ECHE 362

TA Guide for Gas Membrane Laboratory

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In this experiment, the students are asked to do four tasks:

1. Determine the permeances for oxygen and nitrogen and the O2/N2 permeability ratio for the laboratory membrane module.
2. Determine experimentally the effects of the stage cut and retentate pressure on the permeate and retentate concentrations and the permeate flow rate. Over what conditions is the perfect mixing model valid?
3. Model the effect of fiber diameter on the permeate pressure along the length of the fiber. Check the assumption that the bore-side (permeate) pressure drop is negligible.
4. Design a new separation train using the four available modules to meet the oxygen and nitrogen requirements using the perfect mixing model. Optimize the process to yield the maximum production of the oxygen-rich and nitrogen-rich streams given the current compressor’s capabilities. How should the four modules be arranged, and at what pressure and stage cuts should they be operated?

**Tasks 3 and 4 are done outside the lab – the foreman’s report deals with tasks 1 and 2 only.**

**If you are unfamiliar with gas membrane separations and the following terms (permeate, retentate, permeance, stage cut, perfect mixing) please see Dr. Wainright.**

**Operation of Gas Separation System**

The pilot plant has a PRISM separator consisting of four columns, two co-current and two countercurrent. Permeate flow is always down from the top of the column and out the bottom – the top end of the fibers is sealed. The retentate flow over the outside of the fibers starts at the bottom of the left most column, up that column, across and then down (co-current) in the 2nd column from the left, and then up and then down again. The columns can be operated individually, or in groups of two or four using the valves on the permeate outflow at the bottom to shut off the permeate flow. With no permeate flow eventually a column will reach equilibrium; the same pressure and the same O2 concentration for both the permeate and retentate. Under these conditions, no separation occurs and the column effectively does not exist. However, most of the time we run all four columns together, ignoring co- vs counter- current differences. The retentate flow cannot be modified; it always passes through all four columns in series. Gas cylinders of oxygen, nitrogen, and air will be used as feed streams. The oxygen concentration of the permeate and the retentate streams are measured using oxygen analyzers. The permeate and retentate flow rates are measured using rotameters. Two pressure gauges are located on the apparatus (feed and retentate pressure) and needle valves are included to control the retentate flow rate. The permeate flow rate and pressure are not controlled, we always exhaust the permeate to atmospheric pressure.

**Calibration**

Calibration curves are provided for the rotameters. Make sure the originals stay with the lab – the students may borrow them to make copies but they must be returned ASAP.

The oxygen analyzers should be calibrated according to the following procedure:

1. Set the appropriate valves to bypass the columns and send gas directly to the oxygen sensors.
2. The meters should read 20.9% within 5 minutes after flushing the system with air. If not, adjust the meter.
3. Flush with oxygen. If readings within 5 minutes do not stabilize at 100± 2%, adjust the sensor to 100% and repeat step 1.

***Do not calibrate using pure N2 – it’s not necessary and it puts a lot of pure N2 into the air.***

**Experimental Operating Limits:**

Feed Pressure (*P*): 100 – 140 psig

Stage Cut (*θ*): 0.1 – 0.5

**Foreman’s Report**

In the Foreman’s report, the students must outline all the experimental runs required in order to calculate the permeances, and for air separation to determine the effect of the retentate pressure and stage cut. For a given retentate pressure, the permeate flow rate is essentially constant. This means that you control the retentate flow rate to control the stage cut. An Excel spreadsheet is required in the Foreman’s report. The data tables must include all parameters to be measured and any formulas for values they need to calculate.

O2 permeance determination:

The range of air pressures of interest is 100 to 140 psig. At those pressures, the O2 partial pressure is 20-32 psia, or 5 to 18 psig. This is the range of retentate pressures they should use to measure O2 permeance. Since a pure gas is used, the retentate flow rate should be low – don’t let them waste gas. They should calculate the slope of a plot of permeate flow rate vs retentate pressure as soon as they have the data. This slope should be 0.23-0.24 slpm/psi using all four columns.

N2 permeance determination:

N2 pressure should be about 80-120 psia, or 65 to 105 psig. Again, a modest retentate flow rate is sufficient. They should plot permeate flow vs retentate pressure, the slope should be about 0.04 slpm/psi. Next they should calculate the O2/N2 permeability ratio (ratio of the slopes) - it should be about 6 – 6.5 but we have seen values from 5 to as high as 8. Also, the intercept should go through the origin (0,0). Significant non-zero intercept values should be checked – take more data.

***They should not proceed with the rest of the experiment until they have reasonable values for the permeances and the permeability ratio.***

Effect of stage cut and retentate pressure on Air separation:

The students will need at least six different runs for this section. Three should be at a constant pressure (for example 115 psig) with the stage cut varying between 0.2 and 0.4. Three more runs should be at a constant stage cut (0.3 for example) and varying pressure (100, 115, 130 psig for example). They will need to calculate stage cut from the permeate and retentate flow rates – this must be in their Excel spreadsheet.

**Safety Considerations**

While the substances used in this experiment do not normally strike one as dangerous, several precautions must be taken. First of all, you are dealing with high pressure cylinders of gas which can do considerable damage if the stem or regulators are damaged. Therefore all cylinders must always be secured so they cannot fall and be damaged. Good practice is to close the main valve on all cylinders except when that particular gas is in use. All around you in the laboratory are experiments that may contain hazardous chemicals. Therefore, you must always wear your lab jacket and safety glasses. The foreman’s report must include the hazards associated with pure oxygen and pure nitrogen and the physical lay-out of the apparatus***. In particular, they should note where the exhaust ports are – when running pure N2 or air, the retentate exhaust is too dilute in O2 to breathe – no one should stand near the exhaust ports for any period of time.***

Emergency shutdown procedure: close main valve on all tanks.

**Data Acquisition**

Data acquisition for this lab is purely by hand. Make sure the foreman’s Excel spreadsheet has columns for all the data to be collected: feed and retentate pressures, retentate composition and flow rate, permeate composition and flow rate. Calculated values that must be included are the stage cut and the graphs needed to determine the O2 and N2 permeances. Also, the mass balance should be checked for all air results. Instruct the foreman in the proper reading of the rotameters (center of the ball) when they are preparing the foreman’s report.

**Known Hardware Issues/Things to watch our for**

Remind the students to turn valves slowly, especially when de-pressurizing a column – we don’t want sudden pressure changes. A sudden pressure drop or increase puts additional stress on the membranes and could lead to membrane failure.

If the retentate rotameter oscillates – increase the retentate flow rate until the meter settles down. Then decrease the flow to the desired setting.

Don’t let the students waste time and air. If they’re talking, the air flow should be off. It should only take a few minutes to dial in a desired set of conditions (retentate pressure and stage cut) and for the readings to stabilize. It’s very helpful to plot the rotameter calibration curves both ways – 1) reading on the x-axis and actual flow on the y-axis, and 2) with the actual flow on the x-axis and the reading on the y-axis. Now you can set a pressure, determine the permeate flow rate, calculate the necessary retentate flow rate for the desired stage cut, look up the retentate rotameter reading required, and adjust the flow control valve to give that reading.

Mass balance – we typically find that we ‘make’ oxygen. There will appear to be slightly more O2 in the permeate plus retentate than entered in the air. However, the mass balance should be within 10%, and generally within 5%. If the mass balance is reasonable, this is a good indication that steady state has been achieved.

Mass Balance calculation:

O2 in the feed = 0.209 x (sum of retentate and permeate flow rates)

O2 out = {(Permeate concentration)(permeate flow) + (rentenate conc.)(retentate flow)}

%Error = 100\* (O2 out – O2 in)/ (O2 in)

This calculation should be in their spreadsheet.

**TA Pre-lab Responsibilities**

Beginning of each semester:

1. Put new batteries in the oxygen sensors. Check sensor operation, and have Craig order new sensors if needed.
2. Make sure we have full tanks of oxygen, nitrogen and air, and the appropriate regulators. Connect the tanks to the system and check for leaks. If we need a tank – tell Craig and he will order it.

After each lab session:

1. Check the remaining pressure in each tank, especially the air tank. Have Craig order a new tank if below 1000 psig
2. Make sure all tanks are closed, all lines and regulators are vented

Note – the foreman for each group is really responsible for both of the above – you double check to be sure that these have been done.