

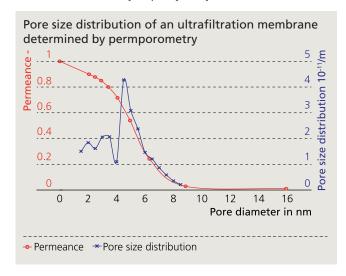
ENVIRONMENTAL AND PROCESS ENGINEERING

MEMBRANE TESTING IN PILOT AND FIELD TESTS

Dr. Marcus Weyd, Dipl.-Ing. Christian Pflieger, Dipl.-Ing. Steffen Wöhner

Membrane processes are widely used as energy-efficient separation methods. They do not need any chemical agents and are usually clearly superior to alternative separation methods in terms of selectivity. Inorganic membranes are characterized by high flow rates. In addition, they can be used under extreme chemical and thermal conditions.

At Fraunhofer IKTS, inorganic membranes for separation processes in liquid, vaporous and gaseous media are developed. These developments mostly aim at improving the separation efficiency and selectivity of the membranes. Further developments target at increasing membrane area per ceramic element or synthesizing and testing novel membrane types for separation tasks. Therefore, new support geometries are also under development, which need to be evaluated concerning material transport. Some types of membranes are also manufactured as prototypes. In addition to the membrane development, the properties of the membranes must be determined as well. Pore sizes of below one nanometer often require a special test procedure. Pore sizes and pore size distributions are determined and layer quality analyzed.



These characterization methods are applied with 1.2 m long industrial-scale membranes as well as with lab samples of only a few centimeters in length. It has to be ensured that the membranes are suitable for the respective process conditions (pressure, temperature, process media and pH). The main methods that are used for membrane characterization are the following:

- Permporometry
- Bubble-point method
- Permeation measurement/clear water flux
- Cut-off determination
- Simulation of material transport and strength
- Burst pressure testing
- Contact angle measurement
- Determination of acid and alkali stability

Crucial for the economic use of a membrane process is the separation efficiency in the industrial process using real media. For the process evaluation, technical data are collected in a multistage process. First principle experiments in lab scale provide first qualitative results for the envisaged membrane processes. In case of positive results, tests with industry-relevant membrane geometries (e.g. multichannel tubes, length 1.2 m) are performed in pilot plants to capture first reliable performance data. IKTS has membrane systems in the laboratory and pilot scale for pervaporation, vapor permeation, gas permeation, micro-, ultra- and nanofiltration and organophilic nanofiltration.

A gas permeation plant for gas separation and drying of gas mixtures at high temperatures and a pilot-scale vapor permeation plant were commissioned in 2014. The vapor permeation plant, which can be equipped with up to 12 industrial-scale membranes in 3 membrane modules, is used for pilot and long-term tests for breaking azeotropes and dewatering solvents at temperatures of up to 220 °C and pressures of up to 25 bar.



Dewatering of ethanol by vapor permeation

| 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 1

For application tests in the field of liquid filtration as well as organophilic nanofiltration, Fraunhofer IKTS is operating an Application Center for Membrane Technology, which is located in Schmalkalden, Germany. The center focuses on membrane characterization, and on testing and developing membrane processes by practical feasibility tests. Mobile systems can be used for customer- and project-specific projects. At the application center, test plants are designed and constructed in the form of prototypes, as well.

In the field of liquid filtration, the following plants are available:

- Table plants (MF, UF, NF; PN16)
- Laboratory plants (MF, UF, NF, UO; PN16/PN100)
- Field test equipment (MF, UF, NF; PN25)
- Mobile explosion-proof plants (MF, UF, NF; PN25/PN40; Ex II 3/2G IIB T3)

Emphasis is put on the fact that each membrane process can also be operated with membranes of an industry-relevant geometry. For membrane characterization regarding the determination of the clear water flux and the retention of test substances, additional filtration equipment is held separately. The determination of the acid or alkali stability takes place in various special plants for the usage of the respective media. Furthermore, static exposure tests of membranes in aggressive media can be carried out at temperatures of up to 150 °C.

For membrane testing in field tests, specialized field test systems were designed and constructed. These systems are char-

acterized by the following features/characteristics:

- Automated batch or feed-and-bleed operation
- Filling and level monitoring
- Regulation of pressure or permeate volume flow
- Intelligent retentate/product discharge
- Heating and cooling function
- Backflush (2 modes)
- Data storage

The field test plants can process a defined number of cycles in batch mode and refill automatically. The temperature is controlled automatically by a thermostat or externally available tempered water. In the feed-and-bleed mode, the product/retentate discharge can be realized proportionally to the permeate flow in order to operate at a given concentration point. The systems can also be integrated into a loop of an industrial plant.

Services offered

- Customer- and application-specific membrane testing and process validation
- Piloting of membrane processes
- Process development for the mentioned membrane processes
- Design and construction of suitable membrane housing
- Development and construction of membrane (test) systems
- Development of membrane cleaning strategies for, e.g., existing customer membrane plants
- Processing and/or monitoring or evaluation of field tests
- Delivery of membrane prototypes
- Concept development for integrating membrane systems/ processes into customer-specific production processes
 - 1 Membrane testing at IKTS pilot plant.
 - **2** Mobile filtration container for treatment of organic solvents.
 - 3 Mobile filtration plant with backflushing device and temperature control unit.
 - 4 Pilot vapor permeation plant at IKTS technical center.