MPE Lab Water Processing
Experiment 2: Pervaporation

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Motivation

Separation processes are for the living nature "basic operations" to be able to complete the metabolism in a necessary manner. These separation processes often proceed through the use of membranes which operate selectively. The extensive usage of these membranes for technical purposes frequently fail because of the production and the still limited applications. An important technical separation process is to overcome the azeotropic point, which is possible with a pervaporation separation operation. Because of the relatively low permeate flow the economic efficiency of a procedure like this is significant. With the following experimental setup the dependence of the permeate flow as a function of the substance mixture , the composition of the mixture as well as the varying permeation temperature will be clarified.

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1. Introduction

Pervaporation is the concentration of liquid mixtures by using a membrane as a separation layer between the supplied liquid phase and the extracted vaporous phase. The main characteristic is that because of the separation layer the thermal equilibrium cannot be defined. Due to the non-porous polymer layer the separation effect is mainly determined by the different solubility of the mixture components. Pervaporation processes gain importance, when traditional methods are too expensive to overcome an azeotrope, lower boiling point differences or remove small amounts from a multicomponent mixture. In many cases the procedure is used in combination with rectification.

1.1. Terms

symbol	dimension	meaning
A	m^2	membrane area
c_F	1	percent by mass (feed flow)
c_P	1	percent by mass (permeate flow)
n_D^ϑ	1	reflactive index at the temperature ϑ
t	s	time
$\dot{m}_P$	g m^{-2} s^{-1}	spezific areal mass flow
m	g	permeate mass
ϑ	°C	temperature

2. Basics

2.1. General description

In the pervaporation a homogenous liquid mixture (feed) is fed into a chamber that is separated by a membrane using normal or increased pressure and a preferably high temperature. It gets separated into a liquid mixture of another concentration on one side of the chamber (retentate) and a gas mixture (permeate) on the other side. Because of the different permeabilities (permeation = solubility + diffusion) in the membrane, water prefers to permeate through hydrophile membranes or organic substance through organophilic membranes; the liquid flow is maintained through the use of the liquid pump, the gas flow by applying a vacuum. To retrieve or if needed to be able to set further steps, the permeate needs to be condensed. Due to the fact that the permeate vaporizes when exiting the membrane, the energy needed for the evaporation enthalpy has to be supplied by the liquid flow.

2.2. The membrane

Generally, all Polymers that can be used to make foils can be used to produce membranes as long as a diffuse and selective material exchange is possible. The membrane must meet the following requirements:

- mechanical resistance,
- chemical resistance,
- thermal resistance,
- high, specific permeate flow,
- reproducible, high selectivity,
- high endurance,
- good cleaning possibilities and
- low costs.

2.2.1. Membrane material, membrane types and membrane modules

- The membrane materials are mainly distinguished in:
  - natural and
  - synthetic materials.

  Important factors are the hydrophily, the temperature stability and the chemical resistance.
- The membrane types can be differentiated after:
  - The surface in: - porous and
    - tighter membranes.
  - The structure in: - symmetrical and
    - asymmetrical membranes.
  - The composition in: - homogenous and
    - inhomogeneous membranes.

  Composite membranes are of technical importance for the pervaporation. Because of the separate production processes of the different layers different characteristics can be combined.
- The membrane modules can be distinguished in:
  - Plate modules,
  - Tubular modules,
  - Spiral wounds and
  - Hollow fiber / capillary modules.

  The packing density and the cleanability are typical, different features.

2.3. Qualitive description of the transport processes

Crucial for the separation exercise is the material transport through the membrane. The following mechanisms are to be considered:

- absorption of the molecules from the liquid to the surface of the active layer
- diffusion of the molecules through the membrane.
- release of the molecules in the gas phase.

Additionally the transport mechanisms in the liquid layer must be noted, because the permeation results in a concentration shift in the retentate along the membrane. In the experimental apparatus a high feed flow makes sure that the concentration shift is low and the supplied energy needed to vaporize the permeate is hardly noticeable. Because of the relatively complicated connections of the total material transport, the quantitative presentation of the transport processes should be dispensed with, as they must be considered separately for each module. In general in a pervaporation procedure the achievable permeate flows for a certain configuration (material, type, module and mixture combination) are determined experimentally and are appropriately scaled up for plant production.

2.3.1. Influence parameters

In order to optimally design pervaporation systems, the knowledge of the influence parameters is of considerable importance; while the permeate stream increases with:

- the applied pressure difference,
- increasing temperature (because of higher diffusion) and,
- decreasing thickness of the membrane.

2.3.2. Conditioning of the membrane

Before each experiment with a new membrane, or a new feed composition the membrane must be conditioned to let the membrane slowly adapt to the new conditions and thus prevent destruction and ensure that stationary transport properties are achieved. For this, the membrane must be in a "cold" condition and, with the use of a vacuum on the permeate side, bathed in the processed solution for about approximately 8 hours.

3. Experimental set-up

3.1. Apperitive Equipment

To carry out the pervaporation experiments, the following apparatuses and devices are available:

- **Pervaporation experimental apparatus** with storage tank, liquid circulation pump, thermostat for temperature control of the feed stream, plate membrane module, diaphragm vacuum pump, vacuum pressure gauge, condensation system with **measuring burette** for the permeate.
- **Cryogenic thermostat** for setting the condensation temperature.
- **Refractometer** with thermostat and **density measurement** to determine the mixture composition.
- **Stopwatch** for experimental time determination.
- **Laboratory balance** for determining the permeate quantity

3.2. Equipment setup

The principal test setup can be found in the RI flow chart attached in the appendix:
The feed flow is taken by a controllable liquid pump out of the thermostatted receiver tank ***B 1*** and is fed centrally into the plate membrane module ***F 1***. The retentate flow is collected via a ring-like channel at the perimeter of the module and fed back to the receiver tank. The vacuum in the permeate chamber is maintained by means of a diaphragm vacuum pump and the accumulating permeate in the heat exchanger ***W 1*** condensates and is caught in the measuring burette ***X 1***. The incoming and outgoing temperature of the fluid flow at the membrane and the pressure of the set vacuum are registered with an industrial recorder.

4. Experimental procedure and evaluation

4.1. Experimental procedure

The following preparations are made by the laboratory staff:

- Filling the composite (composition of the substance is maintained),
- Conditioning of the membrane, inclusive adjusting the volume flow of the feed,
- Adjusting the feed flow,
- Adjusting the cryogenic thermostat.

The experimental procedure includes:

- Adjustment of the test temperature and recording of the temporal temperature profil of the feed and retentate stream,
- Determination of the permeate flux at two different temperatures per group
  Temperature range: 65 °C to 90 °C
- Setting the pressure (PIR 003) to 15 mbar or 30 mbar using valve V11.

4.2. Experimental evaluation

The following data must be evaluated:

- Graphical representation of the temporal temperature curve of feed and retentate stream.
- Tabular and graphical representation of the spezific areal mass flow $\dot{m}_P\ (\vartheta_F, c_F)$ as a function of the feed temperature with the feed concentration c_F as parameter.
- Tabular and graphical representation of the permeate concentration c_P as a function of the feed temperature with the feed concentration c_F as parameter.

5. Literature

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/3/ Ebert, Stefan, Pervaporation, Studienarbeit an der FH Offenburg, 1995

6. RI-flowchart

