Characterization of microfiltration membranes by *in situ* wetting in the ESEM, FT-IR mapping and 3D reconstruction

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Introduction

To enable a spatially resolved investigation of the impact of three typical cleaning agents on PES/PVP based flat microfiltration membranes two microscopic methods were used. The first method deals with the *in situ* wetting and drying of the membranes in an environmental scanning electron microscope (ESEM) [1]. By the combination of simultaneously recorded micro- and macroscopic parameters a layer resolved change of membrane properties caused by the treatment was possible. To confirm these results, IR-maps at cross sections of pristine and treated membranes were recorded. Therewith it was also possible to reveal a spatially resolved change concerning the membrane layers after the treatment.

Additionally, a 3D reconstruction was performed which gives detailed insights into e.g. the pore distribution, their interconnections or enables a fluid dynamic simulation based on a lifelike model [2].

Investigated membranes

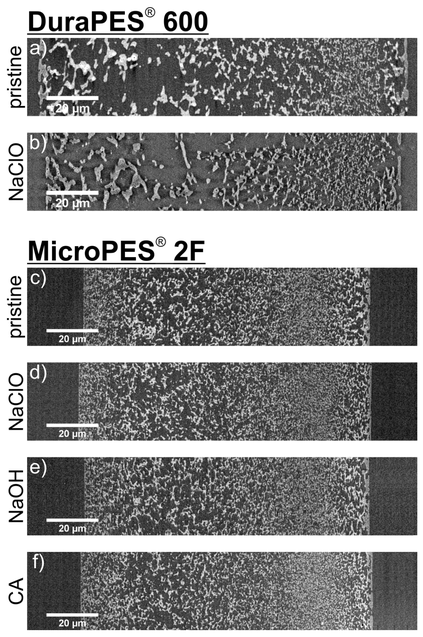

Fig. 1: SEM images of the cross sections of the two investigated membranes from MEMBRANA© GmbH (Wuppertal, DE).

a) Pristine DuraPES® 600 membrane.
b) DuraPES® 600 treated with a 30.000 ppm sodium hypochlorite (NaClO) solution for 24 hours.
c) Pristine MicroPES® 2F membrane.
d) MicroPES® 2F treated with a 30.000 ppm NaClO solution for 24 hours.
e) MicroPES® 2F treated with a 3.000 ppm sodium hydroxide (NaOH) solution for 72 hours.
f) MicroPES® 2F treated with an 2.000 ppm citric acid (CA) solution for 72 hours.

3D reconstruction

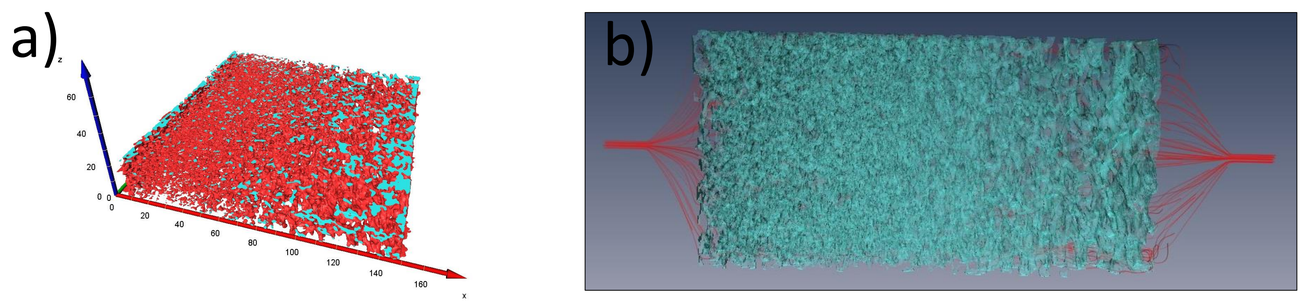

Fig. 2: a) shows a 3D model of a part of the reconstruction over the whole cross section and b) the subsequent fluid dynamic simulation (red lines are possible stream lines).

Serial block-face scanning electron microscopy (SBEM) enables a detailed 3D reconstruction of soft matter material. Figure 2 a) shows the 3D model of a pristine DuraPES® 600 membrane. Therewith various information can be gained, like the overall porosity or its variation over the cross-section, the inner surface structure, the volume fraction. Additional, it enables the simulation with fluid dynamic calculations, to obtain the local flow resistance, see Figure 2 b).

Results of *in situ* wetting in the ESEM

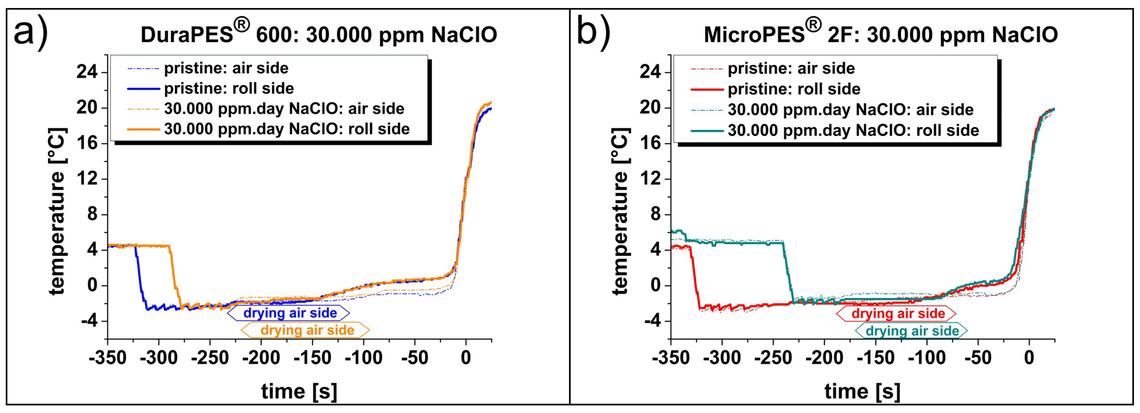

Fig. 3: Comparison of drying characteristics of a) pristine and treated DuraPES® 600 and b) pristine and treated MicroPES® 2F microfiltration membranes; both membranes were treated with 30.000 ppm.day sodium hypochlorite (NaClO) at the same concentration (30.000 ppm).

Both investigated membranes reveal a shortening in the overall drying time after the NaClO treatment. No significant changes were observed in the last drying step representing the drying of the separation layer. Even the time necessary for the drying of the surface pores did not change (see bars). Changes arise only in the first part of the characteristic representing the topmost layer. This indicates that the surface layers become more hydrophobic [3].

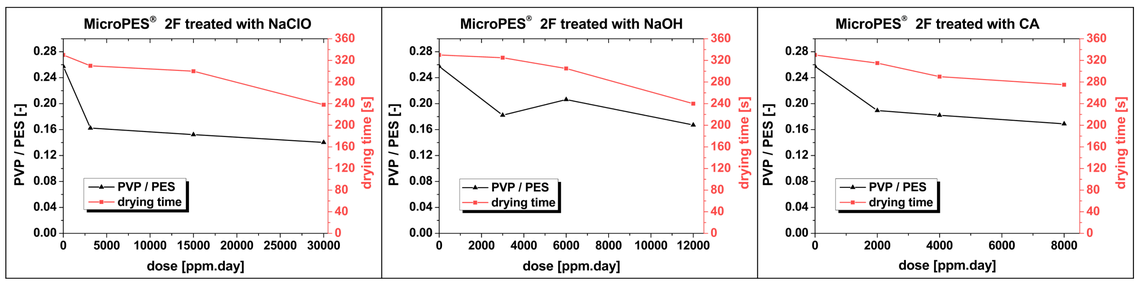

Fig. 4: IR-measurements of PVP/PES ratios and total drying time in dependence on the dose of a MicroPES® 2F treated with a) sodium hypochlorite, b) sodium hydroxide and c) citric acid.

All three cleaning agent treatments (NaClO, NaOH and CA) cause a decrease of the overall drying time, thus changing the membrane behaviour from hydrophilic to more hydrophobic. Additionally the PVP to PES ratios measured by FT-IR at the membrane surfaces reveal a PVP decrease in all three cases. As PVP is responsible for the hydrophilicity of the membranes, a decrease causes a more hydrophobic behaviour.

Results of the IR-measurements

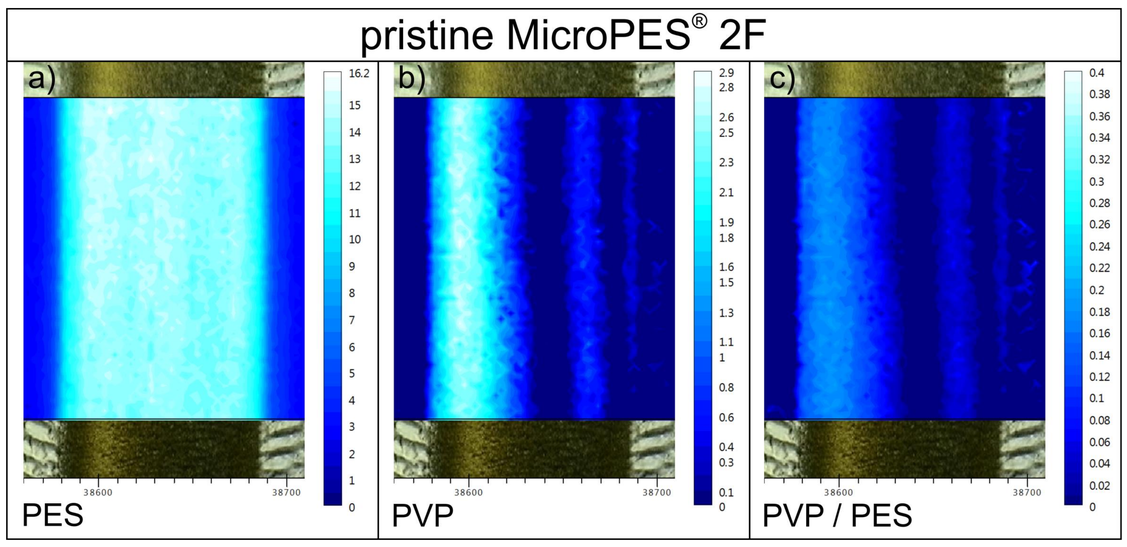

Fig. 5: IR-maps (transmission mode, slice thickness 20 µm) for the main components of a pristine MicroPES® 2F membrane; a) polyethersulfone (PES); b) polyvinylpyrrolidone (PVP); c) PVP/PES map, which compensates for the porosity changes across the cross section.

Fig. 6: IR-maps of the PVP/PES ratios for treated MicroPES® 2F membranes; a) 2.000 ppm CA solution; b) 3.000 ppm NaOH solution; c) 30.000 ppm NaClO solution; the backgrounds show the light microscope images of the respective areas.

The results obtained by the IR-measurements reveal also a PVP decrease at the topmost layers, thus a decrease in the membrane hydrophilicity [2].

References

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