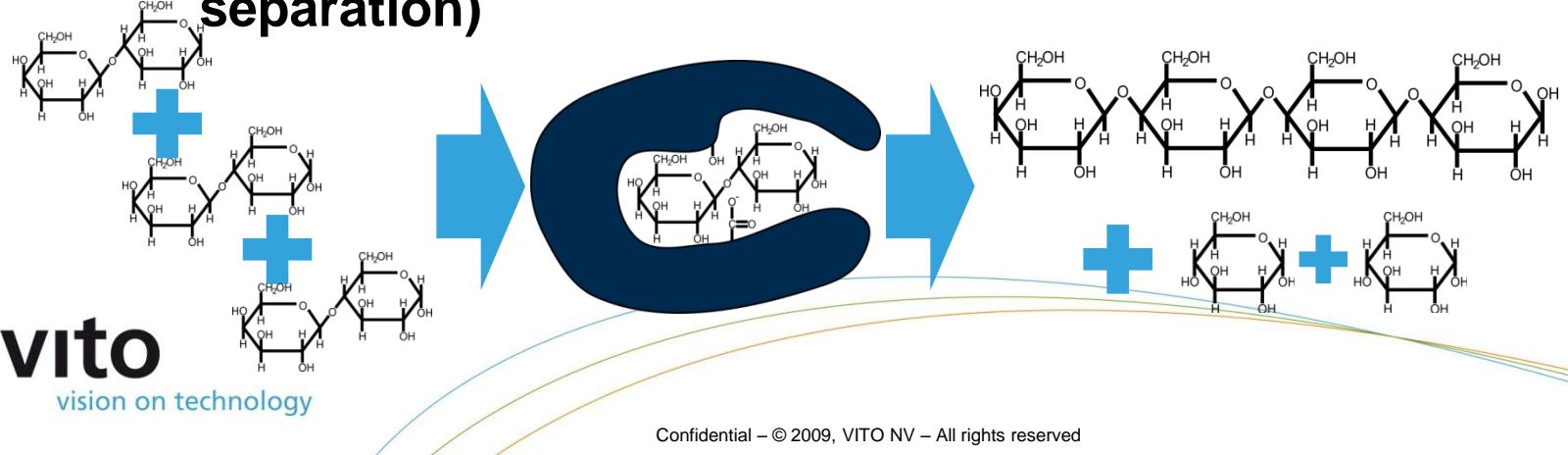


Three applications of enzyme immobilisation @ VITO

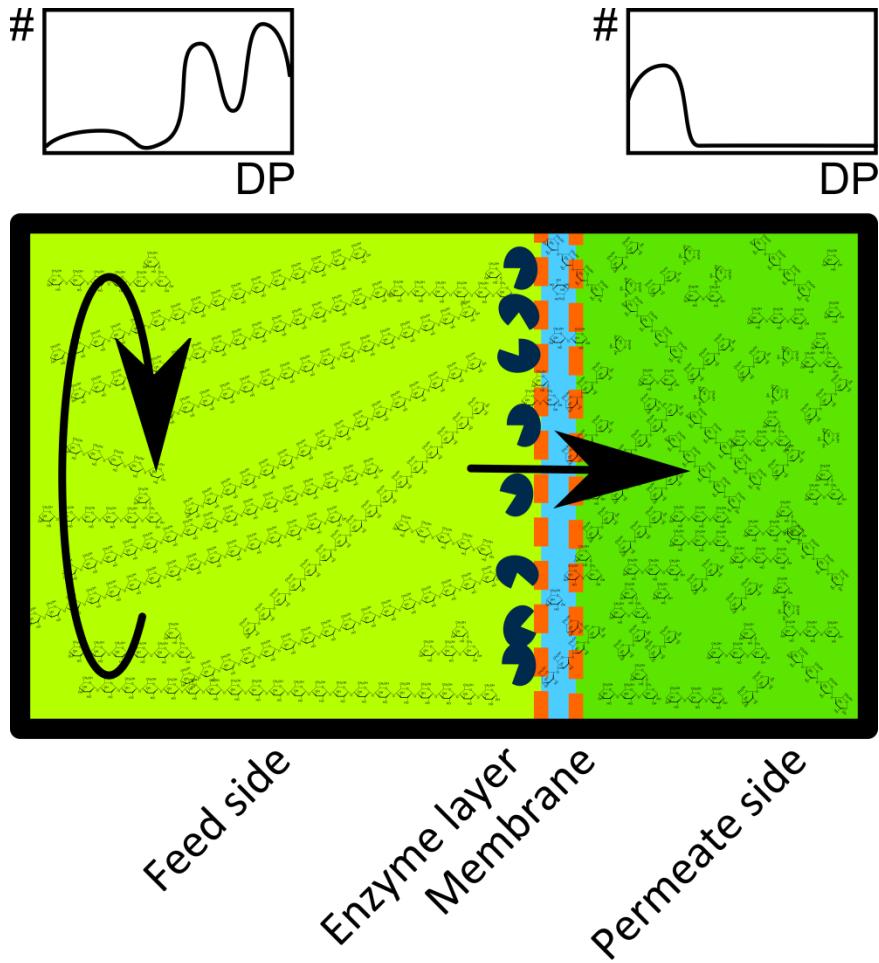
1. Hydrolysis of lactose by immobilized β -galactosidase (re-use enzyme)



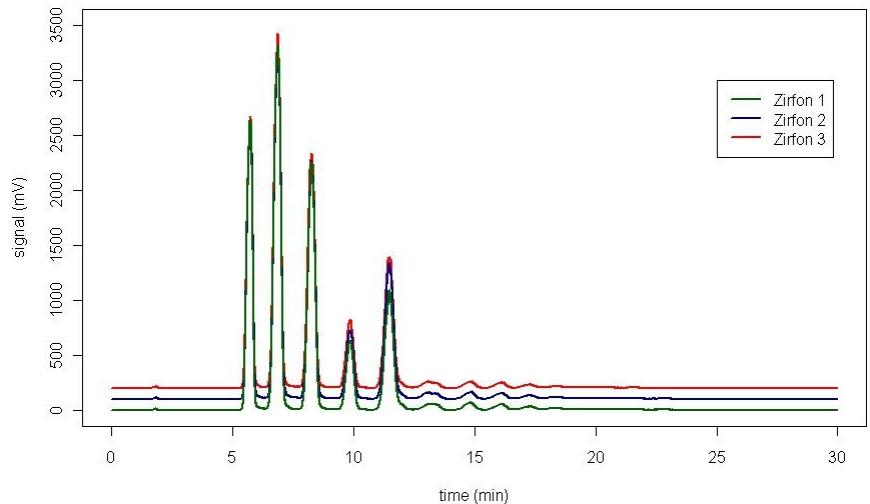
2. Synthesis of galacto-oligosaccharides (GOS) by immobilized β -galactosidase (re-use enzyme and separation)



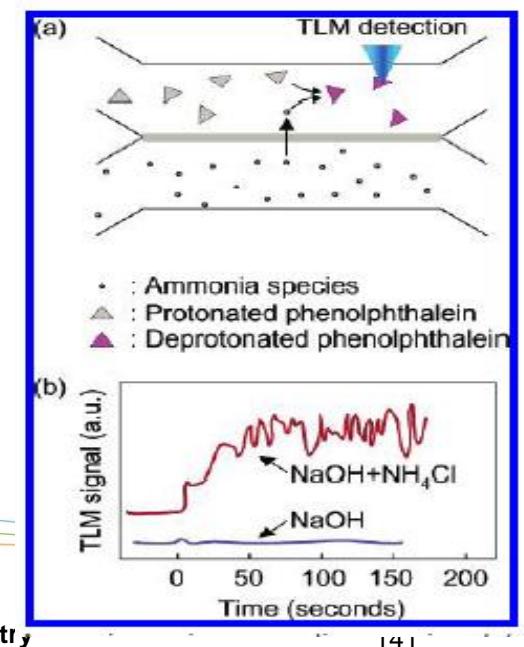
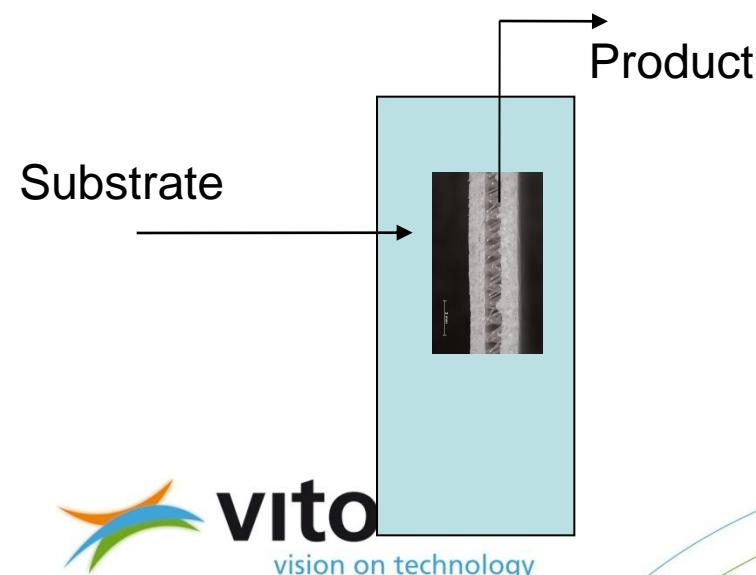
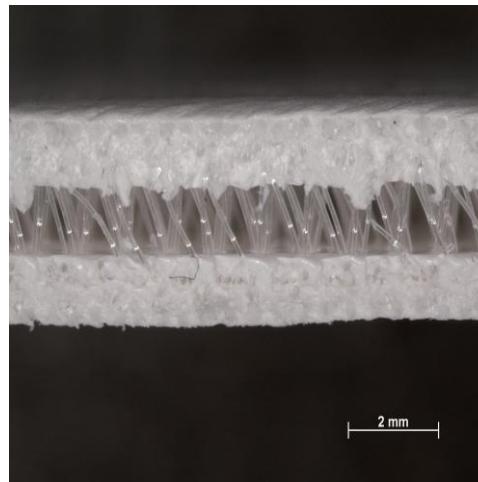
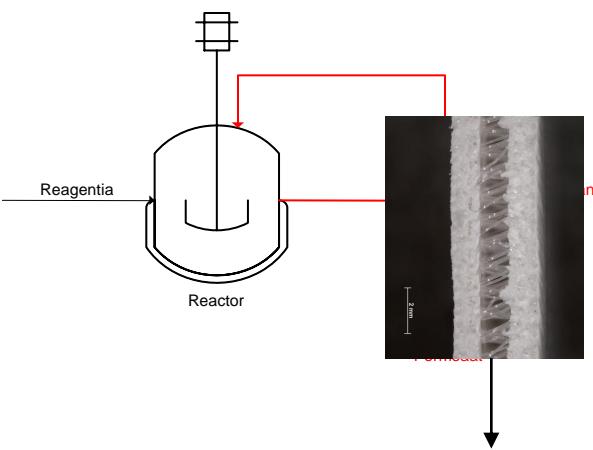
Enzyme immobilisatie



Hydrolysates of starch by alpha-amylase immobilized on zirfon membrane



IPC and membrane (bio)reactors



Solvent nanofiltration : SRNF

- » Cut-off 200 – 1000 Dalton
- » Retention small organic components
- » Charged membrane : retention multivalent ions

MF

0.1 - 3 bar
0.1 - 20 µm

UF

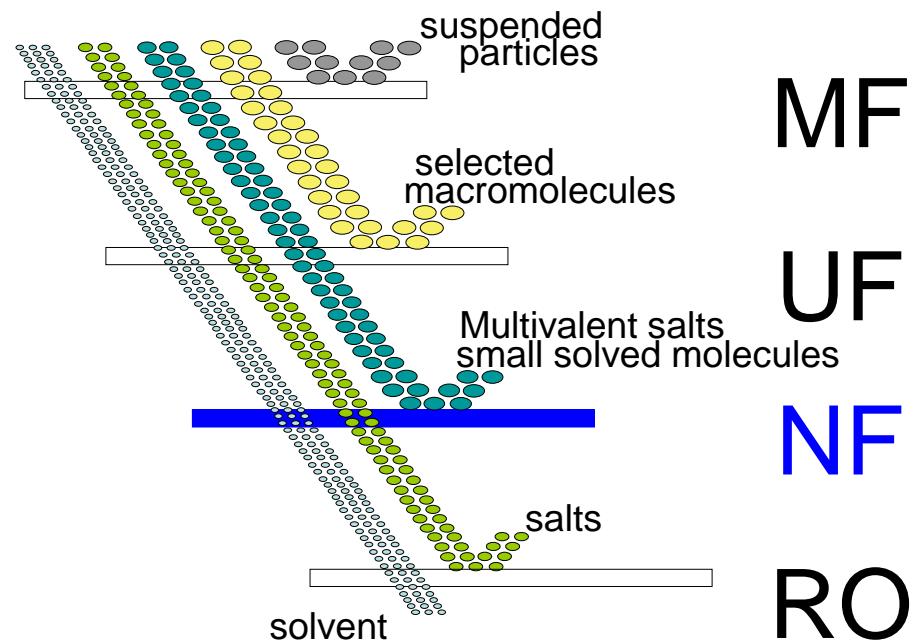
2 - 10 bar
2 nm - 0.1 µm

NF

5 - 30 bar
 \approx 1 nm

RO

10 - 100 bar
0.1 - 1 nm (dicht)



SRNF applications

- » Solvent recovery/recuperation :
 - Aceton in oleochemistry, hexane and ethanol in extractions, solvent in paint and polymer production,....
- » Color removal from waste streams e.g. in textile industry
- » Solvent exchange (high-boiling to low-boiling)
- » Concentration of antibiotics, pharmaceutics, ...
- » Purification of pharmaceuticals or other products :
 - Removal of impurities, higher polymers or other by-products
- » Separation /recovery of homogeneous catalysts
- » Coupling reaction and filtration :
 - By removal of products equilibrium shift of the reaction
- » ...

Implementations of ceramic NF membranes



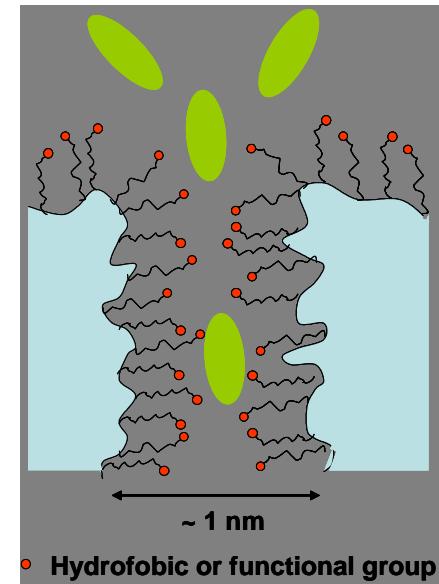
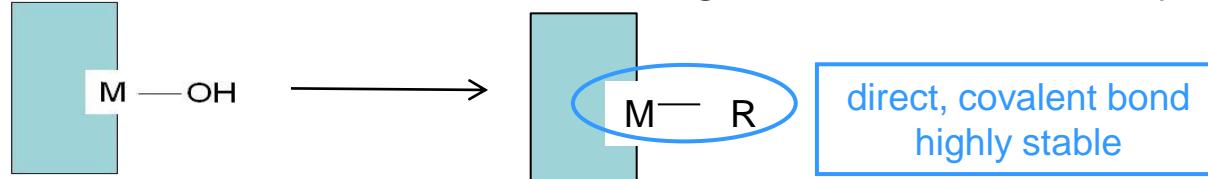
- modified in-line stream
- removal of NMP < 0.1 wt%
- diafiltration with water
- installation 24 m²
- flux 1 m³/h
- no product loss
- re-use of permeate ~100%



- colored textile waste water
- removal of color > 70 à 100 %
- concentration factor 10 à 20
- installation 25 - 65 m²
- flux 5 m³/h
- running since 2002

Need for better separation techniques

- Only rely on energy consuming separation technology?
- Post-modification based on organometal chemistry



- High-selective NF membranes (template assisted synthesis)

Template-assisted SiO_2 -layer
on dense support



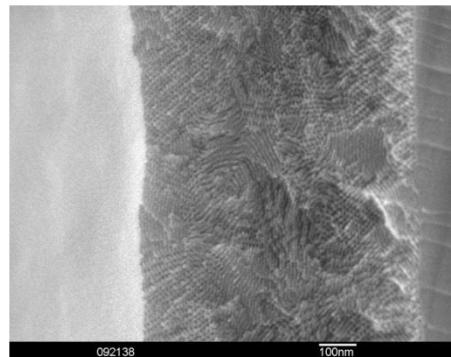
Template-assisted TiO_2 -layer
on porous support



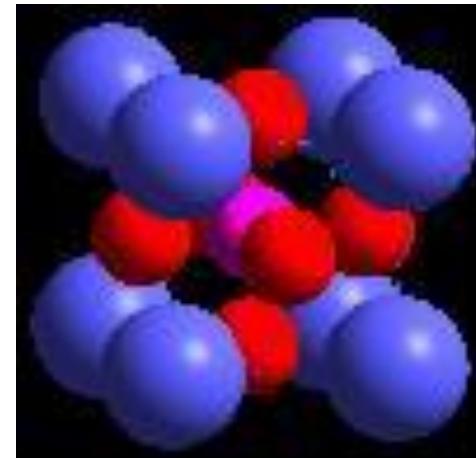
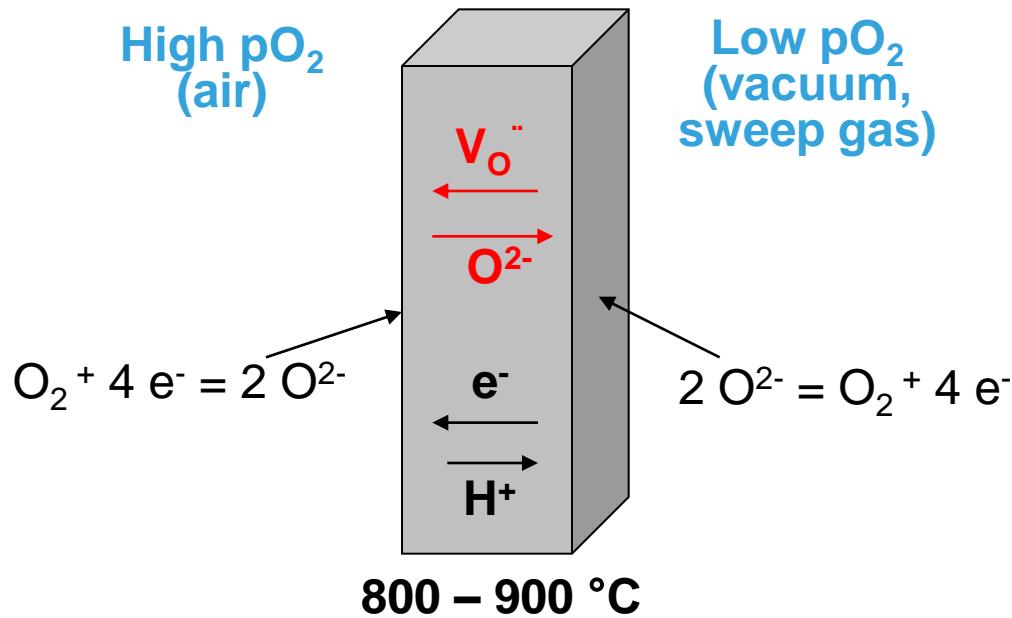
Uniform nano-pores



High-selective NF



Dense mixed ion/electron conductors (MIEC)

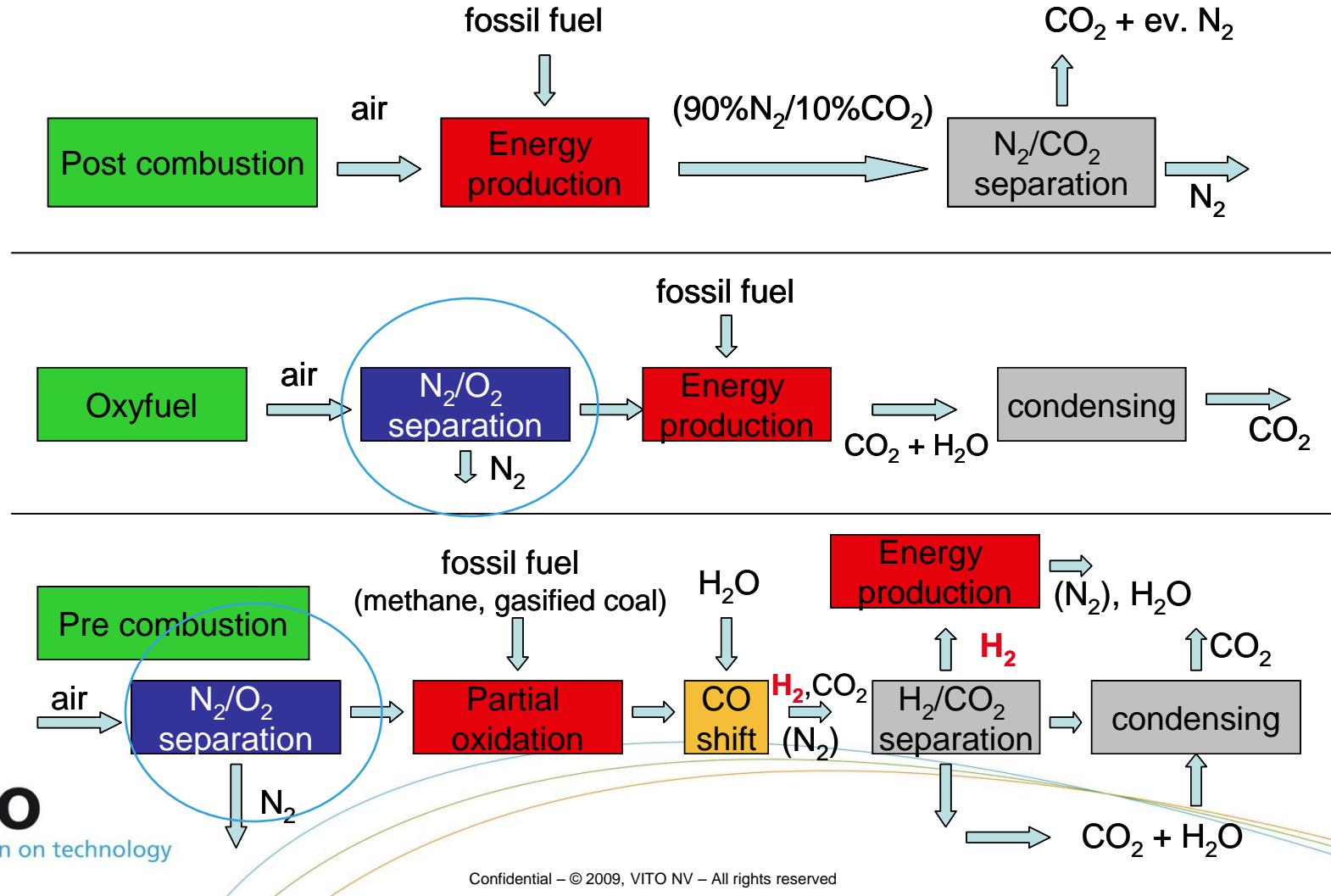


ABO_3 perovskite structure
High values of mixed conductivity

- » Partial substitution of metal cations by cations with lower valencies
 - formation of oxygen vacancies $V_O^{..}$
 - formation of oxygen non-stoichiometry : $\text{AA}^*\text{BB}^*\text{O}_{3-\delta}$
 - possibility of oxygen to be transported over the material
- 100% selectivity
- » Oxygen non-stoichiometry depends on doping, pO_2 and T
- » Multivalent cations ensure high electron conductivity

Energy application of mixed ion electron conductors

- » Gas separation in zero-emission power plants
- » Very large installations necessary!

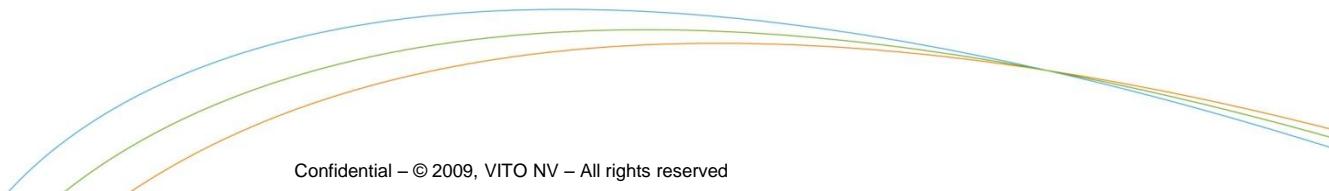


Membrane geometries

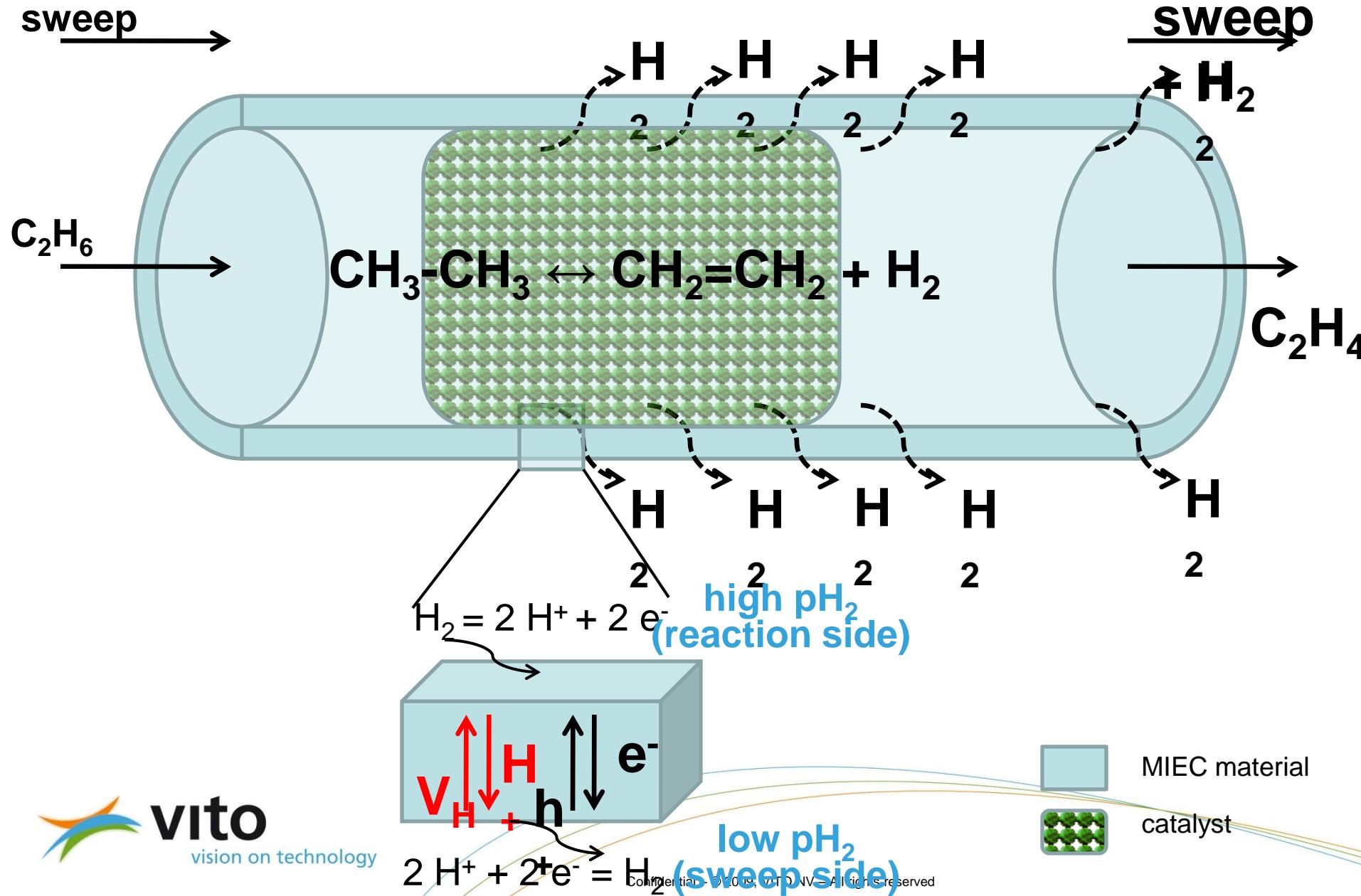


Membrane geometry	Surface/volume ratio
Flat (plate & frame)	$\sim 100 \text{ m}^2/\text{m}^3$
Tubular ($d > 5\text{mm}$)	$100-500 \text{ m}^2/\text{m}^3 (r = 5\text{mm})$
Tubular honeycomb ($d = 2.5\text{mm}$)	$800 \text{ m}^2/\text{m}^3$
Capillaries ($0.5 < d < 5\text{mm}$)	$500-4000 \text{ m}^2/\text{m}^3 (r = 0.5\text{mm})$
Hollow fibres ($d < 0.05\text{mm}$)	$4000-30000 \text{ m}^2/\text{m}^3 (r = 0.05\text{mm})$

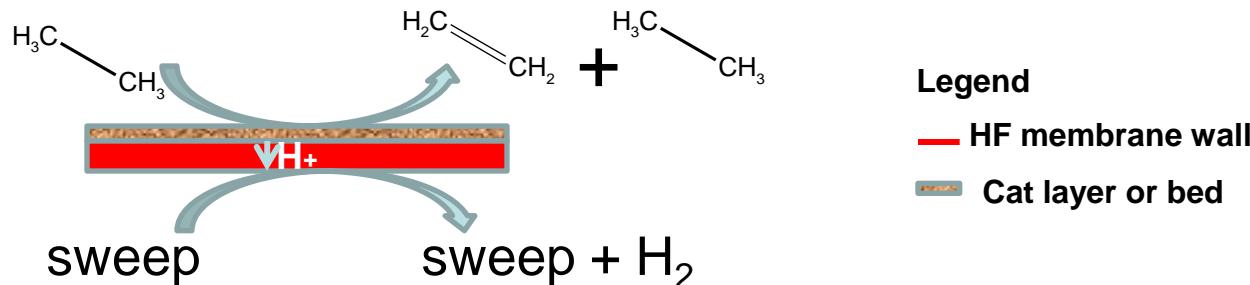
→ interesting for large scale gas separation applications



Concept of MIEC hollow fiber membrane reactor for dehydrogenation reactions



CMR: example for dehydrogenation of ethane (2)



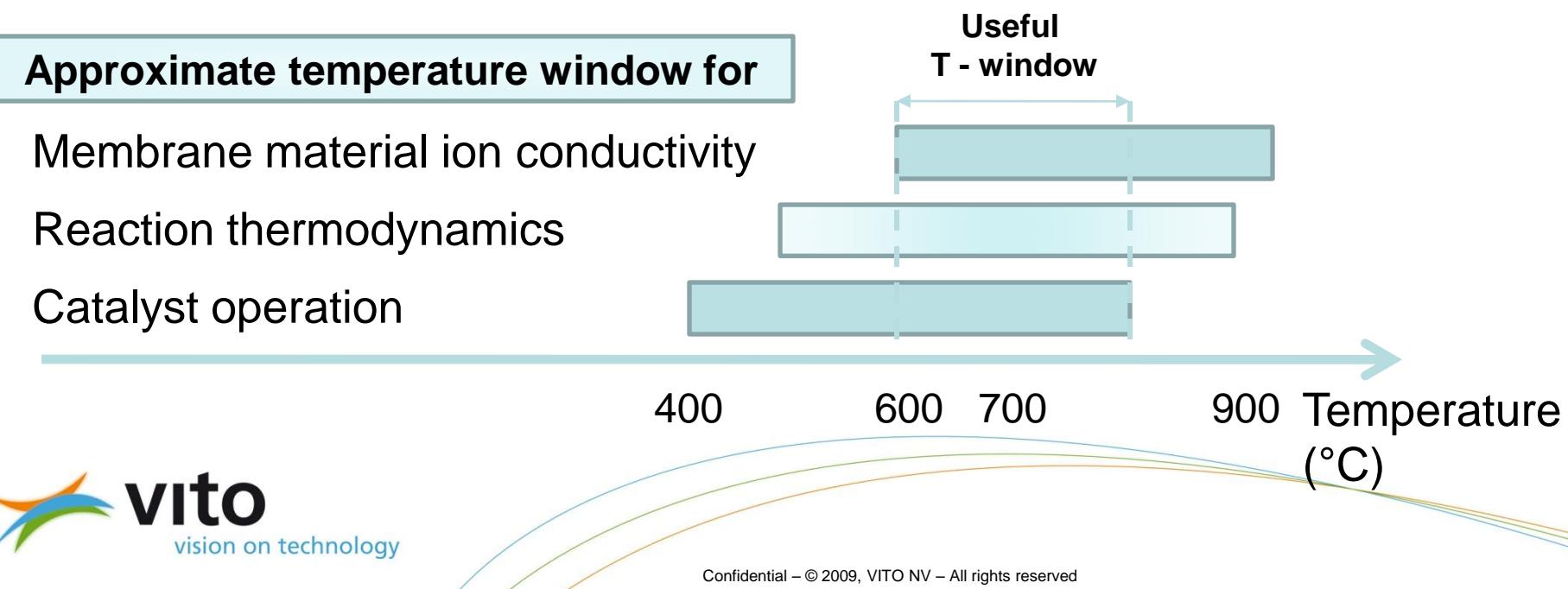
Approximate temperature window for

Membrane material ion conductivity

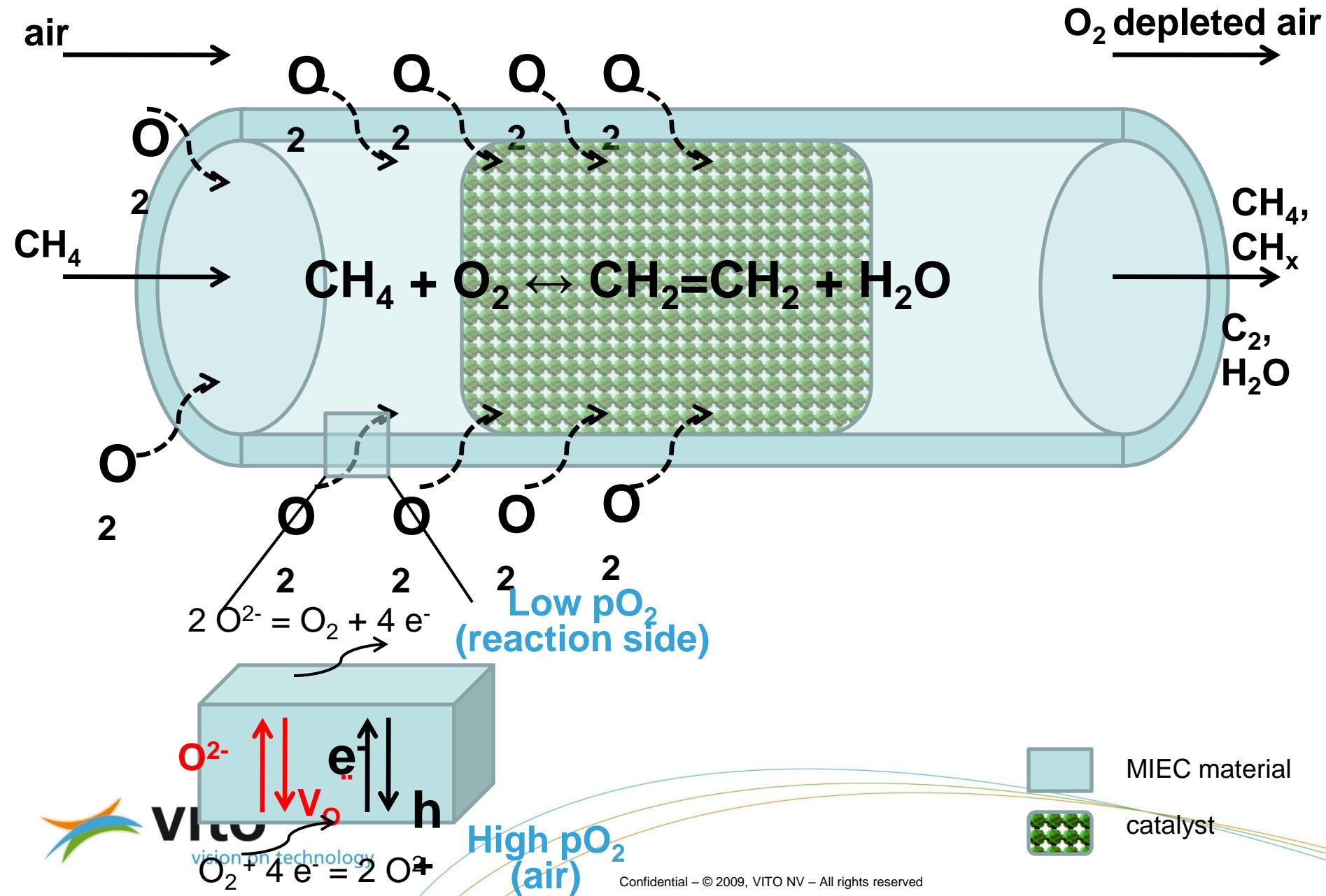
Reaction thermodynamics

Catalyst operation

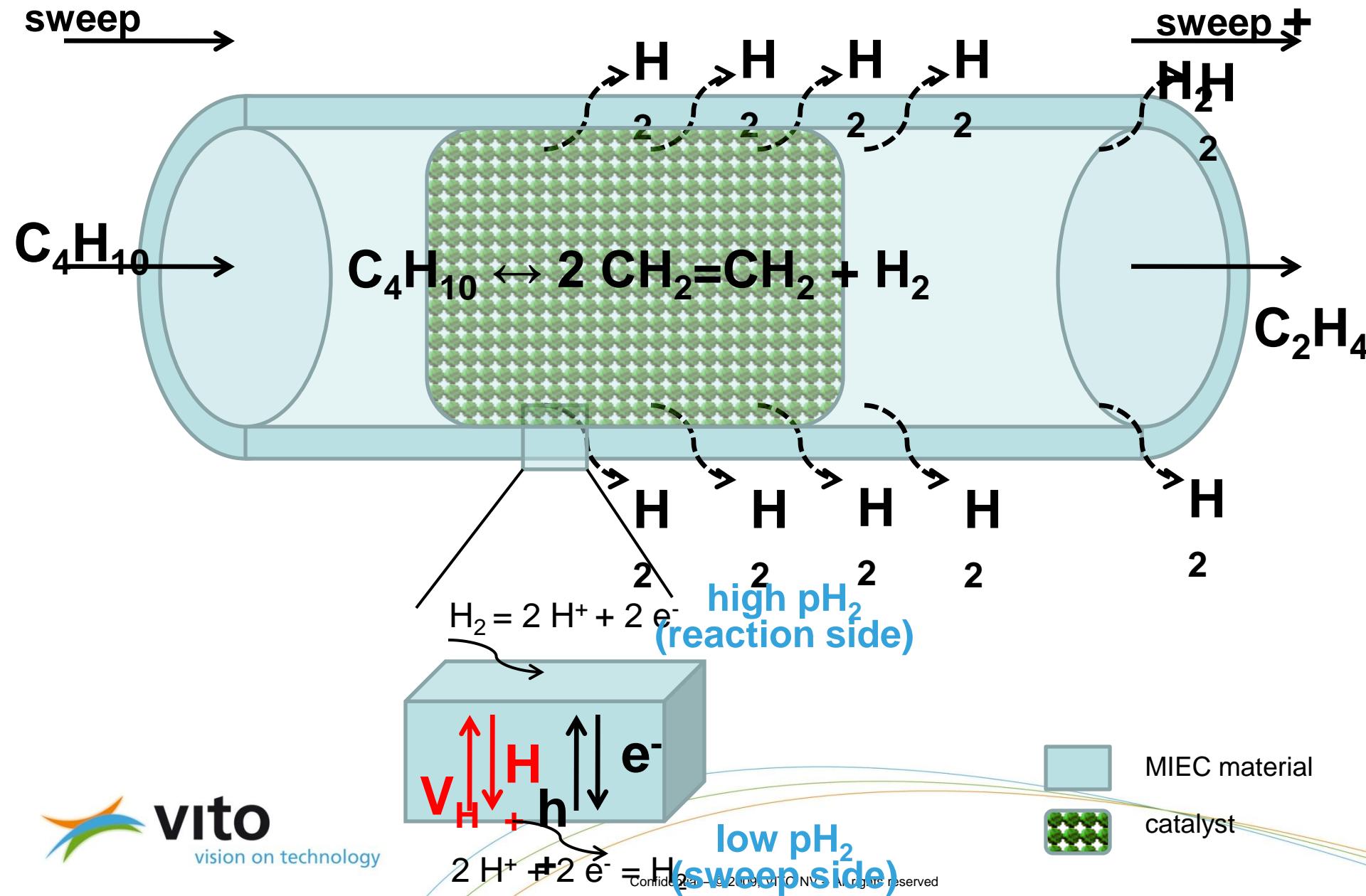
Useful
T - window



Concept of MIEC hollow fiber membrane reactor for oxidative coupling of methane



Concept of MIEC hollow fiber membrane reactor for dehydrogenation/cracking reactions



OUTLINE

1. Inleiding
2. Membranen / Modules en ontwerp
3. Procesparameters en fouling
4. Overzicht waterige membraantoepassingen
5. Case-studies membranen voor waterzuiveringstoepassingen
6. Overzicht en case-studies niet-waterige membraantoepassingen
- 7. Conclusies**

CONCLUSIES

- ✓ Het is technisch mogelijk om quasi elke waterkwaliteit te maken
- ✓ Goedwerkende installaties vragen echter een duidelijke kennis van ontwerp- en applicatieparameters
- ✓ Duidelijke (milieu)voordelen maken van membraantechnologie “best beschikbare-techniek”
- ✓ Technische + economische haalbaarheid: erg gevalspecifiek !
- ✓ Aandacht voor concentraatstromen !
- ✓ Membranen geschikt voor organische solventen
- ✓ Membraanreactoren: combinatie van conversie en scheiding

The background image shows a sunset over a calm body of water. The sky is a gradient from orange at the top to a darker blue at the horizon. Silhouettes of palm trees and other tropical foliage are visible along the far shore. The water's surface is slightly rippled, reflecting the warm colors of the setting sun.

WATER

Our Global Common

Meer info?

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