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Dewatering of ethanol by pervaporation and vapour permeation with industrial scale NaA-membranes

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1. Introduction

Membranes made of the hydrophilic Zeolite NaA (pore size 4.1å) are of high interest for technical drying of organic solvents by pervaporation. Most promising is the drying of bio ethanol for bio fuel application. In cooperation between SMART Chemicals Development Ltd. and inocermic GmbH, a 100% subsidiary of Hermsdorfer Institut fuer Technische Keramik e.V., an industrial NaA-membrane production line was installed in Hermsdorf/Germany [1,2]. Beyond the stable production of high quality membranes the membrane characterisation by pervaporation and vapour permeation with synthetic mixtures and real distillation products from fermentation and the investigation and adjusting of process parameters is quite necessary for the calculation of industrial separation processes.

2. Results and discussion

The NaA-membranes were tested by pervaporation with synthetic mixtures of technical ethanol and water. Process temperature and flow rate were varied to obtain high fluxes and high separation factors (Table 1). Both parameters strongly influence the permeate flux: the higher the temperature and flow rate the higher the permeate fluxes. Nearly no influence on the permeate quality was found. In all cases nearly pure water of >95% permeated through the membranes resulting in high separation factors up to 30,000. The permeate concentration always kept constant until the water content in feed was higher 0.5%. At lower water contents a slightly decreasing of permeate concentration was observed (Fig. 1).

For determining the long term stability membranes were kept in contact to water/ethanol

Table 1
Permeat flux in kg/(m^2 h) of NaA-membranes in pervaporation with ethanol/water mixtures (90 wt%/10 wt%)

Temperature	100°C	110°C	120°C
flow rate			
1.1 m/s	5.5	–	10.5
1.6 m/s	8.2	10.1	12.8
4 m/s	12.5	–	–

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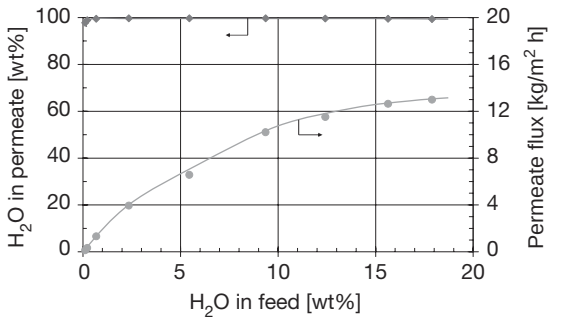

Fig. 1. Pervaporation test (ethanol/water) at 120°C.

mixtures of technical quality over a period of 3 months. During this time these membranes were tested up to 30 times by pervaporation. In all measurements nearly the same separation properties were observed indicating a good chemical stability over long time.

In vapour permeation tests with ethanol/water mixtures at 125°C a separation behavior comparable to the pervaporation tests has been observed (Fig. 2). A permeate flux of 10.5 kg/(m^2 h) (10% water in feed) and a permeate concentration always >95% water were measured. A decrease of the temperature also resulted in a reduced flux as assumed before while the separation factor nearly kept constant. By a test with an acid solution no influence on membrane separation properties was observed.

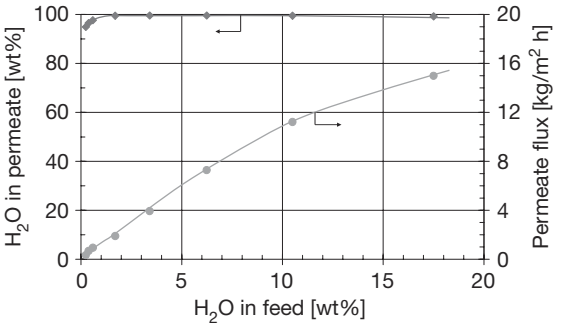

Fig. 2. Vapour permeation test (ethanol/water) at 125°C.

Two typical samples of distilled ethanol from fermentation process were dewatered by pervaporation. One sample came from drinking wine production a second from grain fermentation. The samples were neutralised by adding NaOH. High permeate flux of 10 kg/(m^2 h) (10% water in feed, 120°C, 3 m/s) were found while the permeate concentration in both cases had been >95% and the dewatering was performed up to 99.5% ethanol in feed.

3. Conclusion

Industrial-scale NaA-membranes produced in Hermsdorf/Germany were tested in pervaporation and vapour permeation under different conditions and with different feed mixtures. In all cases the permeate flux was maximised by increasing the process temperature and in case of pervaporation also by accelerating the flow rate. The separation factor was independent of the investigated parameters. Always nearly pure water of >95% permeated through the membrane allowing the dewatering of ethanol from synthetic mixtures as well as from industrial fermentation processes up to purities of >99.5%.

Acid feed solutions were dewatered by pervaporation after neutralisation with NaOH whereas in vapour permeation the neutralisation seems to be not necessary. A long term stability test over a period of 3 months gave a first idea about the good chemical stability of the produced industrial-scale NaA-membranes under pervaporation conditions.

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