NORWEGIAN UNIVERSITY OF SCIENCE AND TECHNOLOGY

# ST6: Air separation with membranes

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## 1. Objective

The goal of this experiment is to get to know some of the aspects of membrane separation by performing a simple air separation process. The students will explore how different operational parameters influence a membrane separation process, and how combinations of several membrane modules can be used to obtain a targeted separation performance.

## 2. Background and theory

A membrane is a permselective barrier or interface between two phases-two gas phases in the case of air separation. Separation occurs because one of the components in the feed gas passes the membrane more easily and quicker than the other components.

Important properties in membrane separation are permeation rate and selectivity. Permeability quantifies how easily a given gas component will pass through the membrane, and selectivity about how easily the same gas component passes through the membrane with respect to another component. This transport of gas through a given membrane material is determined by a combination of diffusivity and solubility.

Today, gas separation by membrane processes is mostly accomplished by use of non-porous polymeric membranes. Some of the most used polymers for this purpose are polysulfone, polyimide, cellulose acetate and polycarbonate.

Membranes can be produced in different morphologies - flat sheet, spiral wound or hollow fibres. In most cases, hollow fibres are preferred since these modules have the highest packing density; i.e. large membrane area packed in a small volume. An illustration of a hollow fibre module is shown in Figure 1.

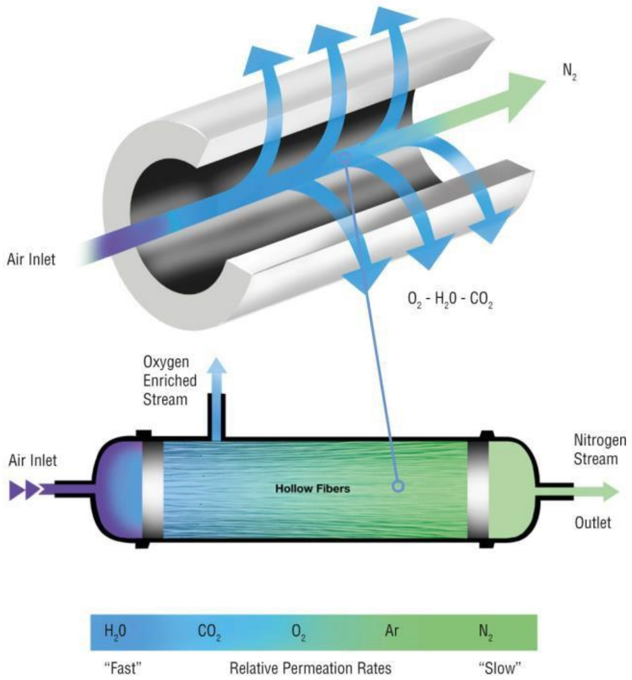


**Figure 1:** Hollow fibre module.

In a membrane process for gas separation there will be different streams; the feed gas – the starting point, the permeate – the gas that has permeated through the membrane, and the retentate – the gas that did not pass the membrane. An illustration of a very simplified transport mechanism for a membrane process is given in Figure 2 (the symbols used correspond to the ones used in the student's prescribed textbook, Geankopolis),

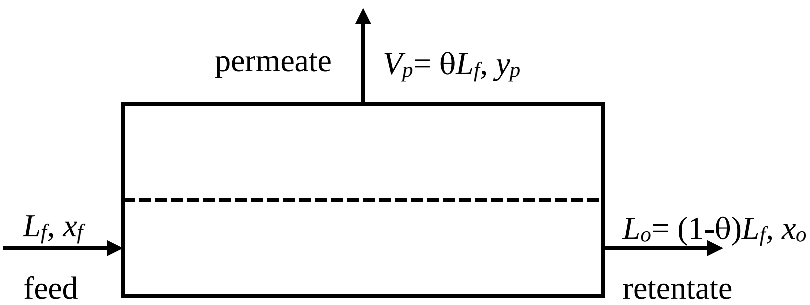


**Figure 2:** Simplified transport mechanism for a membrane process, where $L_f$, $L_o$, and $V_p$ are feed-, retentate- and permeate flow rate, respectively, and $x_f$, $x_o$ and $y_p$ are mole fractions of a component in the feed, retentate, and permeate, respectively.

where $L_f$ is feed flow rate, $x_f$ is mole fraction of a component in the feed, $L_o$ is retentate flow rate, $x_o$ is mole fraction of a component in the retentate, $V_p$ is permeate flow rate and $y_p$ is mole fraction of a component in the permeate.

The overall material balance then becomes:

$$L_f = L_o + V_p \tag{1}$$

A parameter of economic importance in commercial membrane processes is stage cut, or cut rate, $\theta$, given by

$$\theta = \frac{V_p}{L_f} \tag{2}$$

In commercial processes it is essential to choose correct cut rate, because it directly influences the product purity and yield.

Another important parameter is recovery, *R*, for a given gas component, *A*. Recovery relates to the yield of the membrane process, how much of the preferred component is present in the product stream, compared to the amount in the initial feed stream. It indirectly gauges how much of the preferred component is lost. When the preferred product is in the retentate stream, recovery, *R*, is given by

$$R_A = \frac{x_{o,A} L_o}{x_{f,A} L_f} \cdot 100\% \tag{3}$$

For further details on membrane gas separation theory, see chapter 8 Gas Separation in Membrane Technology and Applications by R.W. Baker (handed out).

## 3. The set-up

The set-up to be used in this experiment has modules of Prism membranes (from Air Product AS), where the membrane is made of polysulfone – an oxygen selective membrane. These are commercial modules are used for nitrogen production (up to 98-99% purity) or concentration of oxygen. The set-up has four membrane modules. The modules can be connected in different ways, so it is possible to test different configurations: modules in series, modules in parallel and combination of these. Flow meters are mounted at the exits of the permeate and retentate gas streams, and oxygen analysers (% $O_2$) are placed at the exit of the permeate and retentate stream. Pressure indicators are located in the feed, permeate and retentate streams. All flow controls are automated and controlled by the computer next to the set-up.

# 4. Experimental

## 4.1 Personal Protective Equipment (PPE)

The experimental procedure doesn't involve use of chemicals per se. However, since there are many other experiments that are carried out nearby simultaneously, it is mandated that the students wear lab coat and safety googles throughout the runtime of the experiment. It should be noted that the PPEs are also necessary during operation of high pressure systems.

## 4.2 Procedure

1. Close the Main Feed Valve.
2. Open the gas outlet valve on the wall and set the out pressure at 3 bars.
3. Make the proper connections, with the plastic lines (Check for leaks), to obtain the targeted configuration.
4. Double click on the computer on the icon for tare controllers "tareControllers.py".
5. Double click on "run-py".
6. Set the flow to be tested on the flow controller, click on "Confirm", check value on display of the mass flow controller in retentate stream.
7. Click on "Start Sampling".
8. Open the main feed valve in the set-up slowly.
9. Wait to obtain stable values in the $O_2$ concentration for permeate and retentate streams.
10. Once the run is finished, click on "Stop Sampling".
11. If you want to change the cut rate, double click on "run-py" again, change the set point in the Flow Controller. Click on "Start Sampling". Repeat 8 to 10 as many times as necessary to change the flow rate, cut rate.
12. Once the experiment is finished, click on "Stop Sampling".
13. Close the main Valve.
14. Click on "tareControllers.py".
15. Open the file "FellesLab_Backup"and save your data.
16. Change configuration and restart from point 2.

All the experiments should be run with constant feed pressure at room temperature. Assume that only nitrogen and oxygen is present in the feed gas.

You should run experiments with the following configurations:

1. One, single module. Test all 4 modules
2. Four modules in series, with respect to retentate
3. Three modules in parallel, with respect to retentate
4. Four modules in parallel, with respect to retentate
5. Your own proposed configuration to obtain high nitrogen purity (based on 1, 2, 3 and 4)
6. Your own proposed configuration to obtain high oxygen purity (based on 1, 2, 3 and 4)

Run four cut rates for 2, 3 and 4 configurations.
Calculate Recovery of $O_2$ and $N_2$ in both streams (Retentate and Permeate)

## 5. Work plan

The students should follow the general guidelines for work plans pertaining to the felleslab. In addition, some more specific information for this experiment is given here.

Read the comments on what to include in the report, so that you are prepared to perform the experiment in such a way that you will be able to answer the questions given there.
Based on what you shall do in the experiment, the following should be included in the work plan:

1. Some background and theory
   - Name the three factors that determine the performance of a membrane gas separation system.
   - Include the equations you will use for the calculations in the experiment
   - What is the driving force for the separation?
   - What is the wanted gas component in the permeate and the retentate respectively?
   - What do you expect in counter-current flow, cross-flow and co-current operations in membrane modules?
2. A description of what you will do in the experiment, operating procedure and what do you expect for every configuration.
3. Prepare an excel sheet with the needed input and formulas for the experiments.
   - What will be your inputs – what will you measure?
   - Which formulae do you need – what will be your output?
   - This must help you to calculate all the flow streams, mole fractions, recovery and cut rates. For $O_2$ and $N_2$
4. Name the possible risks for this experiment, and answer the next questions:
   - According to your opinion, what is the largest risk with the experiment?
   - What will you do to try to avoid/reduce this risk?
5. Do an example calculation of cut rate and recovery of $N_2$ from the following data:
   - Retentate flow rate: 500 ml/s
   - Permeate flow rate: 100 ml/s
   - $O_2$ concentration in feed: 35%
   - $O_2$ concentration in permeate: 80%

*There will be a small discussion with **some questions** to students mostly pertaining to the work plan to check the preparedness of the group just before the experiment. NB! The answers to the questions add up to the final grade*

## 6. Report

You should follow the general guidelines for reports pertaining to the felleslab.
***In addition, some more specific information for this experiment*** is given here.

1. Include theory relevant for the experiment, including all equations used in calculations. Summarize the three factors that determine the performance of a membrane gas separation system. Hint on the industrial version of the experiment you have done, including applications, membrane materials and process design. Name some other membrane gas separation applications you find interesting, including membrane materials and process design. **A brief and concise theory section addressing all requirements cited above will be sufficient.**
2. Present your results using **tables and graphs scientifically**. **Describe what happened in the different experiments based on what you have learned from the theory.** Which parameters influenced the separation, and in which way? Compare the results for the different configurations, discuss the differences.
3. Discuss the reasons behind your choices of configurations for high purity nitrogen and high purity oxygen. Discuss purity vs. recovery. Are there any limitations in the set-up towards giving the wanted separation? Do you have any suggestions on what could be done to improve the separation? What are the error sources in this experiment? Use the graphs to explain your conclusions. Remember to include a calculation example as an appendix (**one from the experiment, not the one from the work plan).**