ALMA MATER STUDIORUM
UNIVERSITÀ DI BOLOGNA

Workshop

Geopolymer for Environmental Remediation

ASYMMETRIC MEMBRANES FOR WASTEWATER TREATMENT BY ALKALI ACTIVATION

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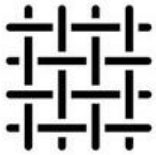
ASYMMETRIC MEMBRANE FOR MICROFILTRATION OF OIL/WATER EMULSIONS

Industries where it is needed to treat oily wastewaters:

Metal finishing

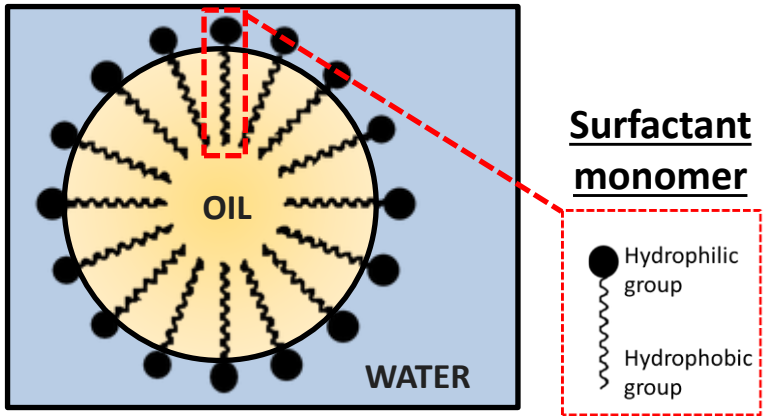
Oil&gas extraction



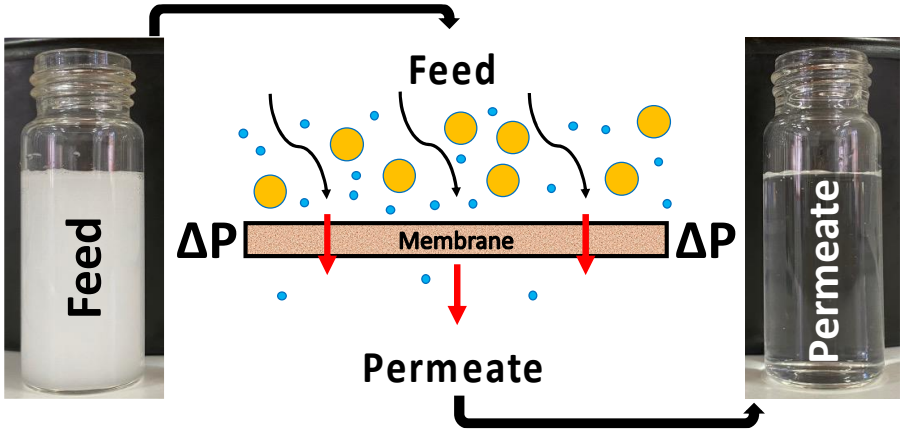
Textile production

Food and beverage

Emulsion: solution of oil and water stabilised by the addition of a surfactant

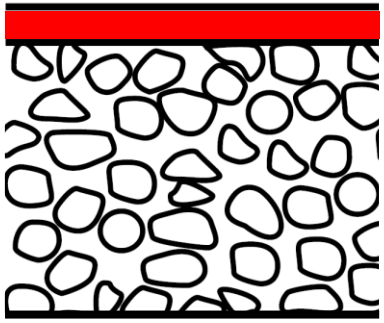


Microfiltration membrane is a cost-effective treatment for emulsions



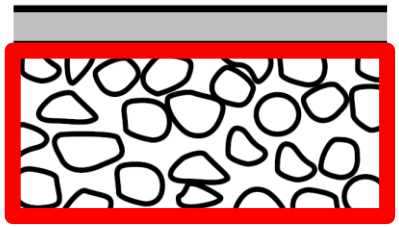
SELECTIVE LAYER
Microfiltration
Pore size: 0.1 – 1 µm

SUPPORT
Porosity: 30 – 65 %
Pore size: 1 – 20 µm

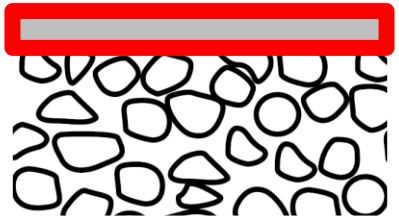


AIM OF THE STUDY

Development of an asymmetric microfiltration membrane that is completely made of geopolymer and is effective for wastewater treatment



Synthesis of a geopolymer support by pressing method



Deposition of a geopolymer selective layer by casting

Testing **Na/Al ratio**

0.7, 0.8, 0.9, 1.0, 1.1

Testing **water content**

8, 10, 12, 15 wt% H_2O

Testing **forming pressure**

0.5, 1, 2, 5, 10, 20 MPa

Testing **Oil/Water emulsions permeability**

Testing **oil rejection**

Comparison of the results with a commercial ceramic membrane

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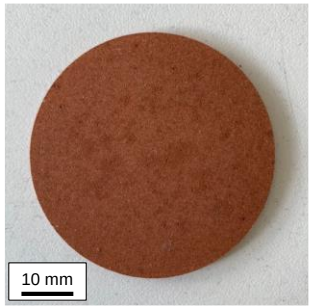
EXPERIMENTAL: Sample preparation

GEOPOLYMER SUPPORT

- **ONE-PART GEOPOLYMER: metakaolin** and **anhydrous sodium silicate** as raw materials
- Shaped by uniaxial **pressing**
- Cured at **70 °C** for **24 hours** and **6 days** at **room temperature**
- Diameter of 45 mm and thickness of 2.2 mm.



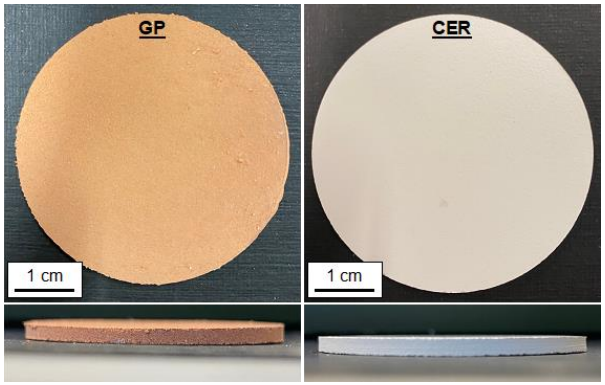
Na/Al ratio = 0.7 - 1.1

H_2O content = 8 - 15 wt%

Uniaxial pressure = 0.5 - 20 MPa

GEOPOLYMER SELECTIVE LAYER

- **TWO-PART GEOPOLYMER: metakaolin, sodium silicate** and **sodium hydroxide** solutions
- **Shaped by casting** and deposited on the support trough spatula deposition
- Cured at **50 °C** for **24 hours** and fired at **400 °C** for **6 min**
- Layer thickness of **40 µm**
- Comparison with a **commercial ceramic membrane**



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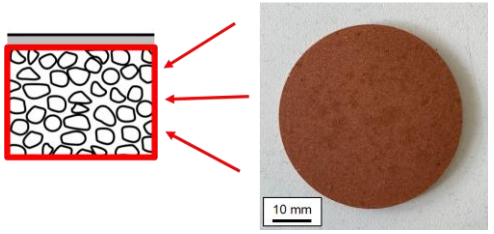
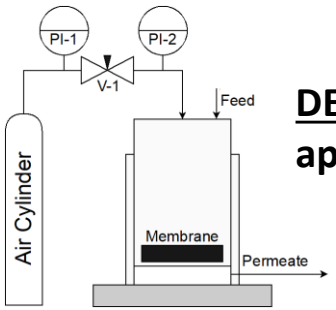
SUPPORT: permeability to pure water & Et-H_2O solution

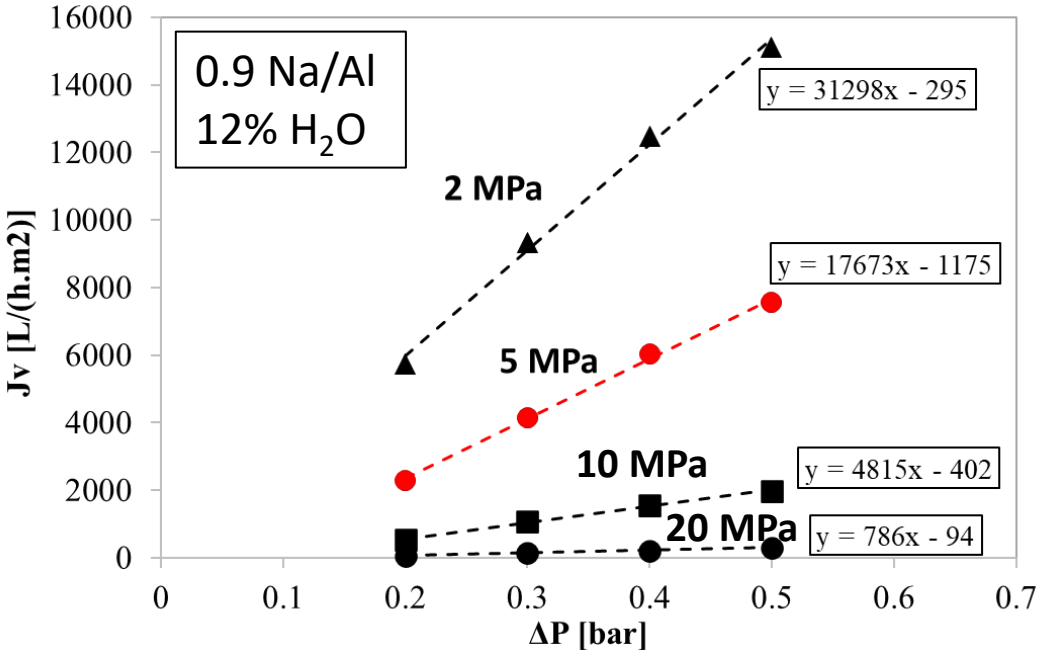




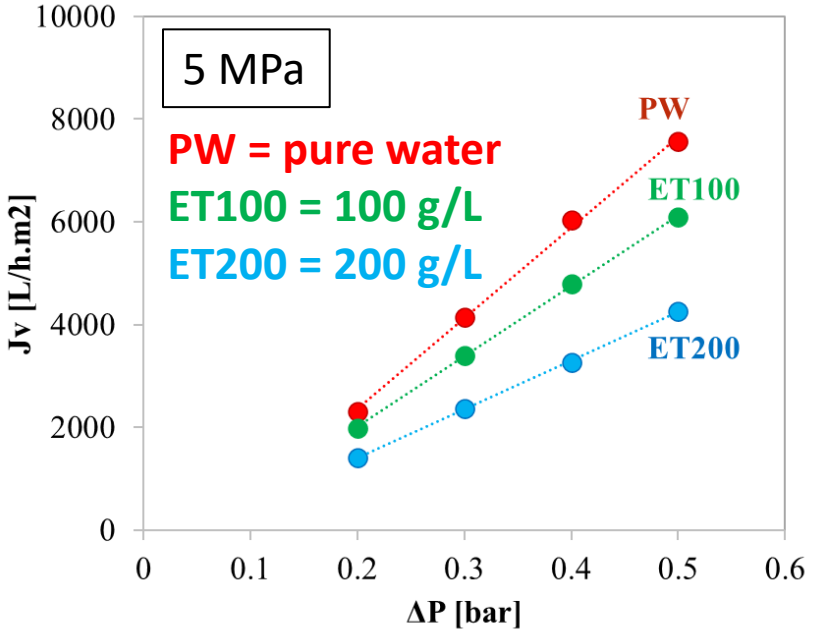
DEAD-END geometry apparatus

Typical permeability values of ceramic supports
7600 – 45000 $L \cdot h^{-1} \cdot m^{-2} \cdot bar^{-1}$

PURE WATER

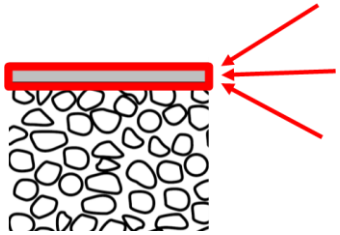


Et-H_2O SOLUTIONS



Increasing the ethanol concentration in the solution leads to a decrease in permeability, highlighting that the geopolymer support has **hydrophilic** behavior

SELECTIVE LAYER: material characterization

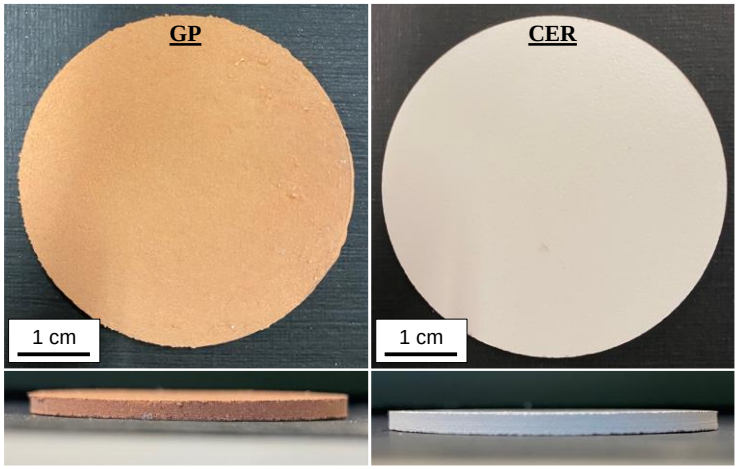


Geopolymeric support:

Na/Al ratio= 0.9

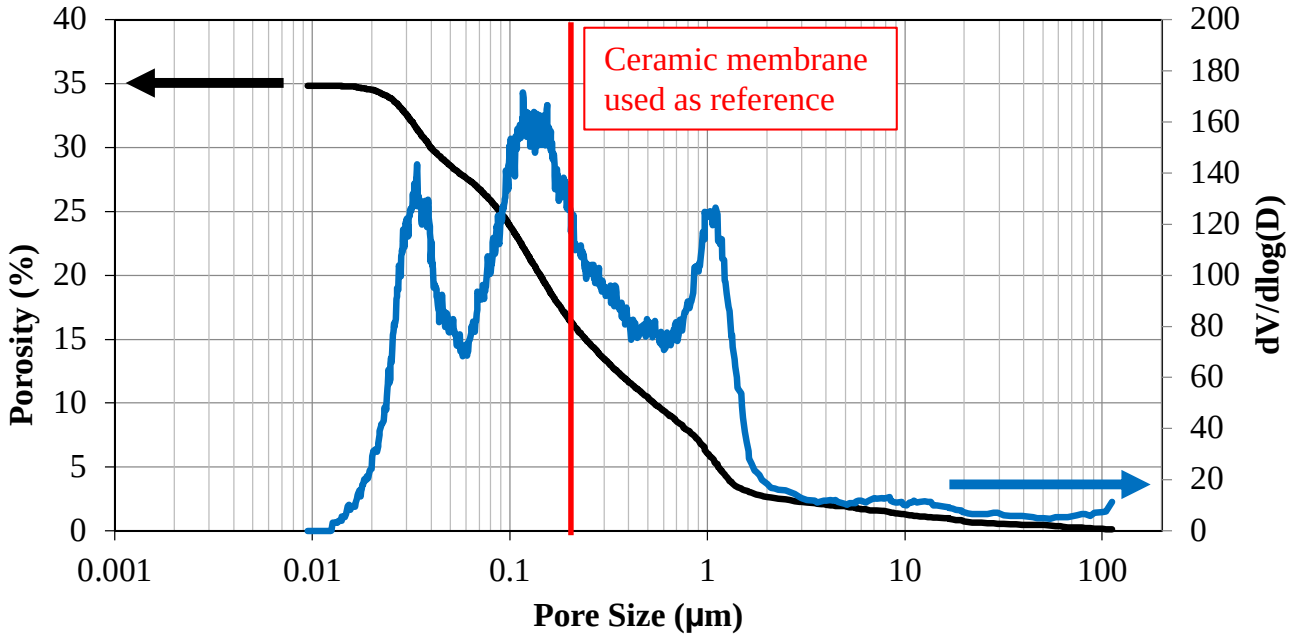
Water content= 12 wt%

Forming pressure= 15 MPa



GP and CER membrane have the **same dimensions** (diameter and thickness)

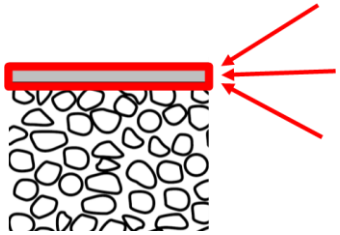
Both membranes are **asymmetric**



GP membrane is in the **microfiltration range**

Average **pore size** of the two membranes are **comparable**

SELECTIVE LAYER: emulsion preparation



Surfactants (1 or 10 CMC):

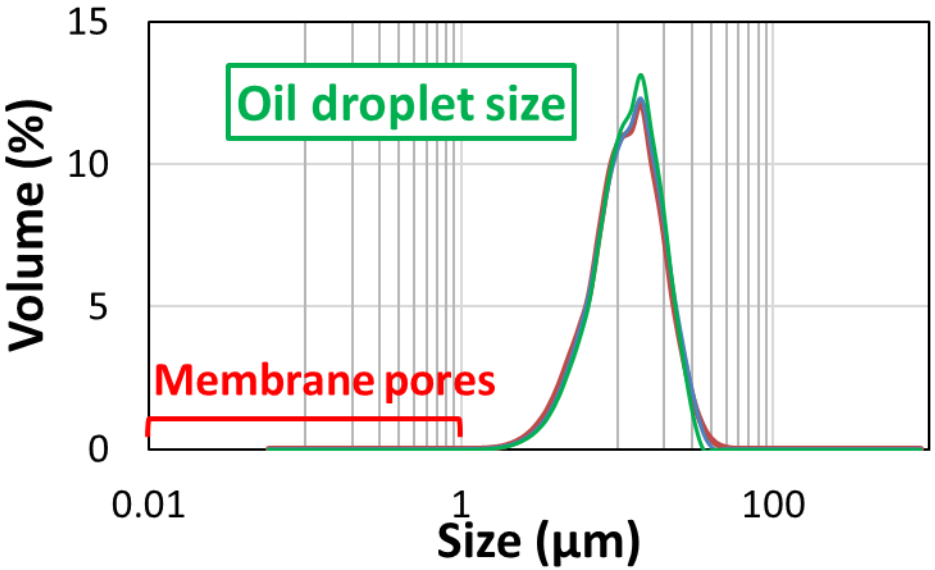
Oleth10 (**anionic**) – 20 mg/L
Brij76 (**non-ionic**) – 200 mg/L
CTAB (**cationic**) – 350 mg/L

Oil: dodecane
3 wt%

Homogeneizer parameters

10k RPM - 2 minutes

Prepared at different pH (2, 5, 8)

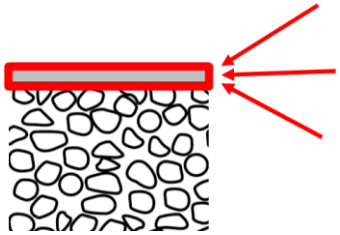


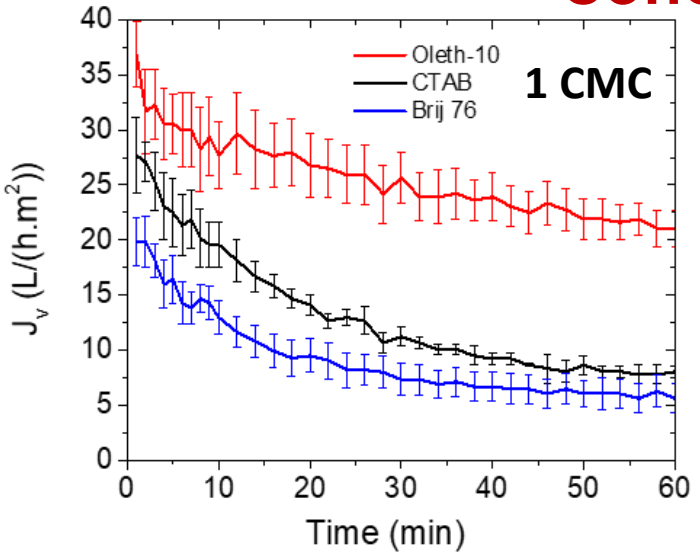
Emulsion | Permeate

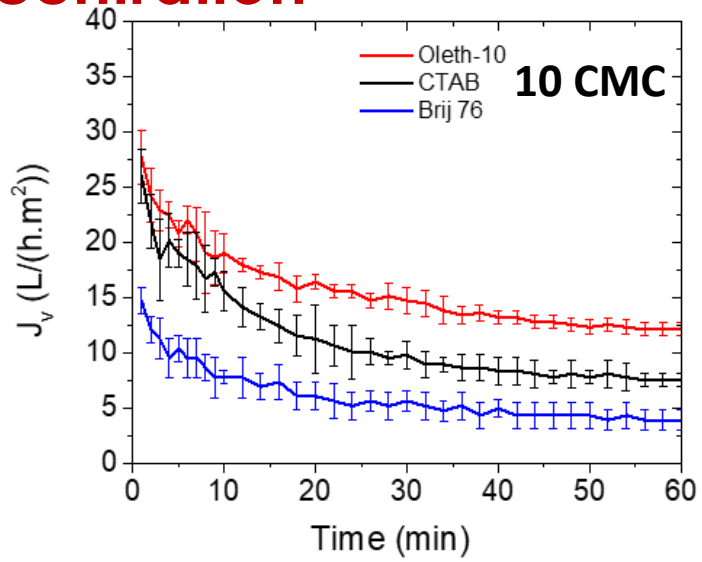
d(50):

Oleth-10 (anionic) ---- 11.15 µm
Brij-76 (non-ionic) ------- 10.93 µm
CTAB (cationic) --------- 11.22 µm

SELECTIVE LAYER: effect of surfactant concentration

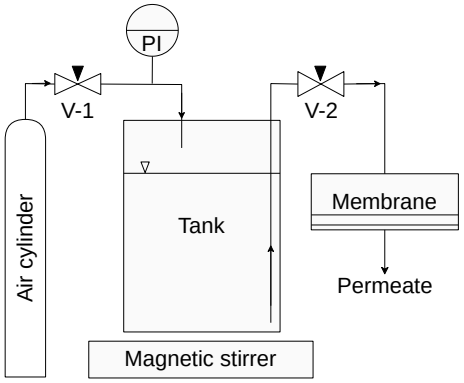


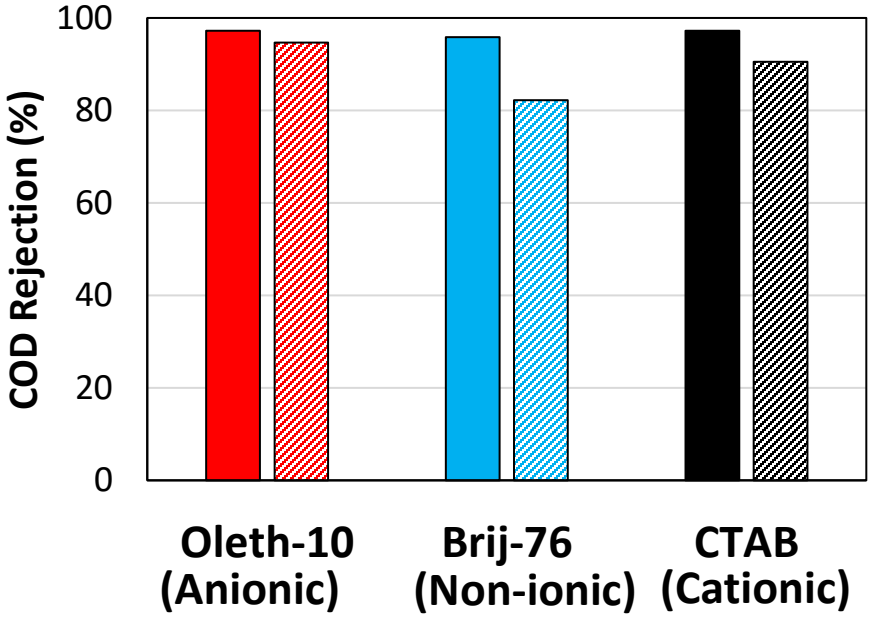




Increasing the **surfactant concentration from 1 to 10 CMC shows a reduction in flux** and a **decrease in rejection**

DEAD-END geometry apparatus





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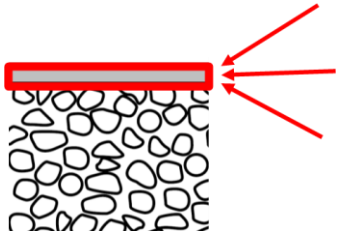
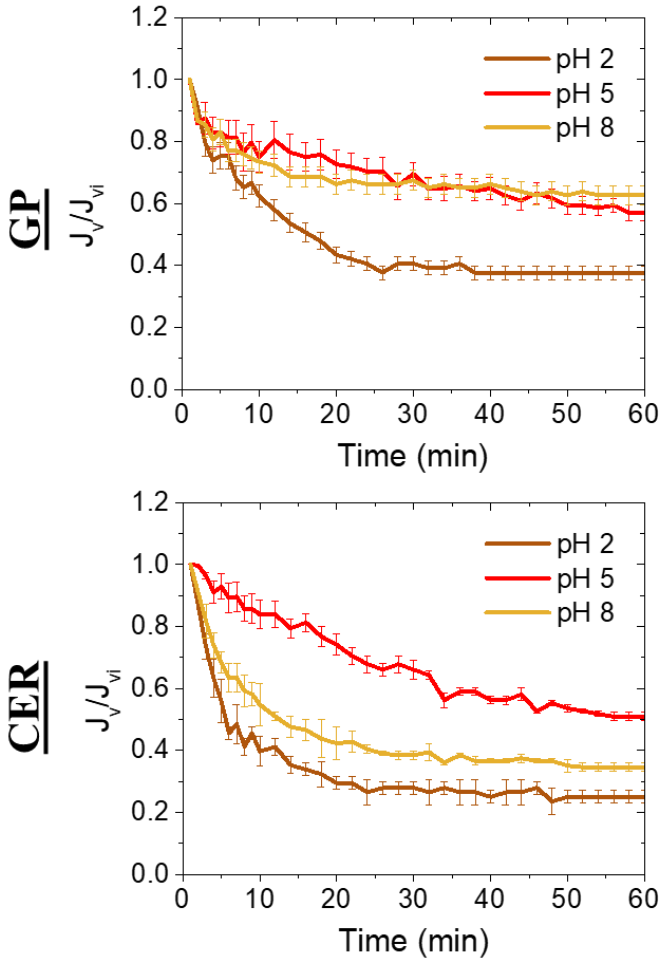
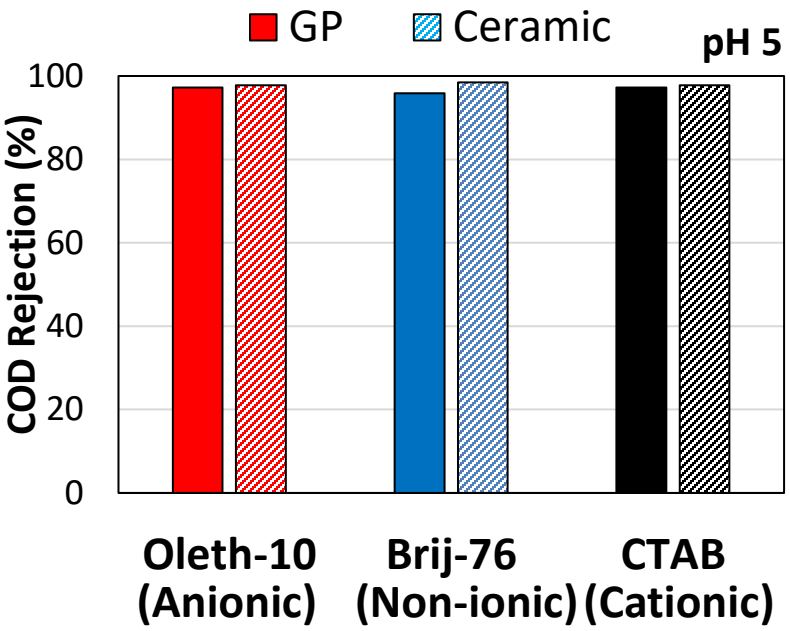
SELECTIVE LAYER: geopolymer vs. ceramic



Oleth-10



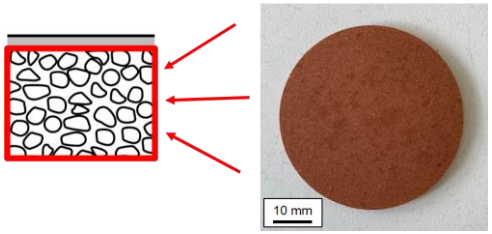


At **pH 5** the two membranes have approximately the same performance in terms of both flux and rejection

At **pH 8** the ceramic membrane performs slightly better

CONCLUSIONS

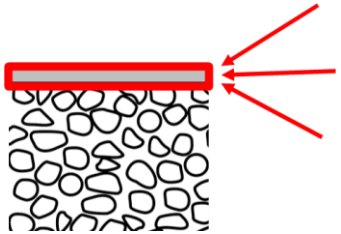
GEOPOLYMER SUPPORT



Highlight the **influence of these mix design parameters** over the final properties of the material

Pressed geopolymer → Support for microfiltration

GEOPOLYMER SELECTIVE LAYER



Geopolymer selective layer **successfully deposited** above the geopolymer support

Excellent results were achieved in terms of **flux** and **rejection**

Performance of geopolymer membrane similar to **performance** of ceramic membrane

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More details in:

- A. Filipponi, G. Masi, M. Matos, J. M. Benito, G. Gutiérrez, M.C. Bignozzi, Development of metakaolin-based geopolymeric asymmetric membrane for oil-in-water emulsion microfiltration, **Ceramics International** 50(12) 2024, 21107-21117, https://doi.org/10.1016/j.ceramint.2024.03.220

- A. Filipponi, G. Masi, S. Bandini, M.C. Bignozzi, Preparation and characterization of metakaolin-based geopolymer membrane supports by facile pressed one-part route, **Ceramics International** 49(4) 2023, 6834-6842, https://doi.org/10.1016/j.ceramint.2022.10.233

- A. Filipponi, G. Masi, M.C. Bignozzi, Pressing metakaolin-based one-part geopolymers: Influence of the mix design on microstructural and physical properties, **Ceramics International** 48(4) 2022, 5814-5823, https://doi.org/10.1016/j.ceramint.2021.11.129

Thank you for your kind attention!

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