MEMBRANE WINTER SCHOOL

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EINDHOVEN

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1
Arkema group
Presentation : A Leader
in Specialty Materials

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20,200
talents

11,020
in Europe

3,660
in North America

5,520
in Asia and the rest of the world

over
10,000
customers

Collaborations with the leading global brands

WOMEN AND MEN AT THE HEART OF OUR DEVELOPMENT

€9,5 BN
sales
in 2021

Presence in

55
countries

141
plants

16
R&D centers

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2
Carling’s plant and acrylic monomers business unit presentation

Video

Research Center building – 30 years old

R&D laboratories

Process department

Analysis laboratories
(R&D and control)

Management building
(GM, HR, Technical departments…)

Continuous Process improvements

On the field
4 reactors of 15 000 to 20.000 tubes

New catalyst tests

Impurities effect

Life cycle enhancement

New operating parameters

On the catalyst hall
5 reactors of 1 tube

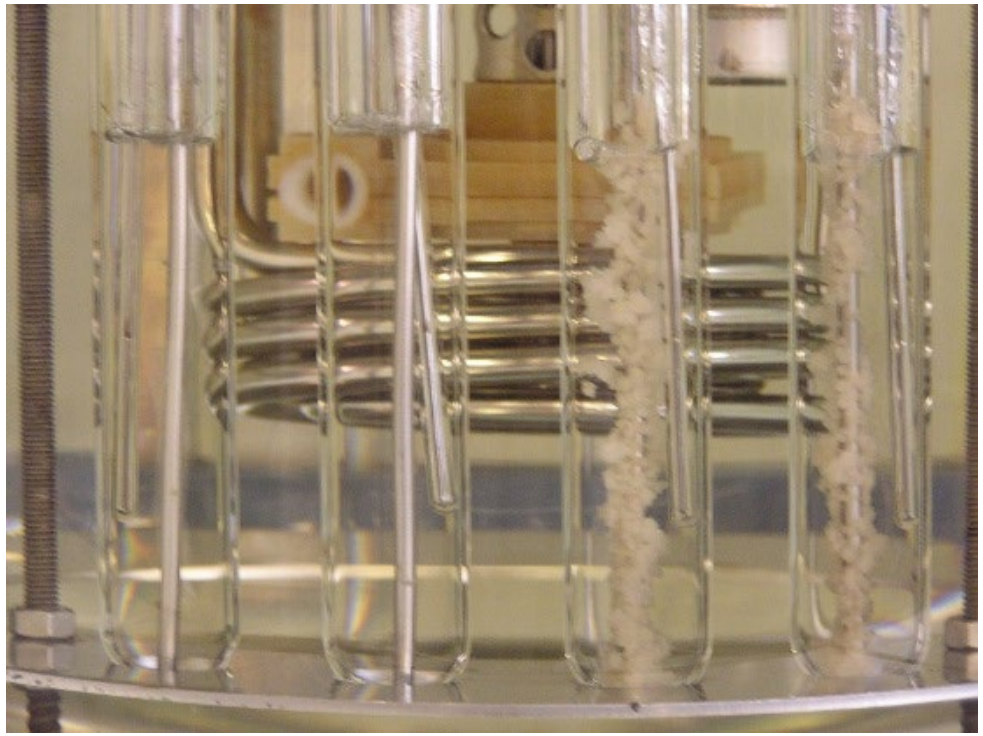
Continuous Process improvements

On the field

Fouling issues

Troubleshooting

Laboratory



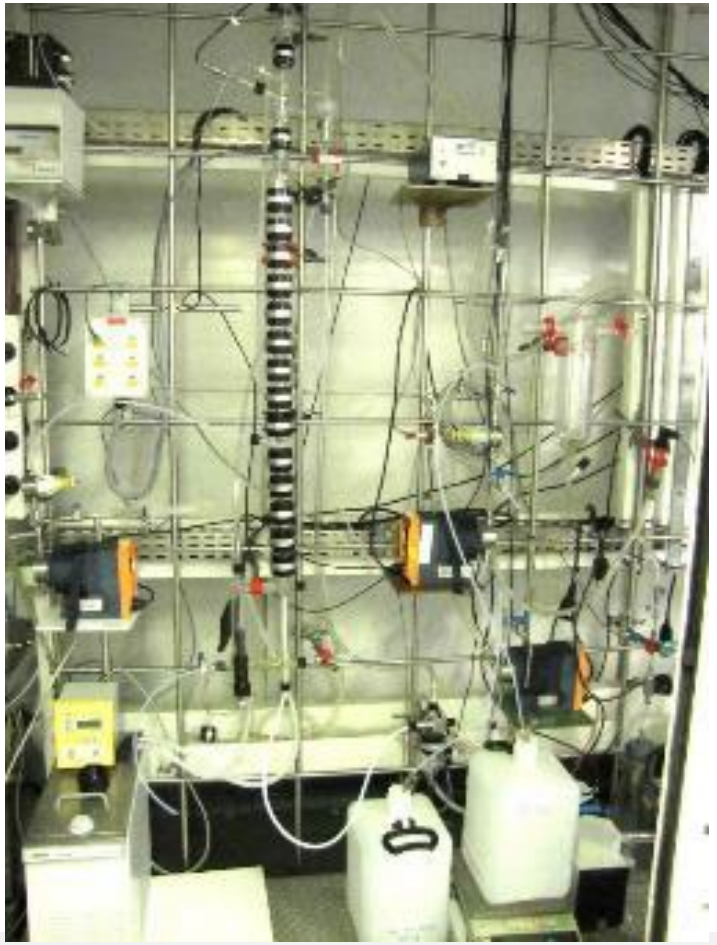
Continuous Process improvements

On the field
800 t/d distillation train

New monomer stabilizations

New operating parameters

In the lab
2 kg/d distillation



Process pilot plant

Link to the industriel unit …

with real reaction gases


3
ALsys – Orelis a expert in filtration

ALSYS
HEADING FOR THE FUTURE

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Group of Specialties:

- Advanced materials
- Process engineering
- Industrial equipment

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In a nutshell

1EASURED

ALSYS
HEADING FOR THE FUTURE

A group of **specialty products** & **services**

5 entities with unequivocal **expertise & know-how**

Experts in **filtration & catalysis technologies**

Focus on **sustainable innovation & growth**

CTI

ORELIS environnement

enerCat Une énergie d'entreprise

CLEARBAKK WATER SOLUTIONS

CeraMem®

5 entities

150 PEOPLE

3 PRODUCTION SITES

40 M€
2018 TURNOVER

PRESENCE IN
14 countries

EACH YEAR
20% invested IN R&D

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Building a Group through organic growth, international development and acquisitions

CTI	ORELIS environnement	enerCat	CLEARBAKK WATER SOLUTIONS	CeraMem®
Salindres (France)	**Salindres (France)**	**Lorient (France)**	**Calgary (Canada)**	**Boston (USA)**
Advanced ceramics & catalytic materials	Membrane materials & filtration processes	Catalytic formulation & advanced materials	Full package & water treatment solutions	Membrane materials & filtration processes
Founded in 1990 Acquisition of CTI by the management team in 2009	Founded early 80's Joined in 2010	Founded in 1990 Joined in 2013	Founded in 2011 Joined in 2016	Founded in 1986 Joined in 2017

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3 Business Units to leverage the Group's ambitions

ALSYS
HEADING FOR THE FUTURE

Business Units	Liquid & Membrane	Gas & Catalysis	Specialty Materials
Entities involved	CTI, ORELIS environnement, CLEARBAKK WATER SOLUTIONS, CeraMem®	CTI, enerCat Une énergie d'entreprise	CTI, enerCat Une énergie d'entreprise

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A wide range of added-value products

Liquid & Membrane

- Ceramic membranes: KLEANSEP™, CERAMEM ®
- Polymeric membranes: PLEIADE ®
- Filtration skids systems: KLEANSEP™, CERAMEM ®
- Waste-water treatment plants: CLEARBAKK
- Polymer makedown / injection plants: CLEARBAKK

Gas & Catalysis

- NOx treatment (NO, NO_2, N_2O)
- Trapping (sulfur S, carbon compounds…)
- Catalytic oxidation (CO, COV…)
- Hot gas filtration
- Catalytic processes & energy production (hydrogen & methane)

Specialty Materials

- Molten metal filters
- Fire bricks
- Antimicrobial agents

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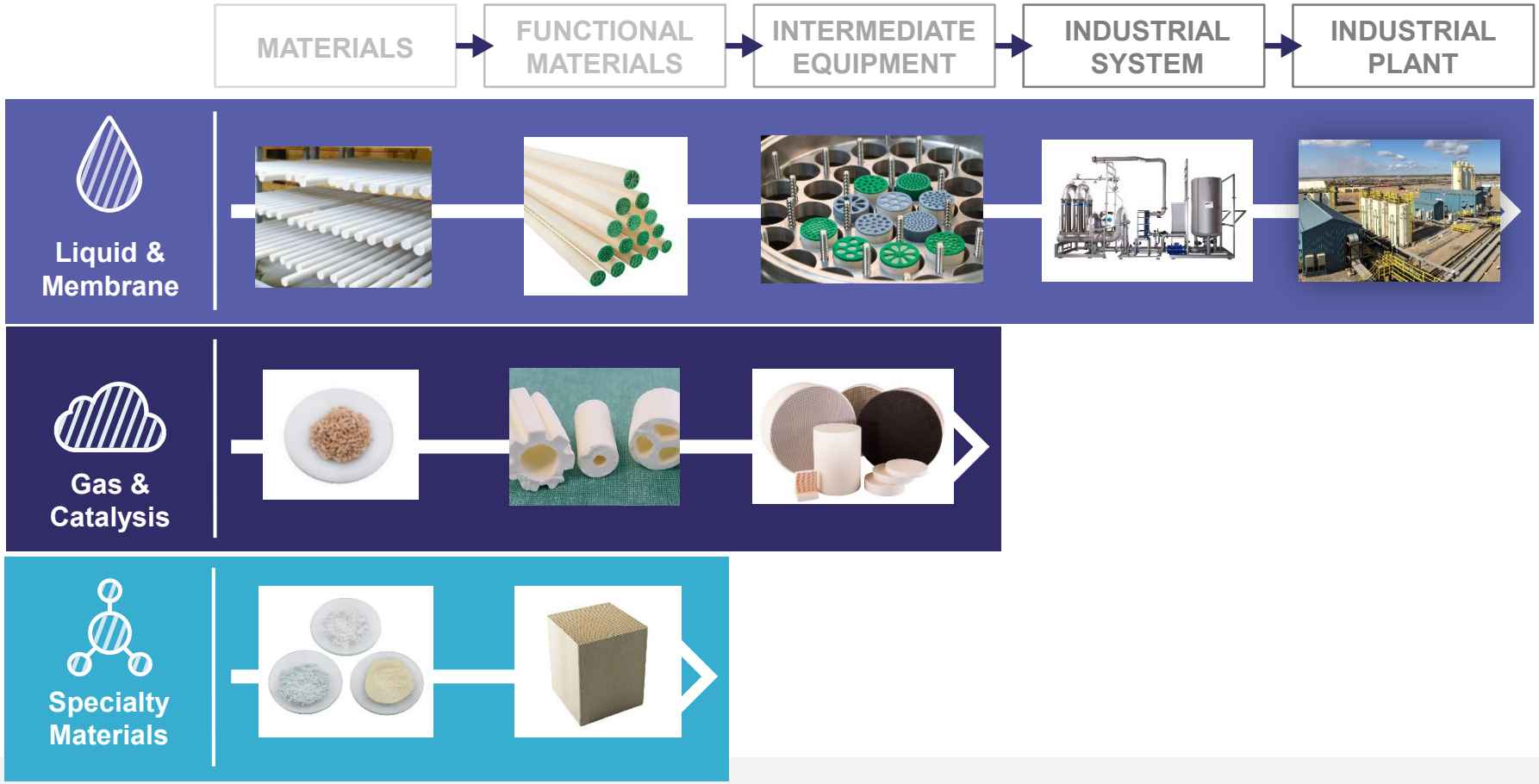
An integrated value chain to ensure competitiveness and sustainable growth

Value chain integration from materials to advanced & complete technologies supporting customer performance



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Liquid & Membrane Business Unit
Key membrane markets

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Liquid & Membrane

Recycle Water and Liquids		Process	
Water	Oily Liquids	Chemicals	Feed & Food
Treatment of industrial, urban waste water through cross-flow filtration technologies		Production of chemicals, pharmaceuticals, animal feed or human food ingredients through cross-flow filtration technologies	
Bio-reactor treatment Landfill leachate, Liquid waste treatment plant …	**Oil & Gas processes** Upstream: Produced water from oil well, … Downstream: Refinery …	**Bio-based formulations and materials** Organic Acids, Diols, Lignin derivatives, Micro-algae …	**Fermentation-based ingredients** Antibiotics, Amino Acids, Vitamins, Enzymes …
Other Water processes Paint bath recycling, Laundry water recycling, Recovery of metals or chemicals …	**Oily Water treatment** Oily emulsions from steel & metal industry, Bilge water …	**Chemicals** Organic pigment, Alcohol dewatering by pervaporation …	**Sugar, Starch & Beverages** Sugar juice, Glucose syrup, High-Fructose Corn syrup, Wine, Milk derivatives …

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Some references of liquid filtration skids

Application: Steel Galvanizing industry wastewater

Feed: Alkaline and/or oily wastewaters, acidic wastewater and cooling water

Two separate lines:
- Chromium removal: 4 skids of 24 membranes. Flow rate of 50 m^3/h (220 gpm)
- Nickel removal: 1 skid of 12 membranes

Location / Customer: Leipzig, Ohio (USA). Pro-Tec (US Steel / Kobe Steel)

Application: Oil & Gas produced water (steam enhanced oil recovery)

Capacity: 50 000 bpd treatment capacity (330 m^3/h – 1450 gpm)

System: 6 skids of 38 membranes each – 228 membranes in total

Location: California (USA)

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Some references of liquid filtration skids

Application: MBR agri-food industry wastewater

Integrated unit into a container :

- **UF system** made of ceramic membranes KLEANSEP™, flow rate of 50 m^3/day (9.2 gpm). The purified water at the outlet is used for cleaning and feeding the gas scrubber
- **RO system** made of polymeric membranes PERSEP™, flow rate of 20 m^3/day (3.7 gpm). The water at the outlet is used to feed a cooling tower and the steam boilers

Location: La Reunion island (Indian ocean)

Application: Laundry waste water recycling

UF system made of ceramic membranes KLEANSEP™. The purified water is used to feed the laundry washer equipment. Flow rate of 540 m3/day (99 gpm).

Location: France (Europe)

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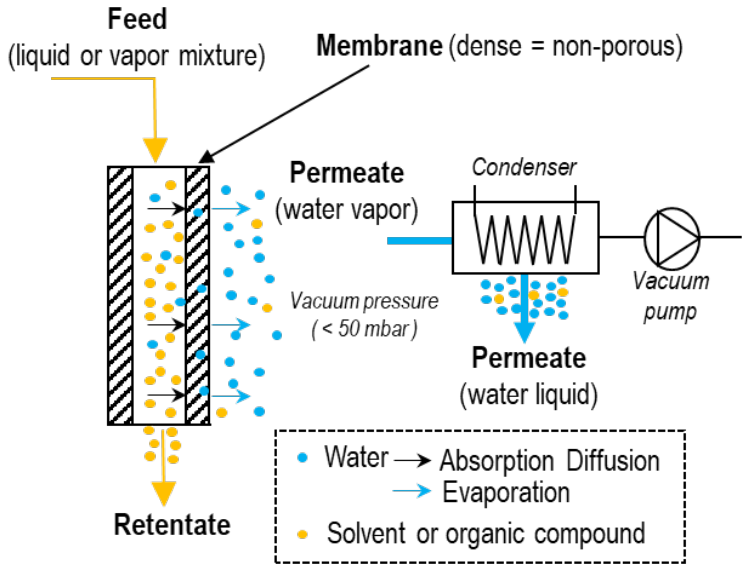
Pervaporation

Innovating for the sustainability and reliability of industrial processes



Main benefits of pervaporation vs. distillation or molecular sieves

- 70% less energy: only latent heat duty for permeate evaporation
- 30% less CAPEX: simple process with compact installation and low foot-print
- No waste generated
- No product contamination
- Low temperature separation possible, compatible with heat sensitive products

Industries	Applications	Benefits
• Fine chemistry • Flavor & Fragrances • Food & Beverage	• Concentration of aromatic mixtures	• Simple process • Unaltered stability of aromatic compounds
• Pharmaceutical • Biofuels • Chemicals • Petrochemicals	• Alcohols dehydration (Ethanol, IPA, Butanol, Acetone, Organic acids…) • Esterification mixtures dehydration • Azeotropic breaking	• High selectivity • Low energy consumption • Minimum process modification • Batch or continuous processes

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Pervaporation

Pervaporation membranes and test performances

- ORELIS provides the **Hybsi® and Zeolite-A** technology on tubular **multichannel ceramic membranes** which offer the highest resistance and permeate flux on the market.
- ORELIS owns a versatile range of **pilot plants** to provide best feasibility and process design studies.

Performances with hydrophilic ceramic membrane

Feed composition // Membrane	Membrane	Temperature (°C)	Flux (kg/h. m²)	Permeate composition
95% n-Butanol, 5% Water	Hybsi®	120	12	3% n-Butanol, 97% Water
88% Ethanol, 5% Methyl isobutyl ketone, 7% Water	Hybsi®	70	2,5	80% Water
92% Ethyl acetate, 2% Ethanol, 2% Toluene, 1% Acetic acid, 3% Water	Hybsi®	70	1,5	87% Water
Ester acrylate, Alcohol, Acrylic acid, 15% Water	Hybsi®	80	12	Ester acrylate, Alcohol, Acrylic acid, 96% Water
Water, 30g/L Polyphenols and aromatic compounds	Hybsi®	40	2,5	Containing traces of organic compounds
95% Ethanol, 5% Water	Zeolite-A	120	4,5	2% Ethanol, 98% Water

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Pervaporation: Versatile offers with multiple choices

- Lab **or** industrial scale
- Pervaporation **or** vapor permeation
- Ceramic Hybsi ® **or** Zeolite-A membrane
- Dehydration at steady conditions **or** extremely quick purification
- Manual **or** automatic control

OneD-Pervaporation Pilot
Tabletop laboratory pilot

- Feed Volume : 2-5L
- Max Temperature : 80°C
- Permeation capacity : 0,5kg/h
- 1 electrical plug 220V, 1,4kW
- No need for liquid N_2

K01 Pervaporation Pilot
Mobile industrial pilot

- Feed Volume : 10-20L
- Max Temperature : 120°C
- Permeation capacity : 3kg/h
- 1 electrical plug 3p-400V 5kW
- Datalogging

Pervaporation Skid Unit
Mobile production skid

- Feed Volume : 20-500L
- Max Temperature : 150°C
- Permeation capacity : 20kg/h
- Automation with touchscreen panel

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Pervaporation: One-D pilot

Technical data

- Feed tank volume: 2 liters (optional: 5 liters)
- Dead volume: 10 ml (undrainable)
- Maximum permeate extraction capacity: 0,55 kg/h

- Maximum feed temperature: +80°C
- Minimum condensation temperature: -20°C
- Minimum permeate pressure: 11 mbar

- Membrane material: HybSi® or Zeolite-A
- Membrane type: short multichannel, inside → outside
- Membrane area: 500 cm² (optional: mono-channel membrane)

- Condensation mode: continuous (optional: N_2 liquid trap)

- Wetted surfaces: SS316L, SS304L, PP, PTFE, FFKM, FKM

- Foot-print (Length x Width x Height): 500 x 700 x 1000 mm
- Weight: 65 kg

- Utility: 1 electrical plug 230 VAC mono, 1,4kW
- Cooling by air

- Instruments: electronic thermometers, flowmeter, manometers, level detector with process security protection

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Pervaporation: K01 pilot

Technical data

- Feed tank volume: 10 liters (optional: 20 liters)
- Dead volume: 50 ml (undrainable)
- Maximum permeate extraction capacity: 3,2 kg/h

- Maximum feed temperature: +120°C
- Minimum condensation temperature: -10°C
- Minimum permeate pressure: 17 mbar

- Membrane material: Hybsi® or Zeolite-A
- Membrane type: long multichannel, inside → outside
- Membrane area for Hybsi: 0.15 m² (optional: 0.25 m², 0.5 m²)
- Membrane area for Zeolite-A : 0.09 m²

- Condensation mode: continuous

- Wetted surfaces: SS316L, PTFE, FFKM, FKM

- Foot-print (Length x Width x Height): 800 x 1300 x 1800 mm
- Weight: 260 kg

- Utility:
  - Electrical plug 400 VAC three phase + neutral + ground, 5kW
  - Cooling by water or by air

- Instruments: electronic thermometers, flowmeter, manometers, level detector with process security protection and datalogger

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Pervaporation: industrial skid unit

Technical data

- Internal feed tank volume: 20 liters (optional: 500L external vessel)
- Dead volume: 1L (undrainable)
- Maximum permeate extraction capacity: 20 kg/h

- Maximum feed temperature: +150°C
- Minimum condensation temperature: -10°C (optional : -50°C)
- Minimum permeate pressure: 20 mbar

- Membrane material: Hybsi® or Zeolite-A
- Membrane type: long multichannel, inside → outside
- Membrane area for Hybsi®: 1.0 m² (optional : 0.15 – 3.0 m^2)
- Membrane area for Zeolite-A : 0.6 m² (optional : 0.09 - 1.9 m^2)

- Condensation mode: continuous

- Wetted surfaces: SS316L, PTFE, FFKM, FKM

- Foot-print (Length x Width x Height): 2900 x 1600 x 2300 mm
- Weight: 700 kg

- Utility:
  - Electrical plug 400 VAC three phase + neutral + ground, 35kW
  - Cooling by water or by air

- Instruments: electronic thermometers, flowmeter, manometers, level detector. Process automation with user-friendly touchscreen panel.

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4

Acrylate monomers

Acrylate esterification chemistry

- Esterification (Commodity acrylate esters)
  - CH_2=CH-COOH + ROH → CH_2=CH-COOR + H_2O

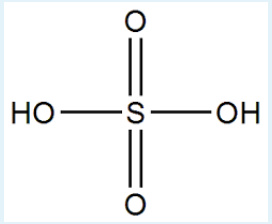
R = methanol, ethanol, butanol, 2-ethyl hexanol

- Transesterification (Specialized acrylate esters)
  - CH_2=CH-$COOR_1$ + R_2OH → CH_2=CH-$COOR_2$ + R_1OH

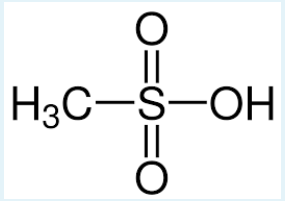
(Light ester) (Heavy alcohol) (heavy ester) (light alcohol)

Ex. Dimethyl aminoethyl acrylate (CH_2=CH-COO-CH2-CH2-N(CH_3)$_2$)
Methoxyethyl acrylate (CH_2=CH-COO-CH2-CH2-O-CH3)

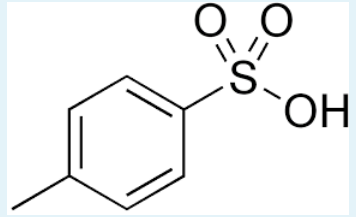
Strong acid catalysts for esterification



Sulfuric acid

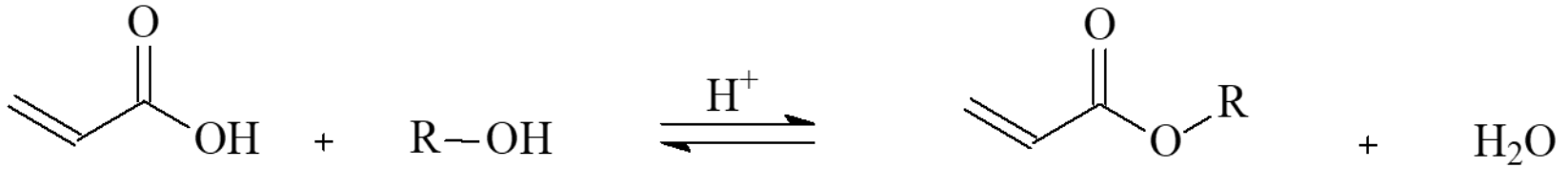


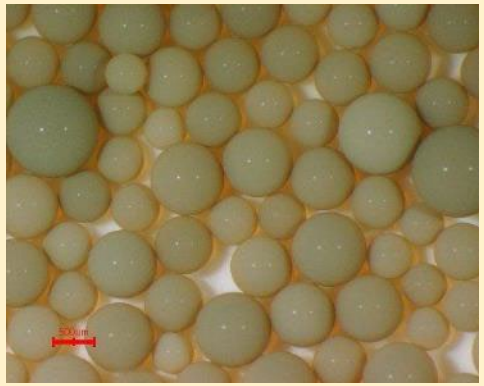
Methylsulfonic acid



Paratoluensulfonic acid

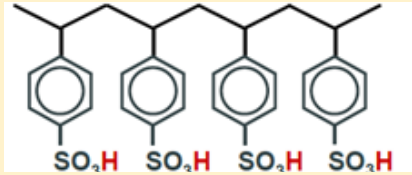
Homogeneous catalyst

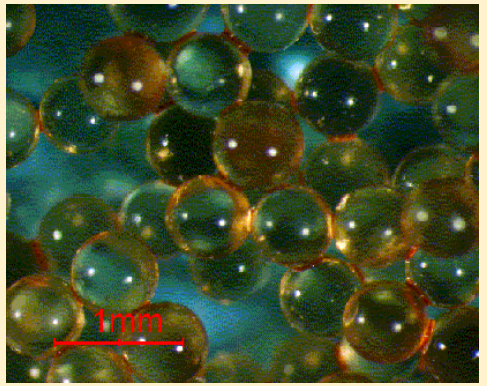




Heterogeneous catalyst

Cationic Exchange resin

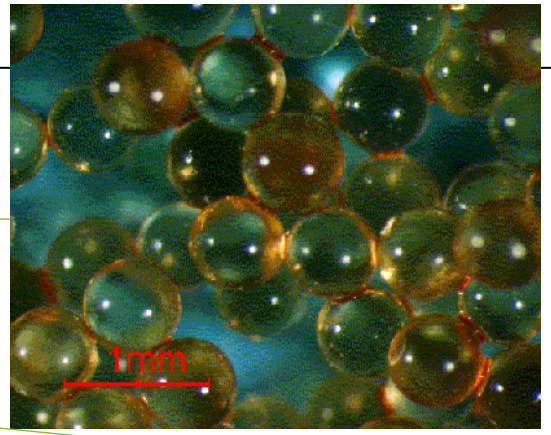




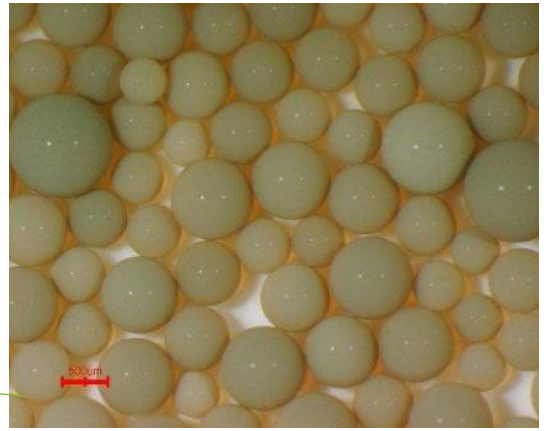
Strong acid catalyst : main commercials characteristics

→ Resin type : gel or macroporous :

without porogene



with porogene



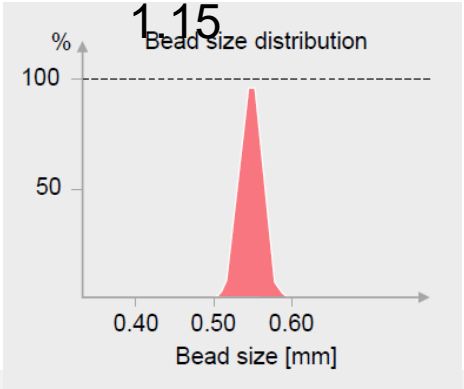
→ Capacity : Nb of H+ of resin

1.1 to 2 eq mole H+ / liter or 4 to 6 eq mole H+ /kg dry

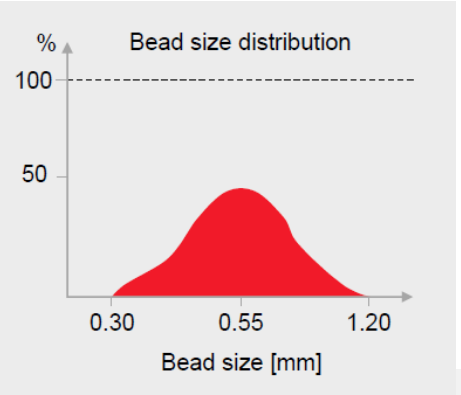
→ Granulometry : (effect on pressure drop)

- **Uniformity coefficient UC**
- **Harmonic mean Diameter about 0.7 mm**
- **Fines : Particle size < 300 microns % ≤ 0.1 %**

Jetted polymerization UC = 1.15



Suspension polymerization UC = 1.6



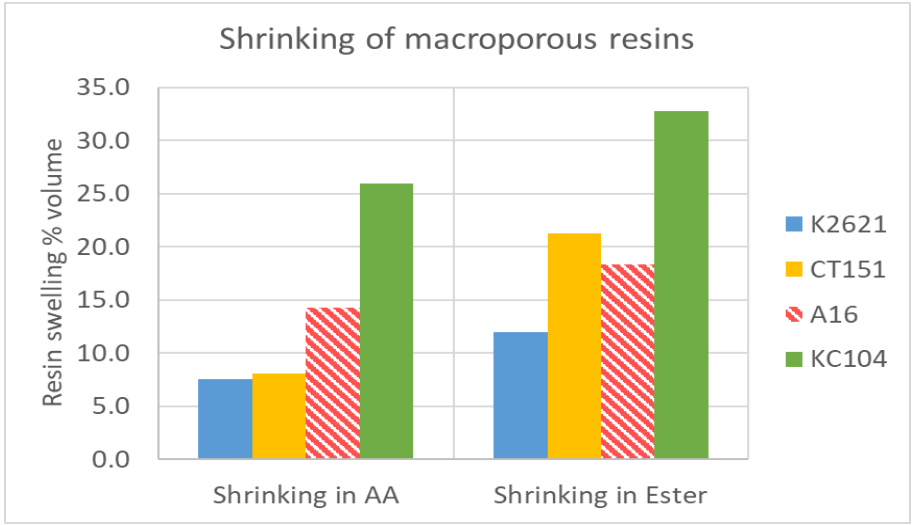
→ Moisture retention

- **50 to 70 % of water**

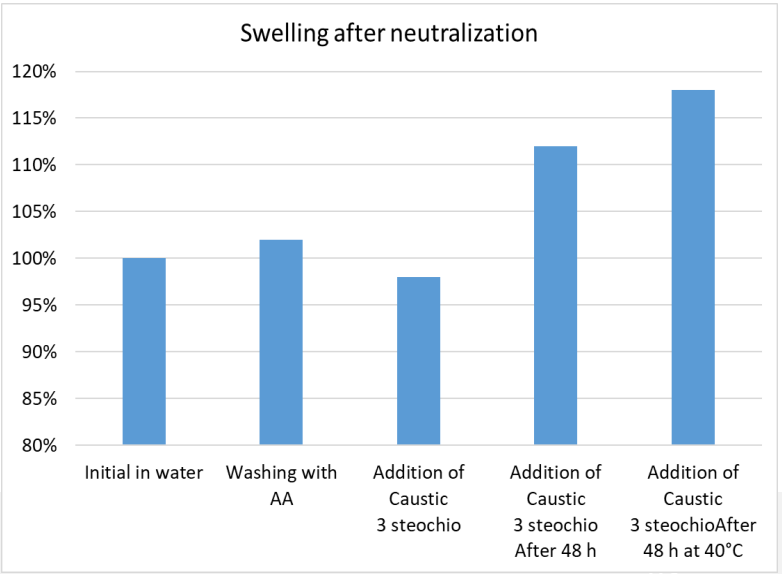
Strong acid catalyst : main usage characteristics

→ Swelling / Shrinking

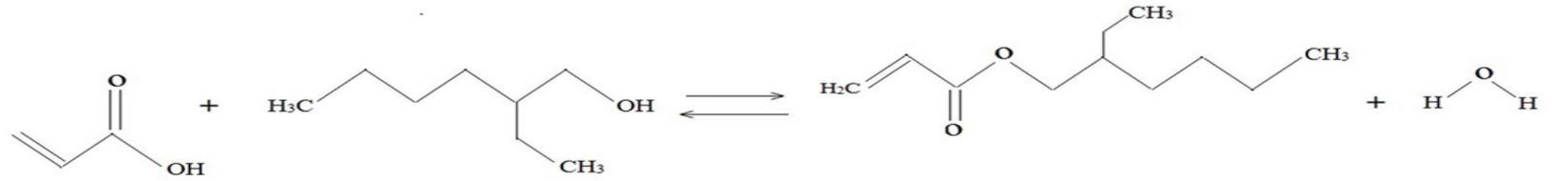
During process



Final neutralization



Reactor volume definition



Acrylic acid (AA) | 2-ethyl-1-hexanol (EHOL) | 2-ethyl-hexyl-acrylate (EHA) | Water

E2H-OH + **CH2=CH-COO-E2H** ⇌ **E2H-O-CH2-CH2-COO-E2H**

Ethyl-2 héxanol — AE2H — OPO

E2H-OH + **CH2=CH-COOH** $\overset{H^+}{\rightleftharpoons}$ **HO-CH2-CH2-COO-E2H**

Ethyl-2 héxanol — AA — E2HHPro

2 CH2=CH-COOH $\overset{H^+}{\rightleftharpoons}$ **CH2=CH-COO-CH2-CH2-COOH** + **E2H-OH** $\overset{H^+}{\rightleftharpoons}$ **CH2=CH-COO-CH2-CH2-COO-E2H**

AA — diAA — Ethyl-2 héxanol — E2HApro : ester du dimère

2 E2H-OH ⇌ **E2H-O-E2H**

Ethyl-2 héxano — DE2HE

E2H-OH $\overset{H^+}{\rightleftharpoons}$ **CH2=CH(C2H5)-(CH2)3-CH3** + **H2O**

Ethyl-2 héxano — 2EHE

Tower heavy fouling

Q

Tower and reboiler heavy fouling

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MEMBRANE SCALE-UP FOR CHEMICAL INDUSTRIES

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PERVAPORATION CHALLENGES

Challenge		Current	Target
Feedstock efficiency	inlet molar ratio acid/alcohol		0.8-1.2
Overall energy consumption	MJ per kg acrylate product		- 20%
Process productivity	kg acrylate product/kg catalyst		+ 20%
Minimise impurities formation (octenes, acid derivatives)	hydroxyl propionic acid concentration at outlet of reactor Octenes concentration at the outlet of the reactor		<100 ppm < 1000 ppm
Operating cost (OPEX)	€/kg acrylate product		-20%
Membrane lifetime in pilot	Months		3

Pervaporation

Innovating for the sustainability and reliability of industrial processes

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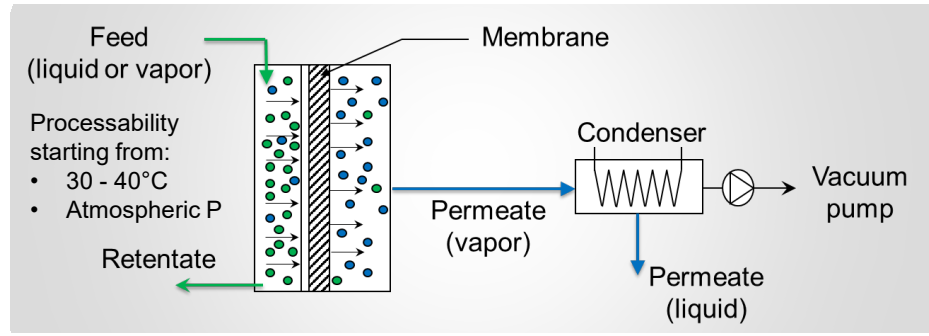
- Pervaporation is a process for the separation of liquid mixtures by partial vaporisation
- ALSYS has access to the Hybsi® technology of ceramic pervaporation membranes which allows water to be separated from solvents and organic compounds
- ALSYS owns a mobile pilot system that can be used for bench-test, on-site pilots, feasibility and validation studies

Principle of pervaporation



Experiences of ALSYS in the field of pervaporation technology

Industries	Applications	Benefits
• Pharmaceutical	• Solvent mixtures recycling by dehydration	• High selectivity • Low energy consumption
• Chemicals • Petrochemicals • Biofuels	• Alcohols dehydration (IPA, Butanol…) • Esterification mixtures dehydration • Azeotropic breaking • Solvent recycling	• Implementation with minimum process modification • Flexible for batch or continuous processes
• Fine chemistry • Flavor & Fragrances • Food & Beverage	• Concentration of aromatic mixtures	• Process intensification

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Orelis pervaporation equipment

(1): Feed tank

Max Temperature	Max Pressure
175°C	8 bar

(2): Membrane module

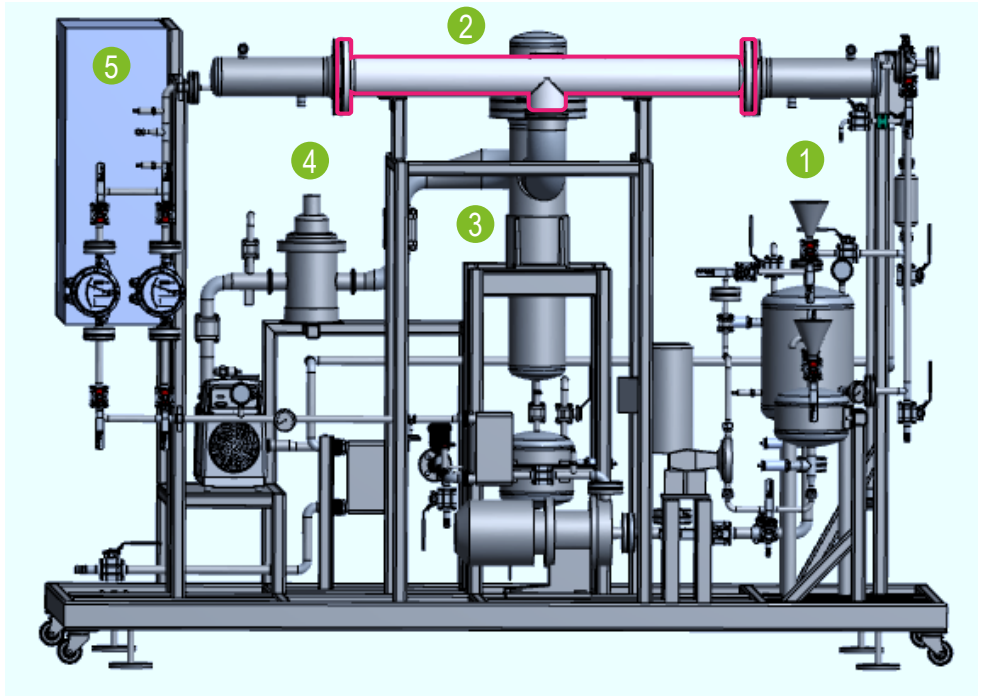
1 membrane	7 membranes
0,15 m²	1,05 m²

(3) & (4): Permeate

Condenser	Cold trap
0 to -50°C	- 180°C

(5): Control panel

- HMI
- Datalogging
- Process control and serucities
- Distance access
- SMS notification



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Pervaporation: expected results

Improved membranes

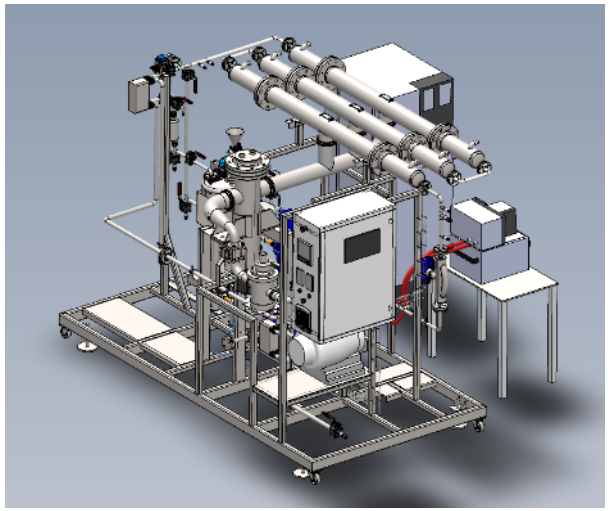
- advanced hybrid silica membranes with H_2O flux > 1.0 kg/m^2/h at operating temperature, and a stability > 90% over 3 months of testing.
- Membranes with acid resistance, increased surface area, long lifetimes and efficient cleaning in place (CIP) strategies (<10% reduction of flux after CIP)

Lowered cost of membranes

- Up-scaled production from 1 to 7 to **a 55-channel tube** reducing costs by from 2100 €/m^2 to 1500 €/m^2 (30% cost reduction).

TRL7 Demonstration

- Currently validated 0.1 m^2 on lab scale
- **1 m^2 of membrane in an industrial setting (ARKEMA industrial production of acrylic ester)**



3D vision of the membrane separation set-up to be integrated in the ARKEMA production plant

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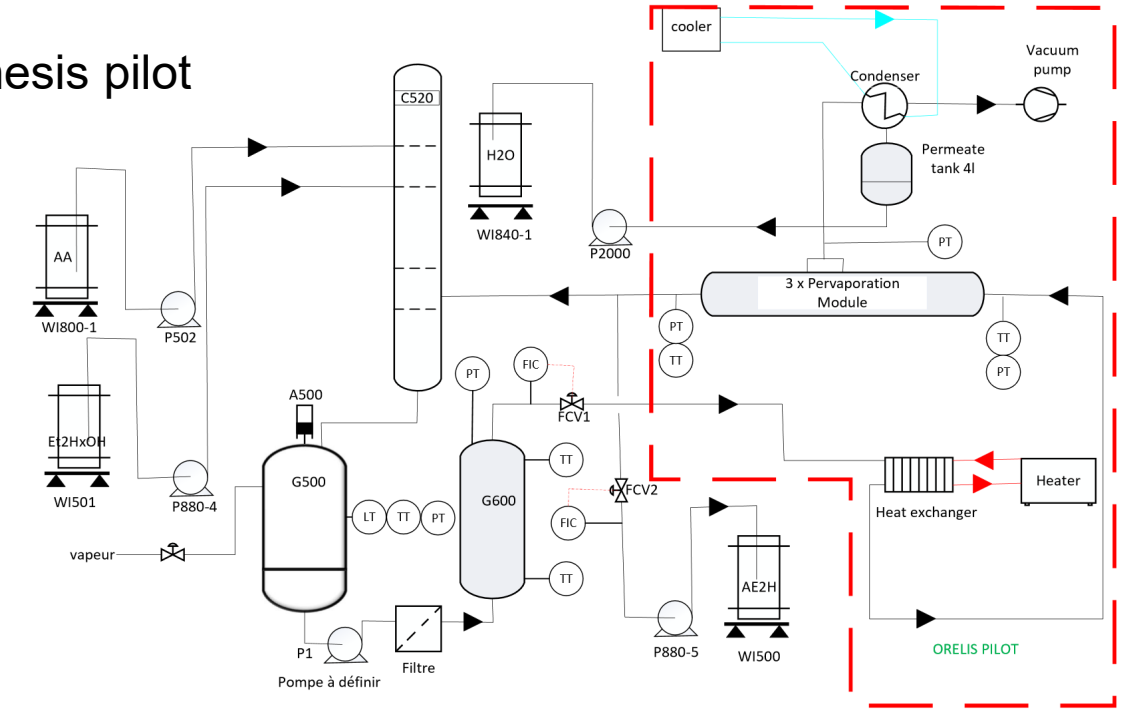
The pervaporation separation integrated in a chemical process

Arkema synthesis pilot



Orelis pervaporation pilot

ARKEMA want to link their reactor G600 (diagram) with pervaporation pilot of ORELIS. The process condition for pervaporation pilot is following:

- Temperature: 80 – 100 °C
- Pressure: 2 bar
- %water in feed: from 1 to 0,4%
- Expected permeate flowrate : 1kg/h of water in permeate.

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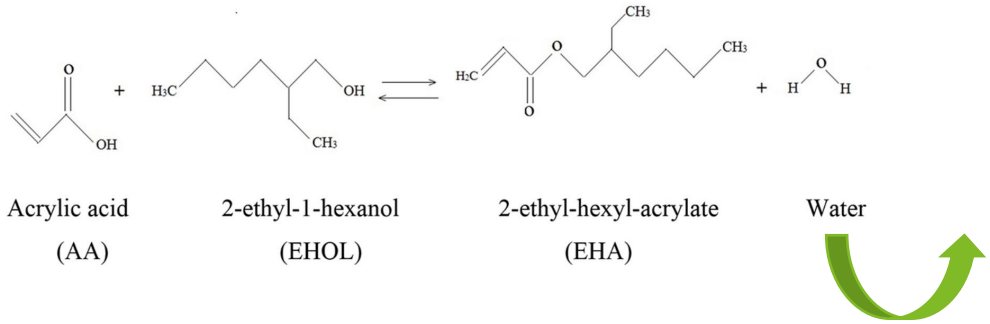
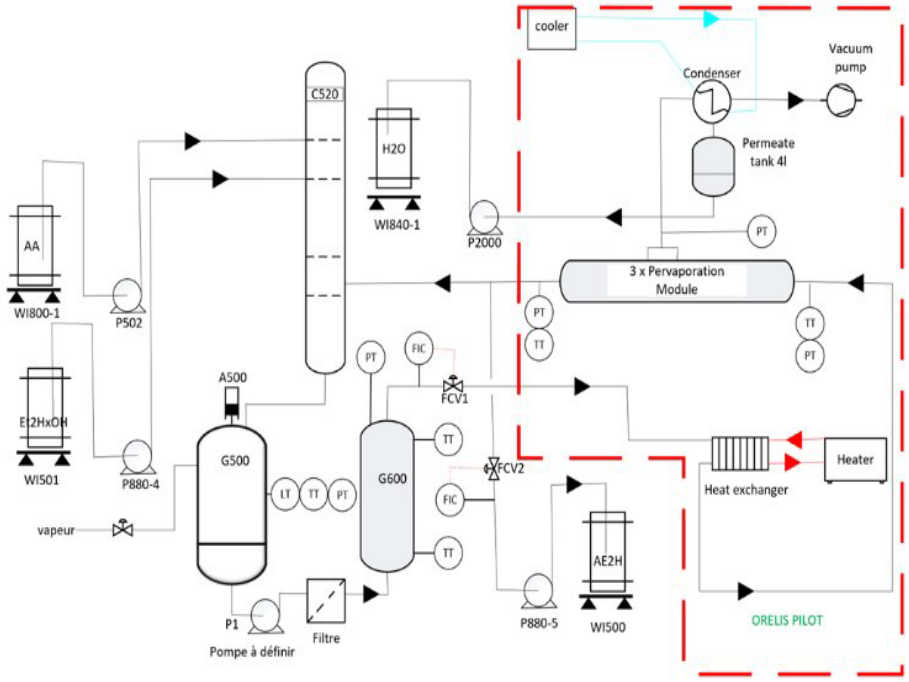
Application of HybSi membranes in esterification reactions

- Esterification reaction for production of 2-ethyl-hexyl-acrylate



- Reaction temperature: 80-100°C,
- Water content: 1-2 wt.% , acrylic acid content: 5-10 wt.%, pH=4
- Boiling point of the mixture = 160°C
- Potential advantages pervaporation:
  - Lower temperature => decrease polymerization and blocking of equipment
  - Faster water removal => decrease undesired byproducts and higher feedstock efficiencies



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WP4.1.1. Pilot demonstration units engineering design and construction

Actual state:

- Number of module : 1
- Number of membrane : 7
- Total membrane area : 1 m^2

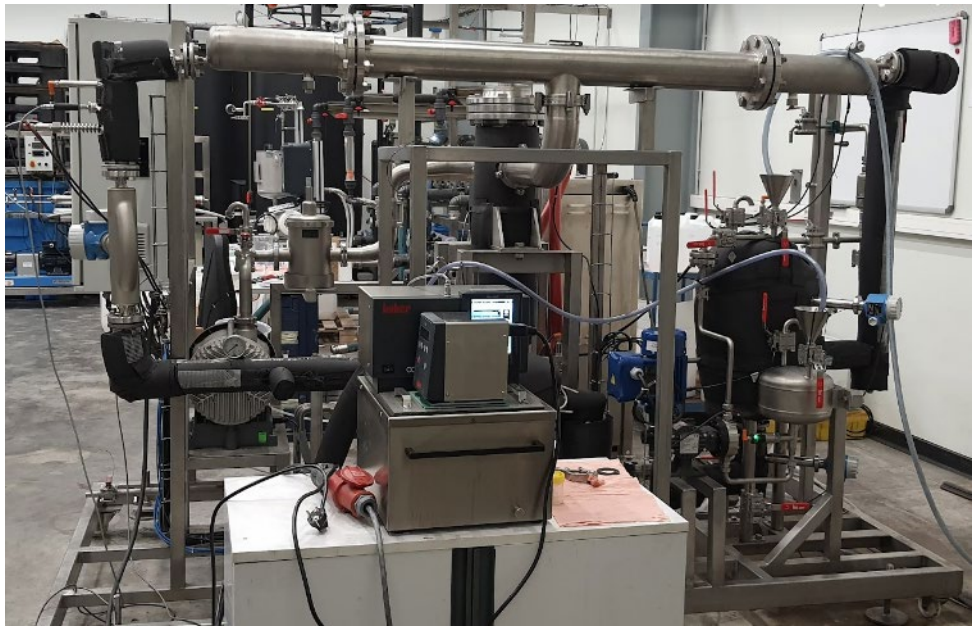
Capacity upgrade:

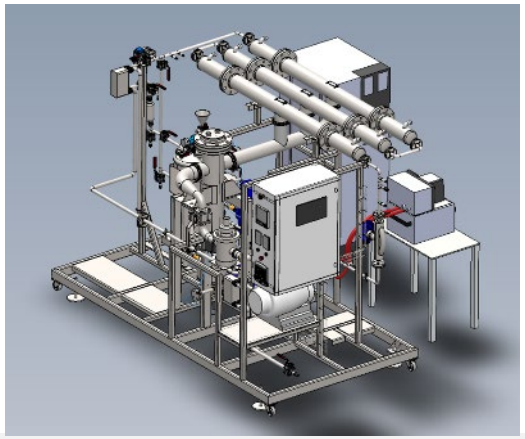
- Number of module : 3
- Number of membrane : 21
- Total membrane area : 3 m^2

Membrane upgrade (if possible):

- Surface per membrane X 3
- Total membrane area : up to 10 m^2

Pervaporation pilot capacity upgrade





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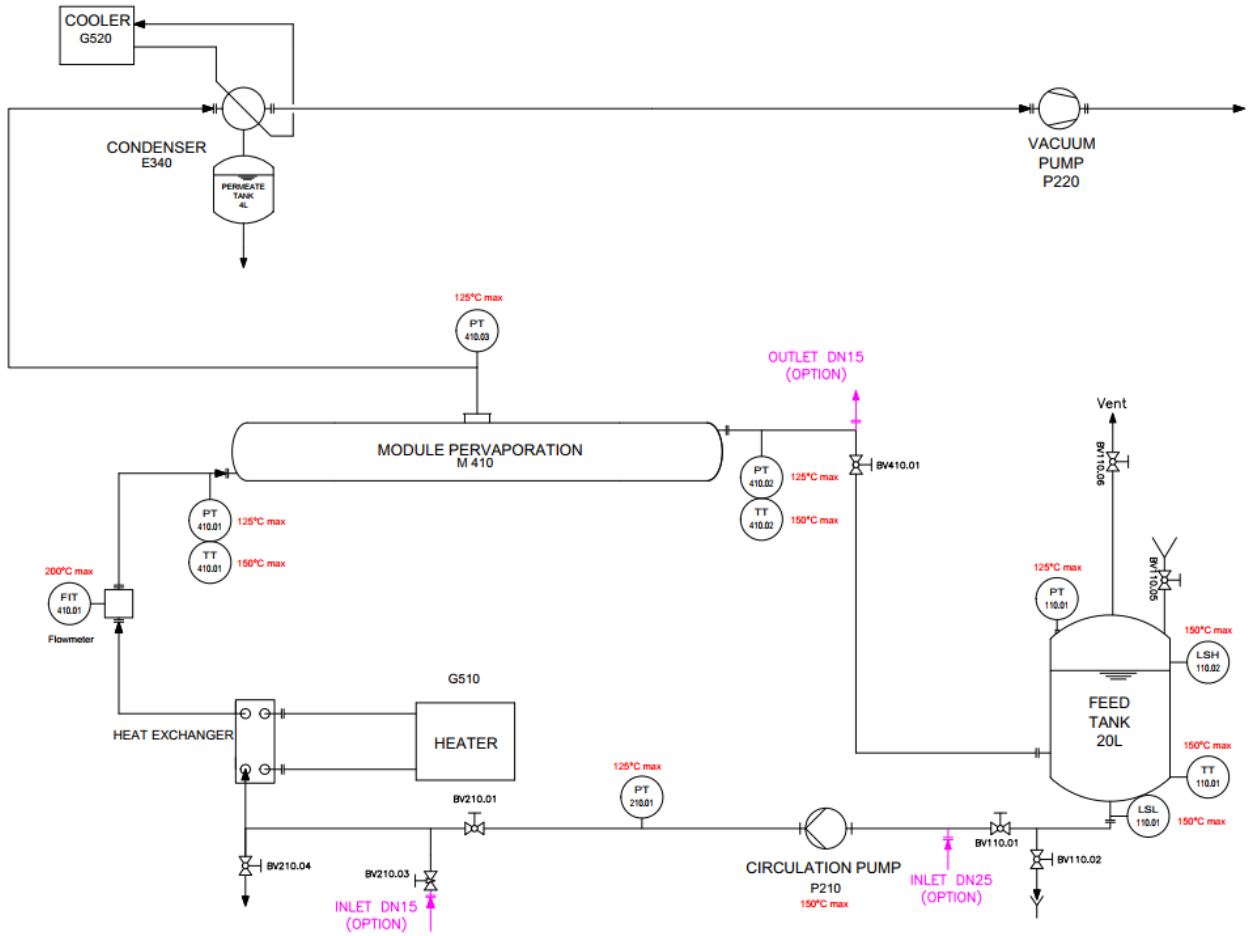
. Pilot demonstration units engineering design and construction

Process parameters

Parameter	Required by application	Designed by Orelis
Temperature	80-100	150°C max
Pressure	1 – 4.5barg	8barg max
Flowrate	Variable	Variable 1400 L/h max
Membrane area	1 – 3 m^2	3 m^2 max
Materials	Compatible with acrylic acide, alcohol, 2-ethyl hexyl acrylate	SS304, SS316, FFKM, PTFE



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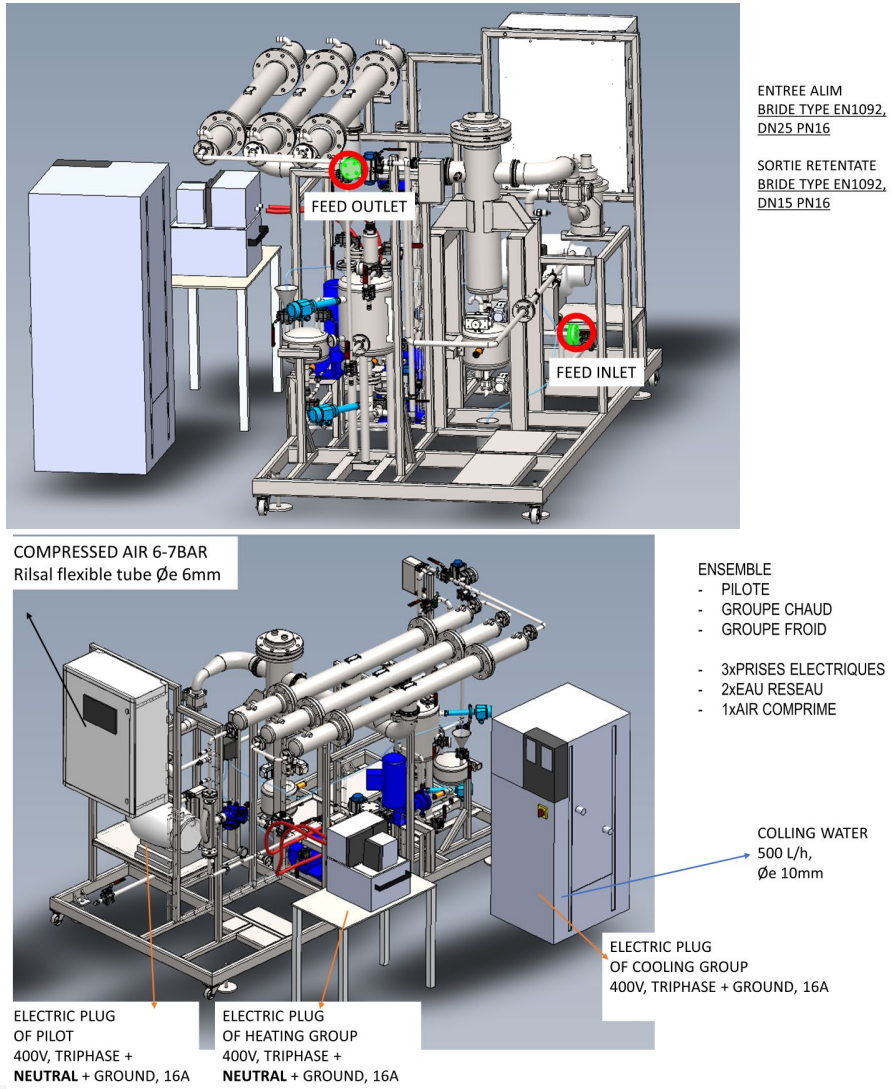
4 WP4.1.1. Pilot demonstration units engineering design and construction

Parameter	Required by Arkema	Designed by Orelis
Feed inlet	OK	DN25
Feed outlet	OK	DN15
Permeate outlet	OK	Flexible hose Ø12mm
Cooling water	OK	Flexible hose Ø10mm
Air Compresseur	OK	6-7 bar, Rilsal flexible hose Øe 6mm
Safety	Shutdown when explosive atmosphere detected	Automated pilot Shutdown with external inlet
Communication	Centralized data logging	Data communication with extension card

Pilot integration in esterification process



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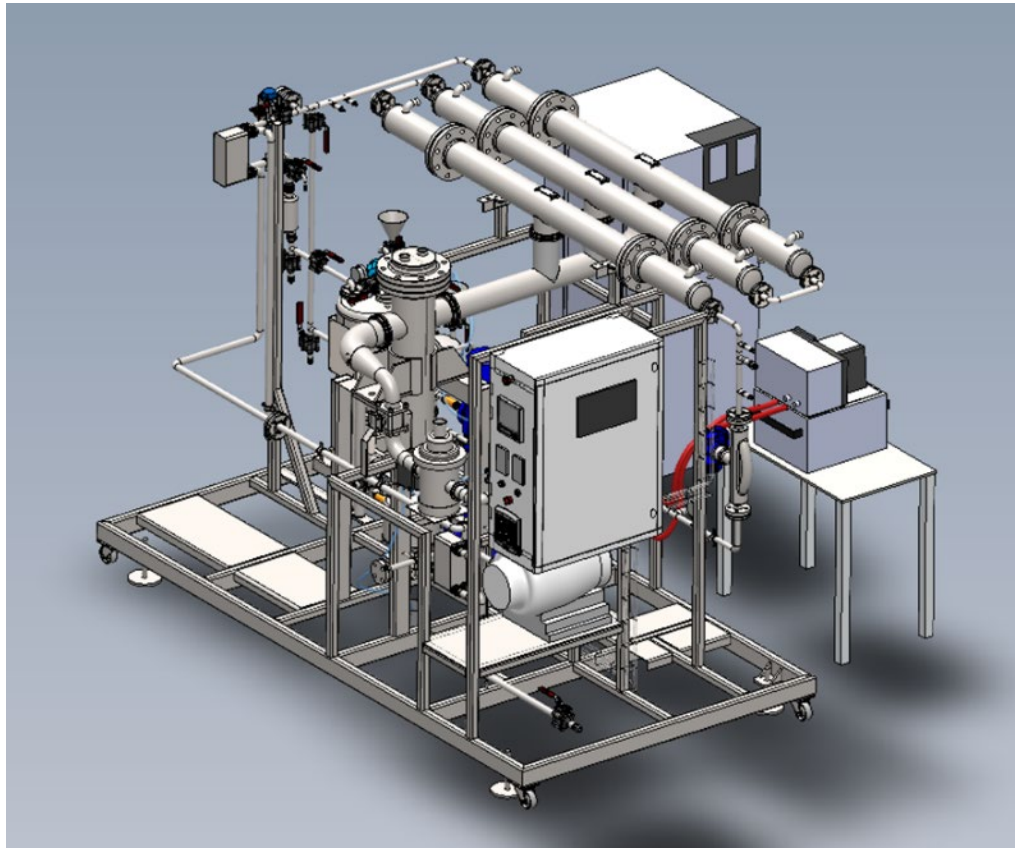
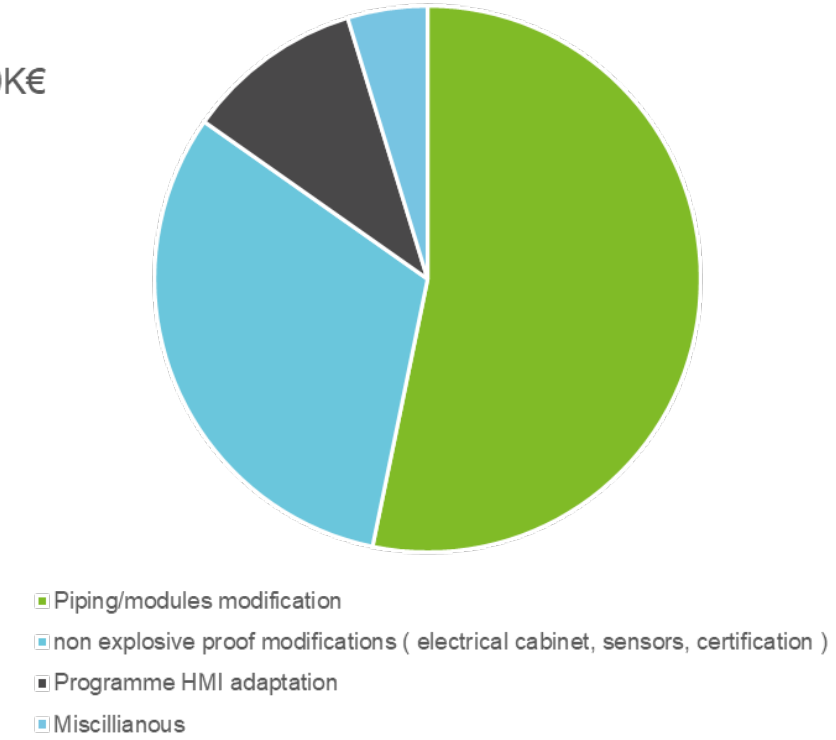
. Pilot demonstration units engineering design and construction



Status of the pilot development/ Budget

Cost : 190K€



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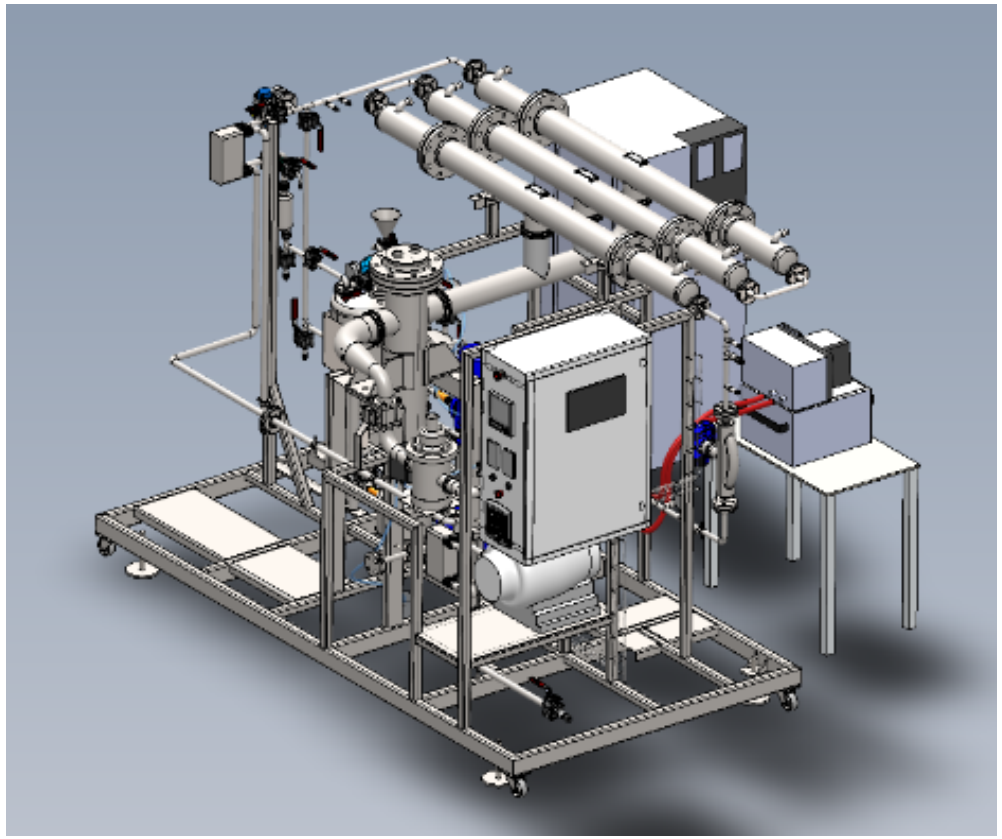
The pervaporation separation integrated in a chemical process

Pervaporation pilot will be installed on Arkema site in August 2025



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THANKS FOR YOUR ATTENTION

MEMBRANE WINTER SCHOOL

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C.Manzas, D.Tournigant CTI
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EINDHOVEN

JANUARY 27-28TH 2025

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