In situ experiments in the ESEM - What can they tell us about polymeric microfiltration membranes?

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Introduction

Multilayered microfiltration membranes made of polymeric materials are used in a great variety of applications such as waste water treatment, the filtration of colloids and particles in the beverage industry …

The increase in the complexity of the structure of these membranes requires also more and more sophisticated characterization methods. Two recently developed methods based on electron microscopy will be presented:

➔ 3D reconstruction by use of automated serial sectioning and imaging in the environmental scanning electron microscope (ESEM). Quantitative determination of many membrane parameters is possible.

➔ Investigation of the wetting and drying of membranes in the ESEM. This method can provide information in which membrane layer degradation occurred.

Membrane structure

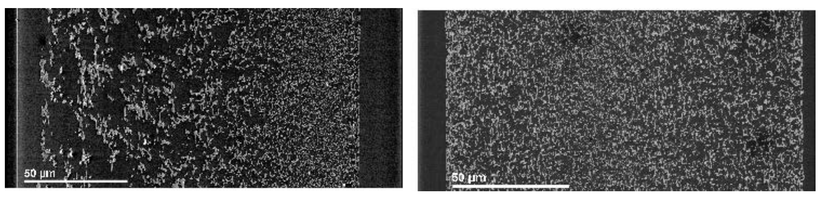

Fig. 1. SEM images (BSE) of the cross sections of two polyethersulfone based membranes: Membrana DuraPES®450 (left) and Sartorius 15406 (right).

➔ Images of the cross sections of membranes recorded by SEM provide quick and rough information about the asymmetry of the layered membrane structure and the pore size distribution.

3D reconstruction of the membrane structure

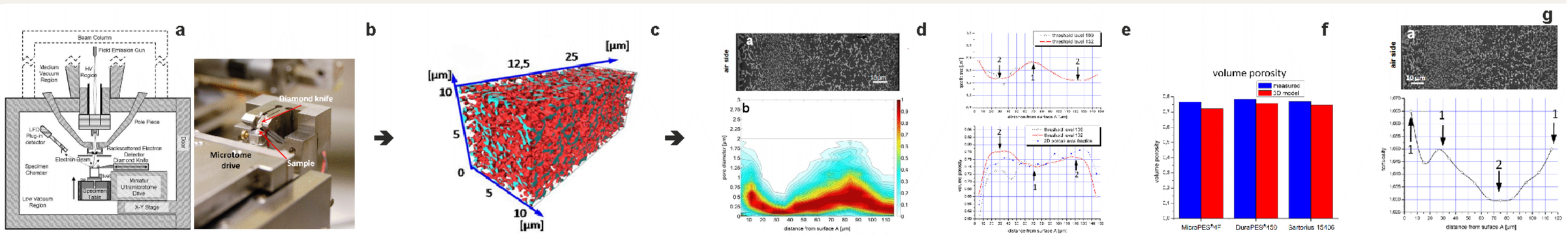

Fig. 2. a: Schematic of the ultramicrotome (Gatan 3View™) mounted in the specimen chamber of the ESEM; b: Image of a part of the microtome; c: 3D reconstruction of the separation layer of the membrane DuraPES®450 (see also Fig. 1); d: pore diameter distribution along the cross section of the membrane MicroPES®4F; e: specific surface area and volume porosity along the cross section of the membrane Sartorius 15406; the dependence of the results on the threshold value chosen for image segmentation is shown; the volume porosity is also compared to the porous area fraction calculated from a 2D image; f: comparison of the calculated volume porosities with the measured values; g: change of the tortuosity along the cross section of the membrane MicroPES®4F. For the 3D reconstructions and the calculation of the parameters the software AVIZO® Fire was used.

➔ Automated serial sectioning and imaging of the block face (maximum area ~ 0.5 x 0.5 µm², minimal slice thickness ~ 30 nm) makes recording of the image stacks for the 3D reconstruction less tedious and time-consuming.

➔ A great variety of parameters characterizing the membrane structure and not available from 2D images can be calculated from the 3D reconstruction (see Fig. 2). Fig. 2f demonstrates that calculated values are in good agreement with measured ones. But from the 3D reconstruction also local variations of the structure can be calculated [1].

Wetting and drying of membranes in the ESEM – Localization of membrane degradation

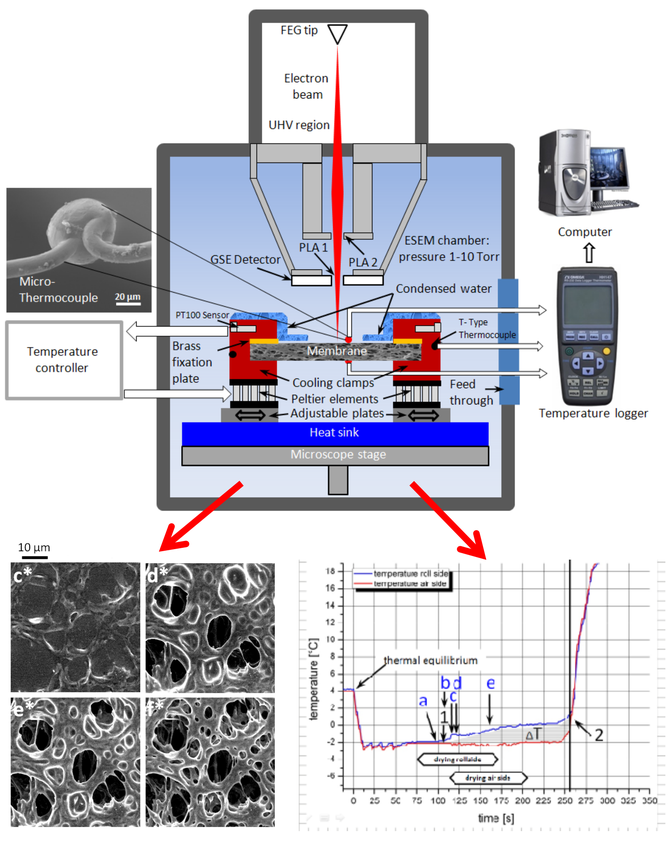

Fig. 3. Top: schematic of the experimental setup; bottom left: Drying of the surface pores as a function of the drying time; bottom right: the temperatures at both membrane surfaces of the cooled membrane (4°C) as a function of the drying time.

➔ The simultaneous recording of images of the drying of the pores at one of the membrane surfaces and of the temperature characteristics at both surfaces is possible. The latter mirror both the membrane structure and provide information about the interaction of the membrane material with water [2].

➔ Changes in the hydrophilicity of a membrane either during operation or caused by membrane cleaning will cause a change both in the time necessary for the drying of the membranes and in the temperature characteristics (Figure 4). Contrary to conventional test methods used for membrane characterization, these changes tell us to which layer the membrane degradation can be mainly attributed [3].

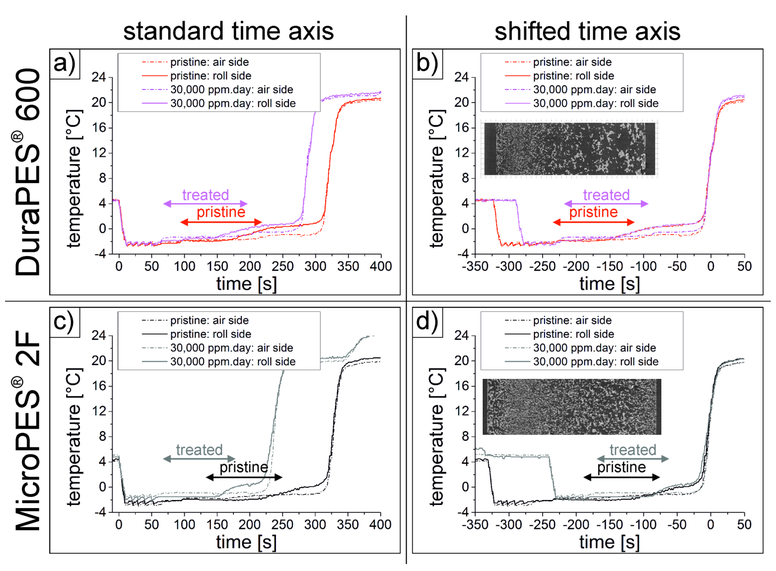

Fig. 4: Temperature profiles of 2 different membranes before and after treatment with a dose of 30,000 ppm.day NaClO with a concentration of 30,000 ppm. The double arrows indicate the start and end of the drying of the surface pores at the air side. Two time axes were used for a better visualisation of the drying behaviour of the membranes. The double arrows mark the start and end of the drying of the surface pores. The insets show a cross section of the respective membrane.

➔ Extensive investigations proved that all tested chemicals used for membrane cleaning also cause membrane damage. The main mechanism seems to be a loss of hydrophilicity directly at the membrane surface. Due to the rather large membrane volume investigated, the results are much more reliable than those gained from analyses at single cross sections.

References

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